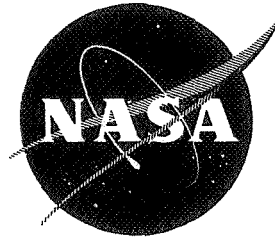


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FINAL REPORT

LOW COST CARBONACEOUS DEVELOPMENT

by

S. H. Cardall

THIOKOL CHEMICAL CORPORATION
WASATCH DIVISION
Brigham City, Utah

Prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

March 1971

CONTRACT NAS3-12034

NASA Lewis Research Center
Cleveland, Ohio
L. A. Burkardt, Project Manager

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FOREWORD

The research and development work described herein was conducted by the Wasatch Division of Thiokol Chemical Corporation under NASA Contract NAS3-12034. The work was done under the management of the NASA Project Manager, Mr. L. A. Burkardt, NASA Lewis Research Center, Cleveland, Ohio.

This program was conducted at the Wasatch Division under the management of Mr. E. L. Bennion with Mr. S. G. Goddard acting as Project Engineer. The principal investigators were Mr. S. H. Cardall and Mr. R. C. Laramée. Motor manufacturing was supervised by Mr. A. W. Pace.

ABSTRACT

Thiokol Chemical Corporation, Wasatch Division, has developed some low cost graphite powder and phenolic resin molding compounds for use in rocket motor nozzle applications. This program was conducted to improve the mechanical properties and reduce the density and thermal conductivity of previously developed low cost carbonaceous materials. The program was structured in three tasks: basic formulation development, fiber reinforcement, and filler additive studies. Four formulations were developed at the completion of the effort. These formulations compare favorably with the original low cost carbonaceous materials and conventional carbon cloth and silica cloth preimpregnated broad goods type nozzle insulators.

TABLE OF CONTENTS

	<u>Page</u>
SUMMARY	1
INTRODUCTION	2
TASK 01 - BASIC FORMULATION DEVELOPMENT STUDIES	3
Resin Selection	3
Formulation Optimization Studies	8
Monsanto RI-4009 Resin	8
Durez 10694 Phenolic Resin	33
DEN 438 Epoxy Novolac	33
Particle Size Distribution Study	55
Material Erosion Tests	63
Monsanto RI-4009 Resin	66
Durez 10694 Phenolic Resin	82
DEN 438 Epoxy Novolac	82
TASK 02 - FIBER REINFORCEMENT STUDY	86
Monsanto RI-4009 Resin	86
Durez 10694 Phenolic Resin	102
TASK 03 - FILLER ADDITIVE STUDIES	111
Monsanto RI-4009 Resin	111
Cork Filler	113
Phenolic Microballoons	113
Wood Filler	113
TU-379 Testing of Formulations Containing Fillers	121
Durez 10694 Phenolic Resin	122
Special TU-379 Nozzle Test	138
FORMULATION OPTIMIZATION MATERIAL SELECTIONS	141
TU-622 MATERIAL EROSION AND CHAR TESTING	141
Erosion	149
Inlet-Throat Erosion	149
Throat-Exit Erosion	152
Char	152
Inlet-Throat Char	152
Throat-Exit Char	155

TABLE OF CONTENTS (Cont)

	<u>Page</u>
MATERIAL CHARACTERIZATION AND COMPARISON	157
Specific Gravity	169
Raw Material Cost	171
Erosion Rate and Char Rate	171
Thermal Conductivity	171
Ultimate Compressive Strength	174
Ultimate Tensile Strength	176
Coefficient of Thermal Expansion	176
Structural Integrity and Ease of Fabrication	179
Material Summary	188
CONCLUSIONS AND RECOMMENDATIONS	191
Conclusions	191
Task 01	191
Task 02	191
Task 03	191
General Conclusions	192
Recommendations	192
REFERENCES	194

LIST OF ILLUSTRATIONS

<u>Figure</u>		<u>Page</u>
1	LCCM Progress Flow Chart	5
2	Tensile Strength of RI-4009 Resin/Graphite Powder	12
3	Compressive Strength of RI-4009 Resin/Graphite Powder	13
4	Torch Test Samples, RI-4009 Resin/Graphite Composite, Mixes 447 thru 449	19
5	Torch Test Samples, RI-4009 Resin/Graphite Composite, Mixes 450 thru 453	20
6	Torch Test Samples, RI-4009 Resin/Graphite Composite, Mixes 454 thru 457	21
7	Torch Test Samples, RI-4009 Resin/Graphite Composite, Mixes 458 thru 461	22
8	Torch Test Samples, RI-4009 Resin/Graphite Composite, Mixes 462 thru 465	23
9	Torch Test Samples, RI-4009 Resin/Graphite Composite, Mixes 466 thru 469	24
10	Torch Test Samples, RI-4009 Resin/Graphite Composite, Mixes 470 thru 473	25
11	Torch Test Samples, RI-4009 Resin/Graphite Composite, Mixes 474 thru 477	26
12	Torch Test Samples, RI-4009 Resin/Graphite Composite, Mixes 478 thru 481	27
13	Torch Test Samples, RI-4009 Resin/Graphite Composite, Mixes 522 and 523	28

LIST OF ILLUSTRATIONS (Cont)

<u>Figure</u>		<u>Page</u>
14	Torch Test Sample, RI-4009 Resin, Mix 534	30
15	Torch Test Sample, RI-4009 Resin, Mix 535	31
16	Torch Test Sample, RI-4009 Resin Plus Flame Retardant, Mix 539	32
17	Torch Test Samples, Durez 10694 Resin/Graphite Composite, Mixes 482 thru 485	37
18	Torch Test Samples, Durez 10694 Resin/Graphite Composite, Mixes 488 thru 491	38
19	Torch Test Samples, Durez 10694 Resin/Graphite Composite, Mixes 492 thru 495	39
20	Torch Test Samples, Durez 10694 Resin/Graphite Composite, Mixes 496 thru 498	40
21	Torch Test Samples, Durez 10694 Resin/Graphite Composite, Mixes 500 thru 502	41
22	Torch Test Samples, Durez 10694 Resin/Graphite Composite, Mixes 503 thru 505	42
23	Torch Test Samples, Durez 10694 Resin/Graphite Composite, Mixes 506 thru 508	43
24	Torch Test Samples, Durez 10694 Resin/Graphite Composite, Mixes 509 thru 511	44
25	Torch Test Sample, DEN 438 Epoxy Novolac/Graphite Composite, Mix 514	47
26	Torch Test Sample, DEN 438 Epoxy Novolac/Graphite Composite, Mix 515	48
27	Torch Test Sample, DEN 438 Epoxy Novolac/Graphite Composite, Mix 516	49

LIST OF ILLUSTRATIONS (Cont)

<u>Figure</u>		<u>Page</u>
28	Torch Test Sample, DEN 438 Epoxy Novolac/Graphite Composite, Mix 517	50
29	Torch Test Sample, DEN 438 Epoxy Novolac/Graphite Composite, Mix 518	51
30	Torch Test Sample, DEN 438 Epoxy Novolac/Graphite Composite, Mix 519	52
31	Torch Test Sample, DEN 438 Epoxy Novolac/Graphite Composite, Mix 520	53
32	Particle Size Distribution, Ro-Tap, GL-1008 Graphite Powder	56
33	Particle Size Distribution, Ro-Tap, GL-1011 Graphite Powder	57
34	Particle Size Distribution, Ro-Tap, GL-1012 Graphite Powder	58
35	Tensile Strength/Particle Size Curve, RI-4009 Resin	61
36	Thermal Penetration/Particle Size Curve, RI-4009 Resin	62
37	TU-379 Materials Screening Motor	64
38	Gas Flow Erosion, RI-4009 Resin	67
39	Gas Flow Erosion, RI-4009 Resin and Durez 10694 Resin	68
40	Gas Flow Erosion, RI-4009 Resin, Durez 10694, and DEN Epoxy Novolac	69
41	TU-379 Nozzle Data, RI-4009 Resin, Motor No. 549.1	70
42	TU-379 Nozzle Data, RI-4009 Resin, Motor No. 549.2	71
43	TU-379 Nozzle Data, RI-4009 Resin, Motor No. 562.1	72

LIST OF ILLUSTRATIONS (Cont)

<u>Figure</u>		<u>Page</u>
44	TU-379 Nozzle Data, RI-4009 Resin, Motor No. 562.2	73
45	TU-379 Nozzle Data, RI-4009 Resin, Motor No. 561.1	74
46	TU-379 Nozzle Data, RI-4009 Resin, Motor No. 561.2	75
47	TU-379 Nozzle Data, RI-4009 Resin, Motor No. 572	76
48	TU-379 Nozzle Data, Durez 10694 Resin, Motor No. 550.1	77
49	TU-379 Nozzle Data, Durez 10694 Resin, Motor No. 550.2	78
50	TU-379 Nozzle Data, Durez 10694 Resin, Motor No. 551	79
51	TU-379 Nozzle Data, DEN 438 Epoxy Novolac, Motor No. 566.1	80
52	TU-379 Nozzle Data, DEN 438 Epoxy Novolac, Motor No. 566.2	81
53	TU-622 Nozzle Erosion and Char Profile, DEN 438 Epoxy Novolac	84
54	Test Panels, RI-4009 Resin, Carbon Fiber Reinforcement . .	89
55	Test Panels, RI-4009 Resin, Glass Fiber Reinforcement . . .	90
56	Test Panels, RI-4009 Resin, Rayon Fiber Reinforcement . . .	91
57	Rayon Fiber, 0.25 In. (0.67 cm)	93
58	Machined TU-379 Nozzle Assembly Showing Banding Effect . .	94
59	TU-379 Nozzle Data, RI-4009 Resin, Motor No. 651	95
60	TU-379 Nozzle Data, RI-4009 Resin, Motor No. 652	96

LIST OF ILLUSTRATIONS (Cont)

<u>Figure</u>		<u>Page</u>
61	TU-379 Nozzle Data, RI-4009 Resin, Motor No. 653	97
62	TU-379 Nozzle Data, RI-4009 Resin, Motor No. 654	98
63	TU-379 Nozzle Sections, Carbon Fiber Reinforcements	99
64	TU-379 Nozzle Data, RI-4009 Resin, Motor No. 670	100
65	Post-test Section, TU-379 Nozzle Assembly, 0.25 In. (0.64 cm) 10 Percent Glass Fiber, Motor No. 670	101
66	TU-379 Nozzle Data, RI-4009 Resin, Motor No. 668	103
67	TU-379 Nozzle Data, RI-4009 Resin, Motor No. 667	104
68	TU-379 Nozzle Pretreated Carbon Fiber Sections	105
69	TU-379 Nozzle Data, Durez 10694, Motor No. 671	109
70	TU-379 Nozzle Section	110
71	Test Panels, Cork Filler, RI-4009 Resin	114
72	Test Panels, Phenolic Microballoon Filler, RI-4009 Resin	115
73	Test Panels, Wood Filler, RI-4009 Resin	116
74	Test Panels, Glass Microballoon Filler, RI-4009 Resin	117
75	TU-379 Nozzle Data, RI-4009 Resin, Motor No. 643	123
76	TU-379 Nozzle Data, RI-4009 Resin, Motor No. 644	124
77	TU-379 Nozzle Data, RI-4009 Resin, Motor No. 645	125
78	TU-379 Nozzle Data, RI-4009 Resin, Motor No. 646	126
79	TU-379 Nozzle Data, RI-4009 Resin, Motor No. 647	127

LIST OF ILLUSTRATIONS (Cont)

<u>Figure</u>		<u>Page</u>
80	Wood Filler (10, 15 and 20 Percent)/RI-4009 Resin	128
81	Microballoon and Cork Fillers/RI-4009 Resin	129
82	TU-379 Nozzle Data, Durez 10694 Resin, Motor No. 673	135
83	TU-379 Nozzle Data, Durez 10694 Resin, Motor No. 672	136
84	TU-379 Nozzle Sections	137
85	TU-379 Nozzle Data, RI-4009 Resin, Motor No. 665	139
86	TU-379 Nozzle Section (Special Test)	140
87	Sectioned TU-622 Test Nozzle, LCCM-2610 (Graphite Phenolic)	142
88	Sectioned TU-622 Test Nozzle, LCCM-4120 (Graphite Phenolic)	143
89	Sectioned TU-622 Test Nozzle, UF-1161, UF-1162, UF-1163, and UF-1164	144
90	UF-1161 Pressure Trace	145
91	UF-1162 Pressure Trace	146
92	UF-1163 Pressure Trace	147
93	UF-1164 Pressure Trace	148
94	TU-622 Nozzle Liner Material Erosion Rate Performance, Inlet-Throat	151
95	TU-622 Nozzle Liner Material Erosion Rate Performance, Throat-Exit	153

LIST OF ILLUSTRATIONS (Cont)

<u>Figure</u>		<u>Page</u>
96	Nozzle Liner Material Char Rate Performance, Inlet-Throat	154
97	Nozzle Liner Material Char Rate Performance, Throat-Exit	156
98	UF-1161 (Parallel to Pressure) Coefficient of Thermal Expansion vs Temperature	161
99	UF-1162 (Parallel to Pressure) Coefficient of Thermal Expansion vs Temperature	162
100	UF-1163 (Parallel to Pressure) Coefficient of Thermal Expansion vs Temperature	163
101	UF-1164 (Parallel to Pressure) Coefficient of Thermal Expansion vs Temperature	164
102	UF-1161 (Perpendicular to Pressure) Coefficient of Thermal Expansion vs Temperature	165
103	UF-1162 (Perpendicular to Pressure) Coefficient of Thermal Expansion vs Temperature	166
104	UF-1163 (Perpendicular to Pressure) Coefficient of Thermal Expansion vs Temperature	167
105	UF-1164 (Perpendicular to Pressure) Coefficient of Thermal Expansion vs Temperature	168
106	Raw Material Cost/Lb (1,000 Lb Quantities)	172
107	Liner Material Thermal Conductivity	173
108	Liner Material Ultimate Compressive Strength and Young's Modulus at Room Temperature (With Grain or With Ply)	175
109	Liner Material Ultimate Tensile Strength and Young's Modulus at Room Temperature (With Grain or With Ply)	177

LIST OF ILLUSTRATIONS (Cont)

<u>Figure</u>		<u>Page</u>
110	Liner Material Coefficient of Thermal Expansion, With Grain and Against Grain or With Ply and Against Ply (at 800°F, 700°K)	178
111	TU-622 Liner Material Structural Integrity and Ease of Fabrication	180
112	Graphite Powder Phenolic Test Application Nozzle	190

LIST OF TABLES

<u>Table</u>		<u>Page</u>
I	LCCM Development Screening Test - Task 01	7
II	RI-4009 Resin Test Matrix, Formulation Optimization Studies	9
III	Durez 10694 Test Matrix, Formulation Optimization Studies	10
IV	DEN 438 Epoxy Novolac Test Matrix, Formulation Optimization Studies	11
V	LCCM Development Optimization Study, Monsanto RI-4009 Resin	15
VI	LCCM Development Optimization Study, Durez 10694 Resin	34
VII	LCCM Development Optimization Study, DEN 438 Epoxy Novolac	46
VIII	LCCM Particle Size Distribution Test Matrix	59
IX	LCCM Development, Particle Size Distribution Study (RI-4009 Monsanto Resin)	60
X	LCCM Development, TU-379 Motor Test Matrix, Erosion Data, and Physical Properties	65
XI	LCCM Development, Thermal Conductivity	85
XII	LCCM Fiber Reinforcement Fabrication Test Matrix	87
XIII	LCCM Development, Physical Properties Fiber Reinforcement Study, RI-4009 Resin	88
XIV	LCCM Development, Physical Properties Fiber Reinforcement Study, Durez 10694, 25 Percent Resin	106

LIST OF TABLES (Cont)

<u>Table</u>		<u>Page</u>
XV	LCCM Development, Physical Properties Fiber Reinforcement Study, Durez 10694, 25 and 40 Percent Resin	108
XVI	LCCM Development, Filler Additives Test Matrix	112
XVII	LCCM Development, Filler Additive Study, Cork Physical Properties	118
XVIII	LCCM Development, Filler Additive Study, Phenolic Microballoon Physical Properties	119
XIX	LCCM Development, Filler Additive Study, Wood Flour Physical Properties	120
XX	LCCM Development, Physical Properties Filler Additive Study, 74 Micron Particle Size	131
XXI	LCCM Development, Physical Properties Filler Additive Study, Phenolic Microballoon Filler	132
XXII	LCCM Development, Physical Properties Filler Additive Study, Cork Filler	133
XXIII	LCCM Development, Physical Properties Filler Additive Study, Wood Filler	134
XXIV	TU-622 Material Performance	150
XXV	Comparison of UF Materials and Original Thiokol Formulations	158

LIST OF TABLES (Cont)

<u>Table</u>		<u>Page</u>
XXVI	Summary of Thermal Expansion Data	160
XXVII	Raw Material Compositions	170
XXVIII	Weighting Factors	182
XXIX	Inlet Material Selection	184
XXX	Throat Material Selection	185
XXXI	Exit Material Selection	187

SUMMARY

This is the final report for Contract NAS 3-12034, Low Cost Carbonaceous Materials (LCCM). The program objective was to improve the physical and mechanical properties of Thiokol's low cost carbonaceous materials, while maintaining their excellent erosion resistant properties. The program was broken into three primary tasks. Task 01 was the Formulation Development Study, wherein the resin-graphite formulations and the graphite particle size were optimized. Under Task 02, Fiber Reinforcement Study, several fiber types were introduced into the resin-graphite combination to increase the mechanical properties of the materials. Task 03, the Filler Additive Study, combined a number of fillers in an attempt to reduce the thermal conductivity and density of the LCCM compounds.

In Task 01, several resin systems and a number of graphite powders were evaluated to optimize the formulations. The resin systems included single stage phenolics, two stage phenolics, polyphenylene, and epoxy-novolac. Four of the resins were acceptable after screening and were used through the task. They were combined with graphite powders and evaluated for mechanical properties, oxyacetylene char penetration, thermal conductivity, and erosion resistance. Particle size distribution studies were made on the several graphite powders combined with the resins to further optimize the formulations. Two resin systems and two graphite particle sizes were selected at the completion of Task 01 and were used in the remaining tasks. Three fibers (carbon, glass, and rayon) were selected to reinforce the resin-carbon formulations in Task 02. The formulations were evaluated for mechanical properties and erosion resistance. The carbon fiber cut to 0.25 in. proved to be best for reinforcing the formulations. Task 03, the Filler Additive Study, was conducted to improve the thermal conductivity and reduce the density without impairing the erosion resistance or mechanical properties. The fillers included cork, glass, and phenolic microballoons, wood flour, and liquid NBR rubber. The wood flour appeared to be superior to the other fillers.

Four LCCM formulations developed from the preceding tasks are identified as UF-1161, UF-1162, UF-1163, and UF-1164. A comparison of these new compounds has been made by an evaluation of individual material characteristics and a tradeoff with the original LCCM compounds and standard carbon and silica cloth reinforced plastic insulators for nozzle applications. Generally, the new UF formulations show improvements over the original LCCM systems. They fall short of the carbon cloth-phenolic ablatives in most areas, but appear to be acceptable for use in large rocket nozzles on the basis of performance and low cost.

INTRODUCTION

Prior to this contract, Thiokol Chemical Corporation had developed a series of low cost carbonaceous materials (LCCM) for use in rocket motor nozzle insulation applications. The LCCM compounds are combinations of phenolic resins and graphite powders which have been cured under heat and pressure. Their erosion resistance to rocket exhausts was equivalent to carbon fiber reinforced phenolic insulations, but the structural properties did not compare favorably with conventional reinforced plastics. They had poorer mechanical properties, higher density, and greater thermal conductivity than materials such as carbon cloth-phenolic. The LCCM's were only a mixture of resin and graphite powder. It was believed that reinforcement in fiber form could be added to improve the mechanical properties, and possibly other ingredients could be added to lower the density and thermal conductivity without reducing the erosion resistance.

The objective of this program was to make the LCCM formulations more desirable for nozzle insulation applications by increasing the structural properties, decreasing the density, and reducing the thermal conductivity while maintaining the previously demonstrated erosion resistance. The program was accomplished in three phases followed by a final characterization of the formulations. The first phase was optimization of the resin-carbon formulations. The second phase was the reinforcing of the formulations using fibers. The third phase was the addition of fillers to reduce the thermal conductivity and density. A final characterization was done on the four best materials to accurately define their physical properties.

TASK 01 - BASIC FORMULATION DEVELOPMENT STUDIES

This task was conducted to optimize the resin-graphite compounds for LCCM formulations. It consisted of evaluating compounds of various resins and graphite powders.

Resin Selection

Seven resin systems, which may be classified into four types, were investigated in this task:

1. Single stage phenolic (Resol) - three resin systems
2. Two stage phenolic (novolac) - two resin systems
3. Polyphenylene - one resin system
4. Epoxy novolac - one resin system

Phenolic resins were selected for evaluation because they have a high cross link density, which tends to produce a firm char that is important in ablative insulations. Both one and two stage phenolics were selected for investigation to determine which is best for LCCM applications.

The polyphenylene resin system has fewer methylene bridges joining aromatic rings in the polymeric system than conventional phenolics do. Consequently, a higher concentration of aromatic rings occur in the polymer. This increases the total carbon content of the polyphenylene system, increases the thermal stability, and reduces the probability of chain cleavage. Therefore, they too were considered candidates for LCCM applications.

The epoxy novolac family of resins was selected for evaluation because the resins cure through an epoxy linkage, but have much of the aromatic structure of phenolic resins and approach the phenolics in char forming properties. They also have the advantage of curing at low pressure.

The basic formulation development studies were initiated with the following seven resin systems:

1. Durez 10694, a liquid phenolic resin, manufactured by Hooker Chemical Corporation } Single staged resin

- | | |
|---|-----------------------|
| 2. Durez 23362, a powder phenolic resin, manufactured by Hooker Chemical Corporation | } Single staged resin |
| 3. SC 1008, a liquid phenolic resin, manufactured by Monsanto Chemical Company | |
| 4. RI-4009, a powder phenolic resin, manufactured by Monsanto Chemical Company | } Two staged resin |
| 5. Durez 14000, a powder phenolic resin, manufactured by Hooker Chemical Corporation | |
| 6. DEN 438, epoxy novolac, manufactured by Dow Chemical Corporation. Methyl nadic anhydride and benzyl-dimethylamine were used to cure the DEN 438 resin. | |
| 7. Polyphenylene, manufactured by Ironside Chemical Company | |

A systematic test matrix was devised to investigate the potential of the seven resin systems. This test program progressed as graphically described in Figure 1. The candidate resin systems were chosen to reflect the broad spectrum of resins available; however, the initial investigation of the seven resin systems was made only to determine the three most promising binders for use in the optimization studies.

The candidate resin systems and graphite powder combinations were mixed and cured into flat panels for test specimens. The mixing was done by two methods. The first was simply to put all the ingredients (dry) into a large plastic bag and shake or knead into a homogeneous powder. The second required mixing the dry graphite powders with liquid resins in a machine such as a Hobart mixer.

Each of the material mixes had a mix ratio of one part resin to three parts graphite powder, with the exception of two epoxy novolac mixes. In these, a resin content of 12.5 percent was used to include an intermediate evaluation since the relative effects of this system as a graphite particle binder were unknown. The graphite particles used throughout the screening study were three parts Great Lakes 1008 and one part 1012 graphite powder.

These basic formulations were evaluated for tensile strength, density, compressive strength, hardness, and thermal penetration rate. In order to assist in the evaluation and subsequent rating of the seven resin systems, the test matrix was expanded to include hardness tests on the virgin material and on the char after subjection to high heat and a thermal penetration rate torch test.

Heat penetration rate was defined for this program in terms of mils/sec and was determined by measuring the time to raise the backside of a flat panel (5 by 7 in.) 0.50 in. thick (12.70 by 17.78 by 1.27 cm) to 200° F (366° K) when heated on the opposite

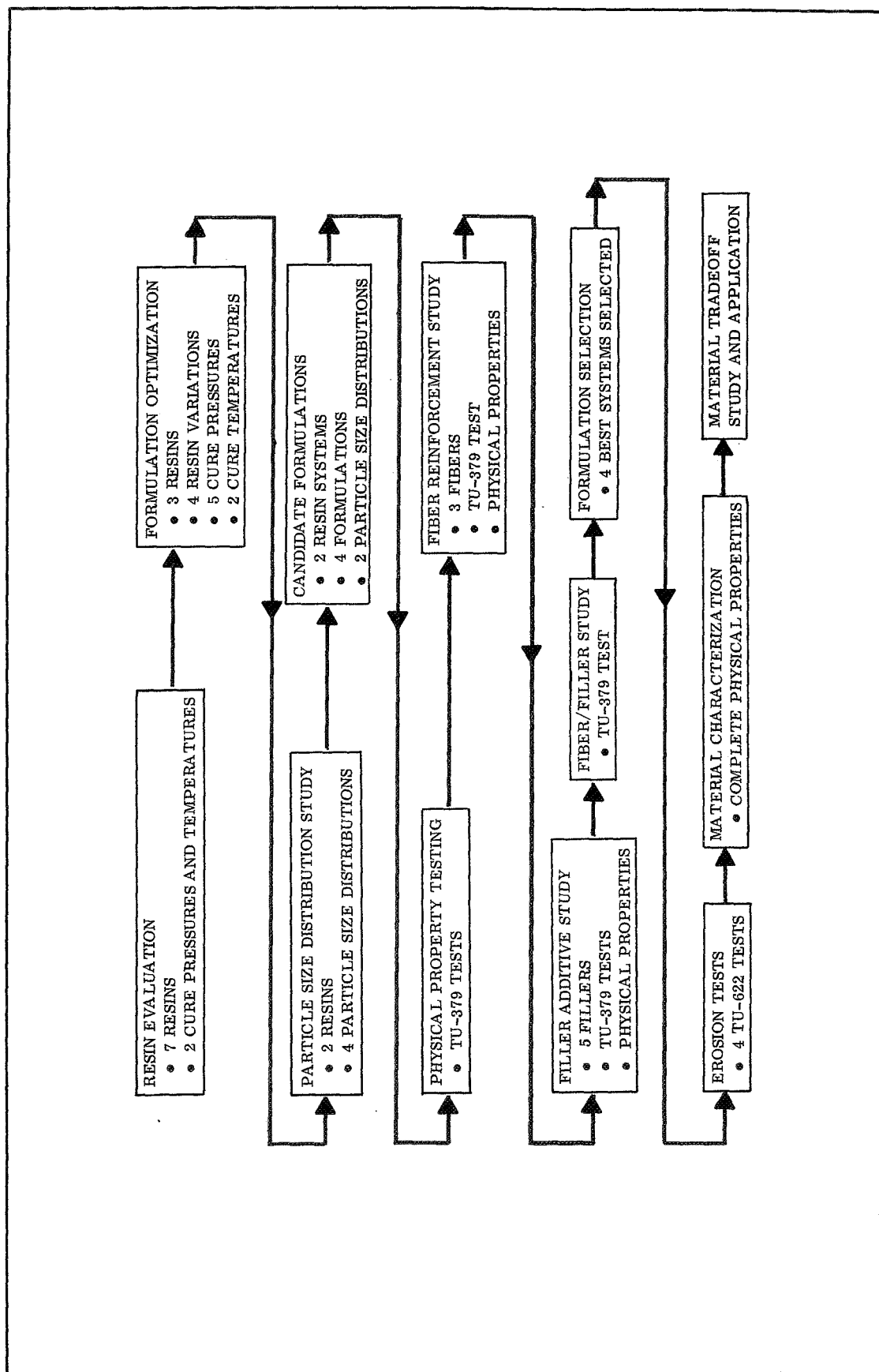


Figure 1. LCCM Progress Flow Chart

side with an oxyacetylene torch operating at a temperature in excess of 5,000°F (3,035°K).

The test panels subjected to the heat penetration rate test were segmented, and the heat affected zones were examined for spalling, cracking, and char hardness. Although the thermal penetration test does not measure the ablative properties of the material, it does indicate how the material will react when subjected to a high heat source.

The Monsanto RI-4009, a two stage powder phenolic resin, was selected over the single stage powdered phenolic, Durez 23362, since the RI-4009 retained 20 percent higher tensile strength and exhibited only moderate spalling when exposed to the torch test. Based on test results, RI-4009 showed higher material structural strengths when cured at high pressure (1,000 psi or 6.9×10^6 N/m²) and temperature (325° F or 436°K).

Durez 10694, a single stage liquid phenolic resin, was selected over the other candidate systems for a number of reasons. It produced castable or trowelable LCCM compounds which cured at low temperature (170° F or 350°K) and low pressure (15 psi or 1.03×10^5 N/m²). In addition, this resin does not require "staging" or resin advancement as does the SC 1008 liquid single stage and the polyphenylene resins. Although the physical properties are lower than the two stage phenolic resins, it exhibits good char and spalling characteristics as indicated by the torch test.

DEN 438 epoxy novolac was selected for follow-on formulation optimization study based upon the advantages this resin offers in respect to low cost large nozzle component application; i.e., it is castable or trowelable, does not require resin advancement, and is a low temperature/pressure cure and non-condensation type polymer. In addition, it has moderate physical and good char and spalling properties.

Based on the test results as shown in Table I, the following three resin systems were selected for evaluation in the formulation optimization phase of the basic formulation development study:

1. RI-4009 (Monsanto Chemical Company)
2. Durez 10694 (Hooker Chemical Corporation)
3. DEN 438 epoxy novolac (Dow Chemical Corporation)

TABLE 1. - LCM DEVELOPMENT SCREENING TEST-TASK 01

Beam Identity	Mix No.	Processing Requirements	Cure Requirements			Tensile Strength		Modulus		Compressive Strength		Density (g/cc)	Hardness Shore D	Penetration Rate (in/hr/sec)	Hardness of Cure After Torch Test Shore D	Torch Test Thickness Dimensional Change		Remarks
			Time (hr.)	Pressure (psi)	Temperature (°F)	(psi)	(N/m ² x 10 ⁶)	(psi x 10 ⁶)	(N/m ² x 10 ⁶)	(psi)	(N/m ² x 10 ⁶)					(in.)	(mm.)	
DUREZ 23362	418	Bag mixing	--	1,000	6.9 x 10 ⁶	325	436	2.68	14.35	14,480	99.91	1.77	80	36.2	50-65	+0.073	1.8642	Moderate to heavy spalling. Good char retention.
One Stage Resin, 25%	421	Bag mixing	--	15	1.03 x 10 ⁶	325	436	2.115	14.59	7,830	54.02	1.43	75	32.0	65	+0.009	0.2286	Slight to moderate spalling.
25% Resin	420	Bag mixing	--	1,000	6.9 x 10 ⁶	170	360	*	--	4,646	32.95	1.48	75	17.3	35	--	--	Heavy spalling.
POLYPHENYLENE Based on 60% Resin in Solution, 25% Resin	422-1 422-2 422-3	Machine mix stage 4 hr 170° F (350° F)	--	1,000	6.9 x 10 ⁶	325	436	--	--	11,996	82.77	1.81	79	58.0	45-60	+0.007	0.1778	Moderate spalling. Good char retention.
			--	15	1.03 x 10 ⁶	395	436	0.516	4.25	4,640	31.91	1.53	63	52.0	33-40	+0.002	0.0508	No noticeable spalling. Good char retention.
			--	1,000	6.9 x 10 ⁶	170	360	--	--	*	--	--	64	Crumbled	20	--	--	Test specimens crumbled. Structurally unstable.
MONSANTO RI-4009 Two Stage Phenolic, 25% Resin	423	Bag mixing	--	1,000	6.9 x 10 ⁶	325	436	1.12	7.72	12,126	83.68	1.75	81	51.5	57	+0.070	1.778	Moderate spalling.
	424	Bag mixing	--	1,000	6.9 x 10 ⁶	170	360	*	--	*	--	*	38	Crumbled	25	--	--	Test specimen crumbled. Structurally unstable.
	425	Bag mixing	--	15	1.03 x 10 ⁶	325	436	0.624	4.37	5,606	38.68	1.37	70	31.4	50-60	+0.002	0.0508	Slight spalling. Good char retention.
MONSANTO RI-4009 Alcohol, 25% Resin	430-1	Machine mixing	--	15	1.03 x 10 ⁶	325	436	2.97	14.27	4,160	28.70	1.48	65	37	44	-0.003	-0.0723	Moderate spalling.
	440	Machine mixing	--	200	1.38 x 10 ⁶	325	436	--	--	7,666	52.89	1.48	72	N/A - malfunction of recorder	55	+0.060	1.27	Moderate spalling.
DEN 438 EPOXY NOVOLAC, 24.5% Resin	432	Machine mixing	2	300	2.97 x 10 ⁶	200	366	--	--	--	--	--	50	Cracked	--	--	--	Specimen cracked during removal from mold.
	436	Machine mixing	15	15	1.03 x 10 ⁶	200	366	6.25	2.26	5,100	35.19	1.55	68	37	40	+0.038	0.969	None.
DUREZ 10094 One Stage Phenolic, 25% Resin	443	Machine mixing	--	15	1.03 x 10 ⁶	170	350	0.75	5.17	8,185	56.47	1.59	75	61	58	+0.018	0.4372	No spalling.
	444	Machine mixing	--	200	1.38 x 10 ⁶	300	422	1.66	11.45	7,968	54.97	1.64	78	49	60	+0.050	1.27	Moderate spalling.
MONSANTO SC-1009 Phenolic, 25% Resin	445	4 hr stage	--	1,000	6.9 x 10 ⁶	320	433	--	--	11,725	80.89	1.81	80	49	55	+0.070	1.778	Heavy spalling.
	446	4 hr stage	--	15	1.03 x 10 ⁶	320	433	1.02	7.04	6,250	43.13	1.55	70	74	60	+0.007	0.1778	No spalling.
DUREZ 14000 Two Stage Phenolic, 25% Resin	486	Machine mixing	--	1,000	6.9 x 10 ⁶	300	422	--	--	9,115	62.89	1.79	--	--	--	--	--	--
	487	Machine mixing	--	15	1.03 x 10 ⁶	300	422	--	--	6,887	47.48	1.40	--	--	--	--	--	--

*Test specimens structurally unsuitable for testing.

--Test data not available.

Formulation Optimization Studies

The three resin systems from the Resin Selection subtask were combined with graphite powders and cured at various temperatures and pressures. The relationship between temperature, pressure, and resin-graphite ratio was evaluated. Physical, structural, and thermal characteristics of these composites were measured.

Tables II, III, and IV reflect the program optimization phase test matrices for the three resin systems that were optimized, Monsanto RI-4009, Durez 10694 and DEN 438 epoxy novolac. The material performance and the evaluations of the developed formulations involving the three resin systems are discussed below.

Monsanto RI-4009 Resin. - Monsanto's RI-4009 resin is a fine grind amber powder. To determine the optimum cure - pressure - resin - graphite combinations, four resin content variations at five cure pressures and at two cure temperatures were tested.

The Monsanto RI-4009 resin levels were evaluated at 20, 25, 30, and 40 percent and at pressures of 200, 500, 800, 1,000, and 2,000 psi, respectively. Flat test panels 12 by 12 by 0.50 in. (30.48 by 30.48 by 1.27 cm) were fabricated with the various percents of resin and graphite powder. The ratio of graphite particles was kept constant: three parts of GL-1008 and one part GL-1012 graphite. Test panels were cured 6 hours.

Tensile, compression and density specimens were machined from the cured panels. In addition, a 5 by 7 by 0.5 in. (12.70 by 17.78 by 1.27 cm) test sample, cut from the original panels, was heated on one side using an oxyacetylene torch operating in excess of 5,000°F (3,035°K). The time to heat the backside of the 0.50 in. (1.27 cm) thick sample to 200°F (366°K) was recorded. Upon cooling, these torch test panels were segmented and the heat affected zones were examined and photographed.

The tensile strength (Figure 2) appears to be at maximum at a cure pressure of 1,000 psi (6.9×10^6 N/m²), followed by a reduction in tensile strength when cured at 2,000 psi (13.8×10^6 N/m²) for each of the resin percentages evaluated. However, when comparing the compressive strength/cure pressure relationship (Figure 3), the compression data peaks out at cure pressure of 2,000 psi (13.8×10^6 N/m²). The compression data (Figure 3) ascends at a cure pressure of 500 psi (3.45×10^6 N/m²) and 800 psi (5.52×10^6 N/m²) (30 and 40 percent resin) and then subsides at 800 psi (5.52×10^6 N/m²) and 1,000 psi (6.9×10^6 N/m²) (30 and 40 percent resin) followed by another ascension at a cure pressure of 2,000 psi (13.8×10^6 N/m²).

TABLE II. - RI-4009 RESIN TEST MATRIX,
FORMULATION OPTIMIZATION STUDIES

Variables

Temperature		Pressure		Resin Content
(° F)	(° K)	(psi)	(N/m ² x 10 ⁶)	(%)
300	422	2, 000	13. 8	20
225	380	1, 000	6. 9	25
		800	5. 52	30
		500	3. 45	40
		200	1. 38	

Number of formulations 40

Test panel size 12 by 12 by 0. 5 in. (30. 48 by 30. 48 by 1. 27 cm)

Number of test panels 40

Tests

Tensile strength	3*
Density	3
Compression	3
Tensile modulus	3
Thermal (torch test)	1
Hardness (virgin and char)	1

*Three tests per formulation

TABLE III. - DUREZ 10694 TEST MATRIX,
FORMULATION OPTIMIZATION STUDIES

Variables

<u>Temperature</u>		<u>Pressure</u>		<u>Resin Content</u>
<u>(° F)</u>	<u>(° K)</u>	<u>(psi)</u>	<u>(N/m² x 10⁶)</u>	<u>(%)</u>
175	352	0		20
300	422	15	0.103	25
		100	0.69	30
		200	1.38	40
Number of formulations		32		
Test panel size		5 by 10 by 1 in. (12.7 by 25.4 by 2.54 cm)		
Number of test panels		64		
Tests				
Tensile strength		3*		
Density		3		
Compression		3		
Tensile modulus		3		
Thermal (torch test)		1		
Hardness (virgin and char)		1		

*Three tests per formulation

TABLE IV. - DEN 438 EPOXY NOVOLAC TEST MATRIX,
FORMULATION OPTIMIZATION STUDIES

Variables

Temperature		Cure Time	Pressure		Resin Content
(°F)	(°K)		(psi)	(N/m ² x 10 ⁵)	
200	366	2	0	0	20
					25
300	422	15	15	1.03	30
					40

Curing agents

Nadic methyl anhydride	87.5 (PHR)
BDMA	1.5 (PHR)

Number of formulations 8

Test panel size 5 by 10 by 1 in. (12.7 by 25.4 by 2.54 cm)

Number of test panels 16

Tests

Tensile strength	3
Density	3
Compression	3
Tensile modulus	3
Thermal (torch test)	1
Hardness (virgin and char)	1

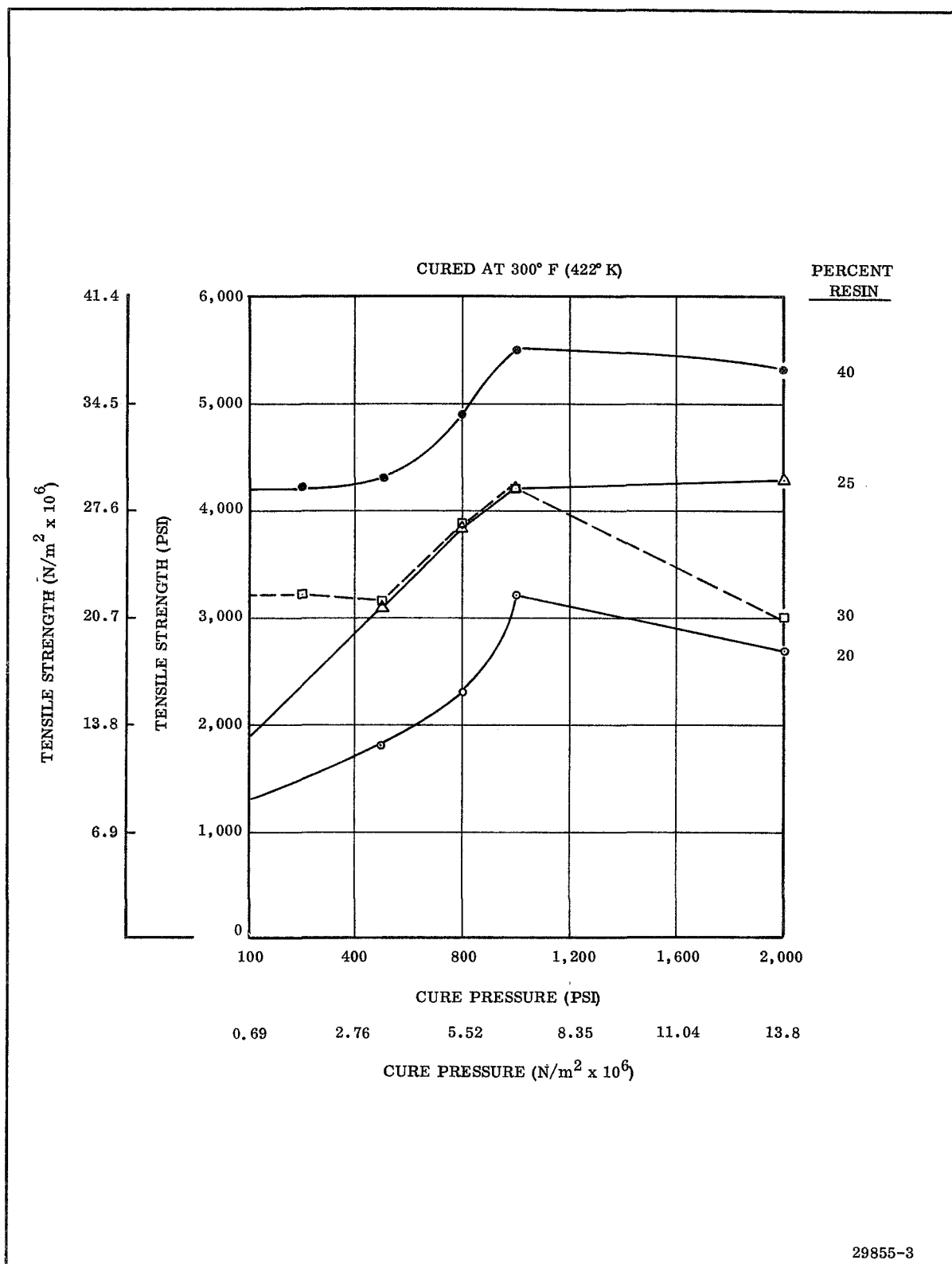


Figure 2. - Tensile Strength of RI-4009 Resin/Graphite Powder

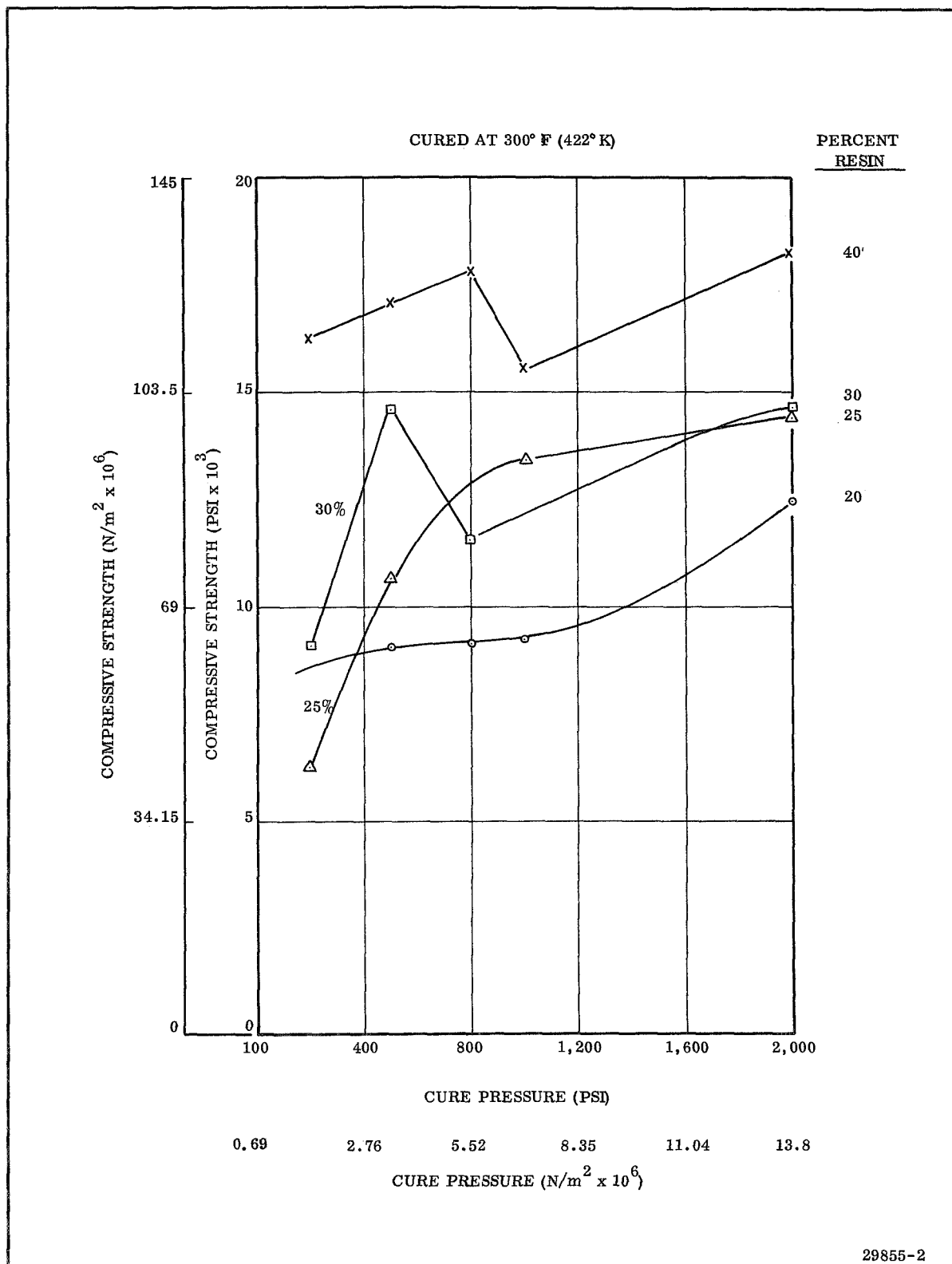


Figure 3. - Compressive Strength of RI-4009 Resin/Graphite Powder

Test results of tensile strength, tensile modulus, density, compression strength, thermal properties, and hardness test are shown in Table V. Figures 2 and 3 show the cure pressure vs tensile and compressive strength relationship of the various resin contents. Figures 4 thru 12 are photographs of the segmented torch test samples and show test results of the heat affected zones.

The overall visual effects of the segmented test samples show less thermal cracking at the lower cure pressures (500 psi, $3.45 \times 10^6 \text{ N/m}^2$) and at the lower resin content (20 percent). However, a firm char was predominant at the higher cure pressures (800 to 1,000 psi, $5.52 \times 10^6 \text{ N/m}^2$ to $6.9 \times 10^6 \text{ N/m}^2$).

As reflected in Figure 2, the tensile strength of each resin level increases abruptly at a cure pressure of 1,000 psi ($6.9 \times 10^6 \text{ N/m}^2$). In order to further determine the maximum progressive strength at this cure pressure, flat test panels (12 by 12 by 0.5 in. or 30.48 by 30.48 by 1.27 cm) were fabricated with 45 and 50 percent RI-4009 resin content, respectively, and cured at 300°F (422°K). Test panels were machined for tensile and compressive strength samples. These properties are presented below:

Mix No.	Tensile Strength		Density (g/cc)	Compressive Strength		Thermal Penetration (mils/sec)	Hardness Shore D	
	(psi)	($\text{N/m}^2 \times 10^6$)		(psi)	($\text{N/m}^2 \times 10^6$)		Virgin	Char
449 (40% resin)	5,370	37.05	1.66	16,960	117.0	18.8	84	45
	5,750	39.67	1.66	14,440	99.6			
	5,450	37.60	1.66	14,940	103.0			
522 (45% resin)	5,675	39.15	1.61	21,060	145.3	18.7	86	35
	5,375	37.08	1.61	20,460	141.2			
	4,710	32.49	1.61	20,540	141.7			
523 (50% resin)	5,125	35.36	1.57	22,300	153.8	16.0	86	35
	5,675	39.15	1.57	22,500	155.2			
	5,850	40.36	1.57	22,550	155.5			

In comparing the properties of material formulations containing 40, 45, and 50 percent resin, the test data indicate that increasing the formulation resin content beyond 40 percent has little effect on tensile strength, it results in approximately a 25 percent increase in compressive strength, and it has a degrading effect on the performance of the material when exposed to high temperature environments (see Figure 13).

It was observed during the optimization studies that a sample containing 25 percent of RI-4009 resin and blended with 75 percent mixture of graphite and cured at 200 psi ($1.38 \times 10^6 \text{ N/m}^2$) and at 300°F (422°K) offered a better visual heat affected zone than a mixture containing a 40 to 60 percent mixture of resin-graphite and cured at 1,000 psi ($6.9 \times 10^6 \text{ N/m}^2$) and 300°F (422°K).

TABLE V. - LCCM DEVELOPMENT OPTIMIZATION STUDY OF MONSANTO RI-4008 RESIN

Mix No.	Percent Resin	Percent Graphite GL-1008	Time (hr)	Cure Requirements			Tensile Strength (psi)	Tensile Modulus (psi x 10 ⁶)	Density (g/cc)	Compressive Strength (psi)	Penetration Rate (mils/sec)	Hardness Shore VIRHM	Chart						
				Pressure (psi)	Temperature (°F)	Temperature (°K)													
423	25	56.25	18.75	4	1,000	6.9	300	422	3,940	27.16	1.14	7.86	1.76	12,400	85.49	51.5	130.8	81	57
							4,390	30.26	1.10	7.58	1.75	11,820	81.49	1.75	11,820	81.49			
							4,740	32.68	1.12	7.72	1.75	12,160	83.84	1.75	12,160	83.84			
							4,356	30.03	1.12	7.72	1.75	12,126	83.60	1.75	12,126	83.60			
447	20	60	20	5	1,000	6.9	300	422	3,620	24.95	2.24	15.44	1.78	9,525	65.87	37.4	94.9	80	57
							3,475	23.95	2.26	15.58	1.76	9,650	66.53	1.76	9,650	66.53			
							2,840	19.58	2.28	15.72	1.79	8,150	56.19	1.79	8,150	56.19			
							3,311	22.82	2.26	15.58	1.77	9,108	62.79	1.77	9,108	62.79			
448	30	52.5	17.5	5	1,000	6.9	300	422	3,540	24.40	2.12	14.61	1.75	12,250	84.46	25.2	64.0	83	55
							4,470	30.81	2.12	14.61	1.75	11,900	82.04	1.75	11,900	82.04			
							5,060	34.68	2.14	14.75	1.76	12,300	84.80	1.76	12,300	84.80			
							4,356	30.03	2.13	14.65	1.75	12,150	83.77	1.75	12,150	83.77			
449	40	45	15	5	1,000	6.9	300	422	3,370	37.02	1.96	13.51	1.66	16,960	116.93	18.8	47.8	84	45
							5,750	39.64	1.93	13.30	1.66	14,440	99.56	1.66	14,440	99.56			
							5,450	37.57	1.94	13.37	1.66	14,940	103.00	1.66	14,940	103.00			
							5,523	38.07	1.94	13.39	1.66	15,446	106.49	1.66	15,446	106.49			
450	20	60	20	15	1,000	6.9	225	380	2,645	18.23	1.78	12.27	1.69	8,650	59.64	37.4	94.9	77	35
							1,750	12.06	1.78	12.27	1.69	7,560	52.12	1.69	7,560	52.12			
							2,255	15.54	1.79	12.34	1.70	8,450	55.33	1.70	8,450	55.33			
							2,216	15.27	1.78	12.29	1.69	8,223	56.69	1.69	8,223	56.69			
451	25	56.25	18.75	15	1,000	6.9	225	380	2,500	17.23	2.47	17.03	1.76	11,400	78.60	23.0	58.3	78	42
							2,255	15.54	2.63	18.13	1.65	12,375	85.32	1.65	12,375	85.32			
							2,415	16.65	2.47	17.03	1.78	18,375	126.69	1.78	18,375	126.69			
							2,390	16.45	2.52	17.39	1.73	14,050	96.87	1.73	14,050	96.87			
452	30	52.50	17.50	15	1,000	6.9	225	380	2,545	17.54	1.89	13.03	1.80	6,975	48.09	23.7	60.2	79	35
							2,500	17.23	1.89	13.03	1.71	9,375	64.63	1.71	9,375	64.63			
							3,155	21.75	1.89	13.03	1.66	12,752	87.92	1.66	12,752	87.92			
							2,733	18.84	1.89	13.03	1.72	9,700	66.86	1.72	9,700	66.86			
453	40	45.00	15.00	15	1,000	6.9	225	380	2,395	16.51	1.93	13.30	1.66	12,600	86.87	13.4	34.1	80	33
							2,675	18.44	2.02	13.92	1.66	13,650	94.11	1.66	13,650	94.11			
							2,890	19.92	2.02	13.92	1.67	12,275	84.63	1.67	12,275	84.63			
							2,653	18.29	1.99	13.71	1.66	12,841	88.53	1.66	12,841	88.53			
454	20	60.00	20.00	5	800	5.52	300	422	2,500	17.23	1.50	10.34	1.68	9,550	65.84	18.8	47.8	75	59
							2,295	15.82	1.52	10.48	1.75	8,790	60.60	1.75	8,790	60.60			
							1,955	13.68	1.52	10.48	1.69	8,460	58.32	1.69	8,460	58.32			
							2,260	15.57	1.51	10.43	1.71	8,933	61.59	1.71	8,933	61.59			
455	25	56.25	18.75	5	800	5.52	300	422	4,106	28.30	2.46	16.96	1.77	13,090	90.25	35.5	90.1	75	55
							3,800	26.20	2.38	16.40	1.78	13,850	96.49	1.78	13,850	96.49			
							4,230	29.16	2.46	16.96	1.74	---	---	1.74	---	---			
							4,045	27.88	2.43	16.77	1.76	13,470	92.87	1.76	13,470	92.87			

TABLE V. - LCCM DEVELOPMENT OPTIMIZATION STUDY OF MONSANTO RI-4009 RESIN (CONT)

Mix No.	Percent Resin	Percent Graphite GL-1003	Time (hr)	Cure Requirements		Tensile Strength (psi)	Tensile Modulus (psi x 10 ⁵)	Density (g/cc)	Compressive Strength (psi)	Penetration Rate (mils/sec)	Hardness Shore D Virgin	Char
				Pressure (psi)	Temperature (°F)							
456	30	52.5	17.5	5	300	422	15.72	1.74	11,500	30.9	82	53
						3,965	27.33	1.74	11,500	78.4		
						4,230	29.16	1.74	11,725			
						3,710	25.57	1.74	11,100			
						3,968	27.35	1.74	11,441			
457	40	45.0	15.0	5	800	422	14.62	1.65	18,200	15.3	83	50
						5,000	34.47	1.66	17,250			
						4,960	34.20	1.68	---			
						4,943	34.08	1.66	17,725			
458	20	60.0	20.0	10	800	380	13.65	1.67	8,660	39.4	73	48
						1,182	8.15	1.67	8,660			
						1,505	10.37	1.75	9,250			
						1,208	8.33	1.74	9,740			
						1,298	8.95	1.72	9,283			
459	25	56.25	18.75	10	800	380	12.68	1.76	9,475	21.3	76	33
						---	---	1.75	13,125			
						---	---	1.76	12,800			
						860	5.93	1.76	11,803			
460	30	52.5	17.50	10	800	380	14.20	1.74	12,050	20.5	76	33
						1,390	9.58	1.74	12,050			
						649	4.47	1.74	12,650			
						1,265	8.72	1.74	15,480			
						1,101	7.59	1.74	13,393			
461	40	45.0	15.00	10	800	380	13.65	1.65	12,400	12.9	77	56
						SAMPLES BROKE	---	1.65	12,400			
						UPON TESTING	---	1.65	13,210			
						---	---	1.65	13,325			
						---	---	1.65	12,978			
462	20	60.0	20.00	4	500	422	9.37	1.64	8,840	39.6	74	61
						1,615	11.13	1.64	8,840			
						2,235	15.40	1.64	9,125			
						---	---	1.64	9,075			
						1,925	13.27	1.64	9,013			
463	25	56.25	18.75	4	500	422	13.10	1.69	19,750	39.0	77	N/A
						3,280	22.61	1.69	19,750			
						3,045	20.99	1.70	12,025			
						2,825	19.47	1.65	12,150			
						3,050	21.02	1.68	14,641			
464	30	52.50	17.50	4	500	422	15.16	1.73	14,700	30.2	82	57
						3,350	23.09	1.73	14,700			
						3,025	20.85	1.73	15,060			
						3,005	20.71	1.73	14,525			
						3,126	21.65	1.73	14,758			
465	40	45.00	15.00	4	500	422	13.03	1.66	19,050	16.9	80	39
						5,376	37.06	1.66	19,050			
						3,845	26.51	1.70	19,350			
						4,335	29.89	1.65	12,425			
						4,515	31.15	1.67	16,941			

TABLE V. - LCCM DEVELOPMENT OPTIMIZATION STUDY OF MONSANTO RI-4009 RESIN (CONT)

Mix No.	Percent Resin	Percent Graphite GL-1008	Time (hr)	Cure Requirements			Tensile Strength (psi)	Tensile Modulus (psi x 10 ⁶)	Density (g/cc)	Compressive Strength (psi)	Penetration Rate (mil/sec)	Penetration Rate (m/sec x 10 ⁻⁵)	Hardness Shore D	
				Pressure (psi)	Temperature (°F)	Temperature (°K)							Virgin	Char
466	20	60.0	20.0	10	500	225	380	2.00	13.78	1.63	7.575	52.22	71	50
								2.00	13.78	1.65	8.150	56.19		
								2.02	13.92	1.67	8.900	61.36		
								2.00	13.78	1.65	8.208	56.59		
467	25	56.25	18.75	10	500	225	380	1.92	13.23	1.65	12.790	88.18	75	45
								1.92	13.23	1.74	15.110	10.41		
								---	---	1.74	15.350	10.58		
								1.92	13.23	1.71	14.416	99.39		
468	30	52.50	17.50	10	500	225	380	2.64	18.20	1.74	10.400	71.70	76	38
								2.62	18.06	1.73	9.575	66.01		
								2.63	18.13	1.74	9.987	68.85		
469	40	45.0	15.00	10	500	225	380	2.22	15.30	1.66	11.370	78.39	80	35
								2.22	15.20	1.66	11.300	78.11		
								---	---	1.66	10.000	98.94		
								2.22	15.30	1.66	10.900	75.15		
470	20	60.0	20.0	4	200	300	422	1.75	12.06	1.53	6.525	44.98	62	57
471	25	56.25	18.75	4	200	300	422	1.75	12.06	1.51	6.400	44.12	71	65
								1.75	12.06	1.48	6.400	44.12		
								1.75	12.06	1.50	6.441	44.40		
472	30	52.50	17.5	4	200	300	422	1.68	11.58	1.54	9.240	63.70	75	38
								1.49	10.27	1.56	9.740	67.15		
								1.49	10.27	1.51	8.520	58.74		
								1.55	10.68	1.54	9.166	63.19		
473	40	45.00	15.0	4	200	300	422	1.88	12.96	1.64	16.700	115.14	82	57
								1.86	12.82	1.64	16.200	111.69		
								1.91	13.16	1.65	15.800	108.93		
								1.88	12.96	1.64	16.233	108.93		
474	20	60.00	20.0	10	200	225	380	1.90	13.10	1.43	4.070	28.06	65	60
								1.88	12.96	1.44	4.550	31.37		
								---	---	1.43	4.580	31.57		
								1.89	13.03	1.43	4.383	30.21		
475	25	56.25	18.75	10	200	225	380	1.45	9.99	1.59	7.720	53.22	72	55
								1.45	9.99	1.55	6.550	45.16		
								1.45	9.99	1.57	7.133	49.18		
								1.45	9.99	1.57	7.133	49.18		
476	30	52.5	17.50	10	200	225	380	1.74	11.99	1.73	10.660	73.49	76	44
								1.72	11.85	1.68	11.625	80.15		
								1.75	12.06	1.67	11.800	81.35		
								1.74	11.99	1.70	11.361	78.33		

TEST SAMPLES NOT AVAILABLE

TABLE V. - ICCM DEVELOPMENT OPTIMIZATION STUDY OF MONSANTO RI-4009 RESIN (CONT)

Mix No.	Percent Resin	Percent Graphite GL-1008	Percent Graphite GL-1012	Cure Requirements				Tensile Strength (psi)	Tensile Modulus (psi x 10 ⁶)	Density (g/cc)	Compressive Strength		Penetration Rate		Hardness Shore D	
				Time (hr)	Pressure (psi)	Temperature (°F)	Temperature (°K)				(psi)	(N/m ² x 10 ⁶)	(mils/sec)	(m/sec x 10 ⁻⁵)	Virgin	Char
477	40	45.0	15.00	10	200	1.38	225	380								
								1,905	1.88	1.59	7,180	49.50	15.4	38.8	77	54
								2,270	1.90	1.64	13,000	89.63				
								2,065	1.91	1.65	13,800	95.14				
								2,080	1.89	1.63	11,326	78.09				
478	20	60.0	20.00	4	2,000	13.8	300	422								
								3,100	2.33	1.86	12,485	85.66	43.5	110.5	80	58
								2,155	2.33	1.87	12,000	82.73				
								3,055	2.33	1.87	12,425	85.66				
								2,770	2.33	1.87	12,283	84.68				
479	25	56.25	17.50	4	2,000	13.8	300	422								
								4,580	2.19	1.82	15,050	103.76	32.1	81.5	80	56
								3,800	2.19	1.83	14,125	97.38				
								4,465	2.21	1.82	13,950	96.18				
								4,285	2.20	1.82	14,375	99.11				
480	30	52.5	17.50	4	2,000	13.8	300	422								
								4,000	2.16	1.77	14,650	101.00	25.0	63.6	77	59
								1,479	2.19	1.77	14,150	97.56				
								3,400	2.18	1.77	14,775	101.87				
								2,959	2.17	1.77	14,525	100.14				
481	40	45.00	15.00	4	2,000	13.8	300	422								
								5,770	2.06	1.68	21,375	147.37	15.9	40.3	84	53
								4,920	2.07	1.68	17,375	119.79				
								5,040	2.07	1.68	17,250	118.93				
								5,243	2.07	1.68	18,666	128.69				

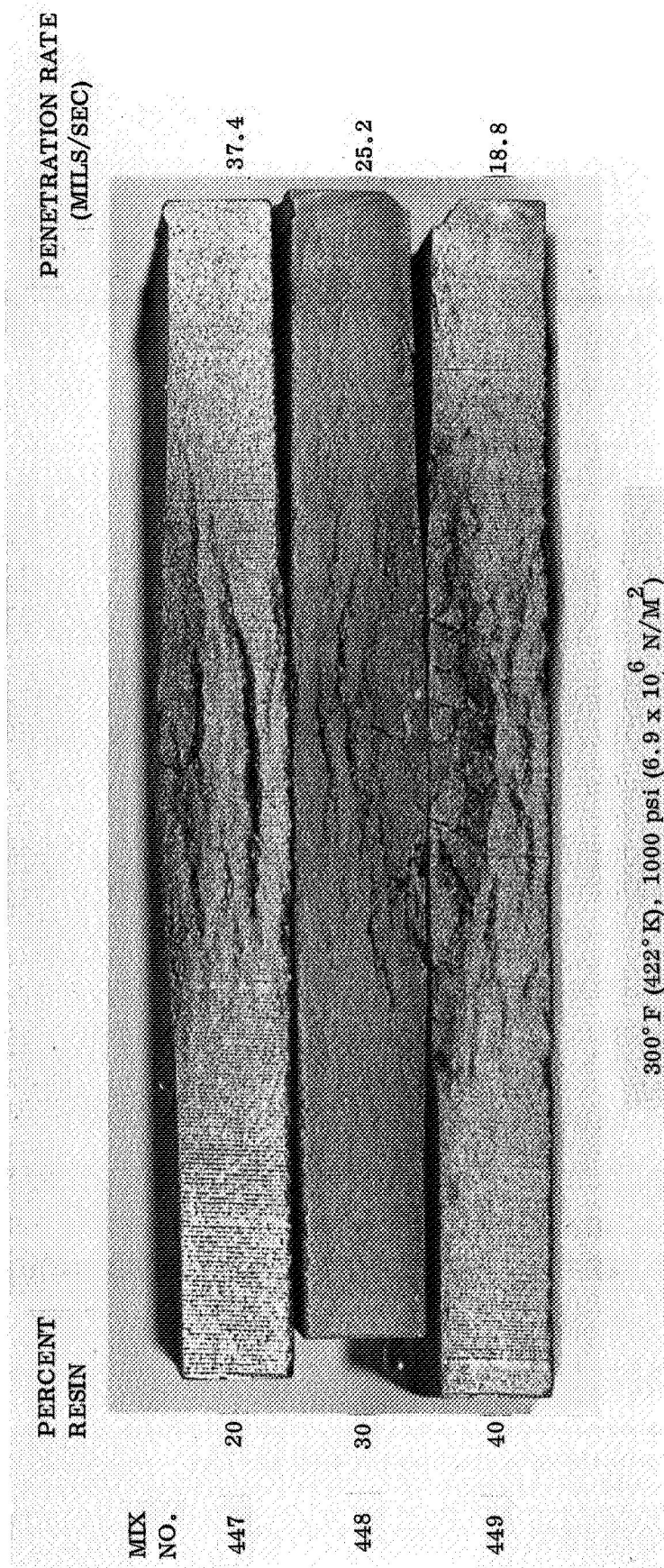


Figure 4.- Torch Test Samples, RI-4009 Resin/Graphite Composite, Mixes 447 thru 449

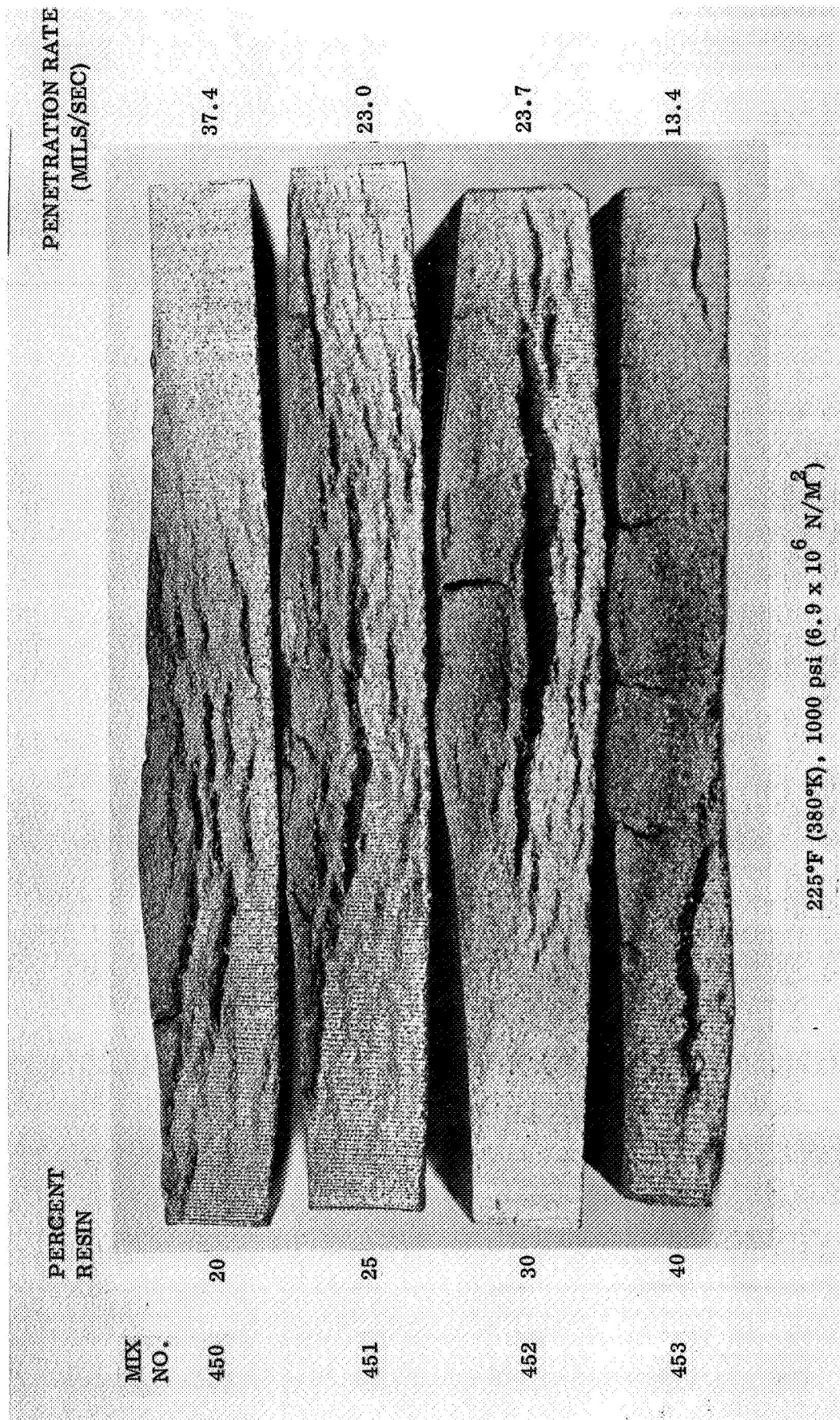


Figure 5.- Torch Test Samples, RI-4009 Resin/Graphite Composite, Mixes 450 thru 453

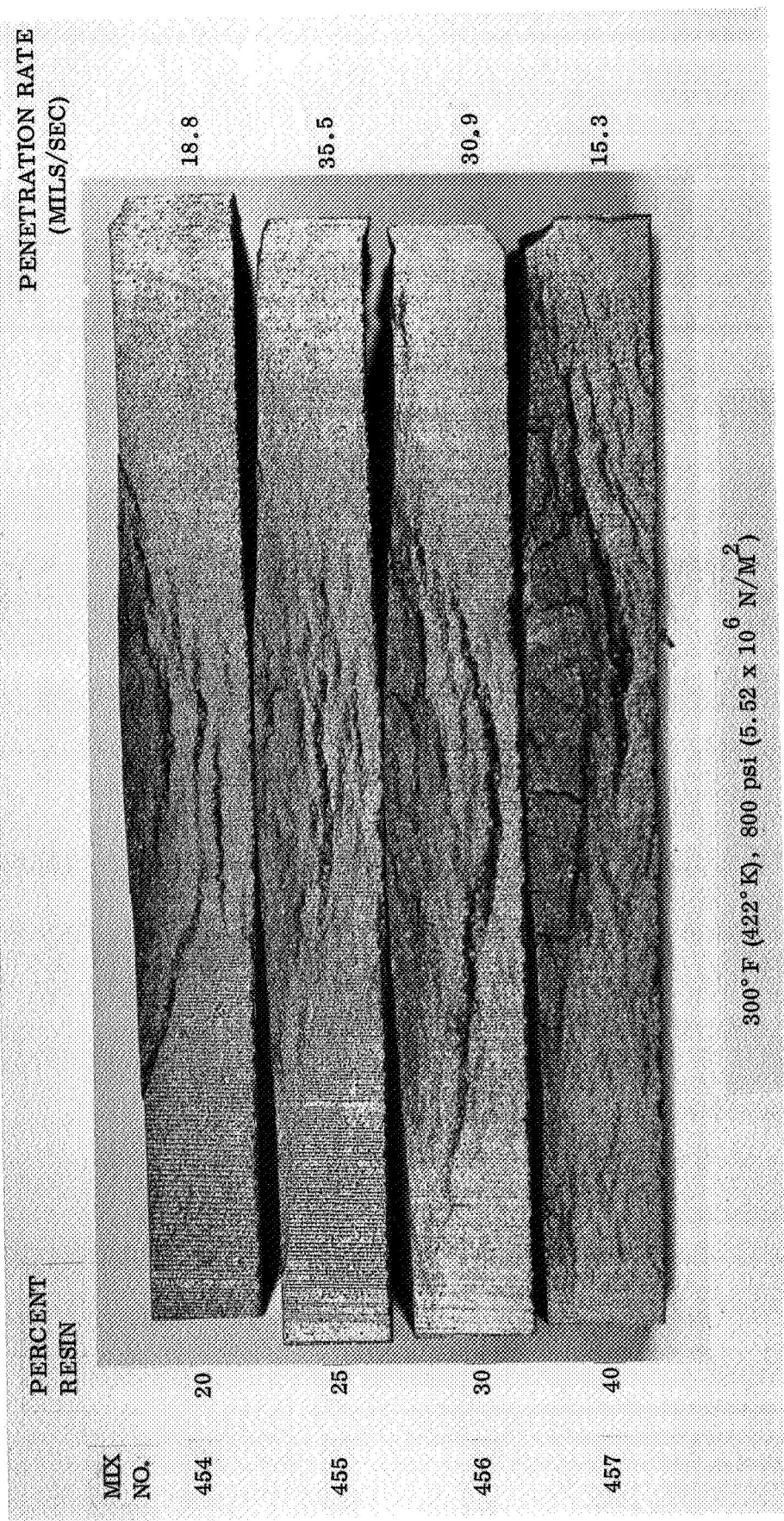


Figure 6.- Torch Test Samples, RI-4009 Resin/Graphite Composite, Mixes 454 thru 457

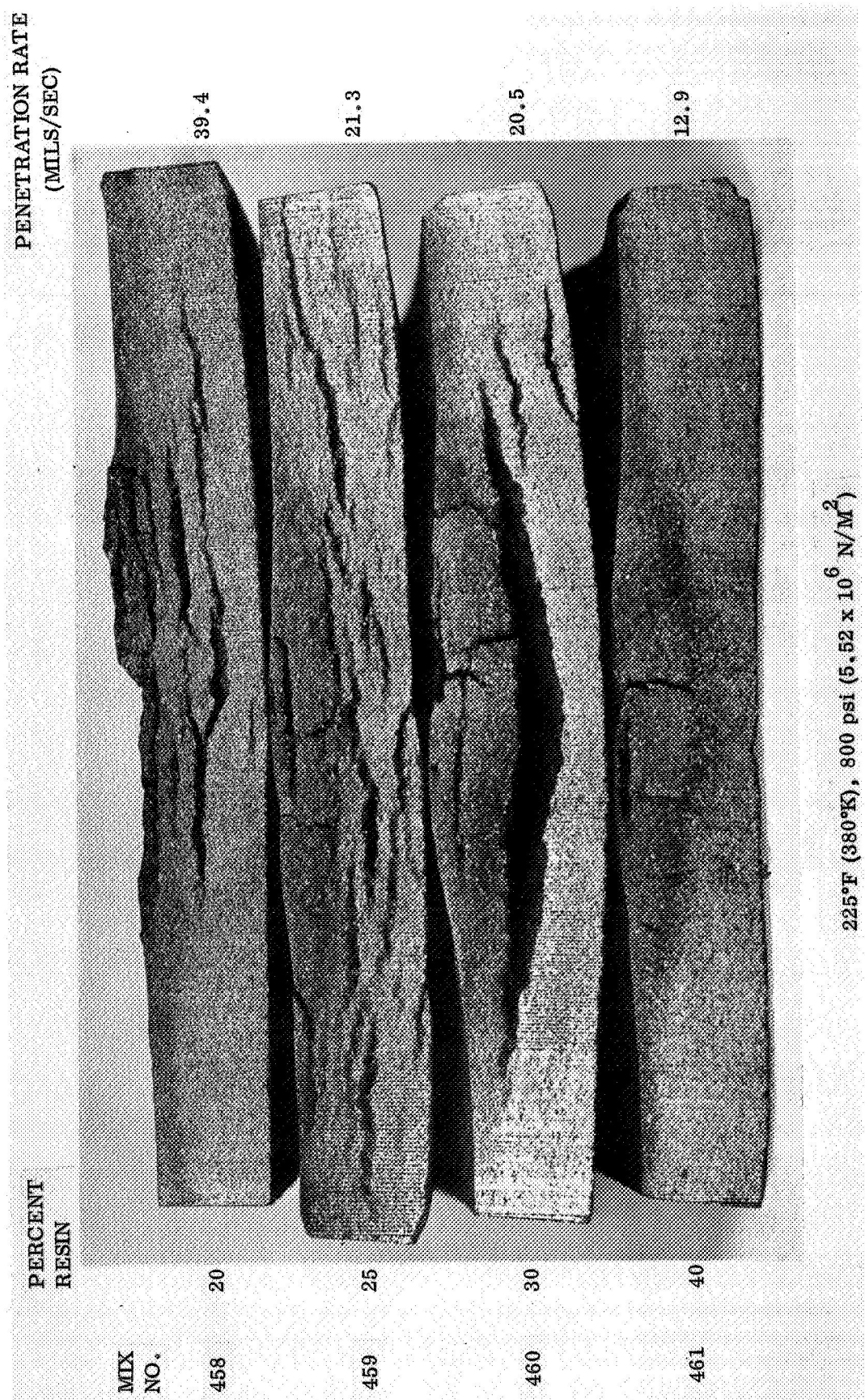


Figure 7. - Torch Test Samples, RI-4009 Resin/Graphite Composite, Mixes 458 thru 461

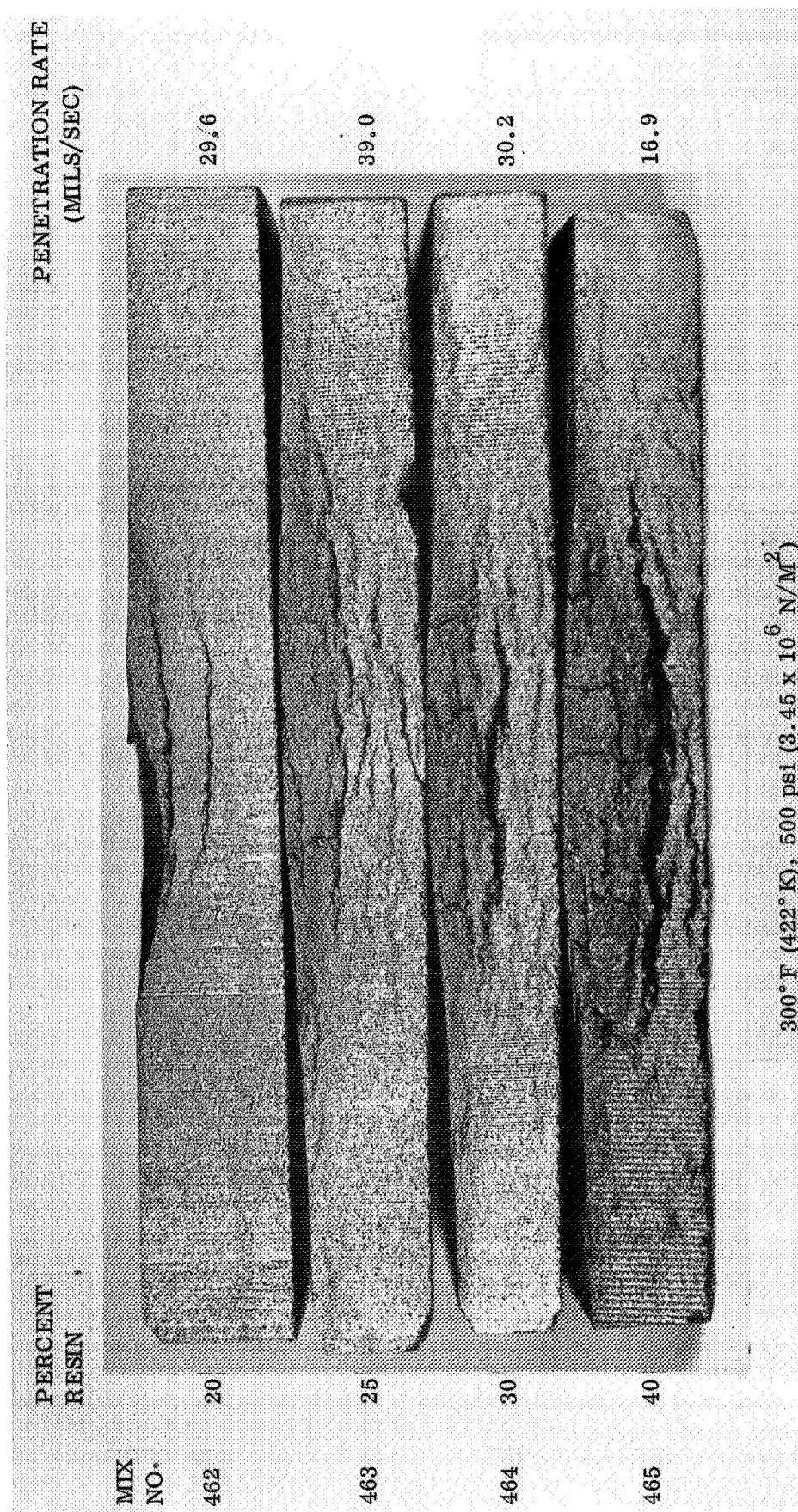


Figure 8. - Torch Test Samples, RI-4009 Resin/Graphite Composite, Mixes 462 thru 465

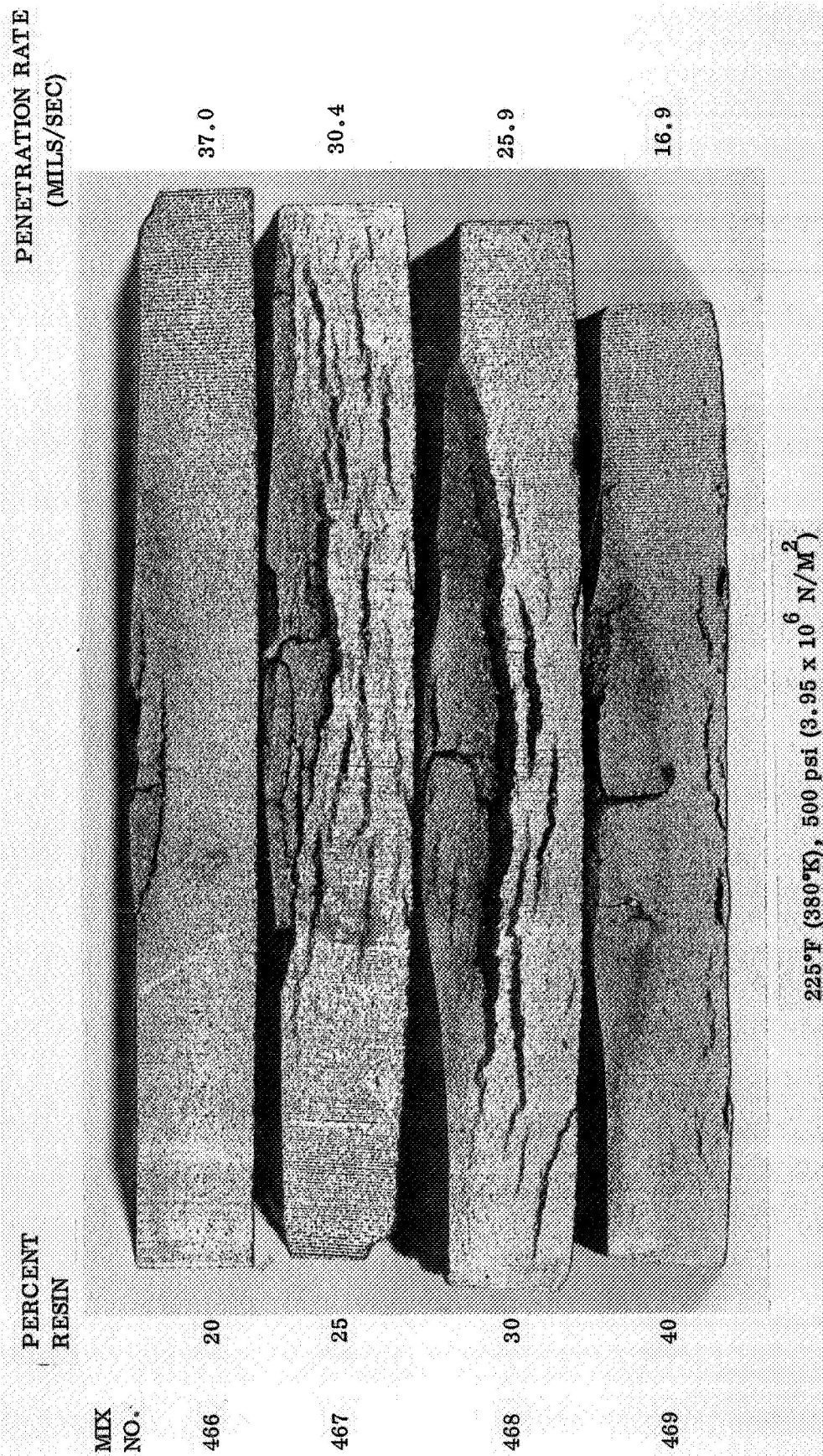


Figure 9. - Torch Test Samples, RI-4009 Resin/Graphite Composite, Mixes 466 thru 469

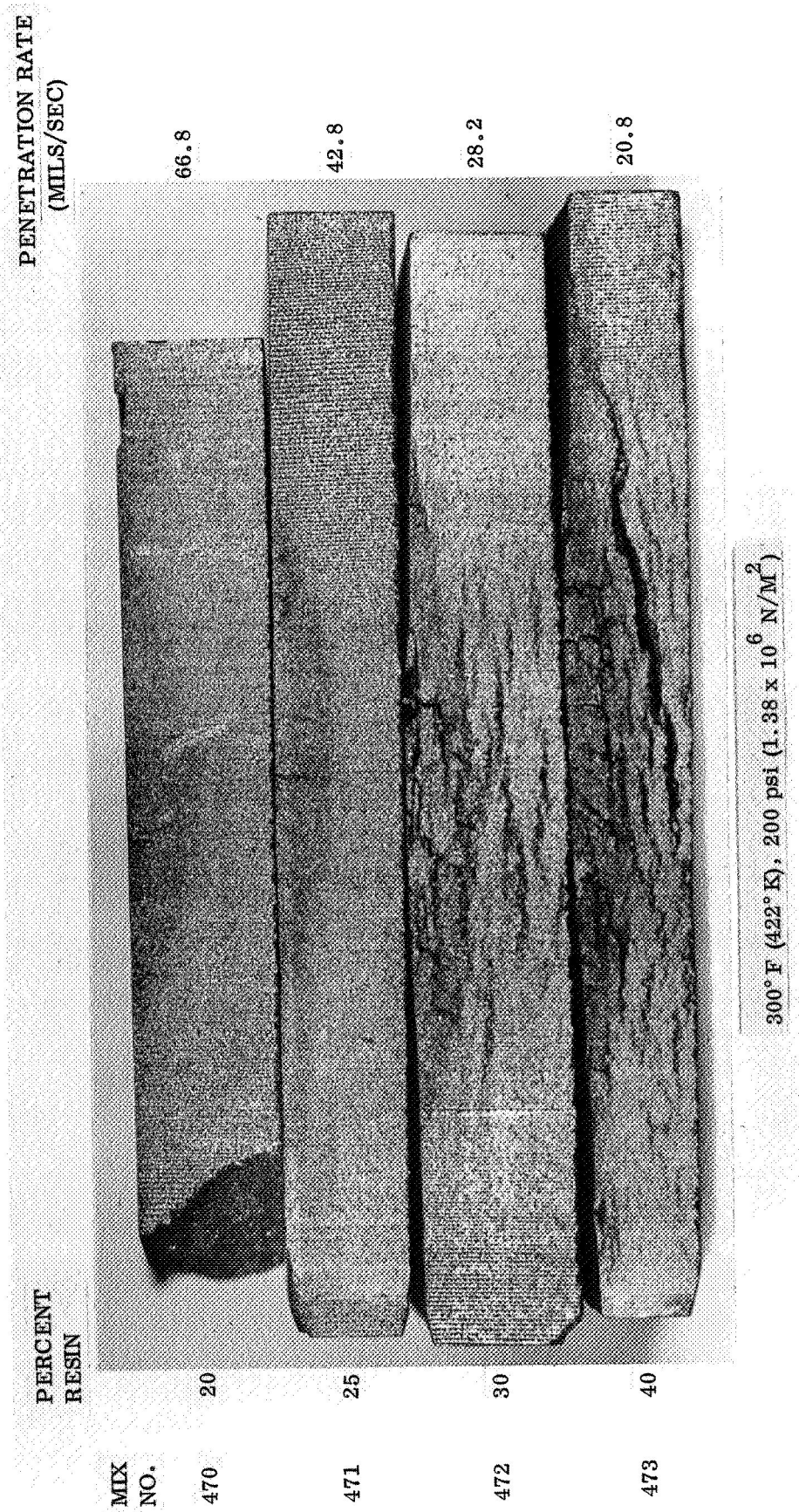


Figure 10. - Torch Test Samples, RI-4009 Resin/Graphite Composite, Mixes 470 thru 473

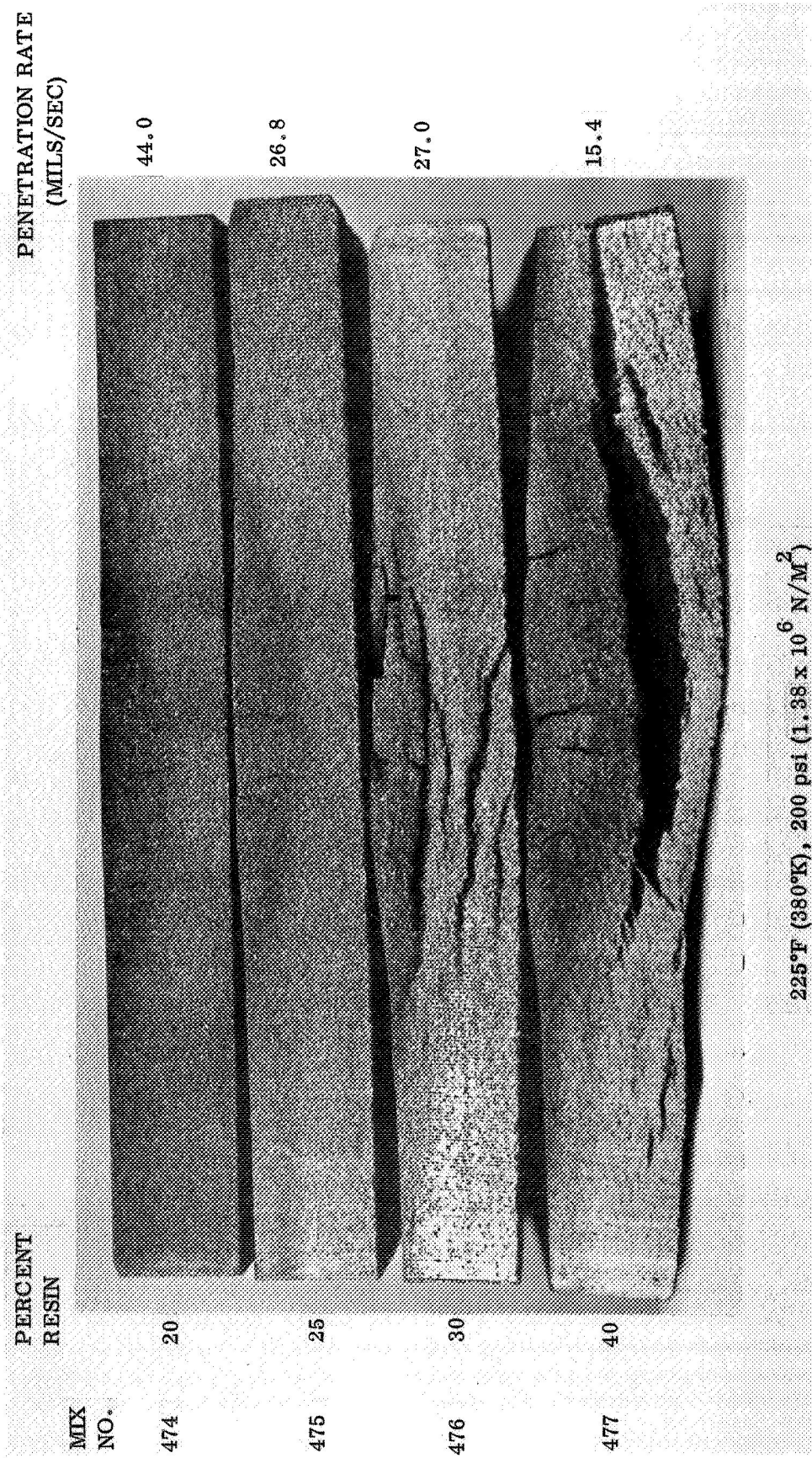


Figure 11. - Torch Test Samples, RI-4009 Resin/Graphite Composite, Mixes 474 thru 477

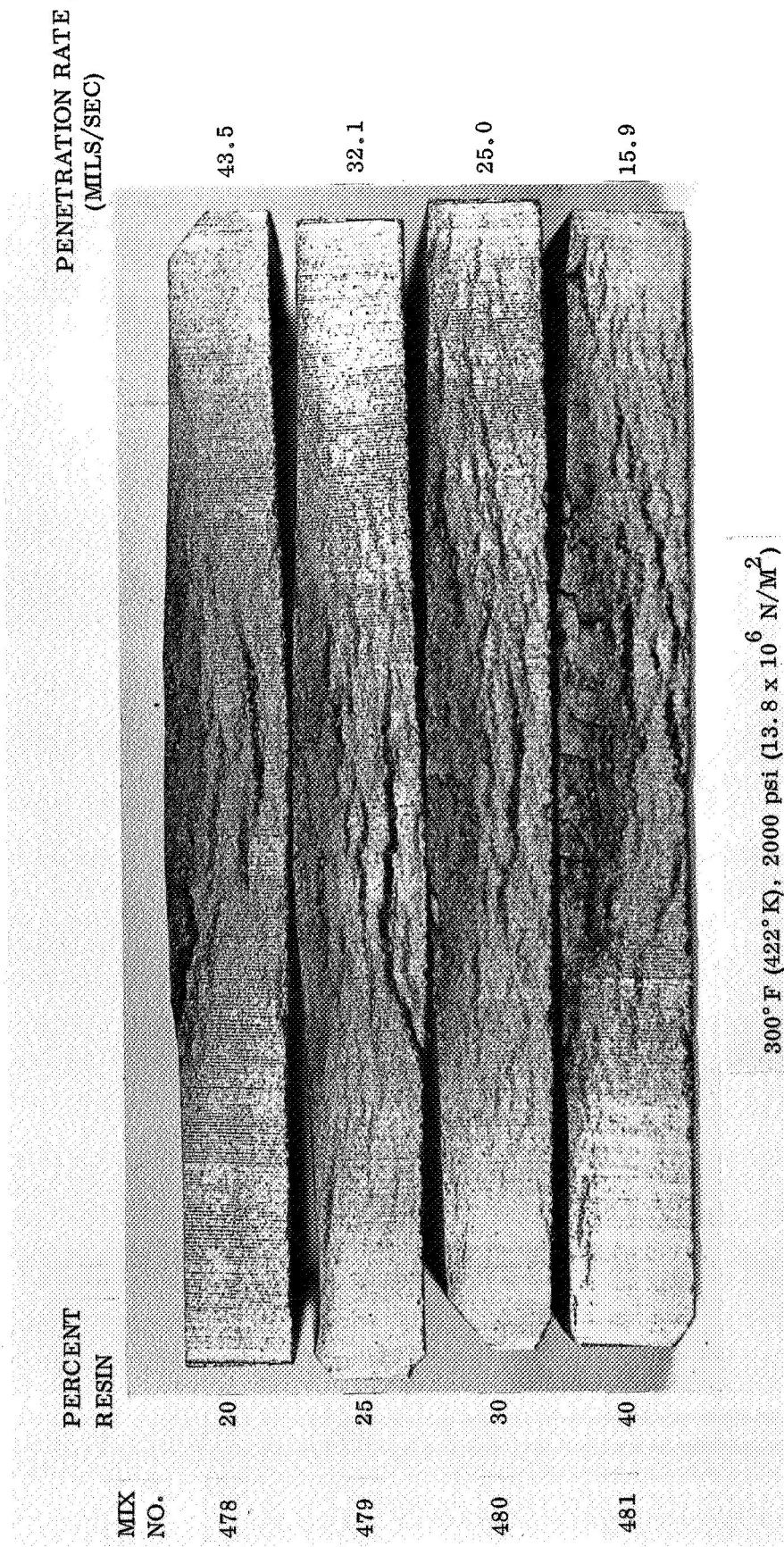


Figure 12. - Torch Test Samples, RI-4009 Resin/Graphite Composite, Mixes 478 thru 481

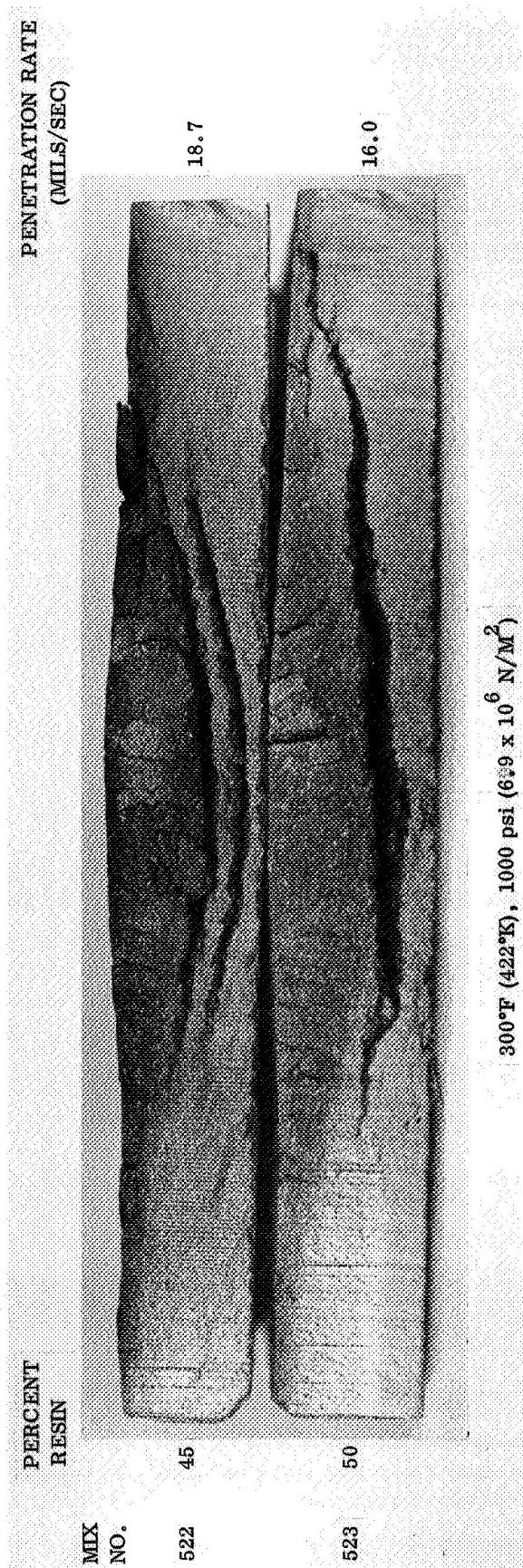


Figure 13. - Torch Test Samples, RI-4009 Resin/Graphite Composite, Mixes 522 and 523

In order to verify the above data and to obtain supplementary information, additional tests of the above materials were made. This study consisted of fabricating the following 5 by 7 by 1 in. (12.70 by 17.78 by 2.54 cm) test panels.

1. Two panels with 25 percent RI-4009 resin; cured at 200 psi (1.38×10^6 N/m²) and 300°F (422°K). Mix No. 534.
2. Two panels with 40 percent RI-4009 resin; cured at 1,000 psi (6.9×10^6 N/m²) and 300°F (422°K). Mix No. 535.
3. Two panels with 40 percent RI-4009 resin, and 10 percent ammonium ortho-phosphate, a flame retardant. The purpose of this test was to demonstrate the effect a flame retardant might have in eliminating thermal cracking. The system was cured at 1,000 psi (6.9×10^6 N/m²) and 300°F (422°K). Mix No. 539.

One test panel of each mix was perforated with 1/8 in. (0.32 cm) diameter holes on 1/2 in. (1.27 cm) centers over an area of 12 sq in. (77.4 sq cm). An oxyacetylene torch operated in excess of 5,000°F (3,035°K) was applied to each panel for 1 minute. The tested and cooled panel was segmented, and photographs were taken of the cross section showing the heat affected zones.

The results of this study indicated that thermal cracking will result in parts fabricated at both 200 (1.38×10^6 N/m²) and 1,000 psi (6.9×10^6 N/m²) of pressure. However, slightly heavier cracking appears to occur at the higher cure pressure. Providing vents for the gases to escape by drilling holes throughout the test panel certainly alleviates thermal cracking as shown in Figures 14 thru 16. The addition of a flame retardant (Figure 16, Mix No. 539) also decreases thermal cracking.

In addition, 1 in. (2.54 cm) cubes of Mixes 534 and 535 were immersed in mineral oil for 24 hours. The test was to determine if the compactness of the material had a direct relationship to oil absorption and thermal cracking. There was no weight gain for Mix No. 535, cured at 1,000 psi (6.9×10^6 N/m²) and a 1 percent gain for the 200 psi (1.38×10^6 N/m²) Mix No. 534.

The 40 percent resin content sample (as shown by the tensile and compression data) was superior to those samples tested at the higher and at lower percent resin contents, regardless of cure pressures.

Based upon the above test results, the following parameters (Mix No. 449) are recommended for curing of the RI-4009 resin system.

- | | |
|---------------------|---|
| 1. Cure temperature | 300°F (422°K). |
| 2. Cure pressure | 1,000 psi (6.9×10^6 N/m ²). |
| 3. Resin content | 40 percent. |

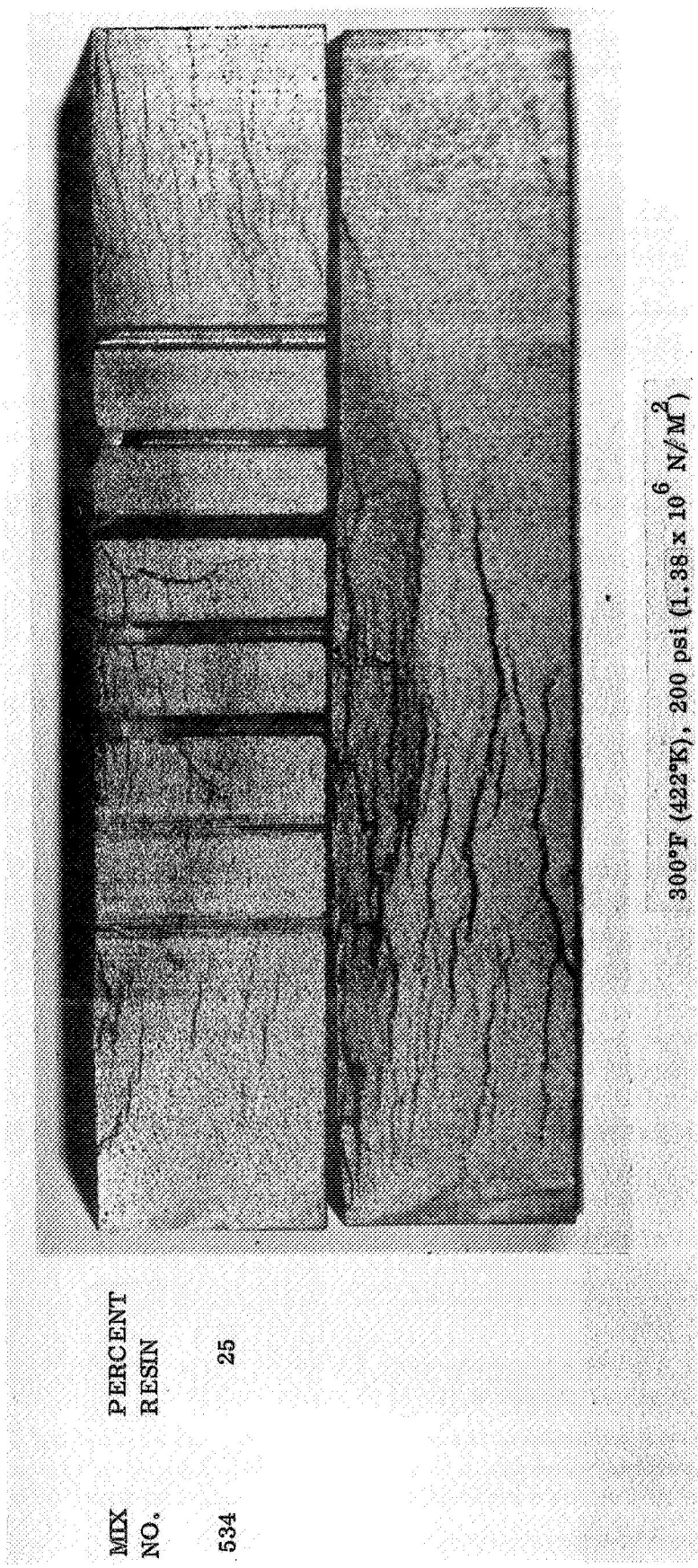


Figure 14.- Torch Test Sample, RI-4009 Resin, Mix 534

MIX
NO. PERCENT
 RESIN

535 40



300°F (422°K), 1000 psi (6.9×10^6 N/M²)

Figure 15. - Torch Test Sample, RI-4009 Resin, Mix 535

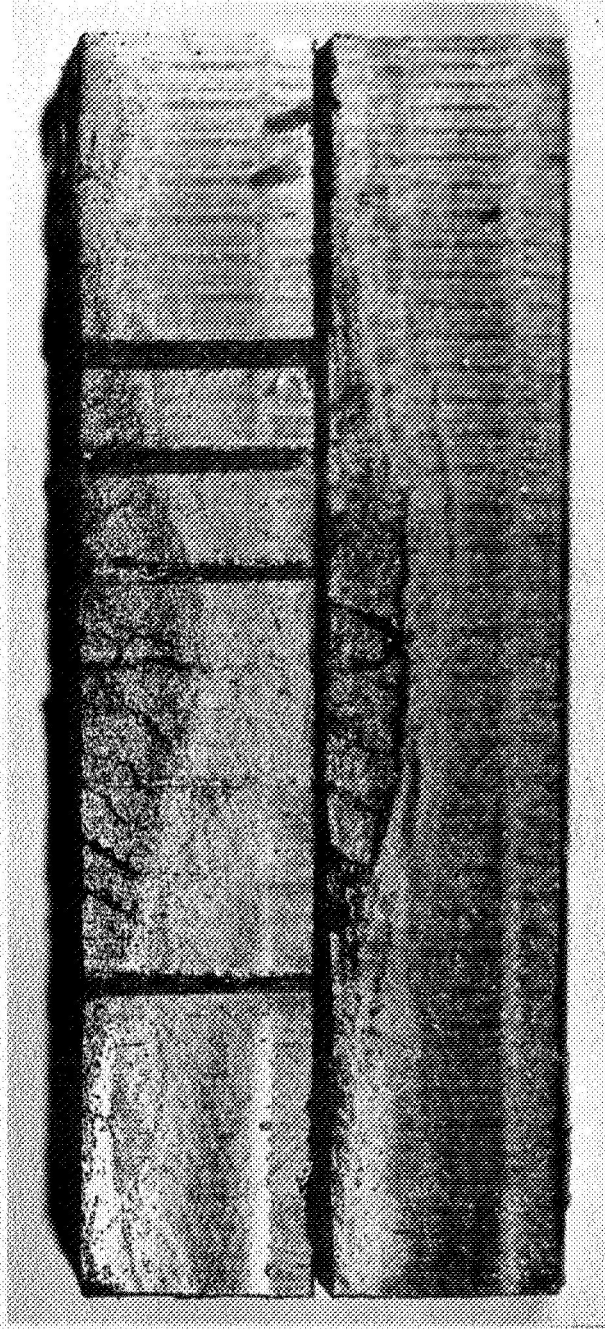
PERCENT
RESIN

40

(PLUS 10
PERCENT
 $(\text{NH}_4)_2\text{HPO}_4$)

MIX
NO.

539



300°F (422°K), 1000 psi (6.9×10^6 N/M²)

Figure 16. - Torch Test Sample, RI-4009 Resin Plus Flame Retardant, Mix 539

Durez 10694 Phenolic Resin. - Durez 10694 is an amber liquid resin with a viscosity of approximately 42.50 centipoise at 25°F (269.4°K). Four resin content variations were evaluated at four cure pressures and two cure temperatures. The Durez 10694 was evaluated at 20, 25, 30 and 40 percent resin and at cure pressures of ambient, 15, 100, and 200 psi (1.03×10^5 , 6.9×10^5 , 1.38×10^6 N/m²). The graphite-resin composites were hand packed into 5 by 10 by 1.0 in. (12.70 by 25.4 by 2.54 cm) molds and cured at 175° and 300°F (352° and 422°K). The ratio of graphite particles was kept constant: three parts of GL-1008 and one part GL-1012 graphite. The data described in Table VI include tensile and compressive strengths, density, and hardness. Test results appear to be sporadic with no definite relationship to resin and cure temperature at the ambient or the 15 psi (1.03×10^5 N/m²) cure pressure. Samples cured at 200 psi (1.38×10^6 N/m²) and 300°F (422°K) offered a superior structural strength to those samples cured at the lower pressures and temperatures, and 25 percent resin content formulations generally presented a superior appearance after exposure to a high temperature environment. In addition, a test sample was heated on one side using an oxyacetylene torch in excess of 5,000°F (3,035°K). The heat penetration rate, the time to heat the backside of the 1/2 in. (1.27 cm) thick sample to 200°F (366°K), was recorded for all panels. Upon cooling, the test panel was segmented, and the heat affected zones photographed and examined for spalling, cracking, and char hardness. Figures 17 thru 24 are photographs of the torch test samples. Test specimens showed none or very little thermal cracking, and the components appeared to be structurally sound. The heat penetration rate is very high with this type of resin system.

The experiments also revealed that at 40 percent level, the resin flowed when subjected to 100 psi (6.9×10^5 N/m²), resulting in resin rich material and plugged vacuum lines. No test data were collected at the 40 percent resin level at either the 100 or 200 psi (6.9×10^5 or 1.38×10^6 N/m²) cure pressures.

The above tests appear to demonstrate that the Durez 10694 resin at the 25 percent resin content offers the greater potential. It may be noted that this resin content does not have the structural strength (1,815 psi or 12.53×10^6 N/m²) as compared with samples fabricated with 30 percent resin (2,551 psi or 17.58×10^6 N/m²). However, the former formulation (25 percent resin) retains a better heat affected zone with little or no radial cracking resulting from the torch test. In addition, material handling during sample fabrication is improved with the use of the lower resin content.

DEN 438 Epoxy Novolac. - This system contains a phenolic type structure with the reactivity of any epoxy resin. Unlike the phenolics, it generally requires no pressure and generates no reaction by-products. However, DEN 438 does require a curing agent. The curing agent chosen is identified as nadic methyl anhydride (NMA), with benzyldimethylamine (BDMA) as an accelerator. This curing agent was selected over other anhydrides and aromatic amines; i. e., DEH 50 methylene diamidine, BF₃ monethylamine complex, Versamid, Shell Z and DMP 30, because it offered a

TABLE VI. - LCCM DEVELOPMENT OPTIMIZATION STUDY OF DUREZ 10634 RESIN (CONT)

Mix No.	Percent Resin	Cure Requirements				Temperature		Tensile Strength		Tensile Modulus		Density (g/cc)	Compressive Strength		Penetration Rate		Hardness Shore D	
		Time (hr)	Pressure (psi)	$(N/m^2 \times 10^5)$	(°F)	(°C)	(psi)	$(N/m^2 \times 10^6)$	(psi)	$(N/m^2 \times 10^9)$	(psi)		(psi)	$(N/m^2 \times 10^6)$	(mil/sec)	$(m/sec \times 10^{-5})$	Virgin	Char
497	25	56.25	18.75	15	0.1035	300	422	295	2.0	0.523	3.6	1.16	--	--	46.0	117.6	45	33
								399	2.8	0.523	3.6	1.25	--	--				
								--	--	--	--	1.22	--	--				
								347	2.4	0.523	3.6	1.21	--	--				
498	30	52.5	17.5	15	0.1035	300	422	612	4.2	0.845	5.8	1.16	3,460	23.9	60.7	154.1	74	52
								452	3.1	0.845	5.8	1.25	4,020	27.7				
								657	4.5	0.845	5.8	1.22	3,880	26.8				
								573	3.9	0.845	5.8	1.21	3,766	26.1				
499	30	52.5	17.5	--	0.1035	300	422	--	--	--	--	TEST SPECIMENS NOT FABRICATED						
500	20	60	20	100	0.69	175	353	432	3.0	1.38	9.5	1.38	2,900	20.01	56.7	143.0	60	30
								932	6.4	1.38	9.5	1.35	2,760	19.04				
								--	--	--	--	1.38	1,150	7.9				
								682	4.7	1.38	9.5	1.37	2,270	15.6				
501	25	56.25	18.75	100	0.69	175	353	683	4.7	1.58	10.9	1.45	3,100	21.39	66.0	167.6	70	57
								1,245	8.6	1.58	10.9	1.54	6,140	42.4				
								--	--	--	--	1.55	6,080	46.1				
								964	6.6	1.58	10.9	1.51	5,306	36.5				
502	30	52.5	17.5	100	0.69	175	353	1,560	10.8	2.08	14.4	1.74	10,325	71.2	31.4	80.2	80	
								1,495	10.3	2.08	14.4	1.74	10,900	75.2				
								762	5.3	2.08	14.4	1.72	10,675	73.7				
								1,272	8.7	2.08	14.4	1.73	10,633	73.3				
503	20	60.0	20.0	6	0.69	300	422	104	0.71	0.89	6.1	1.29	1,205	8.3	74.5	189.2	60	45
								168	1.2	--	--	1.35	2,400	16.6				
								56	0.39	--	--	1.34	2,330	16.1				
								109	0.75	0.89	6.1	1.32	1,978	13.6				
504	25	56.25	18.75	6	0.69	300	422	1,290	8.9	1.34	9.2	1.47	6,940	47.9	64.0	162.6	74	66
								1,330	9.2	1.34	9.2	1.50	5,760	39.7				
								784	5.4	1.34	--	1.53	7,040	48.6				
								1,134	7.8	1.34	9.2	1.50	6,580	45.3				
505	30	52.5	17.5	6	0.69	300	422	667	4.6	1.43	9.9	1.49	8,470	58.4	54.5	139.5	75	65
								1,345	9.3	1.45	10.0	1.51	8,300	57.3				
								1,700	11.73	1.45	10.0	1.55	8,200	56.6				
								1,237	8.5	1.45	10.0	1.52	8,323	57.3				
506	20	60.0	20.0	200	1.38	300	422	484	3.3	1.21	8.3	1.39	2,980	20.6	84.0	213.4	64	54
								693	4.8	1.21	8.3	1.43	3,340	23.1				
								355	2.4	1.21	8.3	1.48	4,730	32.9				
								510	3.5	1.21	8.3	1.43	3,700	25.5				
507	25	56.25	18.75	24	200	1.38	175	1,480	10.2	1.40	9.7	1.58	5,840	40.3	67.0	171.9	71	60
								1,500	10.4	1.40	9.7	1.52	7,920	54.6				
								1,445	10.0	1.40	9.7	1.57	6,980	48.2				
								1,475	10.1	1.40	9.7	1.55	6,913	47.6				
508	30	52.5	17.5	24	200	1.38	175	1,895	13.1	1.69	11.7	1.57	9,390	64.7	32.0	82.0	72	
								1,925	13.3	1.69	11.7	1.62	10,020	69.1				
								2,110	14.6	1.69	11.7	1.64	8,580	59.2				
								--	--	--	--	1.61	--	--				
								1,976	13.6	1.69	11.7	1.61	9,326	64.3				
509	20	60.0	20.0	24	200	1.38	300	366	2.5	1.05	7.2	1.39	3,480	24.0	67.0	171.7	64	47
								775	5.3	1.05	7.2	1.46	4,080	28.2				
								695	4.8	1.04	7.2	1.43	3,240	22.4				
								--	--	--	--	1.43	--	--				
								612	4.2	1.05	7.2	1.42	3,600	24.8				

--No test data available.

TABLE VI. - LCCM DEVELOPMENT OPTIMIZATION STUDY OF DUREZ 10894 RESIN (CONT)

Mix No.	Percent Resin	Percent Graphite GL-1008	Time (hr)	Cure Requirements		Tensile Strength (psi)	Tensile Modulus (psi x 10 ⁶)	Density (g/cc)	Compressive Strength (psi)	Penetration Rate (mils/sec)	Hardness Shore D			
				Pressure (psi)	Temperature (° F)						Virin	Char		
510	25	56.25	18.75	200	300	1,845	1.22	1.53	6,980	75.5	192.0	75	62	
						1,925	--	1.50	4,860					
						1,675	--	1.48	4,720					
511	30	52.5	17.5	200	300	1,815	1.22	1.50	--					
						1,815	--	1.50	5,520					
						2,345	1.63	1.68	10,160	60.6	154.9	80		
						2,675	--	1.59	9,160					
						2,635	--	1.65	9,300					
						2,551	--	1.64	--					
						2,551	11.2	1.64	9,540					

--No test data available.

PENETRATION RATE
(MILS/SEC)
(ROUNDED OFF)

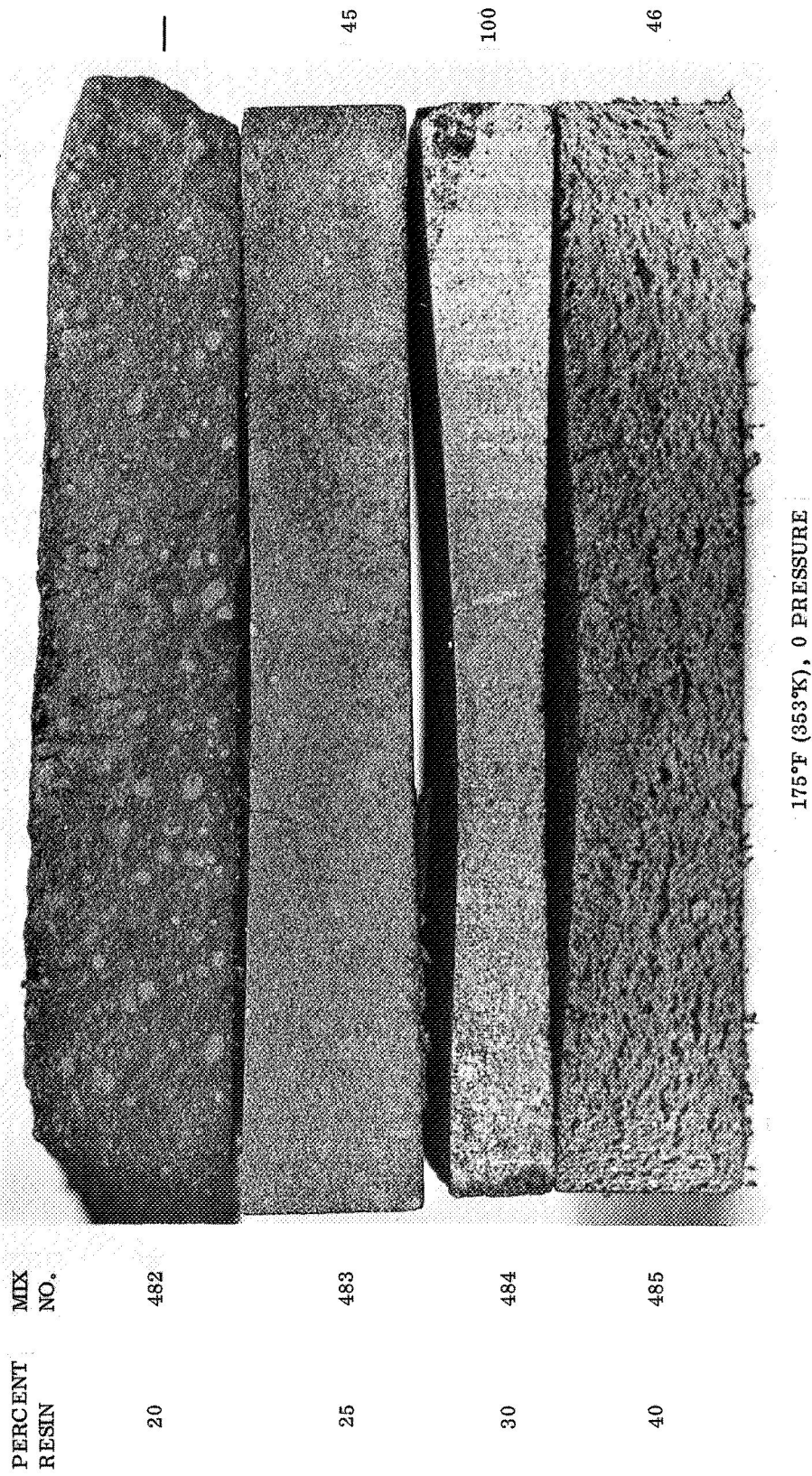


Figure 17. - Torch Test Samples, Durez 10694 Resin/Graphite Composite, Mixes 482 thru 485

PENETRATION RATE
(MILS/SEC)
(ROUNDED OFF)

PERCENT MIX
RESIN NO.

20 488

25 489

30 490

40 491

42

82

37

29

300°F (422°K), 0 PRESSURE

Figure 18. - Torch Test Samples, Durez 10694 Resin/Graphite Composite, Mixes 488 thru 491

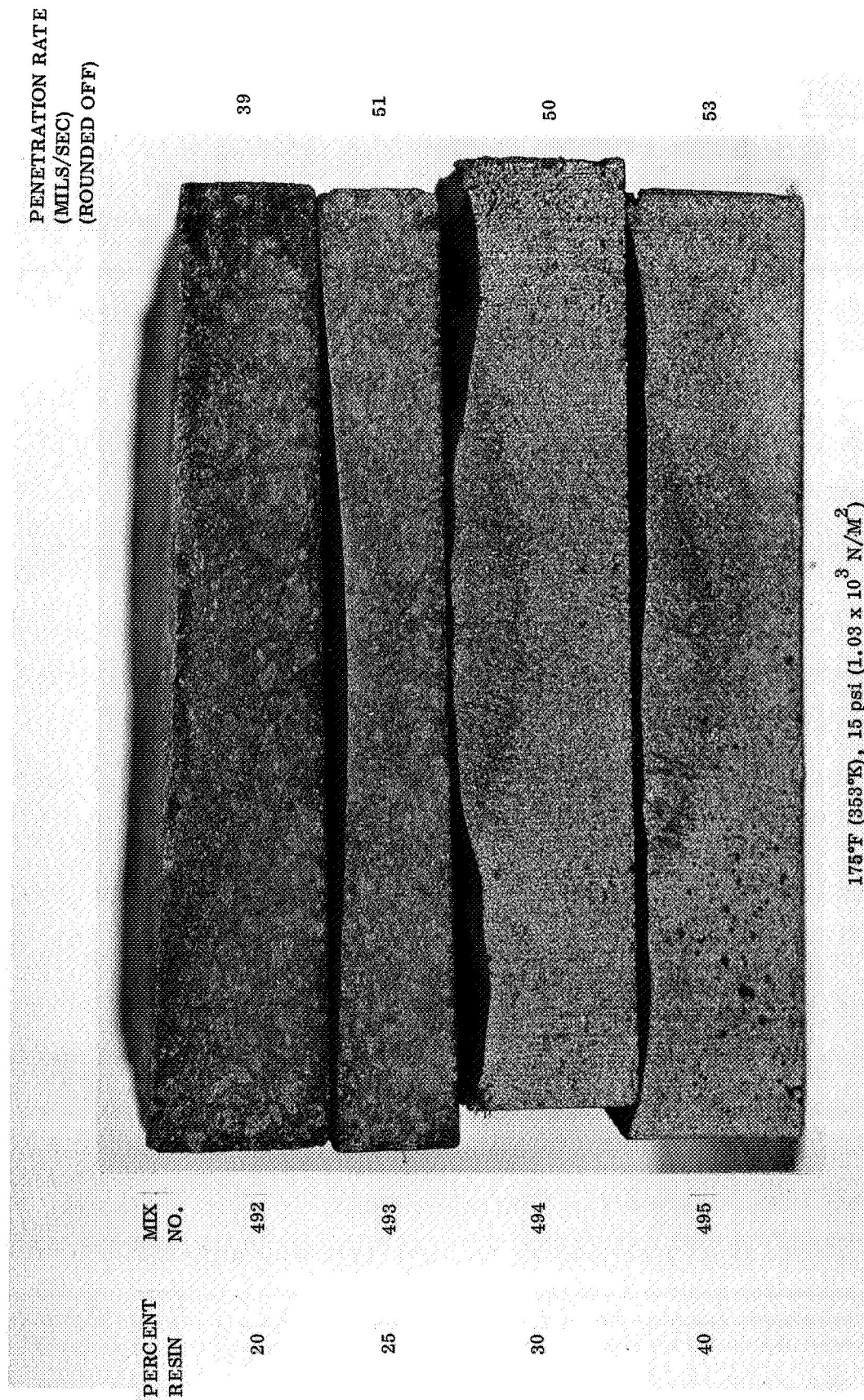


Figure 19. - Torch Test Samples, Durez 10694 Resin/Graphite Composite, Mixes 492 thru 495

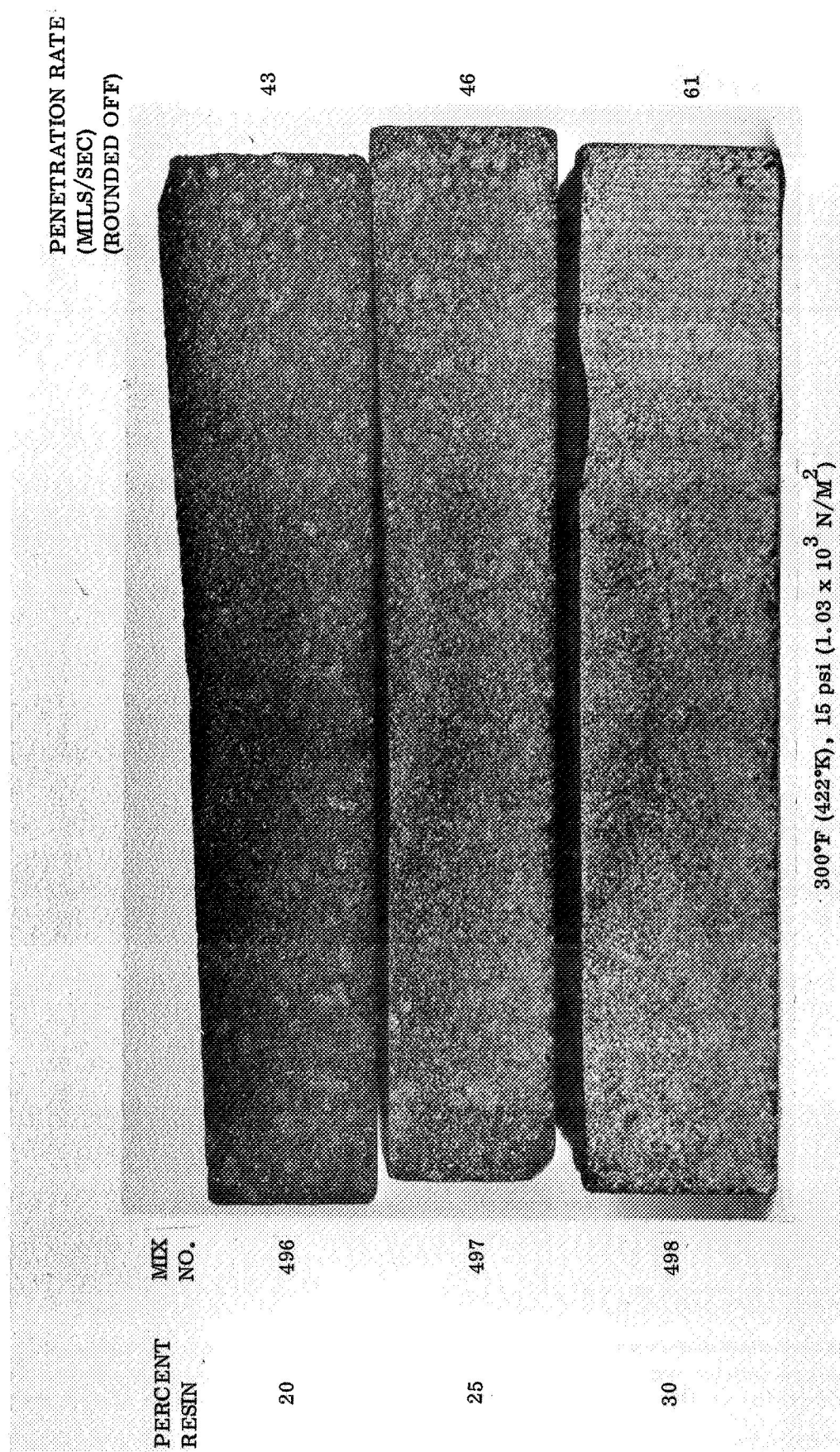


Figure 20. - Torch Test Samples, Durez 10694 Resin/Graphite Composite, Mixes 496 thru 498

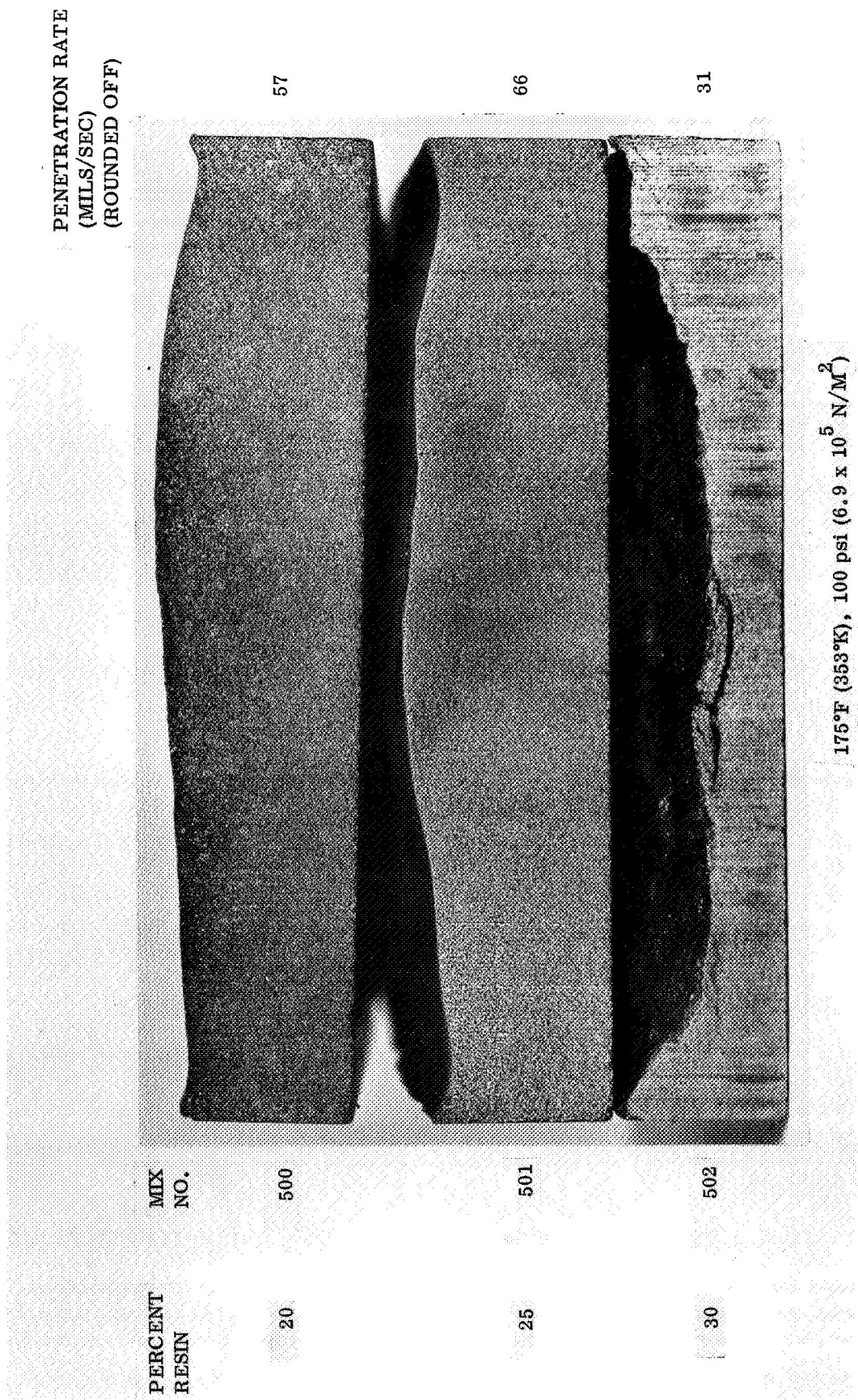


Figure 21. - Torch Test Samples, Durez 10694 Resin/Graphite Composite, Mixes 500 thru 502

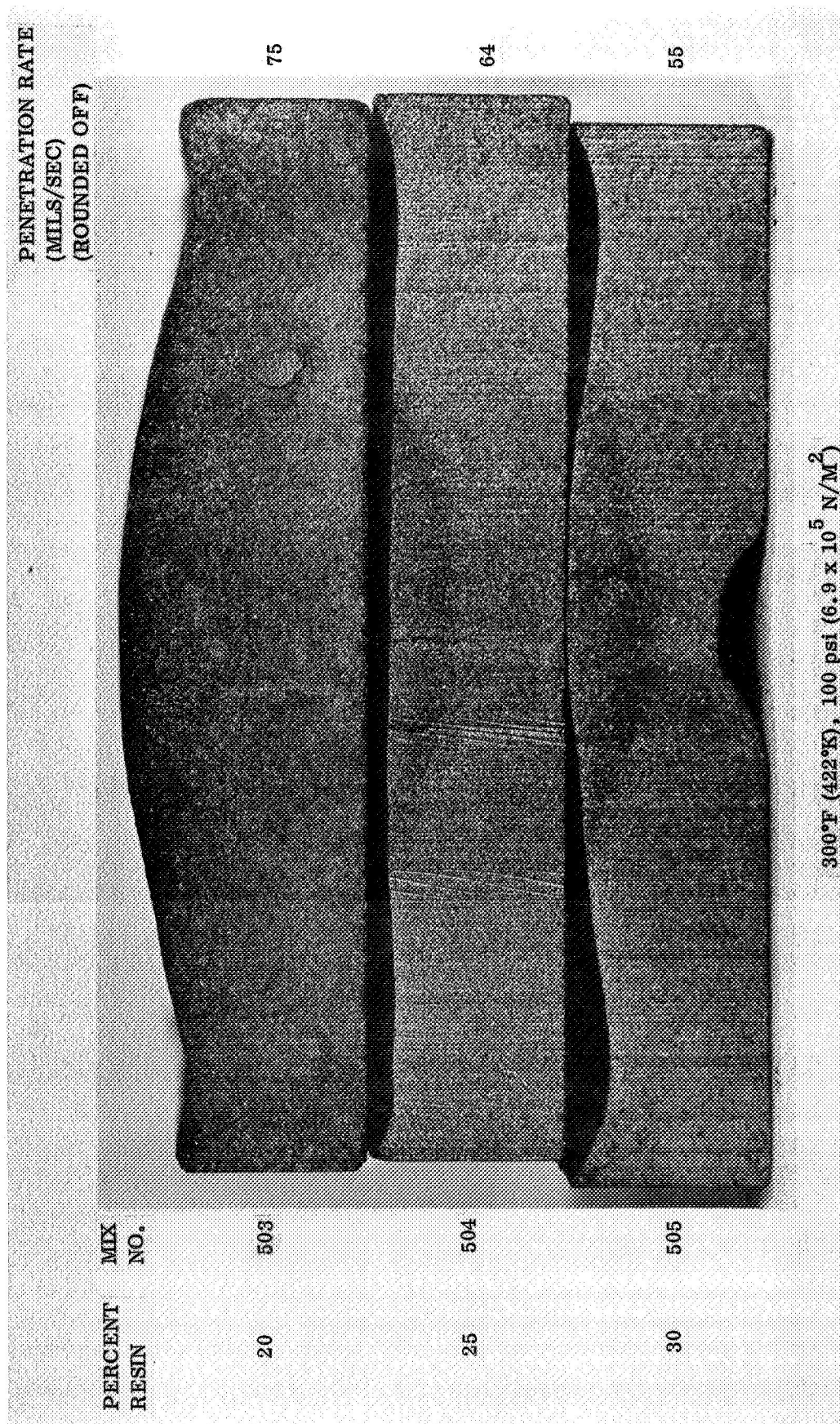


Figure 22. - Torch Test Samples, Durez 10694 Resin/Graphite Composite, Mixes 503 thru 505

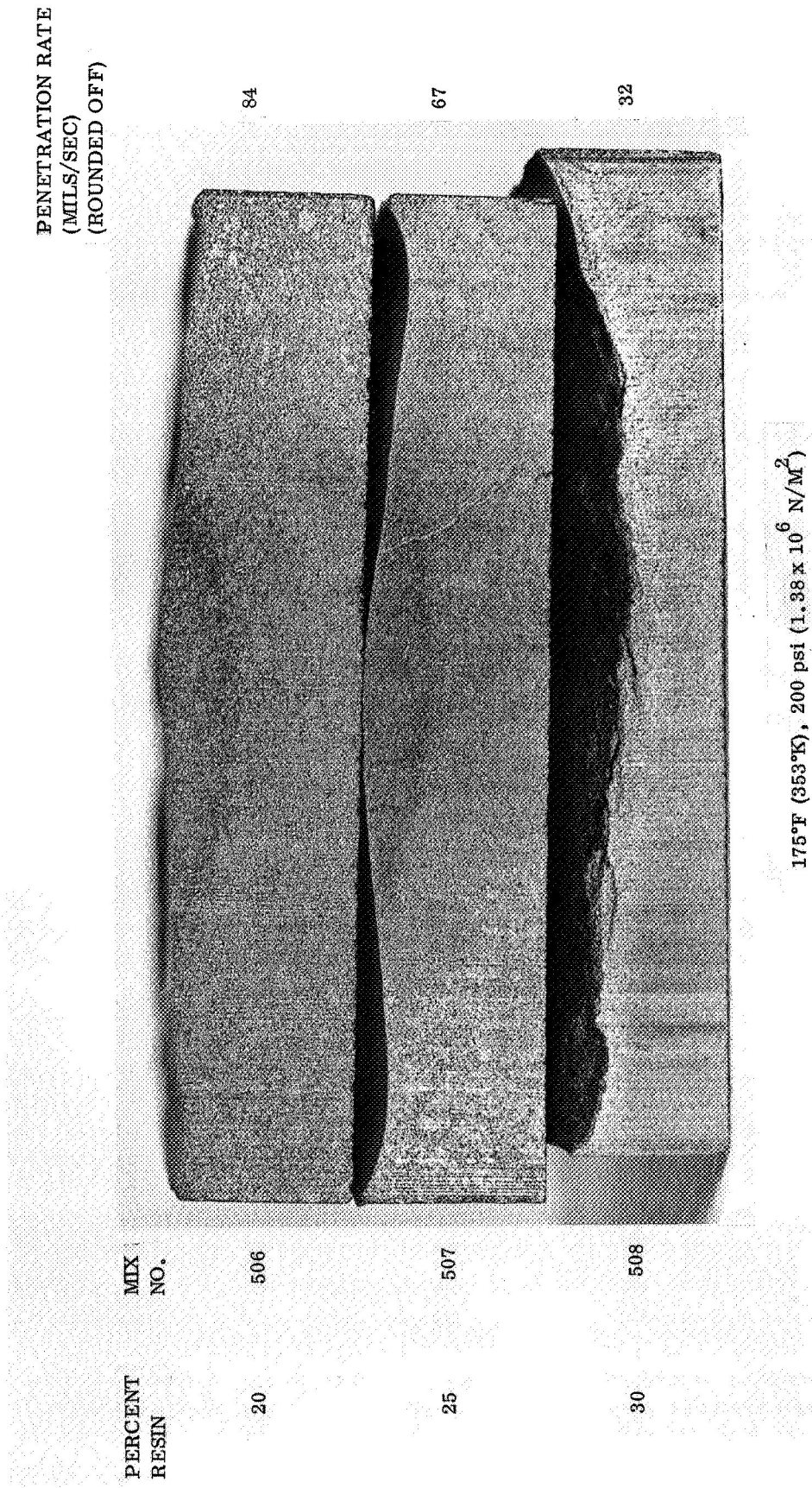
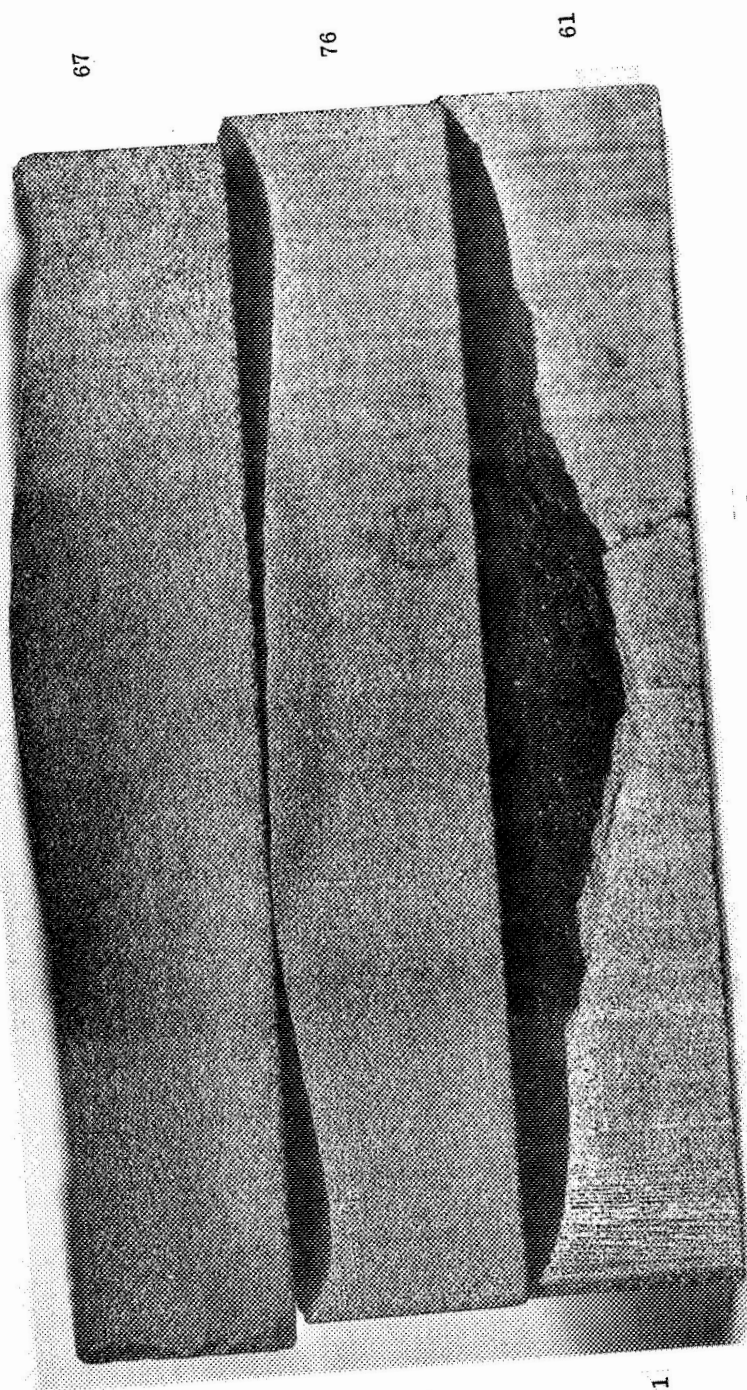


Figure 23. -Torch Test Samples, Durez 10694 Resin/Graphite Composite, Mixes 506 thru 508

PENETRATION RATE
(MILS/SEC)
(ROUNDED OFF)



300°F (422°K), 200 psi (1.38×10^6 N/M²)

MIX
NO.

509

510

511

PERCENT
RESIN

20

25

30

44

Figure 24. - Torch Test Samples, Durez 10694 Resin/Graphite Composite, Mixes 509 thru 511

higher heat distortion temperature, higher flexural and compressive strengths, longer pot life, and ease of handling. This selection was based on technical literature and visual observations of the curing agents evaluated.

The optimization study of this resin system included the evaluation of four formulations cured at two pressure levels and at one temperature cycle.

Table IV outlines the epoxy novolac test matrix.

Test results indicate that the higher pressure (15 psi or $1.03 \times 10^5 \text{ N/m}^2$) aids in structural integrity, and that increases in resin content up to 30 percent improve tensile properties. However, formulations containing a resin content of 40 percent, when cured at 15 psi ($1.03 \times 10^5 \text{ N/m}^2$) pressure, encountered flow problems and were not processible. During the curing cycle, excessive resin was sucked into vacuum line and cured, thus preventing pressure from being applied uniformly throughout the cure.

Test results of tensile strength, density, compressive strength, and thermal properties are shown in Table VII. Figures 25 thru 31 are photographs of the segmented torch test samples and show test results in the heat affected zones. The mix numbers correspond with those listed in Table VII.

In order to determine the effect of vacuum mixing, 44 parts of resin DEN 438 with hardener were mixed with 56 parts of graphite powder. The ratio of graphites were three parts of GL-1008 to one part of GL-1012. The ingredients were vacuum mixed for 15 minutes and cast into molds and cured. Test panels were machined for tensile and compressive samples.

The data indicate that improvements in the density and physical properties of the epoxy novolac material can be obtained by vacuum mixing. Test results appear below.

Mix No.	Condition	Tensile Strength		Tensile Modulus		Density (g/cc)	Compressive Strength	
		(psi)	($\text{N/M}^2 \times 10^6$)	(psi $\times 10^6$)	($\text{N/m}^2 \times 10^9$)		(psi)	($\text{N/m}^2 \times 10^6$)
546	Vacuum mixed, ambient pres- sure cured	4,130	28.47	1.48	10.20	1.62	14,350	98.93
		4,910	33.85	1.44	9.92	1.61	14,625	100.83
		<u>2,810</u>	<u>19.37</u>	<u>1.47</u>	<u>10.14</u>	<u>1.61</u>	<u>14,930</u>	<u>102.93</u>
		3,950	27.23	1.46	10.06	1.61	14,635	100.90
519	Non-vacuum mixed, bag cured at 15 psi (1.03×10^5 N/m^2)	2,690	18.54	1.10	7.59	1.43	9,120	62.88
		2,520	17.27	1.10	7.59	1.41	8,680	57.84
		<u>2,790</u>	<u>19.23</u>	<u>1.09</u>	<u>7.52</u>	<u>1.43</u>	<u>9,200</u>	<u>63.43</u>
		2,666	18.38	1.10	7.58	1.42	9,000	62.05

TABLE VII--LCCM DEVELOPMENT OPTIMIZATION STUDY, DEN 438 EPOXY NOVOLAC

Mix No.	Percent Resin	Percent Graphite GL-1008 GL-1012	Cure Requirements				Tensile Strength (psi)	Tensile Modulus (N/m ² x 10 ⁹)	Density (g/cc)	Compressive Strength		Penetration Rate		Hardness Shore D	
			Time (hr)	Pressure (psi)	Temperature (°F)	Temperature (°C)				(psi)	(N/m ² x 10 ⁶)	(mils/sec)	(in/sec x 10 ⁻⁵)	VIHM	Char
514	20.44	46.04	2	None	200	366	1,155	7.96	1.53	9,060	62.46	16.8	42.67	71	20
	17.89*		15		300	422	589	4.06	1.53	7,440	51.30				
	0.30**						1,705	11.76	1.53	8,410	57.99				
515	20.44	46.04	2	15.1	200	366	1,975	13.62	1.47	8,380	57.78	21.1	53.34	80	40
	17.89*		15		300	422	1,870	12.89	1.47	7,840	54.05				
	0.30**						1,675	11.55	1.47	8,420	58.05				
516	16.97	50.95	2	None	200	366	1,840	12.68	1.47	8,213	56.62	28.1	71.37	52	25
	14.87*		15		300	422	1,75	1.21	1.39	2,800	19.31				
	0.25**						325	2.24	1.42	2,900	19.99				
517	16.97	50.95	2	15.1	200	366	2,86	1.97	1.39	2,000	13.79	15.8	40.13	62	32
	14.87*		15		300	422	358	2.47	1.43	4,140	28.54				
	0.25**						1,330	9.17	1.44	3,980	27.44				
518	23.67	41.43	2	None	200	366	1,065	7.34	1.48	12,240	84.39	23.6	59.94	78	30
	20.71*		15		300	422	2,455	16.93	1.49	12,280	84.67				
	0.35**						2,940	20.27	1.48	12,800	86.25				
519	23.67	41.43	2	15.1	200	366	2,980	17.10	1.48	12,440	85.77	23.2	58.93	78	25
	20.71*		15		300	422	2,690	18.55	1.41	8,680	59.85				
	0.35**						2,520	17.37	1.43	9,200	63.43				
520	29.49	33.18	2	None	200	366	2,666	18.36	1.43	9,000	62.05	8.04	20.42	86	20
	25.81*		15		300	422	2,770	19.10	1.42	13,960	96.26				
	0.44**						2,775	19.13	1.41	13,640	94.04				
							2,808	18.36	1.42	13,873	95.65				

*NMA

**BDMA

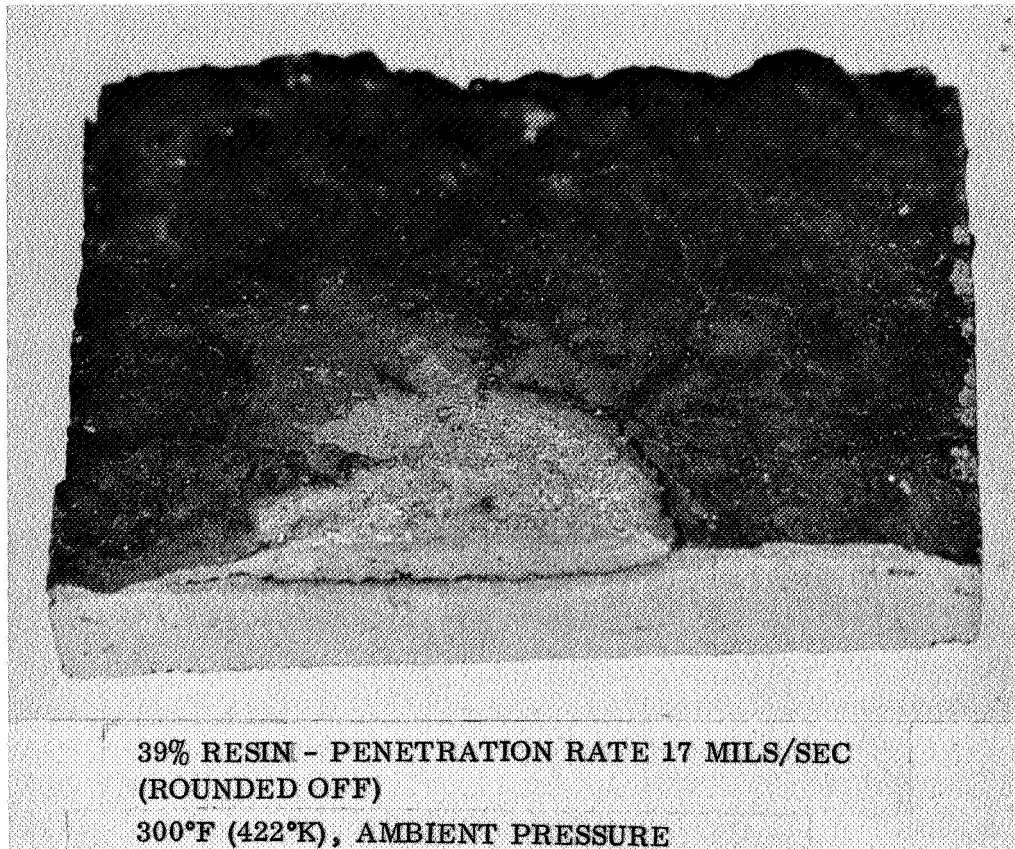


Figure 25. - Torch Test Sample, DEN 438 Epoxy Novolac/Graphite Composite, Mix 514

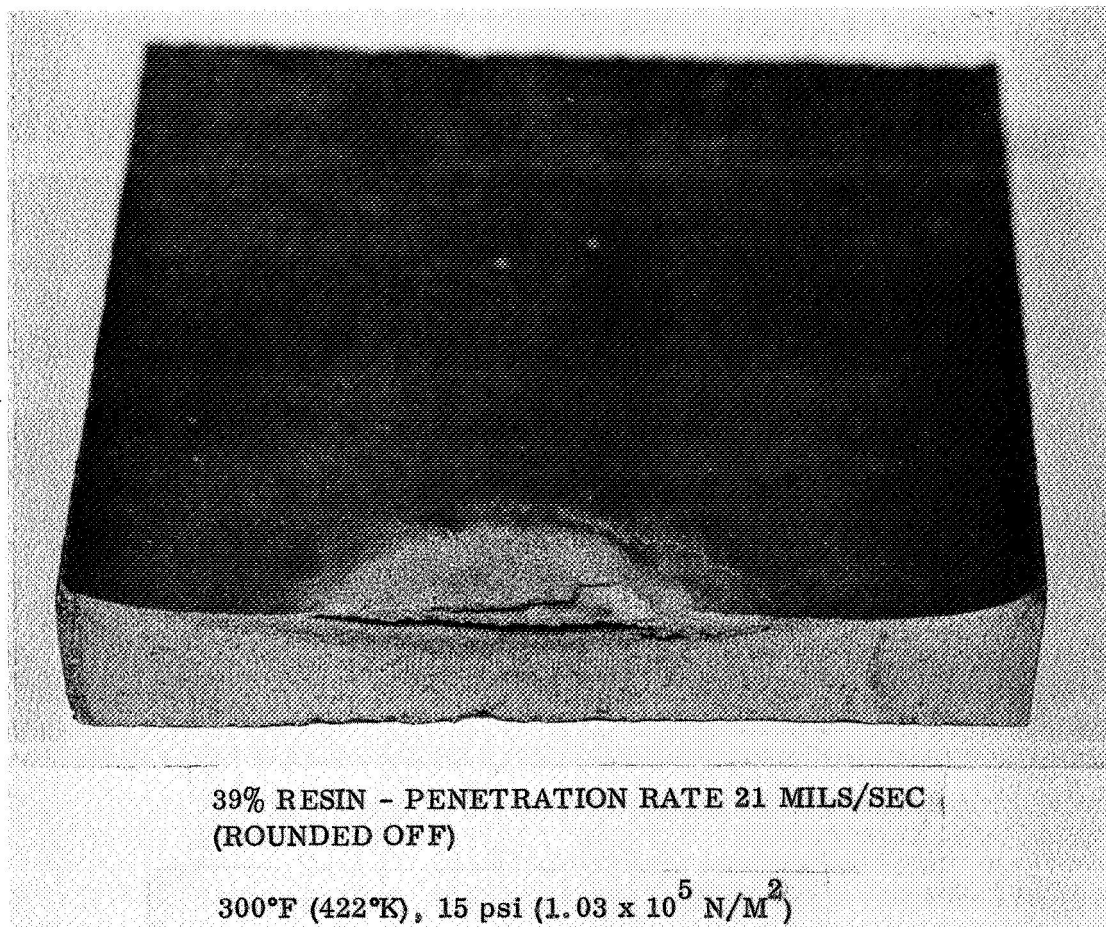


Figure 26. - Torch Test Sample, DEN 438 Epoxy Novolac/Graphite Composite, Mix 515

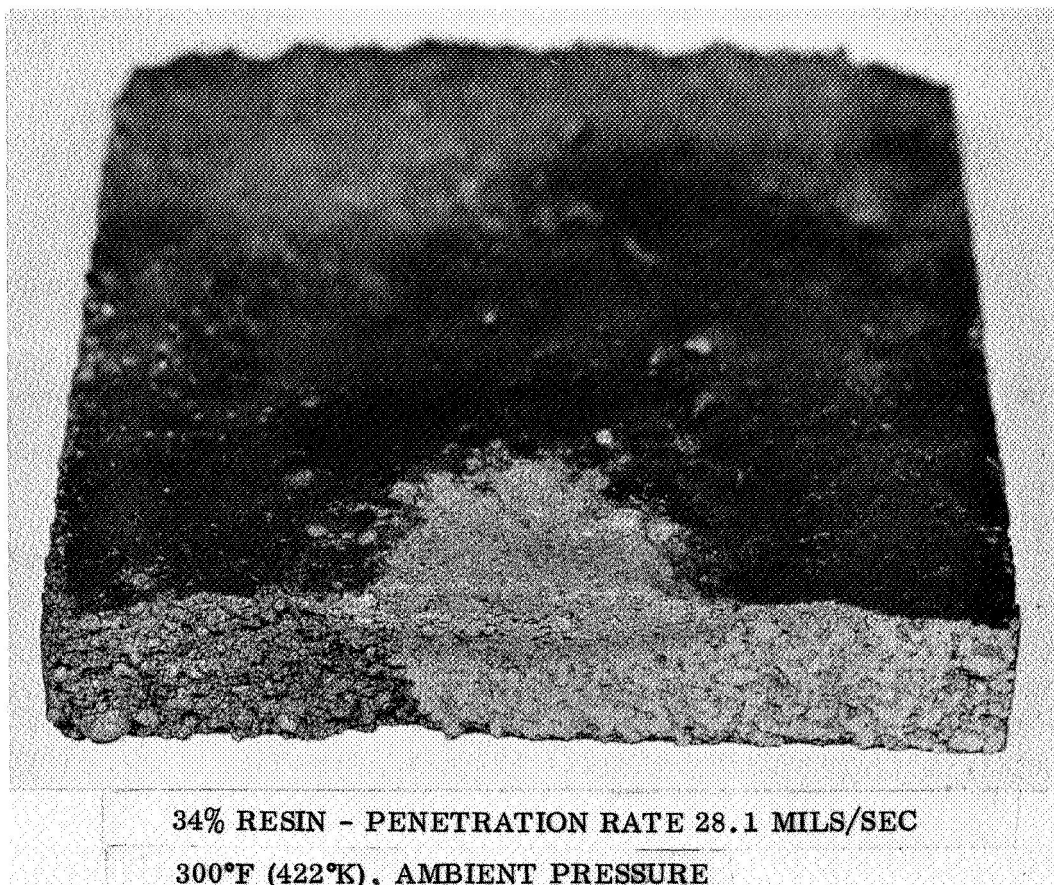


Figure 27. - Torch Test Sample, DEN 438 Epoxy Novolac/Graphite Composite, Mix 516

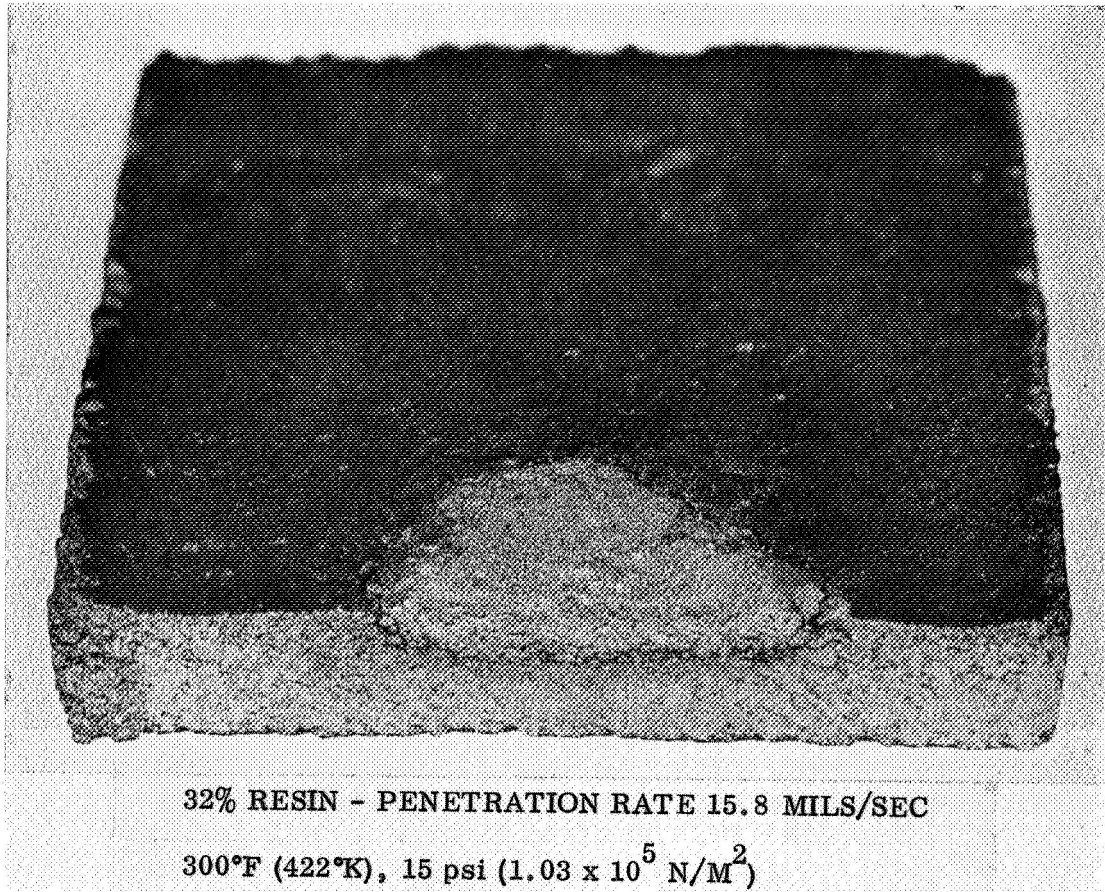


Figure 28. - Torch Test Sample, DEN 438 Epoxy Novolac/Graphite Composite, Mix 517

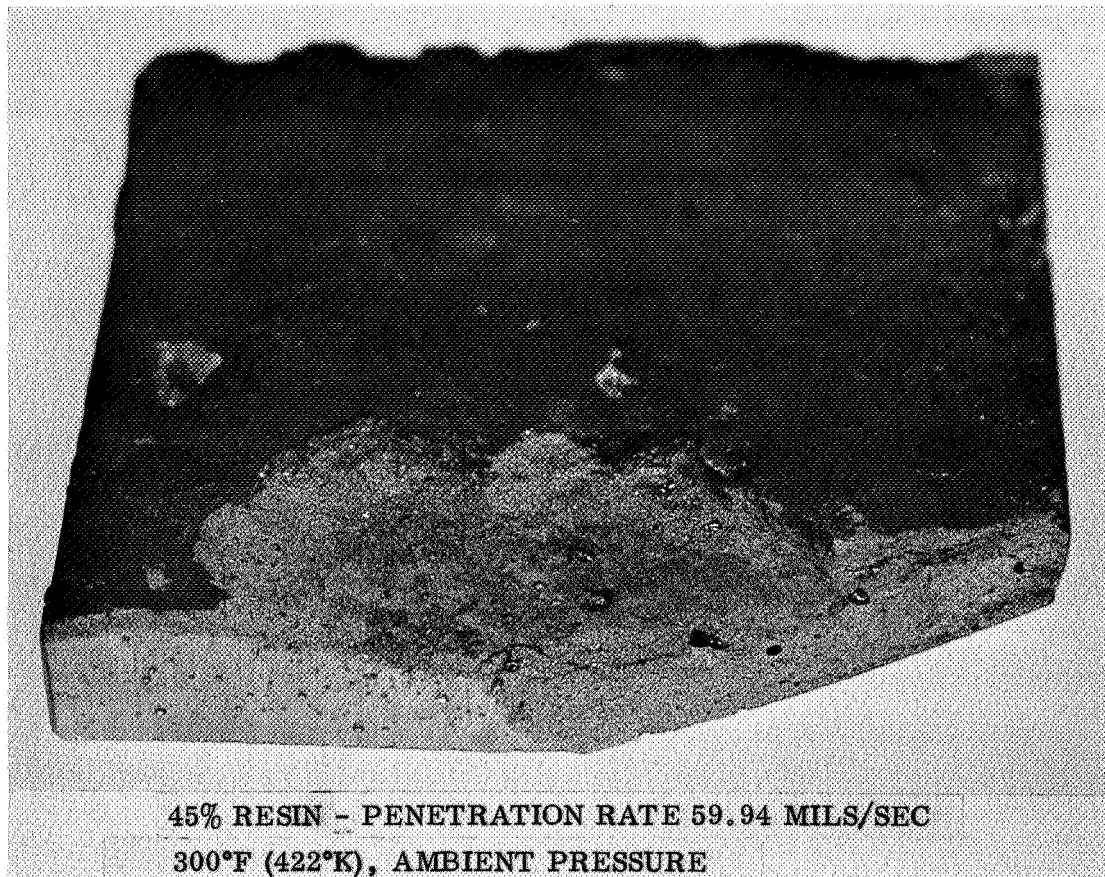


Figure 29. - Torch Test Sample, DEN 438 Epoxy Novolac/Graphite Composite, Mix 518

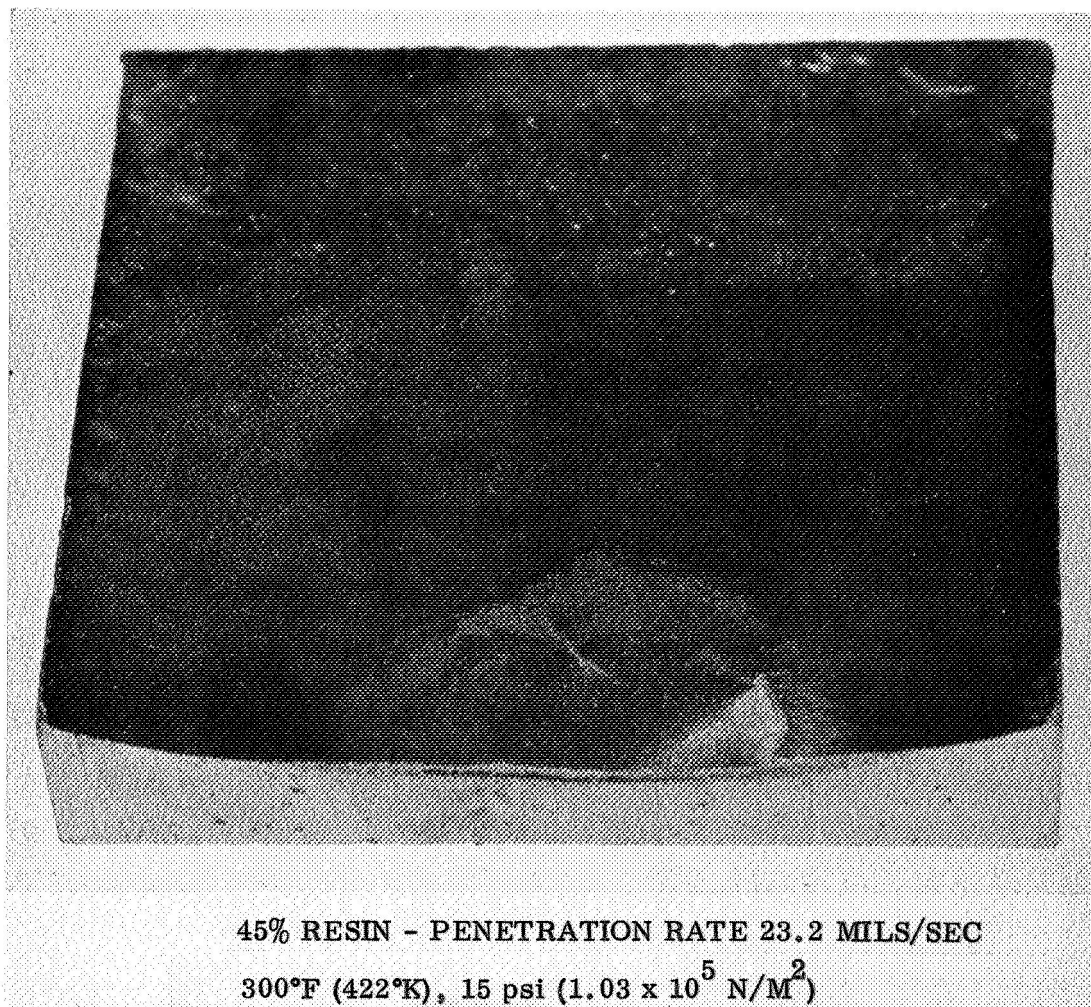


Figure 30. - Torch Test Sample, DEN 438 Epoxy Novolac/Graphite Composite, Mix 519

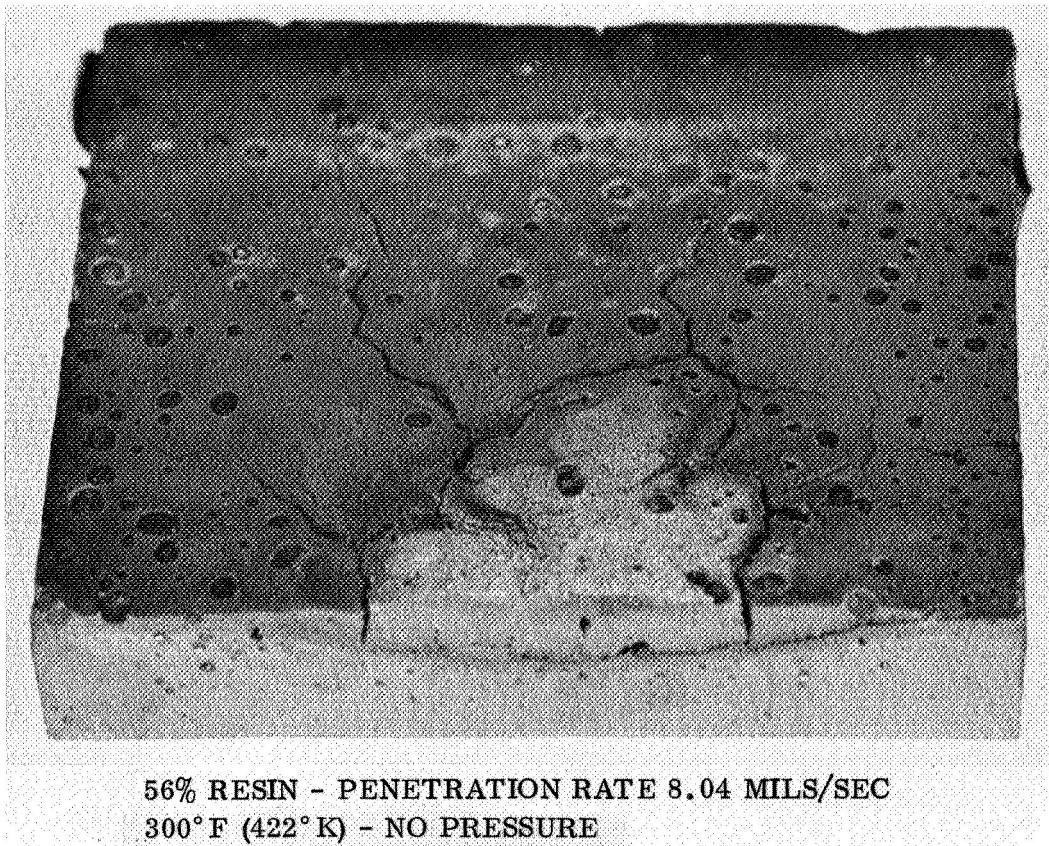


Figure 31. - Torch Test Sample, DEN 438 Epoxy Novolac/Graphite Composite, Mix 520

The data show that vacuum mixing offers superior tensile and compressive properties over the non-vacuum mixed material.

Based upon the above tests, the following parameters are recommended for mixing and curing of the DEN 438 epoxy novolac LCCM formulation.

Resin Mix

DEN 438	100 parts
NMA hardener	87.5 parts
BDMA	1.5 parts

Mix 44.75 percent resin system with 55.25 percent graphite powder mix of three parts GL-1008 and one part GL-1012. Vacuum mix and cure 2 hr at 200°F (366°K) followed by 15 hr at 300°F (422°K).

Particle Size Distribution Study

This study was conducted to determine the effect of graphite particle size on performance and physical properties of the three resin systems investigated in the formulation optimization studies. A particle size distribution study (Ro-Tap) was made on Great Lakes graphite powders: GL-1008, GL-1011, and GL-1012. The results were: (1) 50 percent of GL-1008 was found to be less than 74 microns in diameter, with a particle size range from 50 to 140 microns; (2) 50 percent of the GL-1012 was less than 325 microns, with a particle size range of 50 to 700 microns; and (3) the GL-1011 graphite was very similar to GL-1012 since 50 percent was less than 375 microns, with a range of 50 to 700 microns.

A particle size distribution curve is shown for GL-1008 in Figure 32, for GL-1011 in Figure 33, and for GL-1012 in Figure 34.

The particle size distribution was determined by passing the GL-1008, GL-1011, and GL-1012 graphite through wire sieves with square openings. The sieve sizes were 100, 150, and 200 mesh; i.e., less than 74 microns.

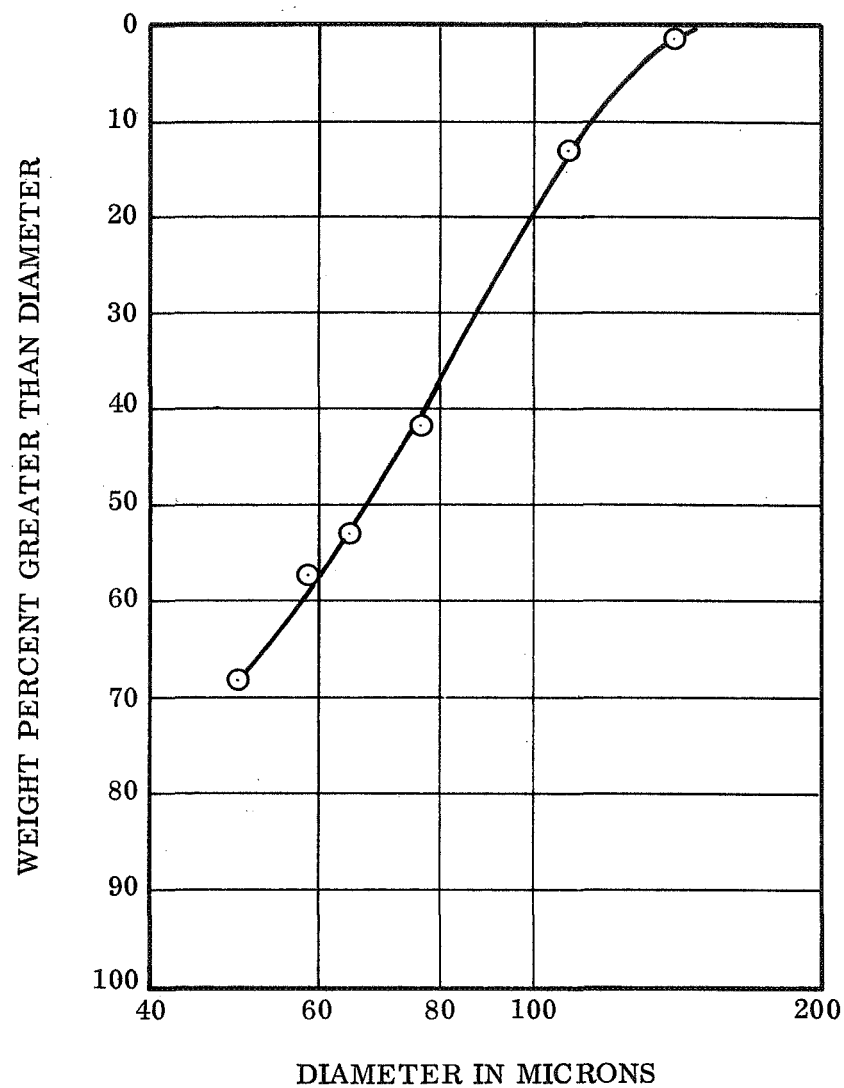
The collected graphite particles were then incorporated into the resin Monsanto RI-4009, Durez 10694, and DEN 438. Table VIII shows the test matrix for the RI-4009 Monsanto resin.

Test results shown in Table IX indicate good structural parts were fabricated from fine particles of graphite. The fine particle size materials produced the highest tensile properties.

Figure 35 shows the tensile strength/particle size relationship of a 40 percent RI-4009 resin and graphite composite. Test samples were cured at 1,000 psi (6.9×10^6 N/m²) and 300°F (422°K).

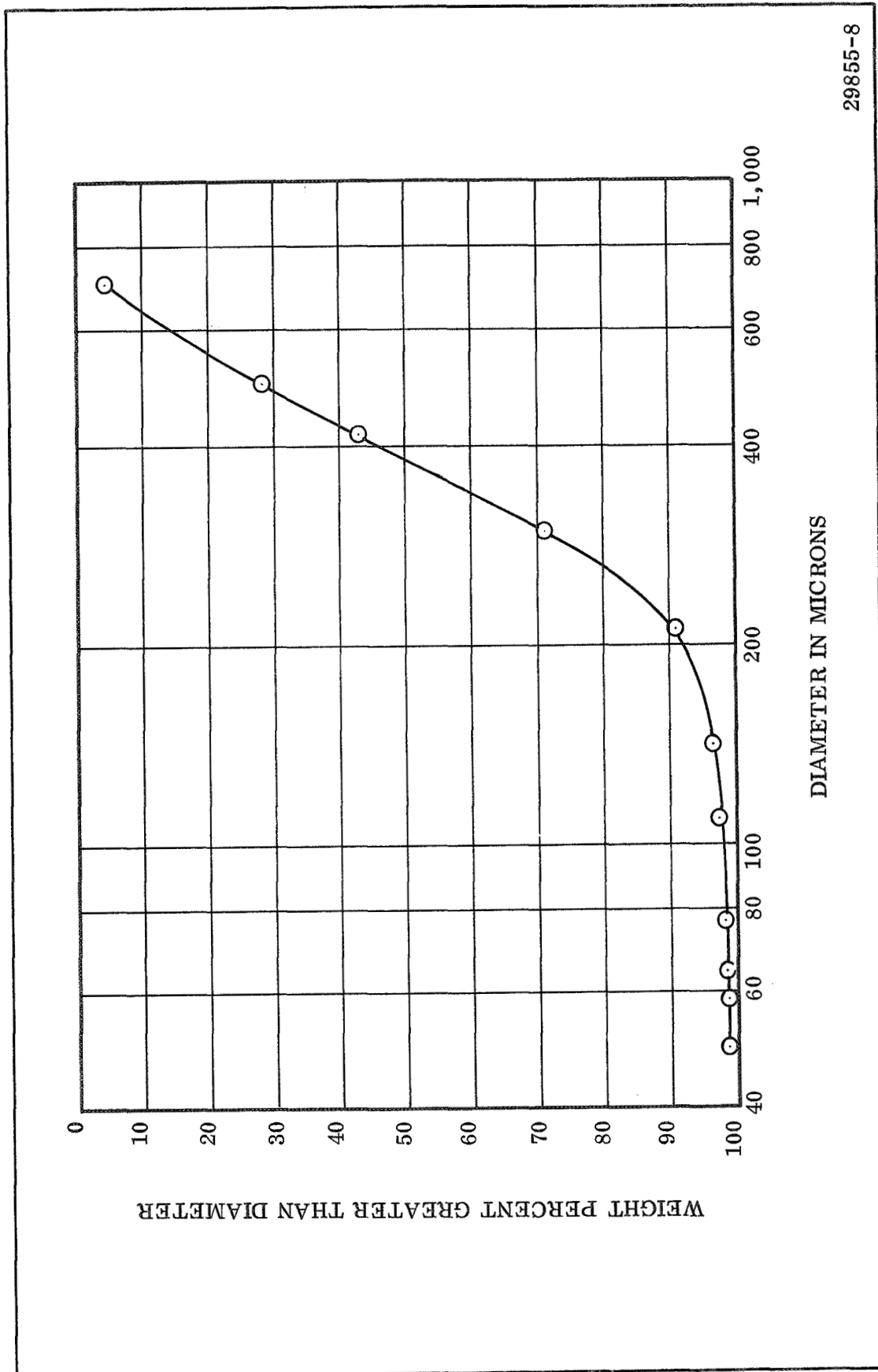
However, it was observed that these fine particle materials were more subject to thermal cracking and spalling as evidenced by the torch test. The thermal penetration rate/particle size relationship resulting from the torch test is shown in Figure 36.

In order to determine the effects of graphite particle size on the Durez 10694 resin system, two sizes of particles were utilized in the evaluation mixes: 74 microns or less, and 325 microns (average). Test panels (5 by 10 by 1.0 in. or 12.7 by 25.4 by 2.54 cm) were fabricated with 25 percent resin and 75 percent graphite. Samples were tested for tensile, density, compressive strength, and thermal properties.



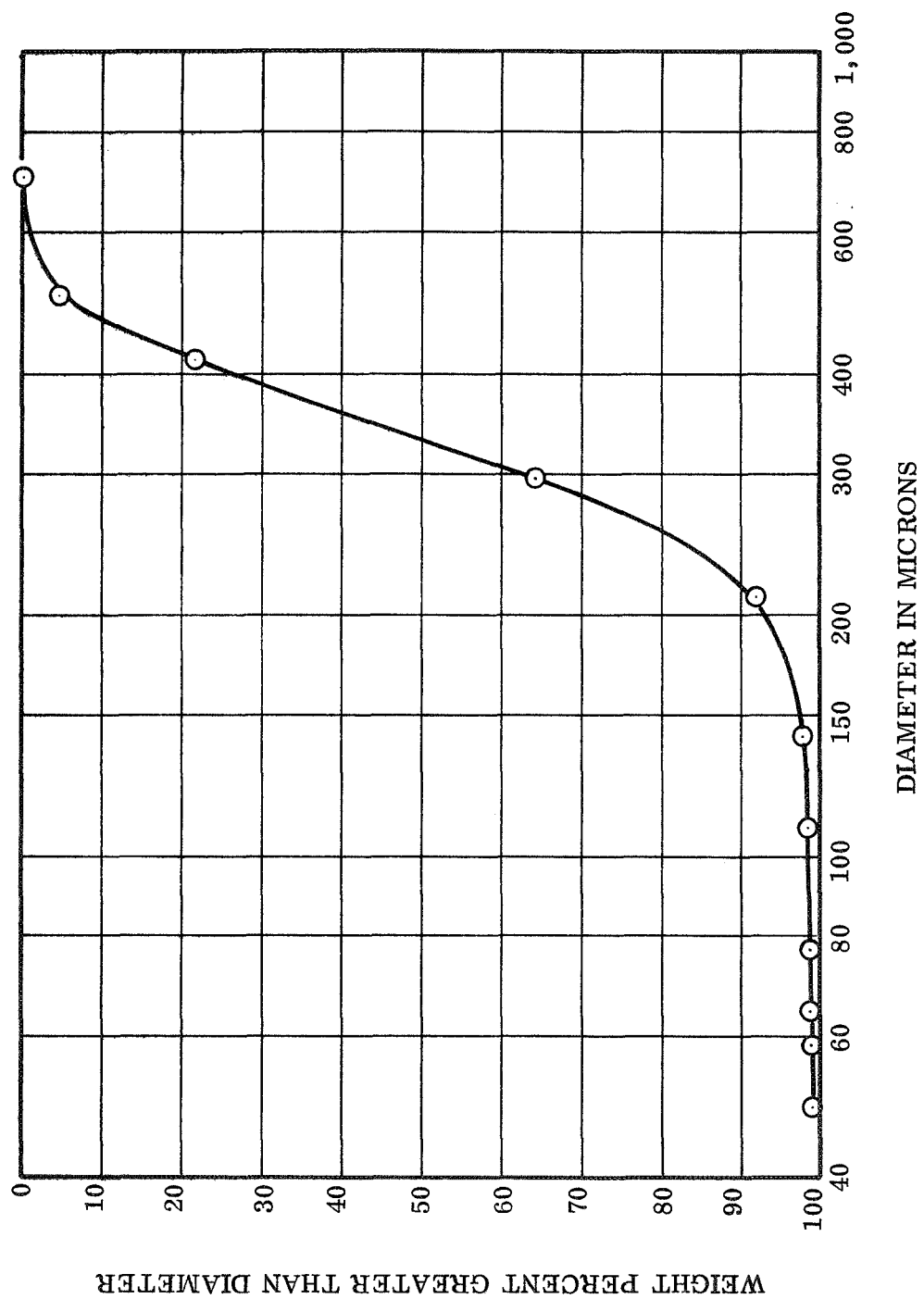
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Figure 32. - Particle Size Distribution, Ro-Tap, GL-1008 Graphite Powder



29855-8

Figure 33. - Particle Size Distribution, Ro-Tap, GL-1011 Graphite Powder



29855-6

Figure 34. - Particle Size Distribution, Ro-Tap, GL-1012 Graphite Powder

TABLE VIII. - LCCM PARTICLE SIZE DISTRIBUTION
TEST MATRIX

Variables

Resin system	RI-4009 (Monsanto)
Percent resin	40
Cure pressure	1,000 psi (6.9×10^6 N/m ²)
Cure temperature	300° F (422° K)

Particle size

Pan	Less than 200 mesh or 74 microns
200 mesh	74 micron
150 mesh	105 micron
100 mesh	149 micron
GL-1008 graphite	50% less than 74 micron
GL-1011 graphite	50% less than 325 micron
GL-1012 graphite	50% less than 375 micron

Tests

Tensile strength
Density
Compression
Tensile modulus
Thermal (torch test)

TABLE XX. - LCCM DEVELOPMENT, PARTICLE SIZE DISTRIBUTION STUDY
(RI-4009 MONSANTO RESIN)

Mix No.	Percent Resin	Percent Graphite	Graphite Particle Size	Tensile Strength (psi)	Tensile Strength (N/m ² x 10 ⁶)	Tensile Modulus (psi x 10 ⁶)	Tensile Modulus (N/m ² x 10 ⁹)	Density (g/cc)	Compressive Strength (psi)	Compressive Strength (N/m ² x 10 ⁶)	Penetration Rate (in./sec)	Penetration Rate (in./sec x 10 ⁻⁵)	Remarks
524	40	60	(Less than 200 mesh) or less than 74 microns	7,075	48.8	2.07	14.3	1.66	18,830	126.5	26.5	67.1	Severe cracking
				6,520	45.0	2.10	14.5	1.66	18,650	128.7			
				7,540	52.0	2.08	14.4	1.66	18,950	130.6			
				7,045	48.6	2.08	14.4	1.66	18,643	128.5			
525	40	60	200 mesh 74 microns	5,400	37.3	1.96	13.5	1.66	17,150	118.3	25.8	65.5	Severe cracking
				5,860	41.1	1.96	13.5	1.66	16,700	115.2			
				5,040	41.7	1.96	13.5	1.65	16,450	113.5			
				5,800	40.0	1.96	13.5	1.66	16,766	115.6			
526	40	60	150 mesh 105 microns	6,180	42.6	2.08	14.3	1.65	19,850	137.0	22.0	55.7	Severe cracking
				5,120	35.3	2.07	14.3	1.65	19,700	136.8			
				5,760	39.7	2.08	14.3	1.65	16,100	111.1			
				5,866	39.1	2.08	14.3	1.65	18,550	127.9			
527	40	60	100 mesh 149 microns	5,760	39.7	2.11	14.5	1.65	17,350	119.7	19.0	48.2	Severe cracking
				5,875	40.5	2.11	14.5	1.65	17,380	119.8			
				6,120	42.2	2.11	14.5	1.65	17,700	122.1			
				5,585	38.5	2.11	14.5	1.65	17,476	120.5			
528	40	60 (GL-1008)	50% of graphite is less than 74 microns. Range - 50 to 140 microns	6,280	43.3	2.07	14.3	1.66	17,750	122.4	23.8	60.5	Moderate cracking
				6,740	46.5	2.07	14.3	1.66	17,700	122.1			
				6,070	41.9	2.07	14.3	1.66	17,600	121.4			
				6,363	43.9	2.07	14.3	1.66	17,683	121.9			
530	40	60 (GL-1012)	50% of graphite is less than 325 microns. Range - 50 to 700 microns	4,140	28.6	1.59	10.96	1.64	16,825	116.1	17.0	43.2	Slight cracking of heat affected zone only
				4,740	32.7	1.59	10.96	1.65	17,025	117.4			
				4,250	29.3	1.62	11.17	1.65	17,150	118.3			
				4,377	30.2	1.60	11.03	1.65	17,000	117.2			
531	40	60 (GL-1011)	50% of graphite is less than 375 microns. Range - 50 to 700 microns	4,200	29.0	1.49	10.3	1.64	15,950	109.9	6.4	16.3 ~	Slight cracking of heat affected zone only
				4,380	30.2	1.49	10.3	1.65	16,275	112.3			
				4,385	30.3	1.48	10.2	1.64	16,250	112.1			
				4,321	29.8	1.49	10.3	1.64	16,158	111.40			

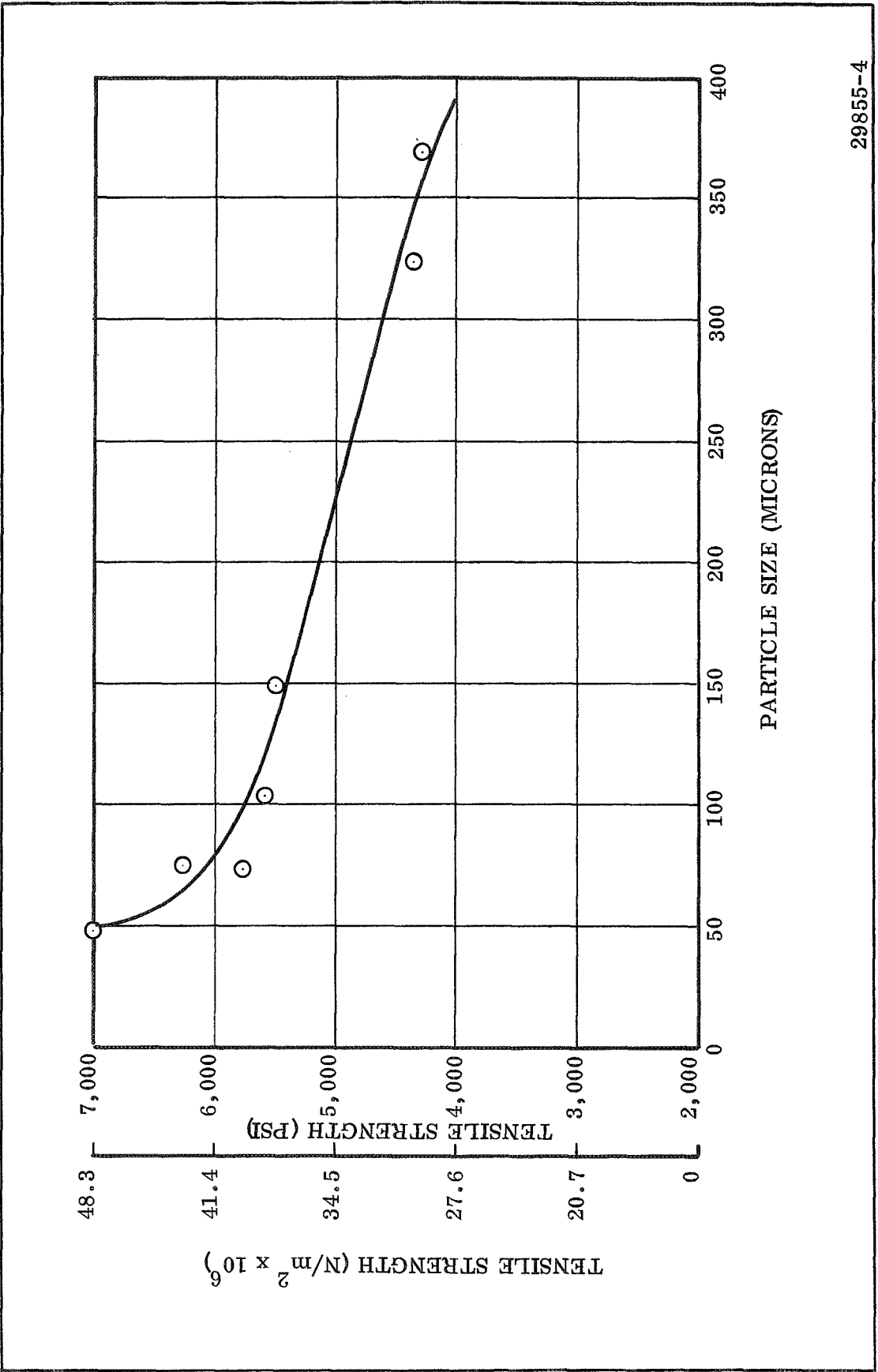


Figure 35. - Tensile Strength/Particle Size Curve, RI-4009 Resin

29855-4

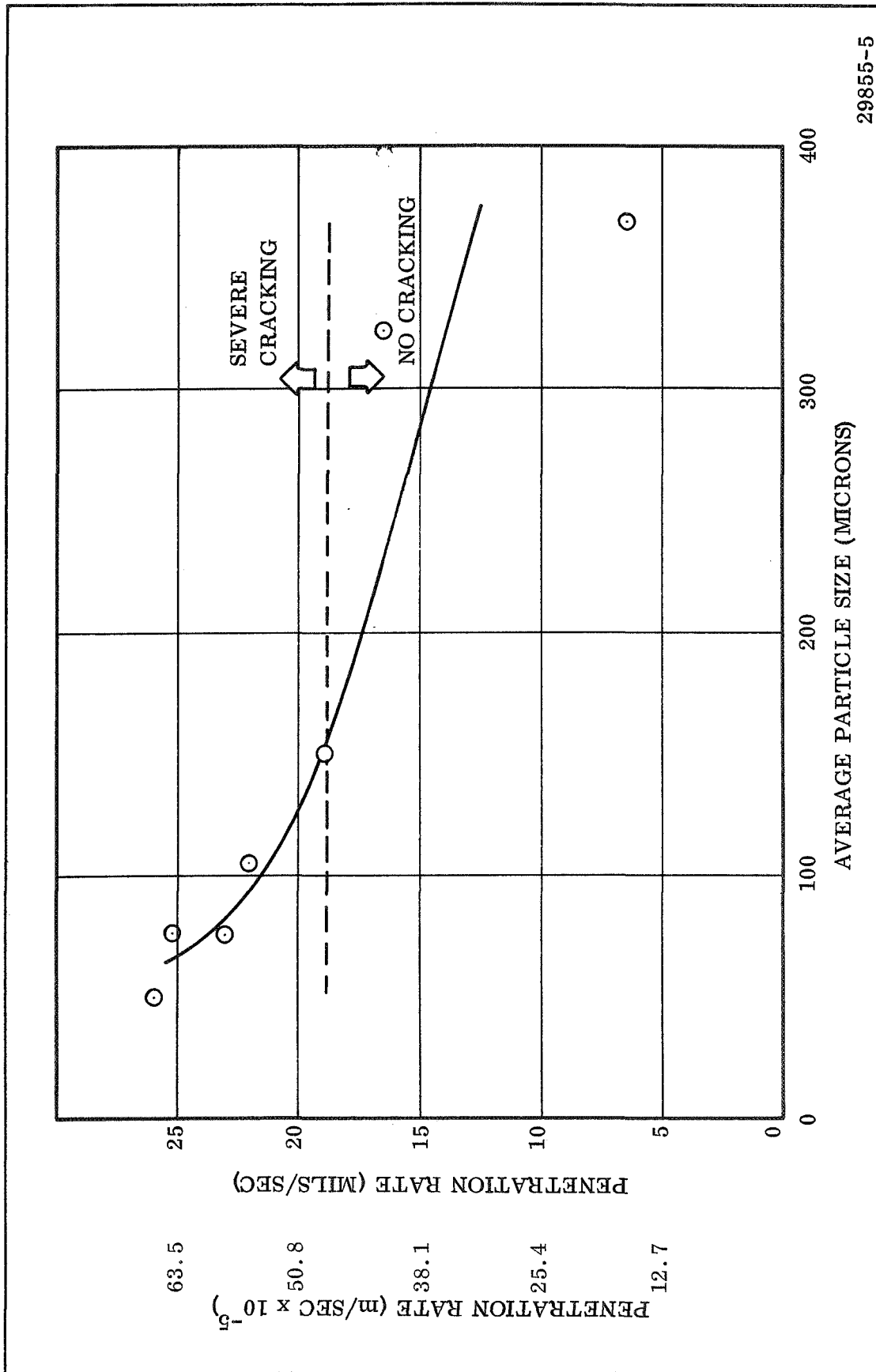


Figure 36. - Thermal Penetration/Particle Size Curve, RI-4009 Resin

29855-5

Test results are shown below.

Graphite Material	Tensile Strength		Tensile Modulus		Density (g/cc)	Compressive Strength	
	(psi)	(N/m ² x 10 ⁶)	(psi x 10 ⁶)	(N/m ² x 10 ⁹)		(psi)	(N/m ² x 10 ⁶)
74 Micron*	1,760	12.14	1.44	9.93	1.56	6,950	47.95
	2,720	18.77	1.44	9.93	1.56	9,570	65.98
	2,480	17.09	1.44	9.93	1.56	10,100	69.69
Average	2,320	16.08	1.44	9.93	1.56	8,873	61.22
325 Micron	1,770	12.21	1.14	7.86	1.47	7,175	49.50
Average	1,945	13.42	1.14	7.86	1.45	7,230	49.89
(GL-1012)**	1,815	12.52	1.14	7.86	1.47	6,120	42.22
Average	1,843	12.71	1.14	7.86	1.47	6,842	47.20

*Mix 547

**Mix 548

No thermal cracking or spalling occurred from the 325 micron material resulting from the thermal penetration tests. Slight cracking and spalling occurred in the 74 micron material when subjected to the same test.

The data show a moderate increase in tensile and compressive strengths with the smaller graphite particles. Density is also increased with the use of the small graphite material.

The effects of graphite particle size on the epoxy novolac DEN 438 were not determined. It was felt that the results collected from the other resin/particle size study could be applied to the epoxy novolac; i.e., decreasing particle size increases tensile and compressive strength with adverse effects in char retention, spalling, and thermal cracking.

Material Erosion Tests

The material formulations developed from the previous studies utilized the following three resin systems: a two stage phenolic resin, Monsanto RI-4009; a single stage resin, Durez 10694; and an epoxy novolac, DEN 438. Optimization studies were conducted on all three resin systems, and the relationship between temperature and pressure was evaluated. In addition, the effect of graphite particle size upon mechanical properties and the effects resulting from thermal tests were determined. The evaluation of these parameters relating to the above properties and processing considerations led to the selection of seven nozzle insulation materials. These candidate insulations were evaluated by erosion testing in 12 nozzles on the TU-379 motor (Figure 37). The formulations developed are shown in Table X. A summary of the erosion data of the 12 TU-379 nozzles and the physical properties is also shown in Table X.

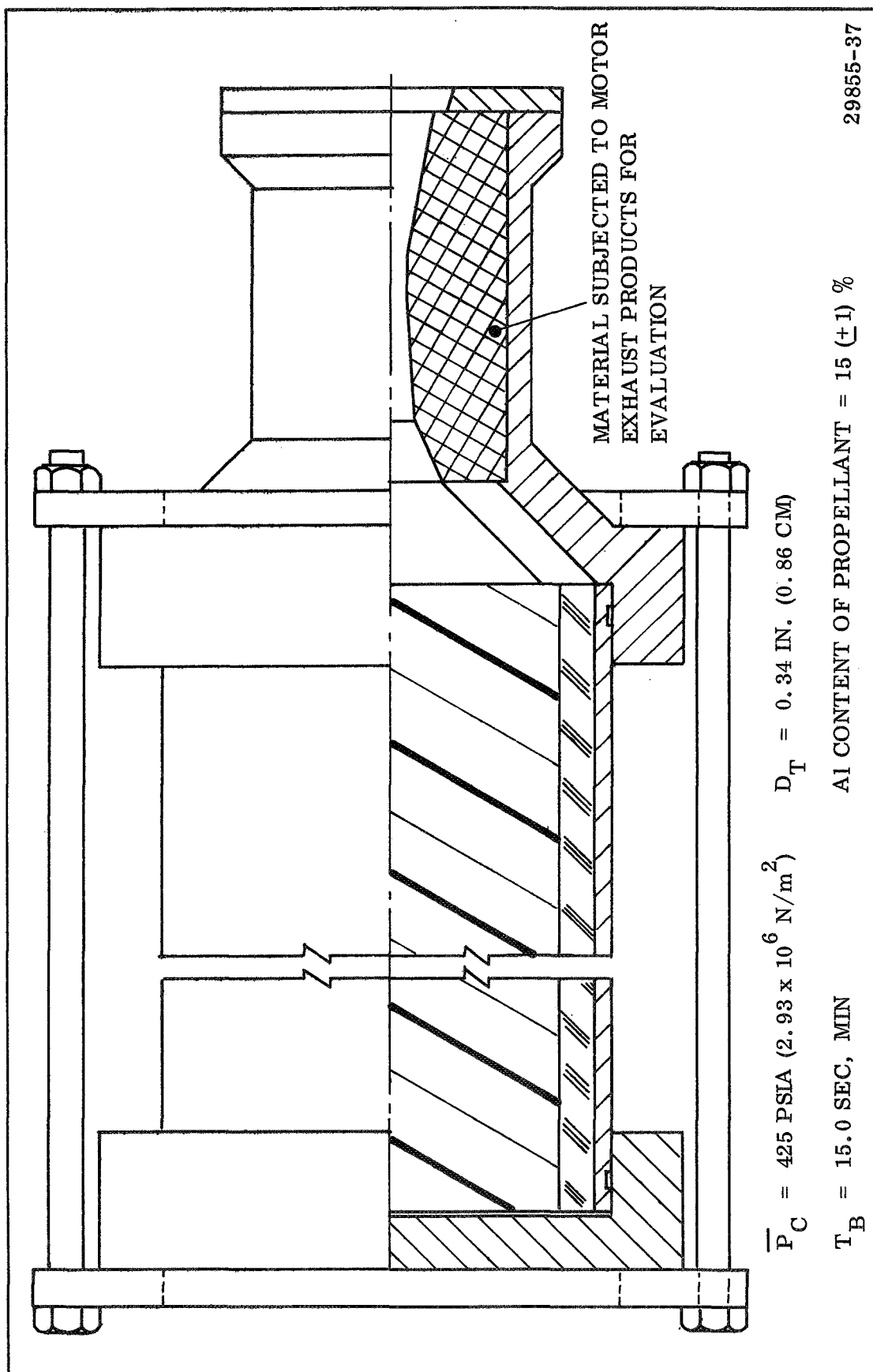


Figure 37. TU-379 Materials Screening Motor

29855-37

TABLE X. - LCCM DEVELOPMENT TU-479 MOTOR TEST MATRIX, EROSION DATA, AND PHYSICAL PROPERTIES

Resin	Percent Grinblite	Graphite Particle Size	Cure Temperature °F	Cure Temperature °C	Cure Pressure (psi)	Motor No.	Maximum Erosion Throat			Burn Time Time (sec)	Pressure Average (psi)	Physical Properties		Density (g/cm ³)		
							Forward Section (in/sec)	Mid Section (in/sec)	Exit Cone (in/sec)			Tensile Strength (psi)	Compressive Strength (psi)			
Monsanto RC-1009	40	60	300	422	1,000	549-1	2.4*	6.10	2.4*	16.77	420	4,140	28.54	16,825	1.64	
						549-2	1.2*	3.06	3.2*	15.48	430	4,740	32.68	17,025	1.65	
	40	60	300	422	1,000	565-1	11.2*	28.45	3.1*	16.12	431	4,376	29.17	17,000	1.66	
						565-2	7.6*	19.30	3.8*	15.85	513	6,520	44.85	18,950	1.66	
25	75		300	422	200	561-1	5.8*	13.97	2.3*	12.66	545	3,76	1,970	13.58	6,525	1.53
						561-2	7.5*	19.05	1.9*	16.08	424	2,92	2,005	14.86	6,400	1.51
25	75		300	422	1,000	572	4.1*	10.41	2.4	16.31	378	2.60	3,940	27.17	12,400	1.70
													4,390	36.27	11,820	1.75
Durez 10684	25	75	300	422	200	550-1	2.1*	5.33	1.0	19.48	247	1.70	1,770	12.26	7,175	1.47
						550-2	2.5	6.35	1.0	19.69	242	1.67	2,720	18.75	7,230	1.45
25	75		300	422	200	551	2.0*	5.08	1.4*	14.05	308	2.12	2,480	12.13	9,950	1.56
													2,790	18.75	9,570	1.56
DEN 438 Epoxy Novolac	25.67 resin 20.71 hardener 0.35 BDMA	55.24	300	422	None	566-1	2.6	6.60	7.8	34.73	N/A	2,480	17.10	10,100	69.54	1.56
			Vacuum mixed			566-2	8.9	9.91	10.5	36.10	N/A	4,130	28.47	14,300	98.94	1.62
												2,510	19.37	14,930	110.94	1.61
												3,550	27.23	14,635	109.96	1.61

*Growth

Each nozzle, after testing, was cut in half and a cross section photograph taken. The photos of the sectioned half are shown in Figures 38 thru 40. Figures 41 thru 52 show the erosion profiles, heat affected zones, and the erosion rates taken at 0.20 in. increments along the centerline of each test nozzle. Motor numbers correspond with those listed in Table X.

The TU-379 motor evaluations and material performances of the developed formulations involving the above resin systems are discussed below.

Monsanto RI-4009 Resin. - Basically, two resin levels were evaluated with the Monsanto RI-4009 resin system.

1. In the first series of experiments, 40 percent resin was blended with 60 percent graphite and cured at 300°F (422°K) and 1,000 psi (6.9×10^6 N/m²). Two different graphite particle sizes were used. One formulation had 74 micron maximum particle size; the other had an average particle size of 325 microns. Two TU-379 motors were tested for each of the two graphite formulations. Test results show those nozzles containing the small particle size graphite, 74 micron (Motor No. 562), exhibited more severe erosion and radial cracking than those manufactured with the large grain graphite (Motors No. 549 and 562). Motor No. 549-1 showed the lowest throat erosion rate of all RI-4009 resin systems tested at 1.2 mils/sec.
2. In the second series of experiments, 25 percent RI-4009 resin was blended with 75 percent graphite. Two mixes of this formulation were fabricated: two test billets cured at 300°F (422°K) and 200 psi (1.38×10^6 N/m²), and one test billet at 300°F (422°K) at 1,000 psi (6.9×10^6 N/m²). The graphite particles were kept constant for each mix: one part of GL-1012 and three parts GL-1008.

Test results indicated that when the materials with the 25 percent resin were cured at 200 psi (1.38×10^6 N/m²) on Motor No. 561 an average throat erosion ratio of 9.4 mils/sec (23.87 m/sec x 10^{-5}) was obtained, compared to 20.1 mils/sec (51.05 m/sec x 10^{-5}) on Motor No. 572 with material cured at 1,000 psi (6.9×10^6 N/m²). The radial cracking of the forward exit cone area of the higher pressure cure test motor, Motor No. 572, appeared to be more severe than the radial cracking of the material cured at the lower pressure, Motor No. 561.

Both formulations of the RI-4009 resin at the 25 and 40 percent levels, regardless of cure pressure or particle size of the graphite, exhibited good char retention. In addition, due to the off gassing from the phenolic resin, a growth was observed in the forward and exit cone area of each nozzle.

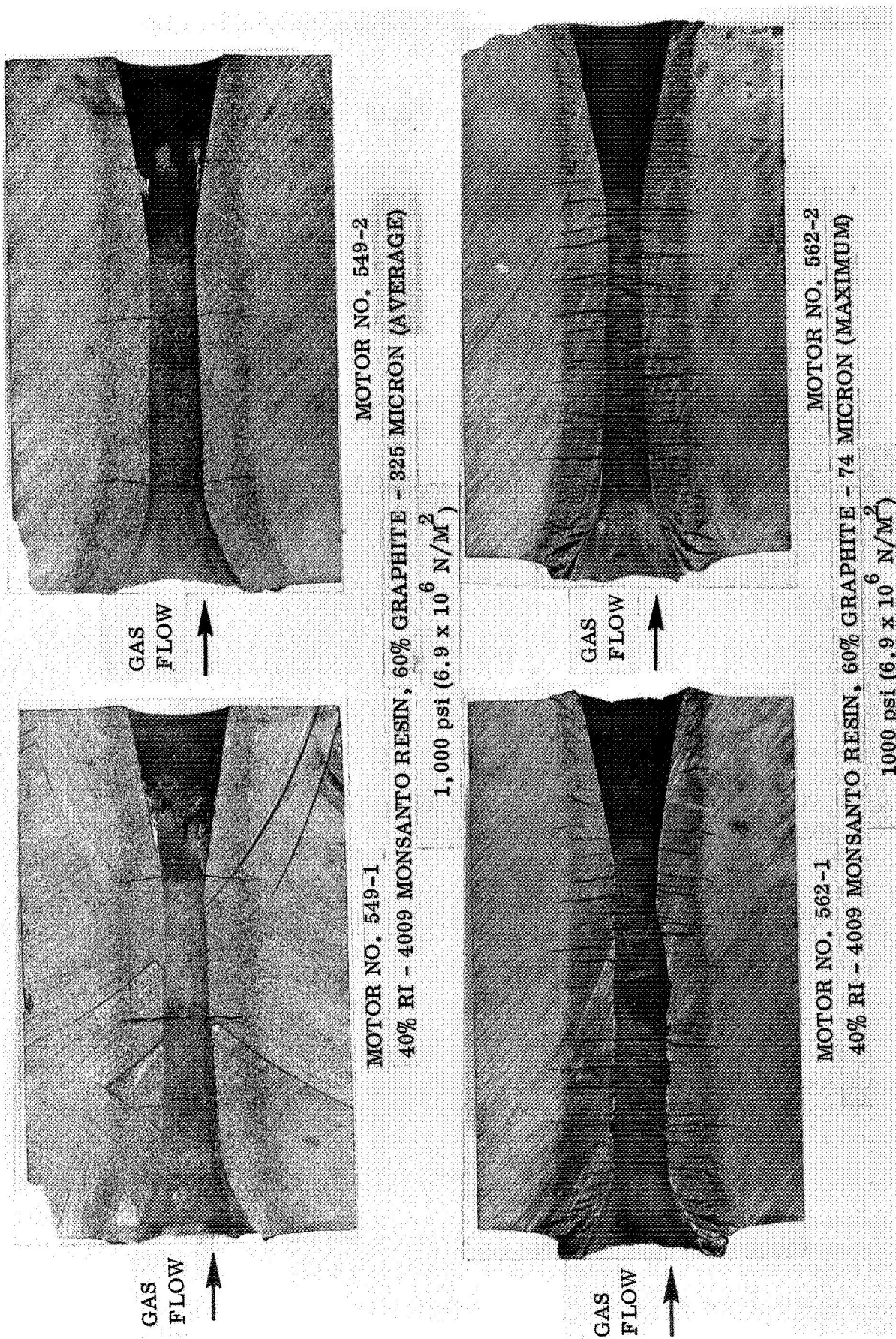
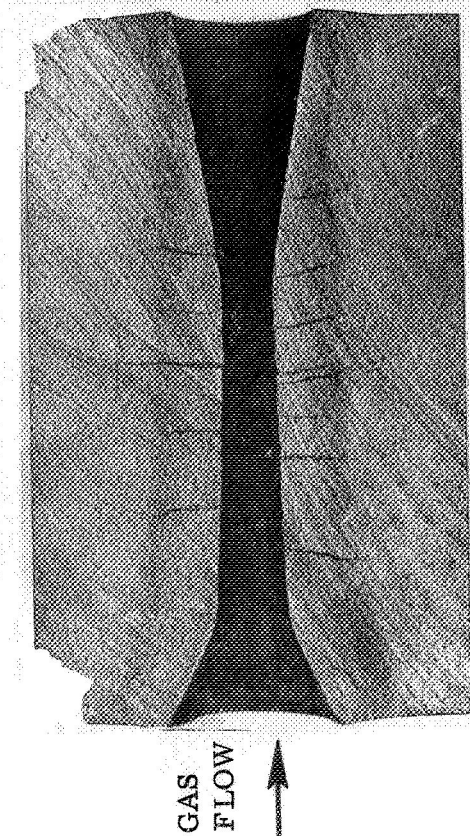


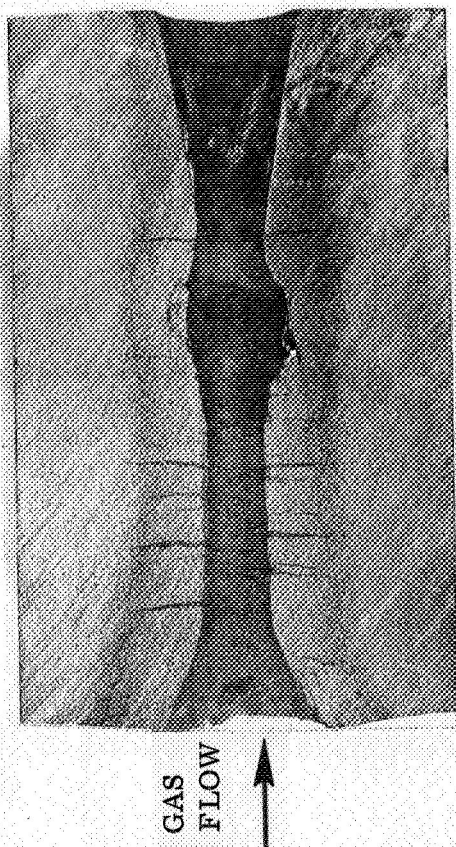
Figure 38. - Gas Flow Erosion, RI-4009 Resin



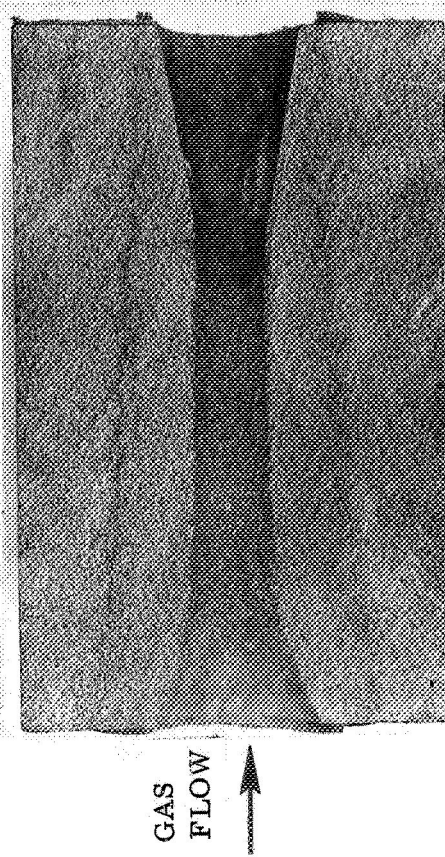
MOTOR NO. 561-1

25% RI - 4009 MONSANTO RESIN

75% GRAPHITE, 200 psi (1.38×10^6 N/M²) CURE



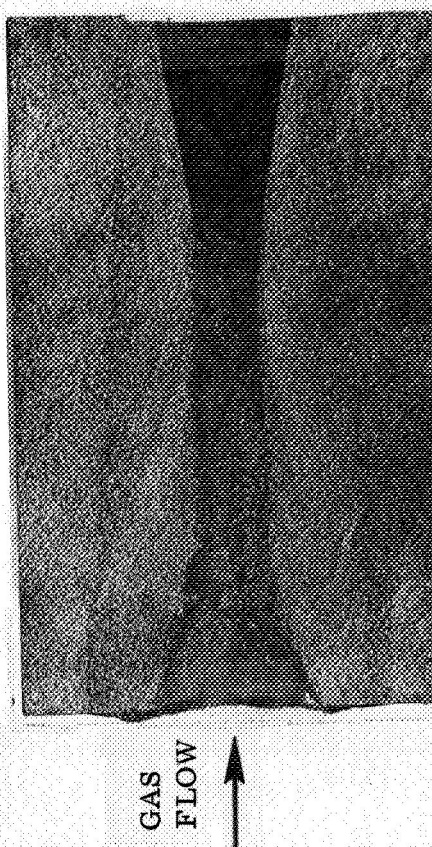
MOTOR NO. 561-2



MOTOR NO. 550-1

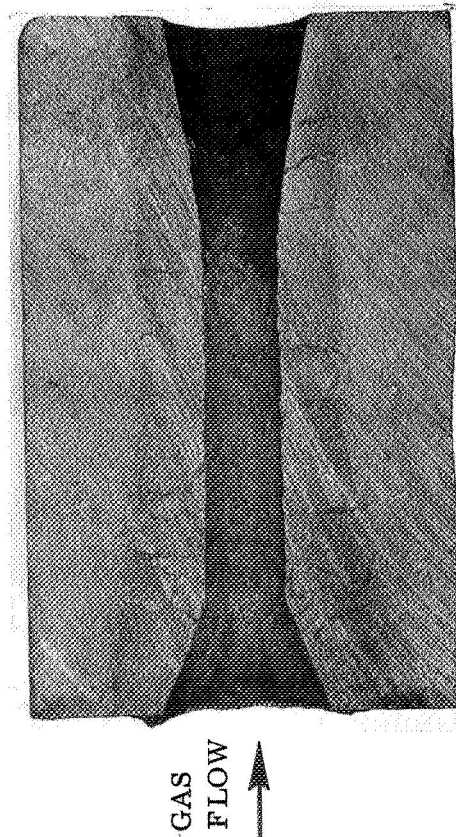
25% DUREZ 10694

75% GRAPHITE, 325 MICRON (AVERAGE) 200 psi (1.38×10^6 N/M²)



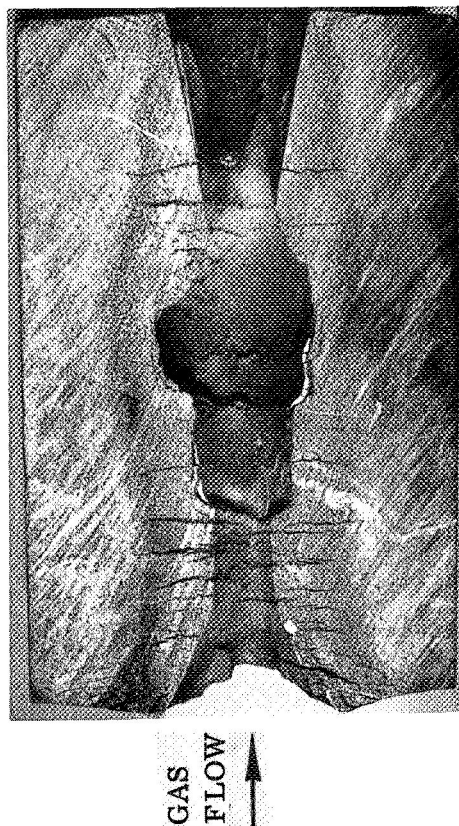
MOTOR NO. 550-2

Figure 39. - Gas Flow Erosion, RI-4009 Resin and Durez 10694 Resin



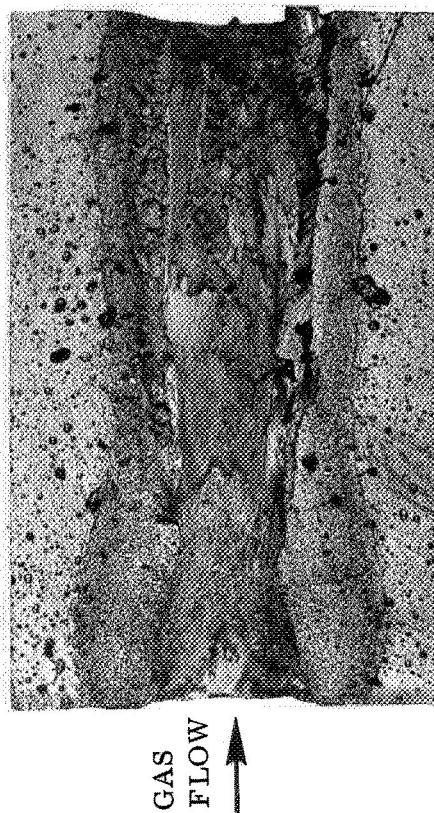
MOTOR NO. 551

75% GRAPHITE, 74 MICRON (MAXIMUM)
200 psi (1.38×10^6 N/M²) 25% DUREZ 10694



MOTOR NO. 572

25% RI - 4009 MONSANTO RESIN
75% GRAPHITE, 1,000 psi (6.9×10^6 N/M²)



MOTOR NO. 566-1

45% DEN 438 EPOXY NOVOLAC 55% GRAPHITE (325 MICRONS)
ZERO PRESSURE



MOTOR NO. 566-2

Figure 40. -Gas Flow Erosion, RI-4009 Resin, Durez 10694, and DEN Epoxy Novolac

40% RI-4009 Resin

60% 325 Micron Graphite (GL-1012)

Cured at 1,000 psi (6.9×10^6 N/m²)

Station No.	Contour				Material Loss		Char Depth		Erosion Rate	
	Initial		Erosion		(in.)	(m x 10 ⁻⁴)	(in.)	(m x 10 ⁻²)	(mils/sec)	(m/sec x 10 ⁻⁵)
	(in.)	(m)	(in.)	(m x 10 ⁻²)						
22	0.92	0.0233	0.94	0.0238	+	+	0.71	0.0180	+1.2	+3.04
21	0.96	0.0243	1.00	0.0254	+	+	0.74	0.0188	+2.4	+6.09
20	0.99	0.0251	1.03	0.0261	+	+	0.77	0.0195	+2.4	+6.09
19	1.04	0.0264	1.07	0.0271	+	+	0.79	0.020	+1.8	+4.57
18	1.08	0.0274	1.10	0.0279	+	+	0.82	0.0208	+1.2	+2.79
17	1.11	0.0281	1.15	0.0292	+	+	0.85	0.0215	+2.4	+6.09
16	1.14	0.0284	1.18	0.0299	+	+	0.85	0.0215	+2.4	+6.09
15	1.18	0.0299	1.20	0.0305	+	+	0.82	0.0208	+1.2	+3.04
14	1.21	0.0307	1.23	0.0312	+	+	0.82	0.0208	+1.2	+3.04
13	1.24	0.0314	1.24	0.0314	0	0	0.82	0.0208	0	0
12	1.24	0.0314	1.24	0.0314	0	0	0.80	0.0203	0	0
11	1.24	0.0314	1.22	0.0314	0.02	0.0005	0.80	0.0203	1.2	3.04
10	1.23	0.0312	1.21	0.0307	0.02	0.0005	0.79	0.020	1.2	3.04
9	1.22	0.0309	1.20	0.0304	0.02	0.0005	0.78	0.0198	1.2	3.04
8	1.22	0.0309	1.20	0.0304	0.02	0.0005	0.77	0.0195	1.2	3.04
7	1.21	0.0307	1.19	0.0302	0.02	0.0005	0.75	0.0190	1.2	3.04
6	1.20	0.0304	1.19	0.0302	0.01	0.0002	0.72	0.0182	0.6	1.52
5	1.19	0.0300	1.18	0.0299	0.01	0.0002	0.70	0.0177	0.6	1.52
4	1.18	0.0299	1.17	0.0297	0.01	0.0002	0.68	0.0172	0.6	1.52
3	1.12	0.0284	1.15	0.0292	+	+	0.64	0.0162	+1.8	+4.57
2	1.04	0.0264	1.08	0.0274	+	+	0.60	0.0152	+2.4	+6.09
1	0.95	0.0241	1.00	0.0254	+	+	0.56	0.0142	+3.0	+7.62
0	0.87	0.0220	0.85	0.0215	0.02	0.0005	0.50	0.0127	1.2	3.04

Legend

—————	Prefiring Surface
-----	Postfiring Surface
-----	Char Depth

Motor Parameters

Burn Time	16.77 sec
Web Pressure	420 psi (2.9×10^6 N/m ²)
Throat Diameter	0.34 in. (0.86 cm)

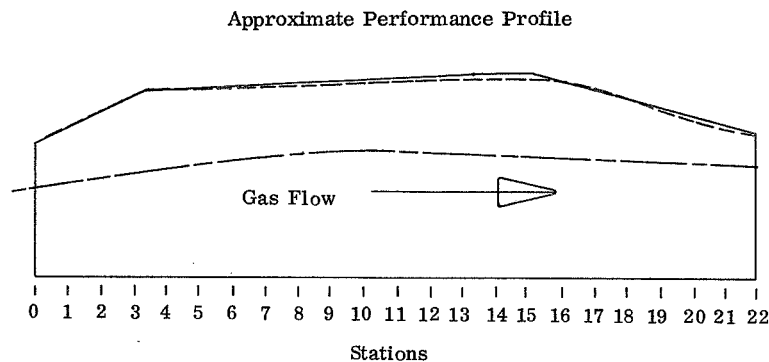


Figure 41. - TU-379 Nozzle Data, RI-4009 Resin, Motor No. 549.1

25% RI-4009 Resin

75% Large and Small Graphite

Cured at 1,000 psi (6.9×10^6 N/m²)

Station No.	Contour		Erosion		Material Loss		Char Depth		Erosion Rate	
	Initial (in.)	(m)	(in.)	(m x 10 ⁻²)	(in.)	(m x 10 ⁻⁴)	(in.)	(m x 10 ⁻²)	(mils/sec)	(m/sec x 10 ⁻⁵)
22	0.92	0.0233	0.96	2.43	+	+	0.80	2.03	+2.4	6.09
21	0.96	0.0243	1.00	2.54	+	+	0.77	1.95	+2.4	6.09
20	0.99	0.0251	1.03	2.61	+	+	0.75	1.90	+2.4	6.09
19	1.04	0.0264	1.08	2.74	+	+	0.72	1.82	+2.4	6.09
18	1.08	0.0274	1.10	2.79	+	+	0.71	1.80	+1.2	3.04
17	1.11	0.0281	1.12	2.84	+	+	0.70	1.77	0.6	1.52
16	1.14	0.0284	1.16	2.94	+	+	0.68	1.72	1.2	3.04
15	1.18	0.0299	1.15	2.92	0.03	7.62	0.63	1.60	1.8	4.57
14	1.21	0.0307	1.08	2.74	0.13	33.02	0.57	1.44	7.7	19.55
13	1.24	0.0314	1.00	2.54	0.24	60.96	0.52	1.32	14.1	35.81
12	1.24	0.0314	0.90	2.28	0.34	86.36	0.56	1.42	20.1	51.05
11	1.24	0.0314	1.03	2.61	0.21	53.34	0.61	1.54	12.4	31.49
10	1.23	0.0312	1.08	2.74	0.15	38.10	0.64	1.62	8.9	22.60
9	1.22	0.0309	1.08	2.74	0.14	35.56	0.68	1.72	8.2	20.82
8	1.22	0.0309	1.07	2.71	0.15	38.10	0.70	1.77	8.9	22.60
7	1.21	0.0307	1.06	2.69	0.15	38.10	0.73	1.85	8.9	22.60
6	1.20	0.0304	1.19	3.02	0.01	2.54	0.75	1.90	0.6	1.52
5	1.19	0.0300	1.19	3.02	0.00	0.00	0.79	2.00	0.0	0.00
4	1.18	0.0299	1.19	3.02	+	+	0.73	1.85	+0.6	+1.52
3	1.12	0.0284	1.16	2.94	+	+	0.68	1.72	+2.4	+6.09
2	1.04	0.0264	1.10	2.79	+	+	0.65	1.65	+3.5	+8.89
1	0.95	0.0241	1.01	2.56	+	+	0.54	1.37	+3.5	+8.89
0	0.87	0.0220	0.93	2.36	+	+	0.44	1.11	+3.5	+8.89

Legend

—————	Prefiring Surface
-----	Postfiring Surface
-----	Char Depth

Motor Parameters

Burn Time	16.91 sec
Web Pressure	379 psi (2.61×10^6 N/m ²)
Throat Diameter	0.34 in. (0.86 cm)

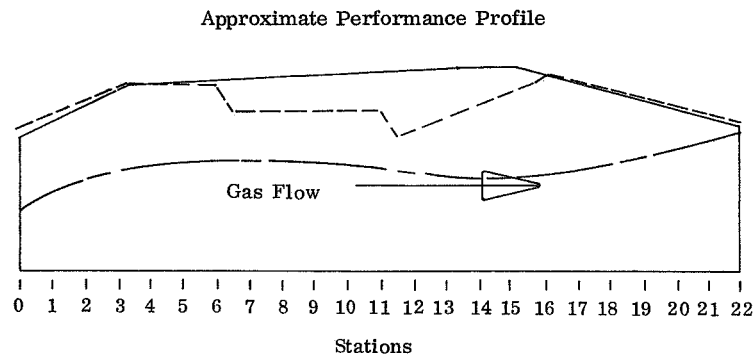


Figure 42. - TU-379 Nozzle Data, RI-4009 Resin, Motor No. 549.2

40% RI-4009 Resin
60% 74 Micron Graphite
Cured at 1,000 psi (6.9×10^6 N/m²)

Station No.	Contour				Material Loss		Char Depth		Erosion Rate	
	Initial		Erosion		(in.)	(m x 10 ⁻⁴)	(in.)	(m x 10 ⁻²)	(mils/sec)	(m/sec x 10 ⁻⁵)
22	0.92	0.0233	0.92	2.33	0	0	0.72	1.82	0	0
21	0.96	0.0243	1.01	2.56	+	+	0.72	1.82	+3.1	+7.87
20	0.99	0.0251	1.04	2.64	+	+	0.73	1.85	+3.1	+7.87
19	1.04	0.0264	1.08	2.74	+	+	0.75	1.90	+2.5	+6.35
18	1.08	0.0274	1.11	2.81	+	+	0.77	1.95	+1.9	+4.82
17	1.11	0.0281	1.14	2.89	+	+	0.78	1.98	+1.9	+4.82
16	1.14	0.0284	1.17	2.97	+	+	0.78	1.98	+1.9	+4.82
15	1.18	0.0299	1.20	3.04	+	+	0.79	2.00	+1.2	+3.04
14	1.21	0.0307	1.19	3.02	0.02	5.08	0.79	2.00	1.2	3.04
13	1.24	0.0314	1.20	3.04	0.04	10.16	0.80	2.03	2.5	6.35
12	1.24	0.0314	1.18	2.99	0.06	15.24	0.80	2.03	3.7	9.39
11	1.24	0.0314	1.14	2.89	0.04	10.16	0.77	1.95	6.2	15.74
10	1.23	0.0312	1.11	2.81	0.12	30.48	0.76	1.93	7.4	18.79
9	1.22	0.0309	1.09	2.76	0.13	33.02	0.76	1.93	8.0	20.32
8	1.22	0.0309	1.14	2.89	0.08	20.32	0.76	1.93	5.0	12.70
7	1.21	0.0307	1.14	2.89	0.07	17.78	0.77	1.95	4.3	10.92
6	1.20	0.0304	1.11	2.81	0.09	22.86	0.76	1.93	5.6	14.22
5	1.19	0.0300	1.14	2.89	0.05	12.70	0.76	1.93	3.1	7.87
4	1.18	0.0299	1.16	2.84	0.02	5.08	0.75	1.90	1.2	3.04
3	1.12	0.0284	1.16	2.84	+	+	0.73	1.85	+2.5	+6.35
2	1.04	0.0264	1.15	2.82	+	+	0.71	1.80	+6.8	+17.27
1	0.95	0.0241	1.09	2.76	+	+	0.66	1.67	+11.2	+28.44
0	0.87	0.0220	1.04	2.64	+	+	0.60	1.52	+10.5	+26.67

Legend		Motor Parameters	
—————	Prefiring Surface	Burn Time	16.12 sec
-----	Postfiring Surface	Web Pressure	431 psi (3.0×10^6 N/m ²)
-----	Char Depth	Throat Diameter	0.34 in. (0.86 cm)

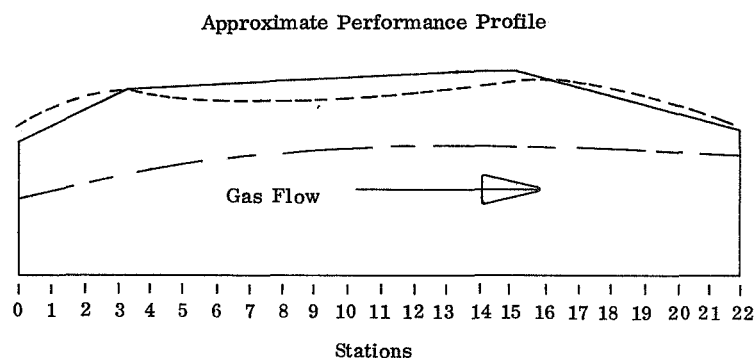


Figure 43. - TU-379 Nozzle Data, RI-4009 Resin, Motor No. 562.1

40% RI-4009 Resin
 60% 74 Micron Graphite
 Cured at 1,000 psi ($6.9 \times 10^6 \text{ N/m}^2$)

Station No.	Contour				Material Loss		Char Depth		Erosion Rate	
	Initial		Erosion		(in.)	(m x 10^{-4})	(in.)	(m x 10^{-2})	(mils/sec)	(m/sec x 10^{-5})
	(in.)	(m)	(in.)	(m x 10^{-2})						
22	0.92	0.0233	0.98	2.48	+	+	0.78	1.98	+3.8	+9.65
21	0.96	0.0243	1.02	2.59	+	+	0.80	2.03	+3.8	+9.65
20	0.99	0.0251	1.05	2.66	+	+	0.80	2.03	+3.8	+9.65
19	1.04	0.0264	1.10	2.79	+	+	0.80	2.03	+3.8	+9.65
18	1.08	0.0274	1.12	2.84	+	+	0.81	2.05	+2.5	+6.35
17	1.11	0.0281	1.16	2.94	+	+	0.82	2.08	+3.2	+8.12
16	1.14	0.0284	1.19	3.02	+	+	0.84	2.13	+3.2	+8.12
15	1.18	0.0299	1.21	3.07	+	+	0.85	2.15	+1.9	+8.82
14	1.21	0.0307	1.22	3.09	+	+	0.85	2.15	+0.6	+1.52
13	1.24	0.0314	1.23	3.12	0.01	2.54	0.87	2.20	0.6	1.52
12	1.24	0.0314	1.22	3.09	0.02	5.08	0.85	2.15	1.3	3.30
11	1.24	0.0314	1.22	3.09	0.02	5.08	0.85	2.15	1.3	3.30
10	1.23	0.0312	1.22	3.09	0.01	2.54	0.84	2.13	0.6	1.52
9	1.22	0.0309	1.21	3.07	0.01	2.54	0.84	2.13	0.6	1.52
8	1.22	0.0309	1.21	3.07	0.01	2.54	0.86	2.18	0.6	1.52
7	1.21	0.0307	1.20	3.04	0.01	2.54	0.83	2.10	0.6	1.52
6	1.20	0.0304	1.19	3.02	0.01	2.54	0.82	2.08	0.6	1.52
5	1.19	0.0300	1.17	2.97	0.02	5.08	0.81	2.05	1.3	3.30
4	1.18	0.0299	1.16	2.94	0.02	5.08	0.78	1.88	1.3	3.30
3	1.12	0.0284	1.16	2.94	+	+	0.74	1.87	+2.5	+6.35
2	1.04	0.0264	1.14	2.89	+	+	0.70	1.77	+6.3	+16.0
1	0.95	0.0241	1.07	2.71	+	+	0.61	1.54	+7.6	+19.30
0	0.87	0.0220	0.98	2.48	+	+	0.51	1.29	+6.9	+17.52

Legend		Motor Parameters	
—————	Prefiring Surface	Burn Time	15.85 sec
-----	Postfiring Surface	Web Pressure	513 psi ($3.6 \times 10^6 \text{ N/m}^2$)
-----	Char Depth	Throat Diameter	0.34 in. (0.86 cm)

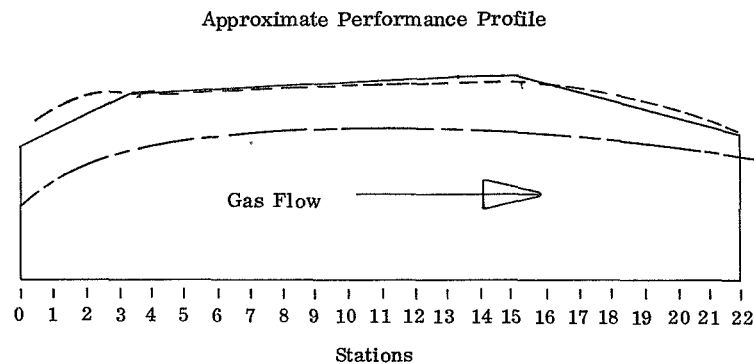


Figure 44. - TU-379 Nozzle Data, RI-4009 Resin, Motor No. 562.2

25% RI-4009 Resin
 75% Large and Small Graphite
 Cured at 200 psi (1.38×10^6 N/m²)

Station No.	Contour				Material Loss		Char Depth		Erosion Rate	
	Initial (in.)	(m)	Erosion (in.)	(m x 10 ⁻²)	(in.)	(m x 10 ⁻⁴)	(in.)	(m x 10 ⁻²)	(mils/sec)	(m/sec x 10 ⁻⁵)
22	0.92	0.0233	--	--	--	--	--	--	--	--
21	0.96	0.0243	0.97	2.46	+	+	0.78	1.98	+0.8	+2.03
20	0.99	0.0251	1.02	2.59	+	+	0.80	2.03	+2.3	+5.84
19	1.04	0.0264	1.06	2.69	+	+	0.86	2.18	+1.6	+4.06
18	1.08	0.0274	1.09	2.76	+	+	0.87	2.20	+0.8	+2.03
17	1.11	0.0281	1.12	2.84	+	+	0.86	2.18	+0.8	+2.03
16	1.14	0.0284	1.16	2.84	+	+	0.84	2.13	+1.6	+4.06
15	1.18	0.0299	1.19	3.02	+	+	0.82	2.08	+0.8	+2.03
14	1.21	0.0307	1.21	3.07	0	0	0.82	2.08	0	0
13	1.24	0.0314	1.22	3.09	0.02	5.08	0.82	2.08	1.6	4.06
12	1.24	0.0314	1.21	3.07	0.03	7.62	0.81	2.05	2.3	5.84
11	1.24	0.0314	1.21	3.07	0.03	7.62	0.80	2.03	2.3	5.84
10	1.23	0.0312	1.20	3.04	0.03	7.62	0.78	1.98	2.3	5.84
9	1.22	0.0309	1.18	2.99	0.04	10.16	0.76	1.93	3.1	7.87
8	1.22	0.0309	1.18	2.99	0.04	1.16	0.76	1.93	3.1	7.87
7	1.21	0.0307	1.18	2.99	0.03	7.62	0.76	1.93	2.3	5.84
6	1.20	0.0304	1.19	3.02	0.01	2.54	0.77	1.95	0.8	2.03
5	1.19	0.0300	1.16	2.84	0.03	7.62	0.74	1.87	2.3	5.84
4	1.18	0.0299	1.15	2.82	0.03	7.62	0.72	1.82	2.3	5.84
3	1.12	0.0284	1.15	2.92	+	+	0.66	1.67	+2.3	+5.84
2	1.04	0.0264	1.06	2.69	+	+	0.61	1.54	+4.7	+11.93
1	0.95	0.0241	1.01	2.56	+	+	0.56	1.42	+4.7	+11.93
0	0.87	0.0220	0.92	2.33	+	+	0.54	1.37	+3.9	+9.90

Legend

———— Prefiring Surface
 - - - - - Postfiring Surface
 - - - - - Char Depth

Motor Parameters

Burn Time 12.66 sec
 Web Pressure 545 psi (3.8×10^6 N/m²)
 Throat Diameter 0.34 in. (0.86 cm)

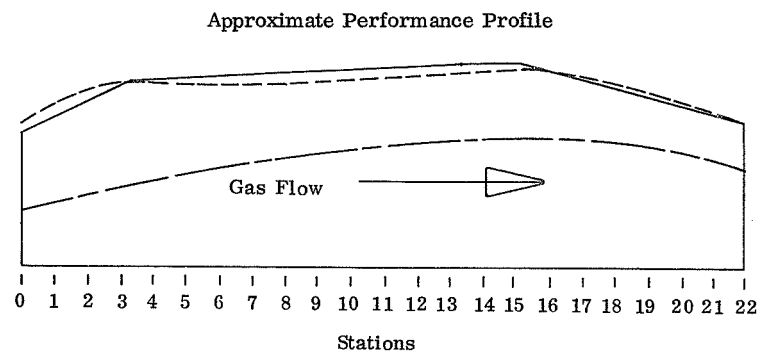


Figure 45. - TU-379 Nozzle Data, RI-4009 Resin, Motor No. 561.1

25% RI-4009 Resin

75% Large and Small Graphite

Cured at 1,000 psi (6.9×10^6 N/m²)

Station No.	Contour		Erosion		Material Loss		Char Depth		Erosion Rate	
	(in.)	(m)	(in.)	(m x 10 ⁻²)	(in.)	(m x 10 ⁻⁴)	(in.)	(m x 10 ⁻²)	(mils/sec)	(m/sec x 10 ⁻⁵)
22	0.92	0.0233	--	--	--	--	--	--	--	--
21	0.96	0.0243	0.99	2.51	+	+	0.70	1.77	+1.9	+4.82
20	0.99	0.0251	1.02	2.59	+	+	0.69	1.75	+1.9	+4.82
19	1.04	0.0264	1.06	2.69	+	+	0.70	1.77	+1.2	+3.04
18	1.08	0.0274	1.09	2.76	+	+	0.71	1.80	+0.6	+1.52
17	1.11	0.0281	1.13	2.87	+	+	0.72	1.82	+1.2	+3.04
16	1.14	0.0284	1.16	2.94	+	+	0.72	1.82	+1.2	+3.04
15	1.18	0.0299	1.18	2.99	0	0	0.72	1.82	0	0
14	1.21	0.0307	1.15	2.92	0.06	15.2	0.71	1.80	3.7	9.39
13	1.24	0.0314	1.07	2.71	0.17	43.1	0.71	1.80	10.6	26.92
12	1.24	0.0314	1.03	2.61	0.21	53.3	0.71	1.80	13.0	33.02
11	1.24	0.0314	1.00	2.54	0.24	60.9	0.71	1.80	14.9	27.84
10	1.23	0.0312	1.16	2.94	0.07	17.7	0.72	1.82	4.4	11.17
9	1.22	0.0309	1.18	2.99	0.04	10.6	0.72	1.82	2.5	6.35
8	1.22	0.0309	1.19	3.02	0.03	7.6	0.72	1.82	1.9	4.82
7	1.21	0.0307	1.21	3.07	0	0	0.72	1.82	0	0
6	1.20	0.0304	1.20	3.04	0	0	0.71	1.80	0	0
5	1.19	0.0300	1.20	3.04	+	+	0.71	1.80	+0.6	+1.52
4	1.18	0.0299	1.19	3.02	+	+	0.70	1.77	+0.6	+1.52
3	1.12	0.0284	1.19	3.02	+	+	0.70	1.77	+4.4	+11.17
2	1.04	0.0264	1.16	2.94	+	+	0.67	1.70	+7.5	+19.05
1	0.95	0.0241	1.07	2.71	+	+	0.59	1.49	+7.5	+19.05
0	0.87	0.0220	0.95	2.41	+	+	0.48	1.21	+4.9	+12.44

Legend

————— Prefiring Surface
 - - - - - Postfiring Surface
 - - - - - Char Depth

Motor Parameters

Burn Time 16.08 sec
 Web Pressure 424 psi (2.9×10^6 N/m²)
 Throat Diameter 0.34 in. (0.86 cm)

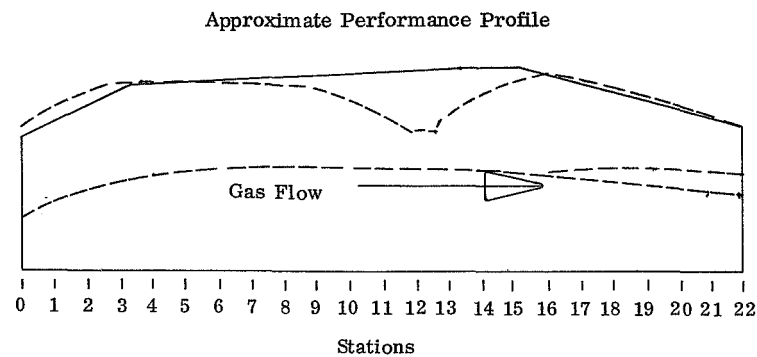


Figure 46. - TU-379 Nozzle Data, RI-4009 Resin, Motor No. 561.2

40% RI-4009 Resin

60% 325 Micron Graphite (GL-1012)

Cured at 1,000 psi (6.8×10^6 N/m²)

Station No.	Contour				Material Loss		Char Depth		Erosion Rate	
	Initial (in.)	(m)	Erosion (in.)	(m x 10 ⁻²)	(in.)	(m x 10 ⁻⁴)	(in.)	(m x 10 ⁻²)	(mils/sec)	(m/sec x 10 ⁻⁵)
22	0.92	0.0233	0.97	2.46	+	+	0.77	1.95	+3.2	+8.12
21	0.96	0.0243	1.00	2.54	+	+	0.76	1.93	+2.5	+6.35
20	0.99	0.0251	1.04	2.64	+	+	0.76	1.93	+3.2	+8.12
19	1.04	0.0264	1.08	2.74	+	+	0.76	1.93	+2.5	+6.35
18	1.08	0.0274	1.11	2.81	+	+	0.78	1.98	+1.9	+4.82
17	1.11	0.0281	1.14	2.89	+	+	0.79	2.00	+2.5	+6.35
16	1.14	0.0284	1.17	2.97	+	+	0.81	2.05	+1.9	+4.82
15	1.18	0.0299	1.20	3.04	+	+	0.76	1.93	+1.2	+3.04
14	1.21	0.0307	1.20	3.04	0.01	2.5	0.77	1.95	0.6	1.52
13	1.24	0.0314	1.21	3.07	0.03	7.6	0.78	1.98	1.9	4.82
12	1.24	0.0314	1.21	3.07	0.03	7.6	0.78	1.98	1.9	4.82
11	1.24	0.0314	1.20	3.04	0.04	10.1	0.76	1.93	2.5	6.35
10	1.23	0.0312	1.19	3.02	0.04	10.1	0.76	1.93	2.5	6.35
9	1.22	0.0309	1.19	3.02	0.03	7.6	0.76	1.93	1.9	4.82
8	1.22	0.0309	1.19	3.02	0.03	7.6	0.76	1.93	1.9	4.82
7	1.21	0.0307	1.19	3.02	0.02	5.0	0.76	1.93	1.2	3.04
6	1.20	0.0304	1.19	3.02	0.01	2.5	0.77	1.95	0.6	1.52
5	1.19	0.0300	1.18	2.99	0.01	2.5	0.74	1.87	0.6	1.52
4	1.18	0.0299	1.18	2.99	0	0	0.69	1.75	0	0
3	1.12	0.0284	1.14	2.89	+	+	0.66	1.67	+1.3	+3.04
2	1.04	0.0264	1.06	2.69	+	+	0.57	1.44	+1.2	+3.04
1	0.95	0.0241	0.96	2.43	+	+	0.46	1.16	+0.6	+1.52
0	0.87	0.0220	0.86	2.18	0.01	2.5	0.40	1.01	+0.6	+1.52

Legend

————	Prefiring Surface
-----	Postfiring Surface
-----	Char Depth

Motor Parameters

Burn Time	15.48 sec
Web Pressure	430 psi (3.0×10^6 N/m ²)
Throat Diameter	0.34 in. (0.86 cm)

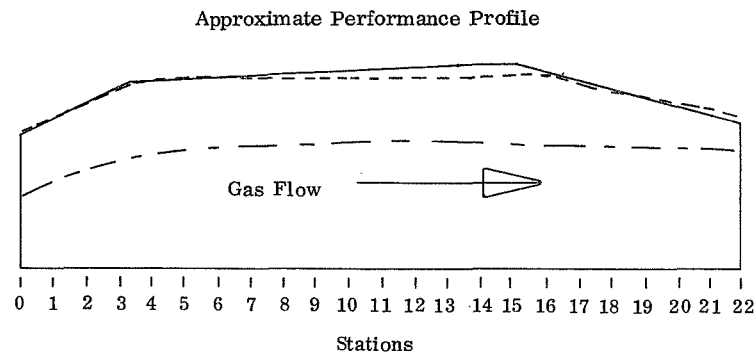


Figure 47. - TU-379 Nozzle Data, RI-4009 Resin, Motor No. 572

25% Durez 10694

75% 325 Micron Graphite GL-1012

Cured at 200 psi (1.38×10^6 N/m²)

Station No.	Contour		Erosion		Material Loss		Char Depth		Erosion Rate	
	Initial (in.)	(m)	(in.)	(m x 10 ⁻²)	(in.)	(m x 10 ⁻⁴)	(in.)	(m x 10 ⁻²)	(mils/sec)	(m/sec x 10 ⁻⁵)
22	0.92	0.0233	--	--	--	--	--	--	--	--
21	0.96	0.0243	0.96	2.43	0.00	0.00	0.68	1.72	0.00	0.00
20	0.99	0.0251	0.99	2.51	0.00	0.00	0.70	1.77	0.00	0.00
19	1.04	0.0264	1.02	2.59	0.02	5.08	0.72	1.82	1.0	2.54
18	1.08	0.0274	1.06	2.69	0.02	5.08	0.73	1.85	1.0	2.54
17	1.11	0.0281	1.09	2.76	0.02	5.08	0.73	1.85	1.0	2.54
16	1.14	0.0284	1.12	2.84	0.02	5.08	0.73	1.85	1.0	2.54
15	1.18	0.0299	1.14	2.89	0.04	10.16	0.73	1.80	2.1	5.33
14	1.21	0.0307	1.13	2.87	0.08	20.32	0.71	1.80	4.1	10.41
13	1.24	0.0314	1.13	2.87	0.11	27.94	0.71	1.80	5.6	14.22
12	1.24	0.0314	1.12	2.84	0.12	30.48	0.71	1.80	6.2	15.74
11	1.24	0.0314	1.14	2.89	0.10	25.40	0.71	1.80	5.1	12.95
10	1.23	0.0312	1.14	2.89	0.09	22.86	0.71	1.80	4.6	11.68
9	1.22	0.0309	1.10	2.79	0.12	30.48	0.71	1.80	6.2	15.74
8	1.22	0.0309	1.14	2.89	0.08	20.32	0.70	1.77	4.1	10.41
7	1.21	0.0307	1.12	2.84	0.09	22.86	0.68	1.72	4.6	11.68
6	1.20	0.0304	1.10	2.79	0.10	25.40	0.63	1.59	5.1	12.95
5	1.19	0.0300	1.10	2.79	0.09	22.86	0.61	1.55	4.6	11.68
4	1.18	0.0299	1.09	2.76	0.09	22.86	0.59	1.49	4.6	11.68
3	1.12	0.0284	1.10	2.79	0.02	5.08	0.54	1.37	1.0	2.54
2	1.04	0.0264	1.04	2.64	0.00	0.00	0.53	1.34	0.0	0.00
1	0.95	0.0241	0.97	2.46	+	+	0.53	1.34	+1.0	2.54
0	0.87	0.0220	0.88	2.23	+	+	0.50	1.27	+0.5	1.27

Legend

———— Prefiring Surface
 - - - - - Postfiring Surface
 - - - - - Char Depth

Motor Parameters

Burn Time 19.48 sec
 Web Pressure 247 psi (1.71×10^6 N/m²)
 Throat Diameter 0.34 in. (0.86 cm)

Approximate Performance Profile

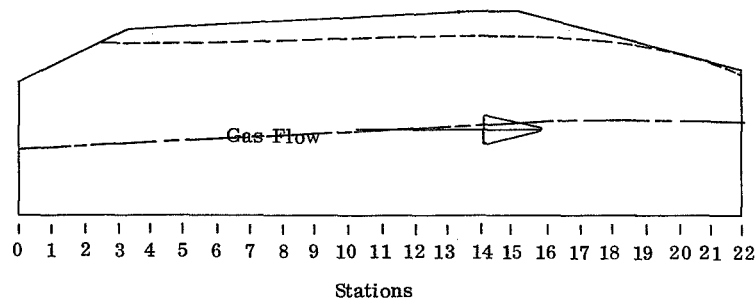


Figure 48. - TU-379 Nozzle Data, Durez 10694 Resin, Motor No. 550.1

25% Durez 10694

75% 325 Micron Graphite (GL-1012)

Cured at 200 psi (1.38×10^6 N/m²)

Station No.	Contour				Material Loss		Char Depth		Erosion Rate	
	Initial		Erosion							
	(in.)	(m)	(in.)	(m x 10 ⁻²)	(in.)	(m x 10 ⁻⁴)	(in.)	(m x 10 ⁻²)	(mils/sec)	(m/sec x 10 ⁻⁵)
22	0.92	0.0233	--	--	--	--			--	--
21	0.96	0.0243	0.95	2.41	0.01	2.54			0.5	1.27
20	0.99	0.0251	0.99	2.51	0.00	0.00			0.0	0.00
19	1.04	0.0264	1.03	2.61	0.01	2.54			0.5	1.27
18	1.08	0.0274	1.06	2.69	0.02	5.08			1.0	2.54
17	1.11	0.0281	1.10	2.79	0.01	2.54			0.5	1.27
16	1.14	0.0284	1.13	2.87	0.01	2.54			0.5	1.27
15	1.18	0.0299	1.15	2.92	0.03	7.62			1.5	3.81
14	1.21	0.0307	1.14	2.89	0.07	17.78			3.6	9.14
13	1.24	0.0314	1.14	2.89	0.10	25.40			5.1	12.95
12	1.24	0.0314	1.12	2.84	0.12	30.48			6.0	15.24
11	1.24	0.0314	1.14	2.89	0.10	25.40			5.1	12.95
10	1.23	0.0312	1.14	2.89	0.09	22.86			4.6	11.68
9	1.22	0.0309	1.15	2.92	0.07	17.78			3.6	9.14
8	1.22	0.0309	1.16	2.94	0.06	15.24			2.0	5.08
7	1.21	0.0307	1.18	2.99	0.03	7.62			1.5	3.81
6	1.20	0.0304	1.17	2.97	0.03	7.62			1.5	3.81
5	1.19	0.0300	1.12	2.84	0.07	17.78			3.6	9.14
4	1.18	0.0299	1.11	2.81	0.07	17.78			3.6	9.14
3	1.12	0.0284	1.11	2.81	0.01	2.54			0.5	1.27
2	1.04	0.0264	1.03	2.61	0.01	2.54			0.5	1.27
1	0.95	0.0241	1.94	2.38	0.01	2.54			0.5	1.27
0	0.87	0.0220	0.84	2.13	0.03	7.62			1.5	3.81

Note: No char was visible on bottom half of nozzle section.

Legend

———— Prefiring Surface
 - - - - - Postfiring Surface
 - - - - - Char Depth

Motor Parameters

Burn Time 19.69 sec
 Web Pressure 242 psi (1.67×10^6 N/m²)
 Throat Diameter 0.34 in. (0.86 cm)

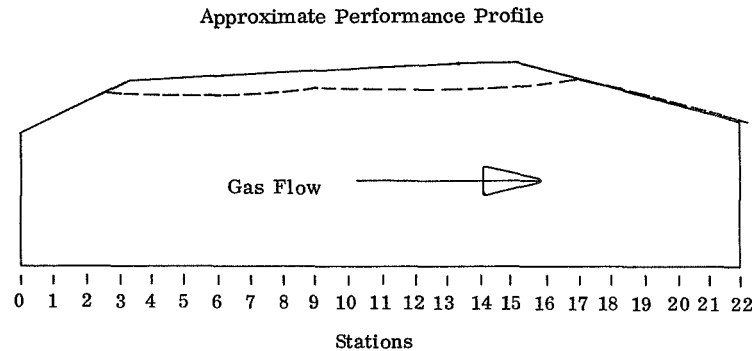


Figure 49. - TU-379 Nozzle Data, Durez 10694 Resin, Motor No. 550.2

25% Durez 10694

75% 74 Micron Graphite

Cured at 200 psi ($1.38 \times 10^6 \text{ N/m}^2$)

Station No.	Contour				Material Loss		Char Depth		Erosion Rate	
	Initial (in.)	(m)	Erosion (in.)	(m x 10 ⁻²)	(in.)	(m x 10 ⁻⁴)	(in.)	(m x 10 ⁻²)	(mils/sec)	(m/sec x 10 ⁻⁵)
22	0.92	0.0233	--	--	--	--	--	--	--	--
21	0.96	0.0243	0.95	2.41	0.01	2.54	0.72	1.82	0.7	1.77
20	0.99	0.0251	1.00	2.54	+	+	0.74	1.87	+0.7	1.77
19	1.04	0.0264	1.02	2.59	0.02	5.08	0.76	1.93	1.4	3.55
18	1.08	0.0274	1.06	2.69	0.02	5.08	0.79	2.00	1.4	3.55
17	1.11	0.0281	1.10	2.79	0.01	2.54	0.80	2.03	0.7	1.77
16	1.14	0.0284	1.13	2.87	0.01	2.54	0.78	1.98	0.7	1.77
15	1.18	0.0299	1.16	2.94	0.02	5.08	0.77	1.95	1.4	3.55
14	1.21	0.0307	1.18	2.99	0.03	7.62	0.78	1.98	2.0	5.08
13	1.24	0.0314	1.18	2.99	0.06	15.24	0.78	1.98	3.4	8.63
12	1.24	0.0314	1.18	2.99	0.06	15.24	0.77	1.95	3.4	8.63
11	1.24	0.0314	1.18	2.99	0.06	15.24	0.76	1.93	3.4	8.63
10	1.23	0.0312	1.18	2.99	0.05	12.70	0.75	1.90	3.4	8.63
9	1.22	0.0309	1.17	2.97	0.05	12.70	0.73	1.85	3.4	8.63
8	1.22	0.0309	1.17	2.97	0.05	12.70	0.72	1.82	3.4	8.63
7	1.21	0.0307	1.17	2.97	0.04	10.16	0.72	1.82	2.7	6.85
6	1.20	0.0304	1.16	2.94	0.04	10.16	0.70	1.77	2.7	6.85
5	1.19	0.0300	1.15	2.92	0.04	10.16	0.68	1.72	2.7	6.85
4	1.18	0.0299	1.15	2.92	0.03	7.62	0.65	1.65	2.0	5.08
3	1.12	0.0284	1.14	2.89	+	+	0.61	1.54	+1.4	3.55
2	1.04	0.0264	1.02	2.59	0.02	5.08	0.59	1.49	1.4	3.55
1	0.95	0.0241	0.98	2.48	+	+	0.53	1.34	+2.0	5.08
0	0.87	0.0220	0.88	2.23	+	+	0.44	1.11	+0.7	1.77

Legend

—————	Prefiring Surface
-----	Postfiring Surface
-----	Char Depth

Motor Parameters

Burn Time	14.66 sec
Web Pressure	308 psi ($2.07 \times 10^6 \text{ N/m}^2$)
Throat Diameter	0.34 in. (0.86 cm)

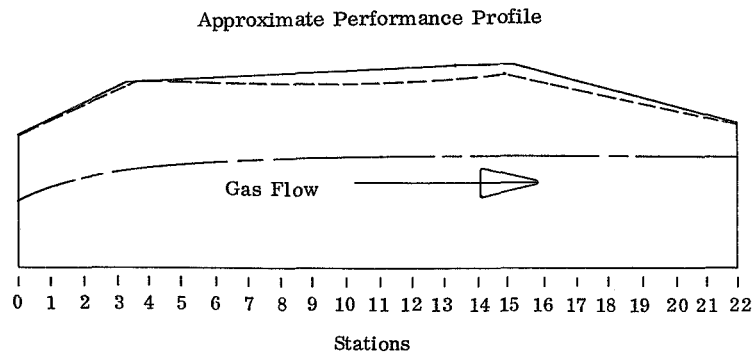


Figure 50. - TU-379 Nozzle Data, Durez 10694 Resin, Motor No. 551

45% DEN 438 Epoxy Novolac

55% 325 Micron Graphite

Cured at zero pressure

Station No.	Contour				Material Loss		Char Depth		Erosion Rate	
	Initial (in.)	(m)	Erosion (in.)	(m x 10 ⁻²)	(in.)	(m x 10 ⁻⁴)	(in.)	(m x 10 ⁻²)	(mils/sec)	(m/sec x 10 ⁻⁵)
22	0.92	0.0233	--	--	--	--	--	--	--	--*
21	0.96	0.0243	0.77	1.95	0.19	48.26	0.53	1.34	6.3	16.00
20	0.99	0.0251	0.78	1.98	0.21	53.34	0.51	1.29	--	--
19	1.04	0.0264	0.81	2.05	0.23	58.42	0.52	1.32	6.6	16.76
18	1.08	0.0274	0.84	2.13	0.24	60.96	0.53	1.34	--	--
17	1.11	0.0281	0.84	3.13	0.27	68.58	0.53	1.34	7.8	19.81
16	1.14	0.0284	0.86	2.18	0.28	71.12	0.53	1.34	--	--
15	1.18	0.0299	0.87	2.20	0.31	78.74	0.54	1.37	9.5	24.13
14	1.21	0.0307	0.88	2.23	0.33	83.82	0.54	1.37	--	--
13	1.24	0.0314	0.90	2.28	0.34	86.36	0.54	1.37	--	--
12	1.24	0.0314	0.92	2.33	0.32	81.28	0.55	1.39	9.2	23.36
11	1.24	0.0314	0.95	2.41	0.29	73.66	0.56	1.42	--	--
10	1.23	0.0312	0.97	2.46	0.26	66.04	0.58	1.47	--	--
9	1.22	0.0309	0.98	2.48	0.24	60.96	0.57	1.44	7.5	19.05
8	1.22	0.0309	1.01	2.56	0.21	53.34	0.56	1.42	--	--
7	1.21	0.0307	1.03	2.61	0.18	45.72	0.57	1.44	--	--
6	1.20	0.0304	1.03	2.61	0.17	43.18	0.50	1.27	--	--
5	1.19	0.0300	1.08	2.74	0.11	27.94	0.40	1.01	5.2	13.20
4	1.18	0.0299	1.10	2.79	0.08	20.32	0.42	1.06	--	--
3	1.12	0.0284	1.03	2.61	0.09	22.86	0.37	0.93	2.3	5.84
2	1.04	0.0264	1.00	2.54	0.04	10.16	0.37	0.93	2.6	6.60
1	0.95	0.0241	0.93	2.36	0.02	5.08	0.36	0.91	1.2	3.04
0	0.87	0.0220	0.86	2.18	0.01	2.54	0.44	1.11	0.3	0.76

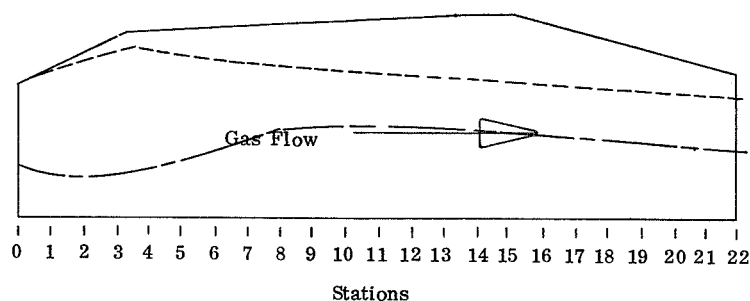
Legend

———— Prefiring Surface
 - - - - - Postfiring Surface
 - - - - - Char Depth

Motor Parameters

Burn Time 34.73 sec
 Web Pressure N/A
 Throat Diameter 0.34 in. (0.86 cm)

Approximate Performance Profile



*In view of poor performance of material, erosion rate was computed on an intermittent basis only.

Figure 51. - TU-379 Nozzle Data, DEN 438 Epoxy Novolac, Motor No. 566.1

45% DEN 438 Epoxy Novolac

55% 325 Micron Graphite

Cured at Zero Pressure

Station No.	Contour				Material Loss		Char Depth		Erosion Rate	
	Initial (in.)	(m)	Erosion (in.)	(m x 10 ⁻²)	(in.)	(m x 10 ⁻⁴)	(in.)	(m x 10 ⁻²)	(mils/sec)	(m/sec x 10 ⁻⁵)
22	0.92	0.0233	--	--	--	--	--	--	--	--*
21	0.96	0.0243	0.67	1.70	0.29	73.66	0.38	0.96	7.2	18.28
20	0.99	0.0251	0.71	1.80	0.28	71.12	0.34	0.86	--	--
19	1.04	0.0264	0.70	1.77	0.34	86.36	0.34	0.86	9.1	23.11
18	1.08	0.0274	0.71	1.80	0.37	93.98	0.35	0.88	--	--
17	1.11	0.0281	0.73	1.85	0.38	96.52	0.37	0.93	10.5	26.67
16	1.14	0.0284	0.78	1.98	0.36	91.44	0.36	0.91	--	--
15	1.18	0.0299	0.74	1.87	0.44	111.76	0.38	0.96	--	--
14	1.21	0.0307	0.81	2.05	0.40	101.60	0.40	1.01	12.2	30.98
13	1.24	0.0314	0.80	2.03	0.44	111.76	0.43	1.09	--	--
12	1.24	0.0314	0.84	2.13	0.40	101.60	0.48	1.21	11.0	27.94
11	1.24	0.0314	0.89	2.26	0.35	88.90	0.49	1.24	--	--
10	1.23	0.0312	0.86	2.18	0.37	93.98	0.49	1.24	--	--
9	1.22	0.0309	0.86	2.18	0.36	91.44	0.50	1.27	8.6	21.84
8	1.22	0.0309	0.90	2.28	0.32	81.28	0.51	1.29	--	--
7	1.21	0.0307	0.92	2.33	0.29	73.66	0.52	1.32	8.0	20.32
6	1.20	0.0304	0.96	2.43	0.24	60.96	0.53	1.34	--	--
5	1.19	0.0300	0.98	2.48	0.21	53.34	0.52	1.32	--	--
4	1.18	0.0299	1.06	2.69	0.12	30.48	0.51	1.29	3.3	8.38
3	1.12	0.0284	1.05	2.66	0.07	17.78	0.48	1.21	1.9	4.32
2	1.04	0.0264	0.90	2.28	0.14	35.56	0.46	1.16	3.9	9.90
1	0.95	0.0241	0.83	2.10	0.12	30.48	0.48	1.21	3.3	8.38
0	0.87	0.0220	0.80	2.03	0.07	17.78	0.48	1.21	1.9	4.82

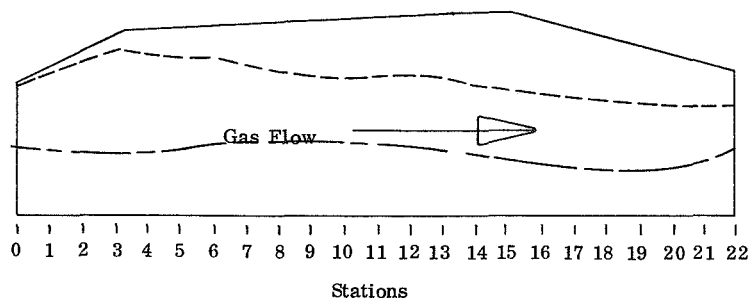
Legend

————— Prefiring Surface
 - - - - - Postfiring Surface
 - - - - - Char Depth

Motor Parameters

Burn Time 36.10 sec
 Web Pressure N/A
 Throat Diameter 0.34 in. (0.86 cm)

Approximate Performance Profile



*In view of poor performance of material, erosion rate was computed on an intermittent basis only.

Figure 52. - TU-379 Nozzle Data, DEN 438 Epoxy Novolac, Motor No. 566.2

Based upon the test results shown in this report, the following basic formulations and curing conditions were recommended to NASA Lewis Research Center and approved for the fiber reinforcement and filler additive studies of Tasks 02 and 03.

Monsanto RI-4009 resin	40.0 percent
Graphite GL-1012	60.0 percent
Cure temperature	300°F (422°K)
Cure pressure	1,000 psi (6.9 x 10 ⁶ N/m ²)

The selection of the above formulation was based upon: (1) the amount of throat erosion was considerably less than formulations evaluated at other resin or pressure levels; (2) the amount of radial cracking was less, as a result of the larger graphite particles; and (3) the growth at the forward section of the nozzle appeared to be minimized with the use of the large (325 microns) graphite size particles and a resin level of 40 percent.

Durez 10694 Phenolic Resin. - Two formulations were developed for the TU-379 motor test: one containing 25 percent resin and 75 percent graphite of the 74 micron (maximum) size (Motor No. 551), and the other containing 25 percent resin and 75 percent graphite of the 325 micron size (GL-1012) in two test billets (Motor No. 550). Both mixes were cured at 200 psi (1.38 x 10⁶ N/m²) and 300°F (422°K). Only one nozzle was evaluated for the Durez 10694 of small particle size graphite (Motor No. 551). Comparing Figures 48 and 50 indicates the erosion profiles of large vs small particle size are nearly identical. Due to the off gassing of the resin, a small growth in the forward section of both systems was noted.

A small amount of radial cracking occurred in the nozzle containing the 74 micron graphite (Motor No. 551); whereas the nozzles containing the larger graphite exhibited no radial cracking (Motor No. 550).

Based upon the above test results, it was determined to use both small graphite particles (74 micron) and the larger graphite (GL-1012) (325 microns) particles with the Durez 10694 resin for the Fiber Reinforcement Studies, Task 02, and the Filler Additive Study, Task 03.

DEN 438 Epoxy Novolac. - One material formulation of the epoxy novolac and graphite composite was fabricated into two component test billets and processed using identical cure conditions. A review of the nozzle cross section photographs (Figure 40) and the erosion profiles (Figures 51 and 52) substantiates that this resin system is the least erosion resistant of the three resin systems evaluated, with a maximum average erosion rate of 10.9 mils/sec at the throat plane. The erosion rate was minimized by the low motor operating pressure as verified by the extended action time of approximately 35 seconds.

The LCCM materials using DEN 438 (epoxy novolac) as the binder were not very successful in the program, but the potential processing characteristics; i. e., low pressure cures, no volatiles, made them appear desirable. Therefore, a composite nozzle end ring 3.5 in. long (8.89 cm) was fabricated and static tested on a TU-622 motor. This static test firing was conducted on a company funded project. The TU-622 motor with a throat diameter of 1.35 in. (3.43 cm) burned for 21.7 sec at 1,013 psi (6.989×10^6 N/m²) as shown in Figure 53. The end ring eroded at a rate of 28.5 mils/sec, which was severe erosion compared with other materials. It was recommended that no further development of the epoxy novolac resin be conducted.

Task 01 of the program was completed with the thermal conductivity determination of each candidate formulation. The Cenco-Fitch heat conductivity apparatus was used for measuring the material thermal conductivity. Table XI shows the cure cycle and thermal conductivity for each of the formulations. The thermal data shown in Table XI represent a baseline study and were used to compare those formulations developed under the Fiber Reinforcement and Filler Additive Studies (Tasks 01 and 02).

The best formulations from Task 01 are listed below with the cure temperature and pressure. These formulations were used as baselines for the reinforcement and filler work in the remaining tasks.

Basic Formulations

<u>Resin System</u>	<u>Percent Resin</u>	<u>Graphite</u>	<u>Percent Graphite</u>	<u>Cure Temperature</u>		<u>Cure Pressure</u>	
				<u>(°F)</u>	<u>(°K)</u>	<u>(psi)</u>	<u>(N/m² x 10⁶)</u>
Monsanto RI-4009	40	GL-1012 325 Micron	60	300	422	1,000	6.9
Durez 10694	25	GL-1012 325 Micron	75	300	422	200	1.38
Durez 10694	25	74 Micron	75	300	422	200	1.38

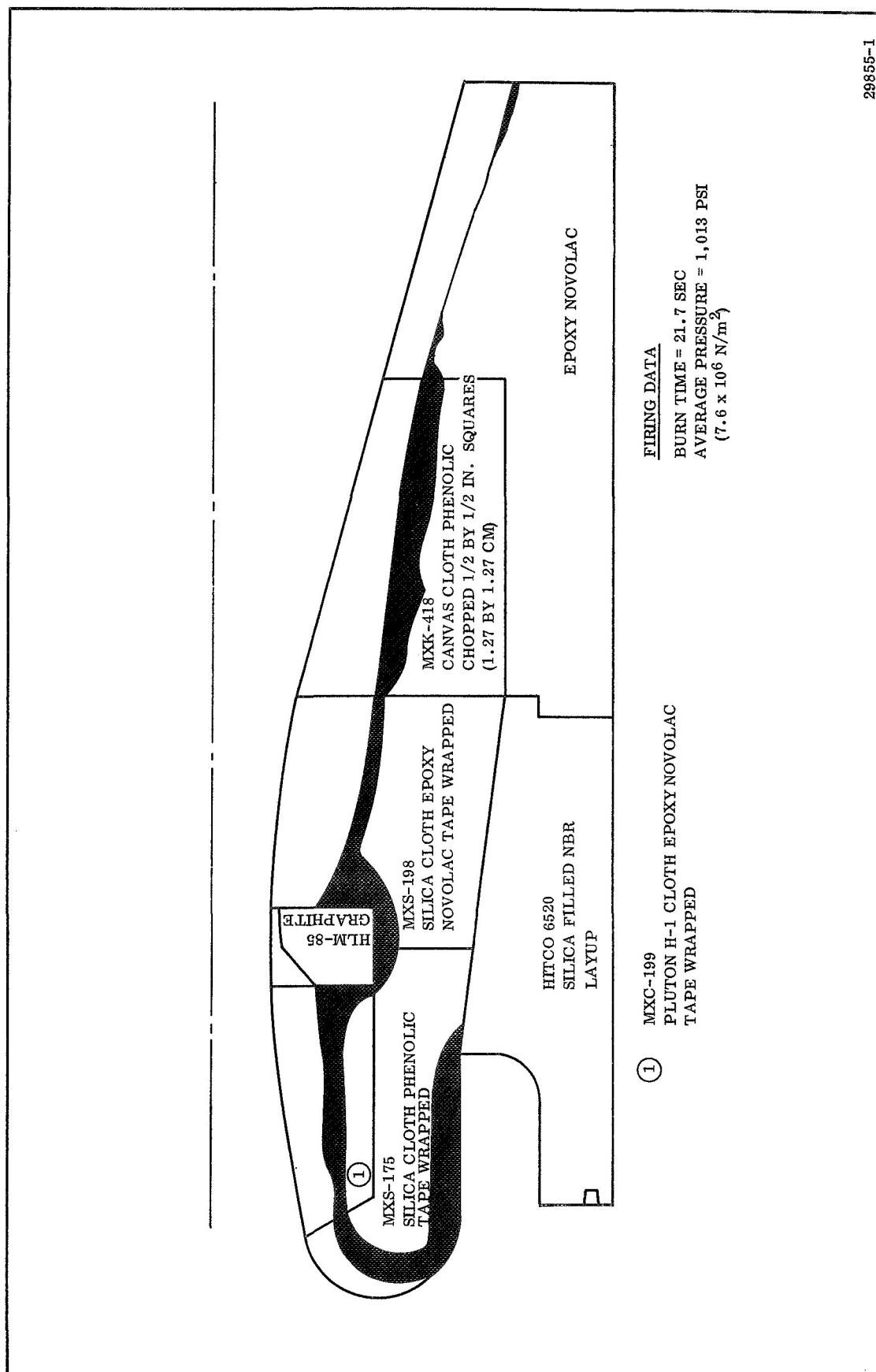


Figure 53. -TU-622 Nozzle Erosion and Char Profile, DEN 438 Epoxy Novolac

TABLE XI. - LCCM DEVELOPMENT, THERMAL CONDUCTIVITY

Mix No.	Resin	Percent Resin	Graphite		Cure Temperature		Cure Pressure		Thermal Conductivity	
			(type)	(%)	(° F)	(° K)	(psi)	(N/m ² x 10 ⁵)	(Btu in./sec ft ² ° F)	(joule/m sec ° K)
556	Monsanto RI-4009	40	74 Micron (max)	60	300	422	1,000	6.9	1.69 x 10 ⁻³	87.68
557	Monsanto RI-4009	40	GL-1012 325 Micron	60	300	422	1,000	6.9	1.88 x 10 ⁻³	97.54
558	Monsanto RI-4009	25	GL-1008, 3 pt GL-1012, 1 pt	75	300	422	1,000	6.9	1.87 x 10 ⁻³	97.02
560	Durez 10694	25	74 Micron (max)	75	300	422	200	1.38	2.14 x 10 ⁻³	111.04
563	Durez 10694	25	GL-1012 325 Micron	75	300	422	200	1.38	2.25 x 10 ⁻³	116.74
564	DEN 438	*	GL-1012 325 Micron	55.25	300	422	Ambient		1.13 x 10 ⁻³	58.63
565	DEN 438	*	74 Micron (max)	55.25	300	422	Ambient		1.53 x 10 ⁻³	79.38

*23.67 percent resin
20.71 hardener
0.35 BDMA

TASK 02 - FIBER REINFORCEMENT STUDY

The three basic formulations derived from Task 01 were reinforced with fibers in an attempt to improve the mechanical properties. In addition, methods of incorporating the fibers in the compounds were developed. The reinforcement materials selected for evaluation were: (1) chopped carbon fiber, 0.25 in. (0.64 cm) length, (2) chopped glass fiber, 0.75 to 1.0 in. (1.71 to 2.54 cm) length, and (3) chopped rayon 0.75 to 1.0 in. (1.71 to 2.54 cm) length.

Various quantities of the reinforcements were mixed with the three basic formulations and molded into flat slabs. Problems in blending in the fibers were encountered with some of the resin systems. These are described in the specific discussions of each formulation. Test specimens were machined from the cured slabs. The fabrication test matrix is shown in Table XII.

Monsanto RI-4009 Resin

No serious problems were encountered in mixing the chopped carbon fibers with the RI-4009 resin and graphite powder compound. However, the rayon and glass fibers did present problems at the 20 percent fiber level. The bulk of the fiber at the 20 percent level was so coarse that it caused separation of the resin and fiber. The mixture appeared to have no cohesive properties.

Test panels were fabricated from the Monsanto RI-4009/fiber reinforcement billets and tested for tensile and compressive strength, density, and thermal conductivity. Test results are shown in Table XIII. In addition, test specimens were torch tested at 5,000° F (3,035° K) for 60 sec, cut in half, and examined. Photographs of the Monsanto RI-4009/fiber reinforced test samples are shown in Figures 54 thru 56.

Data comparison of the carbon fiber reinforcement studies at the 5 and 10 percent levels with those of rayon and glass fiber shows tensile and compressive strengths and densities are comparable. The thermal conductivity of the rayon fiber reinforcement is equal to that of the glass fiber at both the 5 and 10 percent level and is lower than the carbon fiber reinforcement. However, the char hardness of the carbon fiber reinforcement is significantly higher (Shore D of 60) than that of the rayon or glass fiber (Shore D of 42) formulations.

Additional attempts were made to reevaluate the chopped glass and rayon fibers. Fiber lengths were cut to 0.25 in. (0.64 cm) instead of the 1.0 in. (2.54 cm) fiber length used previously. A 10 percent mixture of each fiber was blended with 40 percent of the RI-4009 dry phenolic resin and 50 percent GL-1012 graphite powder.

TABLE XII. - LCCM FIBER REINFORCEMENT FABRICATION TEST MATRIX

Resin	Percent Resin	Percent Graphite	Cure Temperature		Cure Pressure		Fiber Percent		
			(° F)	(° K)	(psi)	(N/m ² x 10 ⁶)	Rayon (0.75 in.) (1.91 cm)	Glass (0.75 in.) (1.91 cm)	Carbon (0.25 in.) (0.64 cm)
Monsanto RI-4009	40	GL-1012 325 Micron (average)	300	422	1,000	6.9	5	5	5
							10	10	10
									20
									30
Durez 10694	25	GL-1012 325 Micron (average)	300	422	200	1.38	5	5	5
Durez 10694	25	74 Micron (max)	300	422	200	1.38	5	5	5

Physical Property Tests

Tensile strength
Compressive strength
Density
Hardness

Note: Percent graphite = 100 - (percent fiber + percent resin)

TABLE XIII. - LCCM DEVELOPMENT, PHYSICAL PROPERTIES FIBER REINFORCEMENT STUDY, RI-4009 RESIN

Resin	Percent Resin	Fiber		Cure Conditions*	Mix No.	Tensile Strength		Density (g/cc)	Compressive Strength		Hardness Shore D		Thermal Conductivity	
		Type	Percent			(psi)	(N/m ² x 10 ⁶)		(psi)	(N/m ² x 10 ⁶)	Virgin	Char	(Btu in./sec ft ² °F)	(joule/m sec °K)
RI-4009	40	Carbon (0.25 in., 0.64 cm)	5		619	4,270 4,560 3,330 4,053	29.5 31.5 23.0 28.0	1.65 1.64 1.66 1.64	16,140 16,800 15,120 16,020	111.4 115.9 104.3 110.5	83	60	1.50 x 10 ⁻³	77.83
RI-4009	40		10		620	3,895 3,373 4,510 3,926	26.9 23.3 31.1 27.1	1.63 1.62 1.64 1.63	16,320 17,020 16,660 16,666	112.4 117.4 115.0 115.0	83	60	1.34 x 10 ⁻³	69.52
RI-4009	40		20		621	4,960 4,520 4,830 4,770	34.2 31.2 33.3 32.9	1.56 1.58 1.58 1.57	20,200 20,140 20,440 20,260	139.4 138.9 141.0 139.8	88	67	0.97 x 10 ⁻³	50.33
RI-4009	40		30		622	5,265 4,525 6,220 5,336	36.3 31.2 42.9 36.8	1.51 1.53 1.54 1.53	21,940 19,160 24,000 21,700	151.4 132.2 165.6 149.7	88	65	5.44 x 10 ⁻³	28.22
RI-4009	40	Rayon (1.0 in., 2.54 cm)	5		623	3,880 3,360 3,815 3,685	26.8 23.2 26.3 25.4	1.62 1.68 1.66 1.65	15,700 16,150 14,825 15,223	108.3 104.5 102.3 105.0	76	42	1.23 x 10 ⁻³	63.82
RI-4009	40		10		624	4,110 3,015 4,190 3,771	28.4 20.8 28.9 26.0	1.59 1.57 1.63 1.60	17,800 17,950 15,350 17,033	122.8 123.9 105.9 117.5	83	42	1.07 x 10 ⁻³	55.51
RI-4009	40	Glass (1.0 in., 2.54 cm)	5		627	4,030 4,840 4,675 4,513	27.8 33.4 32.3 31.1	1.68 1.67 1.67 1.67	16,850 16,850 15,650 16,116	116.3 109.4 108.0 111.2	82	50	1.23 x 10 ⁻³	63.82
RI-4009	40		10		628	4,190 4,370 4,000 4,186	28.9 30.2 27.6 28.9	1.71 1.69 1.69 1.70	16,300 17,400 16,100 17,266	112.5 120.0 124.9 119.1	82	45	1.25 x 10 ⁻³	64.86

*1,000 psi (6.9 x 10⁶ N/m²) at 300° F (422° K)

THERMAL CONDUCTIVITY

(Btu in. / sec ft² °F)

PERCENT FIBER
REINFORCEMENT

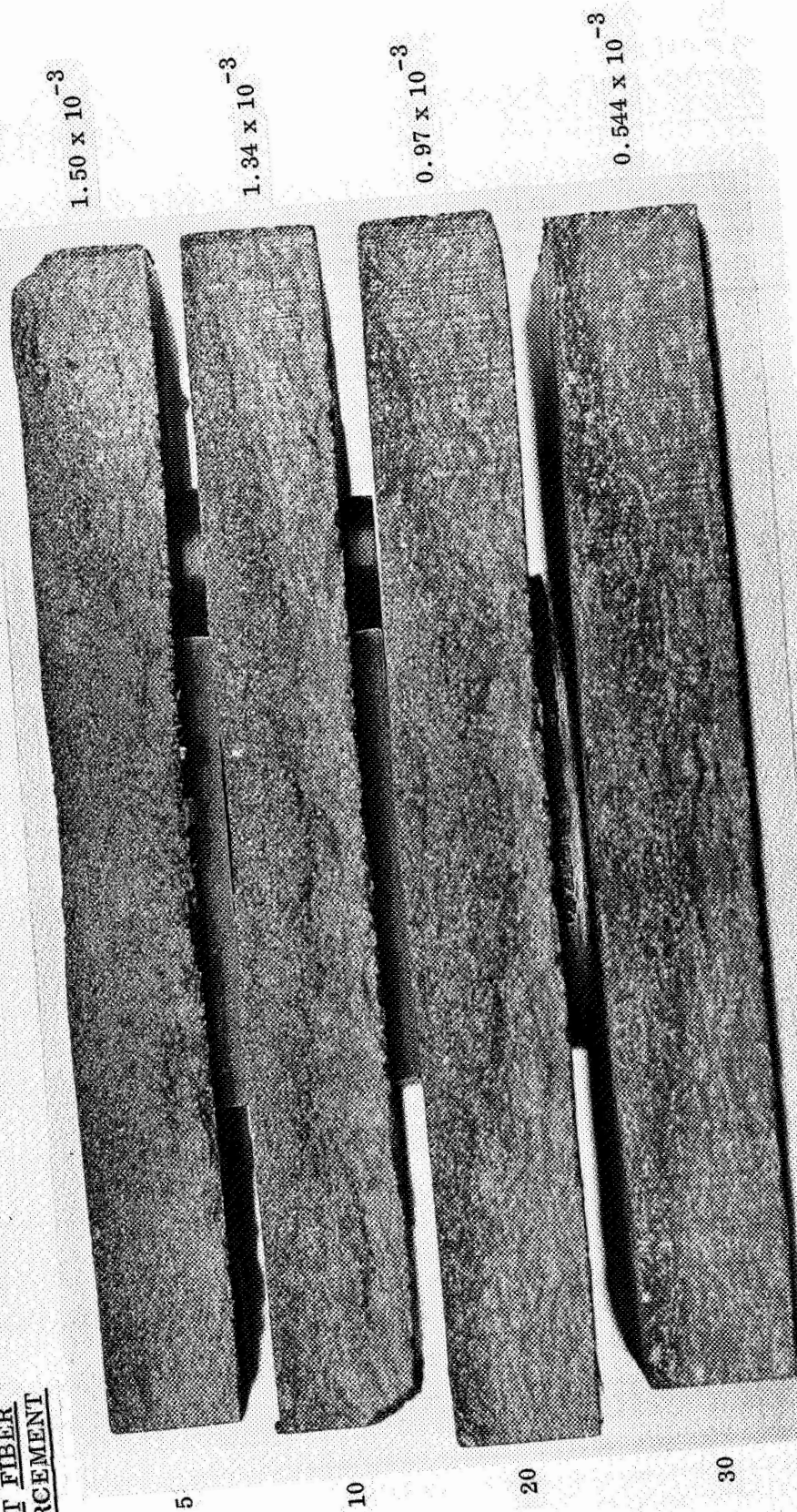


Figure 54. - Test Panels, RI-4009 Resin, Carbon Fiber Reinforcement

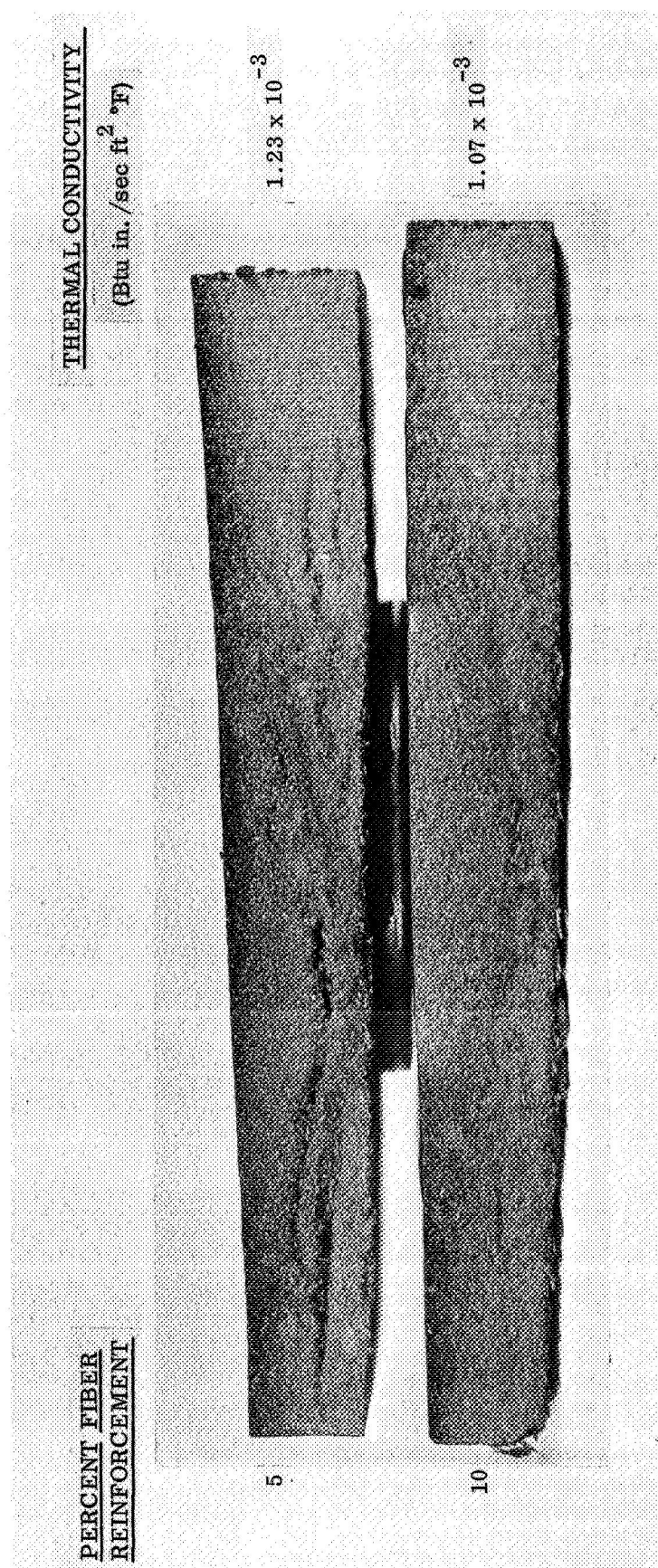


Figure 55. -Test Panels, RI-4009 Resin, Glass Fiber Reinforcement

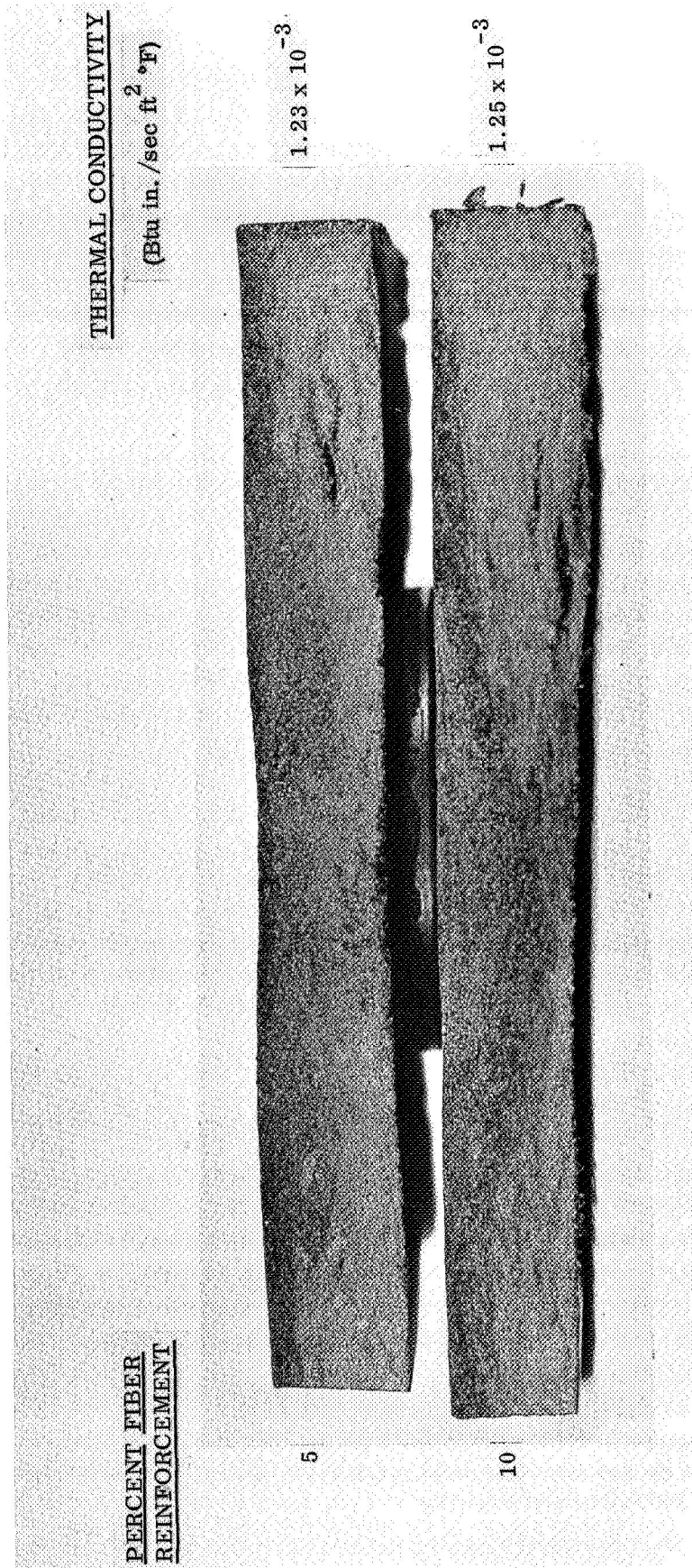


Figure 56. -Test Panels, RI-4009 Resin, Rayon Fiber Reinforcement

Again it was not possible to mix the rayon fibers homogeneously with the resin and graphite powder. The rayon fibers had a tendency to "roll up" like small cotton balls during the mixing cycle, thus causing incomplete mixing of resin and graphite powder. A photograph of this phenomenon is shown in Figure 57. Due to the incomplete mixing of ingredients, no structural components were fabricated.

From all visual observations, complete mixing was accomplished with the glass fibers, and a billet for a TU-379 nozzle was fabricated. However, upon machining the component, a banding effect of glass fibers was noted, indicating incomplete mixing of fiber and resin and graphite particles. Figure 58 shows the banding effect in the machined TU-379 nozzle.

Four TU-379 motor nozzles were manufactured and static fired with material consisting of carbon fiber. The material in each nozzle was compounded with a different level (5, 10, 20, and 30 percent by weight) of fiber concentrations. Good erosion uniformity was obtained with the lower percentages (5 and 10 percent) of carbon fiber on nozzles 651 and 652. However, excessive gouging and heavy erosion occurred at the higher levels of carbon fiber (20 to 30 percent). A uniform swelling was noted in the forward and exit section of all four TU-379 nozzles. Complete test results are shown in Figures 59 thru 62. Photographs of the post-fired sections are shown in Figure 63.

Static test results showed that the glass fiber nozzle maintained a firm char, a good erosion profile, and very little radial cracking. It was recommended that no further development work be performed with the glass fiber at this time, due to the failure of the mixing to disperse the fiber uniformly throughout the formulation. However, with further fiber-resin mixing studies, the material could be promising. Erosion performance results of the TU-379 glass fiber nozzle are shown in Figure 64. A photograph of the sectioned post-test nozzle is shown in Figure 65.

The unimpressive erosion performance of the 0.25 in. (0.64 cm) chopped carbon reinforcement at the 15 and 20 percent levels with the Monsanto RI-4009 resin system may have been the result of insufficient "wetting" or resin permeation of carbon fibers. To determine if a carbon fiber with more resin on it will improve the erosion resistance of the LCCM, a preimpregnated chopped carbon fiber was used in place of the dry carbon. The fibers were impregnated with a single stage liquid phenolic varnish, Monsanto SC-1008 and staged several hours at 170° F (350° K) then mixed with the powder resin and graphite. The combination (Mix No. 668) was cured at 1,000 psi (6.9×10^6 N/m²) at 300° F (422° K). The sum of the two phenolic resins equaled 40 percent of the total LCCM mixture. Another mix (No. 667) was also cured at 1,000 psi (6.9×10^6 N/m²) and 300° F (422° K). This formulation contained 10 percent chopped carbon fiber MX-4926, 35.5 percent RI-4009, 4.5 percent polyamide, and 50 percent GL-1012 graphite. TU-379 nozzle components were fabricated from the above two materials.

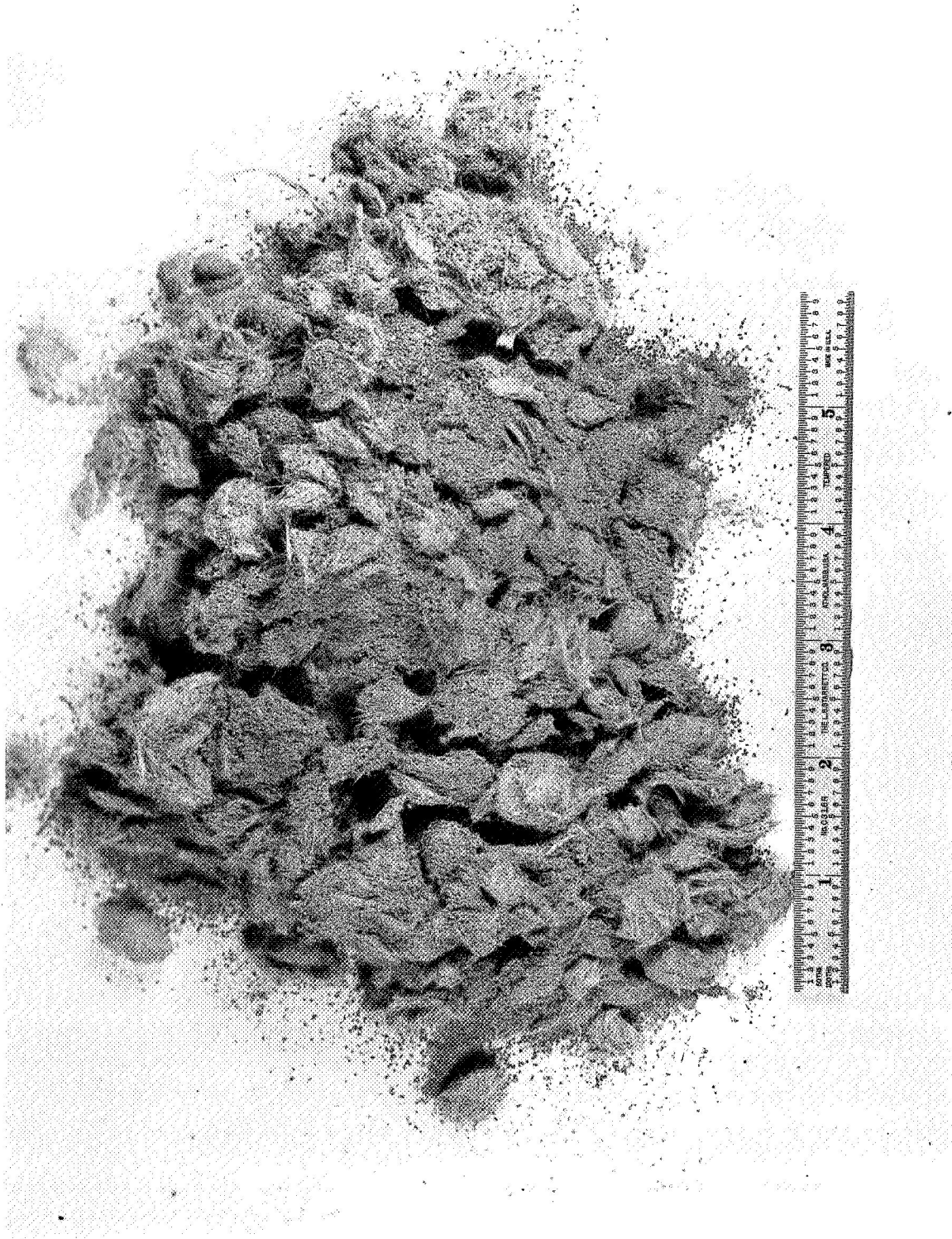


Figure 57. -Rayon Fiber, 0.25 in. (0.67 cm)

MD-7382



Figure 58. - Machined TU-379 Nozzle Assembly Showing Banding Effect

RI-4009 Resin
5% Chopped Carbon Fiber

Station No.	Contour				Material Loss		Char Depth		Erosion Rate	
	Initial (in.)	(m)	Erosion (in.)	(m x 10 ⁻²)	(in.)	(m x 10 ⁻²)	(in.)	(m x 10 ⁻²)	(mils/sec)	(m/sec x 10 ⁻⁵)
22	0.92	0.0233	0.96	2.44	+		0.80	2.04	--	--
21	0.96	0.0243	1.00	2.54	+	--	--	--	--	--
20	0.99	0.0251	1.03	2.62	+	--	--	--	--	--
19	1.04	0.0264	1.07	2.71	+	--	0.77	1.96	--	--
18	1.08	0.0274	1.10	2.80	+	--	--	--	--	--
17	1.11	0.0281	1.16	2.94	+	--	0.80	2.04	--	--
16	1.14	0.0284	1.17	2.96	+	--	--	--	--	--
15	1.18	0.0299	1.18	3.00	0.00	0.00	--	--	--	--
14	1.21	0.0307	1.21	3.06	0.00	0.00	--	--	--	--
13	1.24	0.0314	1.20	3.04	0.04	0.101	0.81	2.06	2.45	6.25
12	1.24	0.0314	1.20	3.04	0.04	0.101	--	--	2.45	6.25
11	1.24	0.0314	1.21	3.06	0.03	0.076	--	--	1.83	4.63
10	1.23	0.0312	1.21	3.06	0.02	0.058	0.76	1.94	1.22	3.11
9	1.22	0.0309	1.18	3.00	0.04	0.101	--	--	2.45	6.25
8	1.22	0.0309	1.18	3.00	0.04	0.101	--	--	2.45	6.25
7	1.21	0.0307	1.18	3.00	0.03	0.076	0.72	1.83	1.84	4.70
6	1.20	0.0304	1.14	2.84	0.06	0.153	--	--	3.67	9.40
5	1.19	0.0300	1.14	2.84	0.05	0.127	--	--	3.06	7.75
4	1.18	0.0299	1.14	2.84	0.04	0.101	0.69	1.76	2.45	6.25
3	1.12	0.0284	1.11	2.79	0.01	0.025	--	--	0.61	1.57
2	1.04	0.0264	1.06	2.69	+	--	--	--	--	--
1	0.95	0.0241	0.98	2.48	+	--	--	--	--	--
0	0.87	0.0220	0.89	2.25	+	--	0.50	1.27	--	--

Legend

———— Prefiring Surface
 - - - - - Postfiring Surface
 - - - - - Char Depth

Motor Parameters

Burn Time 16.31 sec
 Web Pressure 377 psi (2.6 x 10⁶ N/m²)
 Throat Diameter 0.34 in. (0.86 cm)

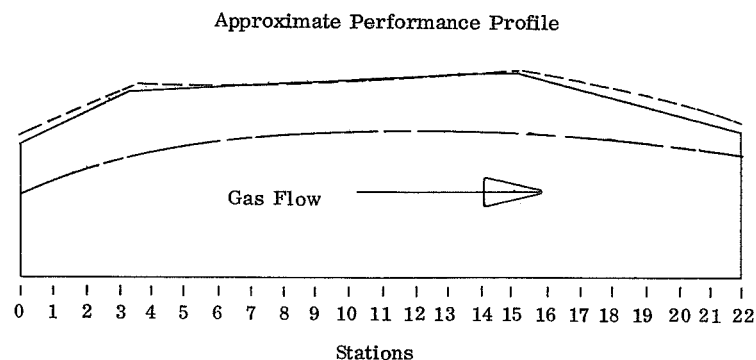


Figure 59. - TU-379 Nozzle Data, RI-4009 Resin, Motor No. 651

RI-4009 Resin
10% Chopped Carbon Fiber

Station No.	Contour				Material Loss		Char Depth		Erosion Rate	
	Initial		Erosion							
	(in.)	(m)	(in.)	(m x 10 ⁻²)	(in.)	(m x 10 ⁻⁴)	(in.)	(m x 10 ⁻²)	(mils/sec)	(m/sec x 10 ⁻⁵)
22	0.92	0.0233	0.96	2.43	+	--	0.75	1.90	--	--
21	0.96	0.0243	1.01	2.56	+	--	--	--	--	--
20	0.99	0.0251	1.04	2.64	+	--	--	--	--	--
19	1.04	0.0264	1.08	2.74	+	--	--	--	--	--
18	1.08	0.0274	1.11	2.81	+	--	0.77	1.95	--	--
17	1.11	0.0281	1.14	2.89	+	--	--	--	--	--
16	1.14	0.0284	1.18	2.99	+	--	--	--	--	--
15	1.18	0.0299	1.21	3.07	+	--	--	--	--	--
14	1.21	0.0307	1.22	3.09	+	--	0.74	1.87	--	--
13	1.24	0.0314	1.22	3.09	0.02	5.08	--	--	1.24	3.15
12	1.24	0.0314	1.20	3.04	0.04	10.16	--	--	2.48	6.30
11	1.24	0.0314	1.19	3.02	0.05	12.70	--	--	3.10	7.87
10	1.23	0.0312	1.16	2.94	0.07	17.78	0.76	1.93	4.35	11.05
9	1.22	0.0309	1.19	3.02	0.03	7.6	--	--	1.86	4.72
8	1.22	0.0309	1.19	3.02	0.03	7.6	--	--	1.86	4.72
7	1.21	0.0307	1.18	2.89	0.03	7.6	0.76	1.93	1.86	4.72
6	1.20	0.0304	1.17	2.97	0.03	7.6	--	--	1.86	4.72
5	1.19	0.0300	1.16	2.94	0.03	7.6	--	--	1.86	4.72
4	1.18	0.0299	1.16	2.94	0.02	5.08	--	--	1.24	3.15
3	1.12	0.0284	1.13	2.87	+	--	0.72	1.82	--	--
2	1.04	0.0264	1.07	2.71	+	--	--	--	--	--
1	0.95	0.0241	1.00	2.54	+	--	--	--	--	--
0	0.87	0.0220	0.92	2.33	+	--	0.42	1.05	--	--

Legend

----- Prefiring Surface
 - - - - - Postfiring Surface
 - - - - - Char Depth

Motor Parameters

Burn Time 16.09 sec
 Web Pressure
 Throat Diameter 0.34 in. (0.86 cm)

Approximate Performance Profile

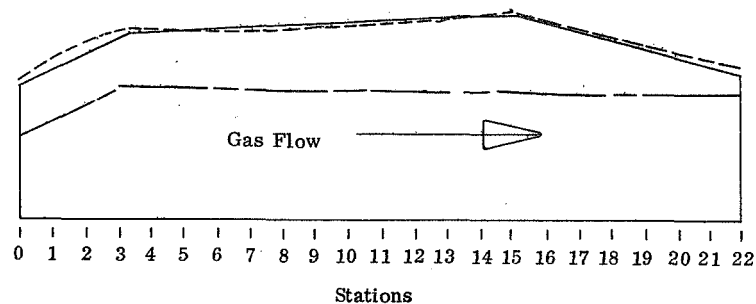


Figure 60. - TU-379 Nozzle Data, RI-4009 Resin, Motor No. 652

RI-4009 Resin
20% Chopped Carbon Fiber

Station No.	Contour				Material Loss		Char Depth		Erosion Rate	
	Initial		Erosion		(in.)	(m x 10 ⁻⁴)	(in.)	(m x 10 ⁻²)	(mils/sec)	(m/sec x 10 ⁻⁵)
22	0.92	0.0233	0.98	2.48	+	--	0.68	1.72	--	--
21	0.96	0.0243	1.02	2.59	+	--	--	--	--	--
20	0.99	0.0251	1.04	2.64	+	--	--	--	--	--
19	1.04	0.0264	1.06	2.69	+	--	--	--	--	--
18	1.08	0.0274	1.09	2.76	+	--	--	--	--	--
17	1.11	0.0281	1.13	2.87	+	--	--	--	--	--
16	1.14	0.0284	1.18	2.99	+	--	--	--	--	--
15	1.18	0.0299	1.13	2.87	0.05	12.70	0.68	1.72	2.74	6.96
14	1.21	0.0307	1.07	2.71	0.14	35.52	--	--	7.68	19.51
13	1.24	0.0314	1.03	2.62	0.21	53.34	--	--	11.52	29.26
12	1.24	0.0314	1.01	2.56	0.23	58.36	--	--	12.62	32.05
11	1.24	0.0314	1.02	2.59	0.22	55.88	--	--	12.07	30.66
10	1.23	0.0312	1.01	2.56	0.22	55.88	0.65	1.65	12.07	30.66
9	1.22	0.0309	1.01	2.56	0.21	53.34	--	--	11.52	29.26
8	1.22	0.0309	1.01	2.56	0.21	53.34	--	--	11.52	29.26
7	1.21	0.0307	1.12	2.84	0.09	22.86	--	--	4.93	12.52
6	1.20	0.0304	1.14	2.89	0.06	15.24	--	--	3.29	8.36
5	1.19	0.0300	1.19	3.02	0.00	0	0.72	1.82	0.00	0
4	1.18	0.0299	1.19	3.02	+	--	--	--	--	--
3	1.12	0.0284	1.15	2.92	+	--	--	--	--	--
2	1.04	0.0264	1.07	2.71	+	--	--	--	--	--
1	0.95	0.0241	0.98	2.48	+	--	--	--	--	--
0	0.87	0.0220	0.89	2.26	+	--	0.45	1.13	--	--

Legend

—————	Prefiring Surface
-----	Postfiring Surface
-----	Char Depth

Motor Parameters

Burn Time	18.22 sec
Web Pressure	203 psi (1.4 x 10 ⁶ N/m ²)
Throat Diameter	0.34 in. (0.86 cm)

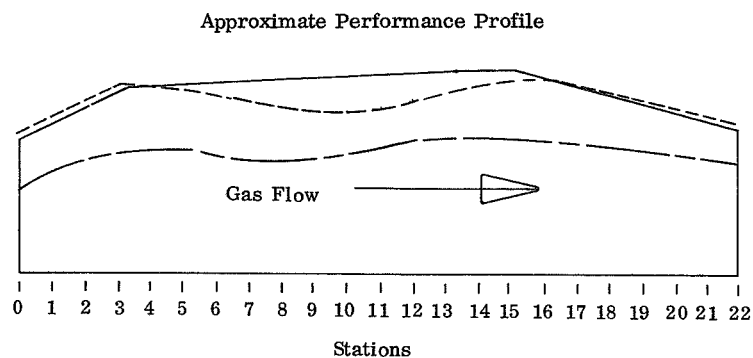


Figure 61. - TU-379 Nozzle Data, RI-4009 Resin, Motor No. 653

RI-4009 Resin
30% Chopped Carbon Fiber

Station No.	Contour				Material Loss		Char Depth		Erosion Rate	
	Initial (in.)	(m)	Erosion (in.)	(m x 10 ⁻²)	(in.)	(m x 10 ⁻⁴)	(in.)	(m x 10 ⁻²)	(mils/sec)	(m/sec x 10 ⁻⁵)
22	0.92	0.0233	0.95	2.41	+	--	0.74	1.87	--	--
21	0.96	0.0243	1.00	2.54	+	--	--	--	--	--
20	0.99	0.0251	1.04	2.64	+	--	0.70	1.77	--	--
19	1.04	0.0264	1.07	2.69	+	--	--	--	--	--
18	1.08	0.0274	1.04	2.64	0.04	10.2	--	--	2.35	5.97
17	1.11	0.0281	1.03	2.62	0.08	20.3	--	--	4.70	11.94
16	1.14	0.0284	1.03	2.62	0.11	27.94	--	--	6.47	16.43
15	1.18	0.0299	1.04	2.64	0.14	35.52	0.69	1.75	8.23	20.90
14	1.21	0.0307	1.04	2.64	0.17	43.1	--	--	9.99	25.37
13	1.24	0.0314	1.05	2.67	0.21	53.34	--	--	12.35	31.37
12	1.24	0.0314	1.04	2.64	0.20	50.8	--	--	11.76	29.87
11	1.24	0.0314	1.03	2.62	0.21	53.34	--	--	12.35	31.37
10	1.23	0.0312	1.00	2.54	0.23	58.36	0.67	1.70	13.52	34.34
9	1.22	0.0309	1.06	2.69	0.16	40.6	--	--	9.41	23.90
8	1.22	0.0309	1.08	2.74	0.14	35.52	--	--	8.23	20.90
7	1.21	0.0307	1.18	2.99	0.03	7.6	--	--	1.76	4.47
6	1.20	0.0304	1.18	2.99	0.02	5.08	--	--	1.17	2.97
5	1.19	0.0300	1.18	2.99	0.01	2.54	0.80	2.03	0.59	1.49
4	1.18	0.0299	1.17	2.97	0.01	2.54	--	--	0.59	1.49
3	1.12	0.0284	1.15	2.92	+	--	--	--	--	--
2	1.04	0.0264	1.08	2.74	+	--	--	--	--	--
1	0.95	0.0241	1.02	2.59	+	--	--	--	--	--
0	0.87	0.0220	0.88	2.23	+	--	0.43	1.09	--	--

Legend

———— Prefiring Surface
 - - - - - Postfiring Surface
 - - - - - Char Depth

Motor Parameters

Burn Time 17.00 sec
 Web Pressure 267 psi (1.84 x 10⁶ N/m²)
 Throat Diameter 0.34 in. (0.86 cm)

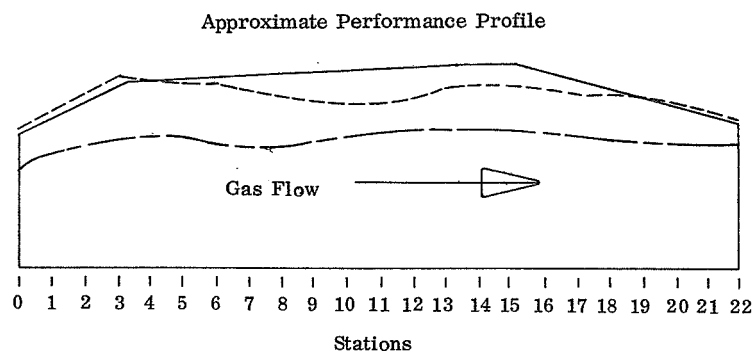
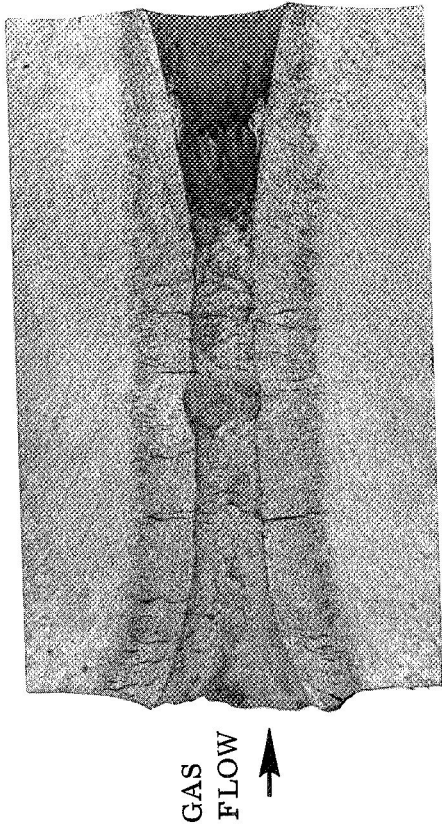
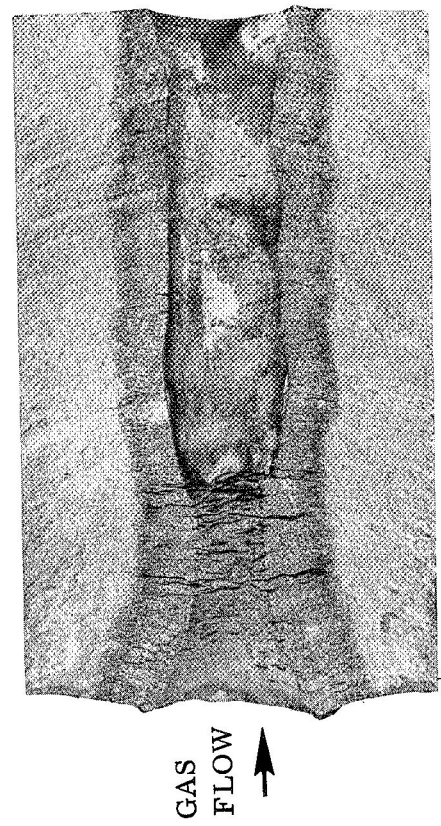


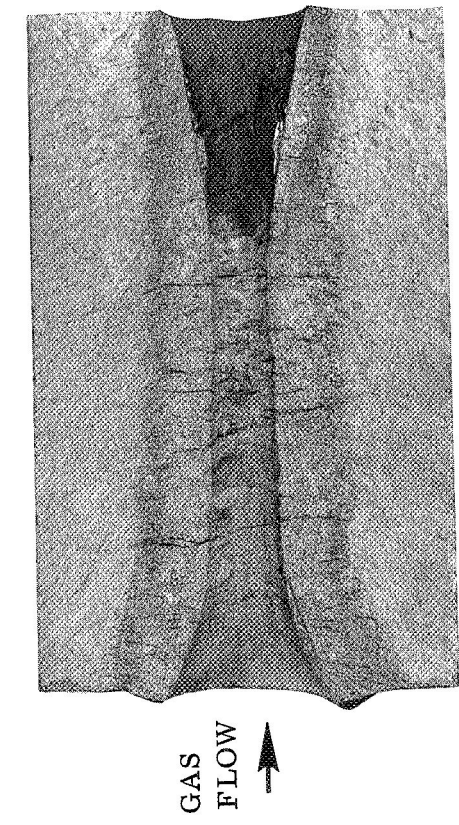
Figure 62. - TU-379 Nozzle Data, RI-4009 Resin, Motor No. 654



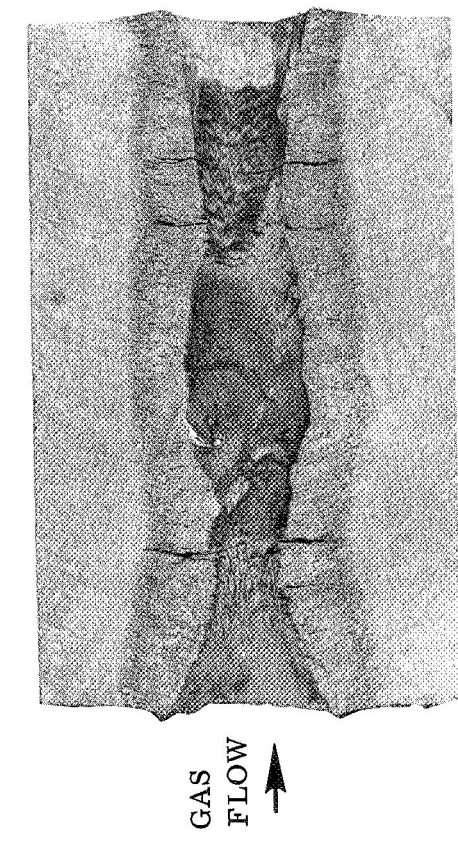
Nozzle 652, 10% Fiber



Nozzle 654, 30% Fiber



Nozzle 651, 5% Fiber



Nozzle 653, 20% Fiber

Figure 63. -TU-379 Nozzle Sections, Carbon Fiber Reinforcements

RI-4009 Resin

10% Chopped Glass

(0.25 in., 0.64 cm)

Station No.	Contour		Erosion		Material Loss		Char Depth		Erosion Rate	
	(in.)	(m)	(in.)	(m x 10 ⁻²)	(in.)	(m x 10 ⁻²)	(in.)	(m x 10 ⁻²)	(mils/sec)	(m/sec x 10 ⁻⁵)
22	0.92	0.0233	0.96	2.44	+	--	0.72	1.83	--	--
21	0.96	0.0243	1.00	2.54	+	--	--	--	--	--
20	0.99	0.0251	1.03	2.62	+	--	0.73	1.86	--	--
19	1.04	0.0264	1.06	2.69	+	--	--	--	--	--
18	1.08	0.0274	1.11	2.79	+	--	--	--	--	--
17	1.11	0.0281	1.14	2.84	+	--	--	--	--	--
16	1.14	0.0284	1.17	2.96	+	--	--	--	--	--
15	1.18	0.0299	1.19	3.10	+	--	0.76	1.94	--	--
14	1.21	0.0307	1.17	2.96	0.04	0.101	--	--	2.39	6.07
13	1.24	0.0314	1.17	2.96	0.07	0.178	--	--	4.19	10.61
12	1.24	0.0314	1.18	3.00	0.06	0.153	--	--	3.59	9.10
11	1.24	0.0314	1.18	3.00	0.06	0.153	--	--	3.59	9.10
10	1.23	0.0312	1.16	2.94	0.07	0.178	0.76	1.94	4.19	10.61
9	1.22	0.0309	1.14	2.84	0.08	0.204	--	--	3.79	9.65
8	1.22	0.0309	1.16	2.94	0.06	0.153	--	--	3.59	9.10
7	1.21	0.0307	1.12	2.83	0.09	0.228	--	--	5.39	13.80
6	1.20	0.0304	1.14	2.84	0.06	0.153	--	--	3.59	9.10
5	1.19	0.0300	1.13	2.86	0.06	0.153	0.71	1.80	3.59	9.10
4	1.18	0.0299	1.12	2.83	0.06	0.153	--	--	3.59	9.10
3	1.12	0.0284	1.10	2.80	0.02	0.058	--	--	1.20	3.04
2	1.04	0.0264	1.03	2.62	0.01	0.025	--	--	0.56	1.42
1	0.95	0.0241	0.98	2.48	+	--	--	--	--	--
0	0.87	0.0220	0.88	2.24	+	--	0.48	1.24	--	--

Legend

—————	Prefiring Surface
-----	Postfiring Surface
-----	Char Depth

Motor Parameters

Burn Time	16.67 sec
Web Pressure	330 psia
Throat Diameter	0.34 in. (0.86 cm)

Approximate Performance Profile

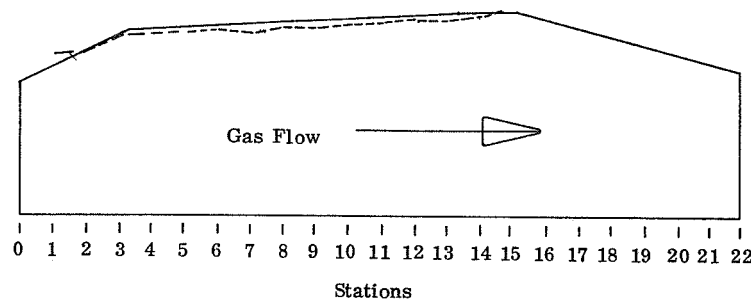


Figure 64. - TU-379 Nozzle Data, RI-4009 Resin, Motor No. 670

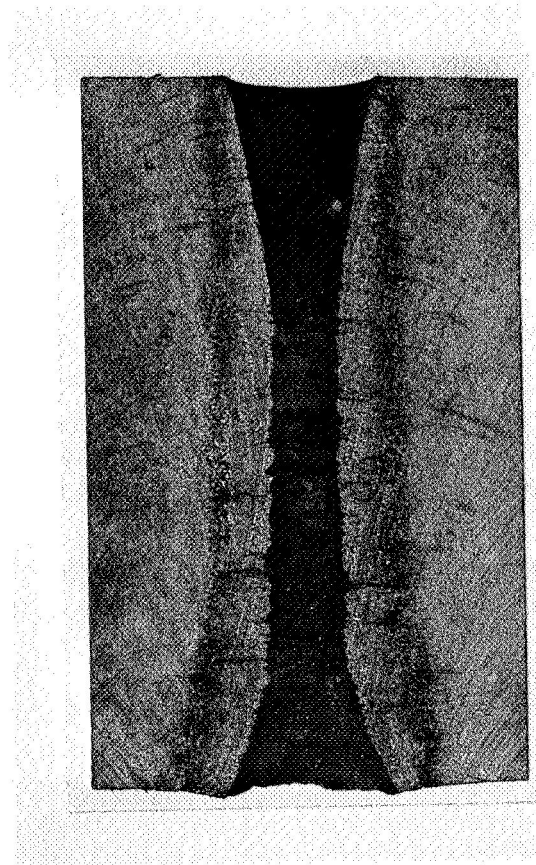


Figure 65. - Post-test Section, TU-379 Nozzle Assembly, 0.25 In. (0.64 cm)
10 Percent Glass Fiber, Motor No. 670

Test results of the TU-379 erosion test (Motor No. 668) indicate chopped carbon fiber impregnated with SC-1008 resin prior to the addition of other ingredients had higher erosion rates than the nontreated chopped carbon fiber (Motor No. 652, page 96). The erosion performance of chopped carbon fiber MX-4926, Motor No. 667, evaluation was limited to a visual observation since the erosion station data were unavailable due to loss of pressure time trace.

Complete test results are shown in Figures 66 and 67. Photographs of the sectioned halves are shown in Figure 68.

Durez 10694 Phenolic Resin

Problems were encountered with the mixing of the fiber reinforcement in the Durez 10694 LCCM compounds. The two basic graphite particle formulations (large GL-1012 and small particle (74 micron) graphite) containing the single stage Durez 10694 phenolic resin have the distinct advantages of curing at low pressure and of being either castable or moldable. However, incorporation of any one of the three fiber reinforcements, even at a 2.5 percent by weight level, made the mix very dry such that it could not be hand packed into the 5 x 10 x 1.0 in. (12.70 x 25.40 x 2.54 cm) material slab mold. Alcohol was added to the mixtures to lower the viscosity in order to add the remaining 2.5 percent by weight fiber, to bring the reinforcement level up to the programmed 5 percent, and to increase the wetting and packing consistency of the formulations. No attempts were made to fabricate slabs from formulations containing fiber above the 5 percent level.

The material slabs were machined into test specimens and were tested for tensile strength, density, compressive strength, and thermal conductivity.

The physical properties of the two Durez 10694 basic formulations containing fibers at the 5 percent level were disappointing. Of the three reinforcement materials evaluated, the carbon fiber evaluations appeared to provide the superior properties. However, the property values of these specimens were inferior to the properties obtained from specimens produced from the basic formulations without fiber reinforcement. The data also indicated that the fibers had a greater property degradation effect on the specimens produced from the 74 micron (maximum) size graphite particles than they did on the specimens produced from the 325 micron (average) size graphite particles. Test results are shown in Table XIV.

After taking into consideration program objectives, physical property tests, and results of the material manufacturing evaluations, the 5 percent carbon fiber reinforcement was selected for TU-379 motor erosion testing over the other two reinforcement candidates. It is noted that the material compounds containing the carbon fibers were less difficult to mix and form into test slabs than were the other compounds.

RI-4009 Resin,
 10% Chopped Carbon Fiber
 (0.25 in., 0.64 cm)
 Impregnated with SC-1008 Resin

Station No.	Contour				Material Loss		Char Depth		Erosion Rate	
	Initial		Erosion							
	(in.)	(m)	(in.)	(m x 10 ⁻²)	(in.)	(m x 10 ⁻⁴)	(in.)	(m x 10 ⁻²)	(mils/sec)	(m/sec x 10 ⁻⁵)
22	0.92	0.0233	0.96	2.43	+	--	0.74	1.88	--	--
21	0.96	0.0243	0.99	2.51	+	--	--	--	--	--
20	0.99	0.0251	1.02	2.59	+	--	0.74	1.88	--	--
19	1.04	0.0264	1.06	2.69	+	--	--	--	--	--
18	1.08	0.0274	1.10	2.79	+	--	--	--	--	--
17	1.11	0.0281	1.12	2.84	+	--	--	--	--	--
16	1.14	0.0284	1.17	2.97	+	--	--	--	--	--
15	1.18	0.0299	1.20	3.04	+	--	0.74	1.88	--	--
14	1.21	0.0307	1.18	2.99	0.03	7.6	--	--	1.72	4.37
13	1.24	0.0314	1.14	2.89	0.10	2.34	--	--	5.74	14.58
12	1.24	0.0314	1.10	2.79	0.14	35.56	--	--	8.03	20.39
11	1.24	0.0314	1.08	2.74	0.16	40.64	--	--	9.18	23.31
10	1.23	0.0312	1.13	2.87	0.10	25.4	0.70	1.78	5.74	14.58
9	1.22	0.0309	1.14	2.89	0.08	20.32	--	--	4.59	11.66
8	1.22	0.0309	1.15	2.92	0.07	17.78	--	--	4.02	10.21
7	1.21	0.0307	1.15	2.92	0.06	15.24	--	--	3.44	8.74
6	1.20	0.0304	1.18	2.99	0.02	5.08	--	--	1.15	2.92
5	1.19	0.0300	1.16	2.94	0.03	7.6	0.76	1.93	1.72	4.37
4	1.18	0.0299	1.16	2.94	0.02	5.08	--	--	1.15	2.92
3	1.12	0.0284	1.14	2.89	+	--	--	--	--	--
2	1.04	0.0264	1.06	2.69	+	--	--	--	--	--
1	0.95	0.0241	0.99	2.51	+	--	--	--	--	--
0	0.87	0.0220	0.87	2.21	0.00	0	0.48	1.22	0.00	0

Legend		Motor Parameters	
—————	Prefiring Surface	Burn Time	17.4 sec
-----	Postfiring Surface	Web Pressure	268 psi (2.35 x 10 ⁶ N/m ²)
-----	Char Depth	Throat Diameter	0.34 in. (0.86 cm)

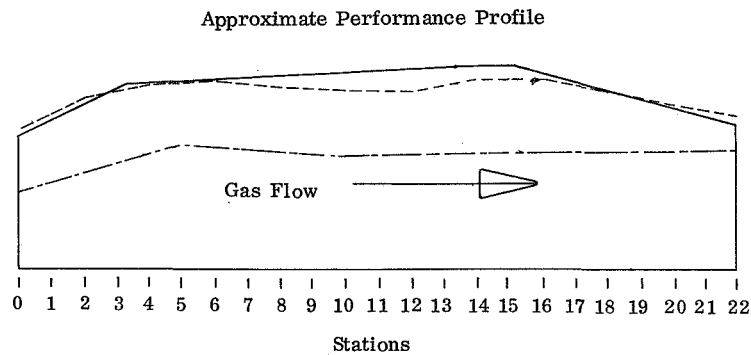


Figure 66. - TU-379 Nozzle Data, RI-4009 Resin, Motor No. 668

RI-4009 Resin

10% Chopped Carbon Fiber
(0.25 in., 0.64 cm)

Impregnated with MX-4926 Resin

Station No.	Contour				Material Loss		Char Depth		Erosion Rate	
	Initial (in.)	(m)	Erosion (in.)	(m x 10 ⁻²)	(in.)	(m x 10 ⁻⁴)	(in.)	(m x 10 ⁻²)	(mils/sec)	(m/sec x 10 ⁻⁵)
22	0.92	0.0233	0.94	2.38	+	--	0.70	1.77	N/A	N/A
21	0.96	0.0243	0.99	2.51	+	--	--	--		
20	0.99	0.0251	1.02	2.59	+	--	0.78	1.98		
19	1.04	0.0264	1.07	2.71	+	--	--	--		
18	1.08	0.0274	1.10	2.79	+	--	--	--		
17	1.11	0.0281	1.13	2.87	+	--	--	--		
16	1.14	0.0284	1.16	2.94	+	--	--	--		
15	1.18	0.0299	1.19	3.02	+	--	0.79	2.0		
14	1.21	0.0307	1.22	3.09	+	--	--	--		
13	1.24	0.0314	1.22	3.09	0.02	5.08	--	--		
12	1.24	0.0314	1.22	3.09	0.02	5.08	--	--		
11	1.24	0.0314	1.21	3.07	0.03	7.6	--	--		
10	1.23	0.0312	1.20	3.04	0.03	7.6	0.79	2.0		
9	1.22	0.0309	1.20	3.04	0.02	5.08	--	--		
8	1.22	0.0309	1.19	3.02	0.03	7.6	--	--		
7	1.21	0.0307	1.18	2.99	0.03	7.6	--	--		
6	1.20	0.0304	1.17	2.97	0.03	7.6	--	--		
5	1.19	0.0300	1.18	2.99	0.01	2.54	--	--		
4	1.18	0.0299	1.16	2.94	0.02	5.08	0.76	1.93		
3	1.12	0.0284	1.13	2.87	+	--	--	--		
2	1.04	0.0264	1.06	2.69	+	--	--	--		
1	0.95	0.0241	1.00	2.54	+	--	--	--		
0	0.87	0.0220	0.88	2.23	+	--	0.45	1.14		

Legend

———— Prefiring Surface
 - - - - - Postfiring Surface
 - - - - - Char Depth

Motor Parameters

Burn Time N/A
 Web Pressure N/A
 Throat Diameter 0.34 in. (0.86 cm)

Approximate Performance Profile

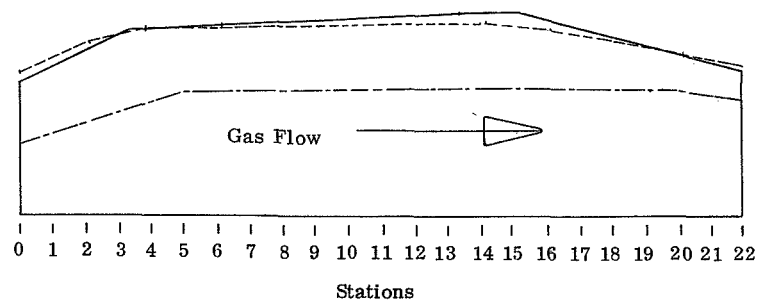
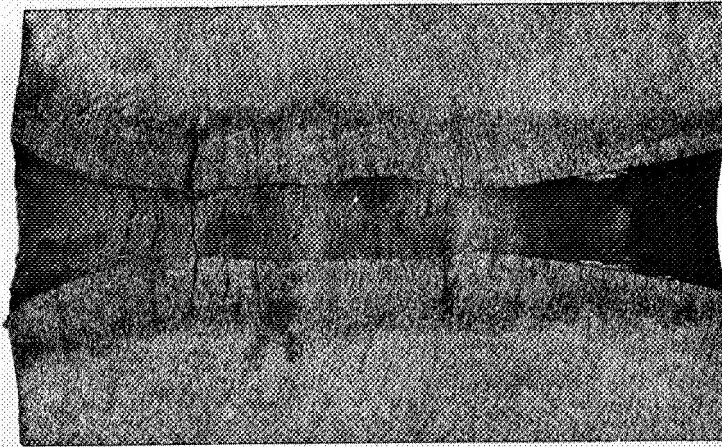
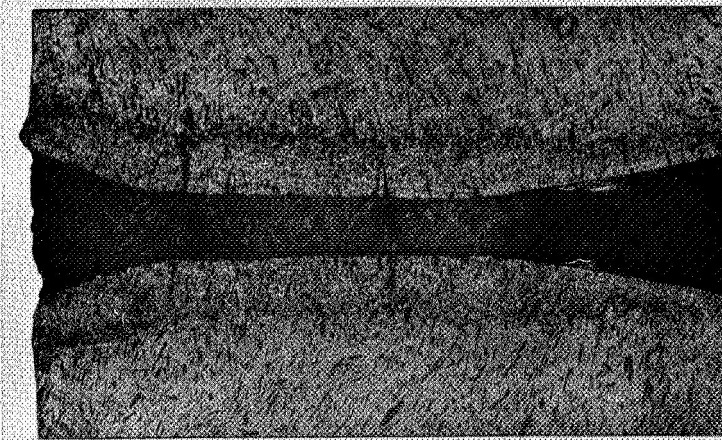


Figure 67. - TU-379 Nozzle Data, RI-4009 Resin, Motor No. 667



MOTOR NO. 668

SC-1008, PRETREATED FIBER AND RI-4009 RESIN



MOTOR NO. 667

MX-4926 FIBER AND RI-4009 RESIN

Figure 68. - TU-379 Nozzle Pretreated Carbon Fiber Sections

TABLE XIV. - ICCM DEVELOPMENT, PHYSICAL PROPERTIES FIBER REINFORCEMENT STUDY, DUREZ 10694, 25 PERCENT RESIN

Resin	Percent Resin	Fiber		Cure Conditions***	Mix No.	Tensile Strength		Density (g/cc)	Compressive Strength	
		Type	Percent			(psi)	(N/m ² x 10 ⁶)		(psi)	(N/m ² x 10 ⁶)
Durez 10694*	25	Rayon (1 in., 2.54 cm)	5		631	664 840 520 674	4.58 5.80 3.59 4.65	1.33	3,160 3,100 3,390 3,216	21.8 21.4 23.4 22.2
Durez 10694*	25	Glass (0.75 to 1.0 in., 1.9 to 2.54 cm)	5		633	755 1,080 -- 912	5.21 7.45 -- 6.29	1.51	5,080 3,915 6,800 5,265	35.1 27.0 46.9 36.3
Durez 10694*	25	Carbon (0.25 in., 0.64 cm)	5		635	1,765 1,760 2,120 1,881	12.18 12.14 14.63 12.9	1.38	7,080 6,620 7,310 7,000	48.9 45.7 50.4 48.3
Durez 10694**	25	Rayon (1.0 in., 2.54 cm)	5		637					
Durez 10694**	25	Glass (0.75 to 1.0 in., 1.9 to 2.54 cm)	5		639	206 240 -- 223	1.42 1.7 -- 1.5	1.31	1,835 1,260 2,200 1,765	12.7 8.7 15.2 12.2
Durez 10694**	25	Chopped Carbon Fiber (0.25 in., 0.64 cm)	5		641	411 528 467 468	2.83 3.64 3.22 3.22	1.15	1,680 1,580 1,580 1,613	11.6 10.9 10.9 11.1

SAMPLE BROKE DURING MACHINING

*GL-1012 graphite

**Small graphite, less than 200 mesh.

***30 psi (0.21 x 10⁶ N/m²) at 300° F (422° K)

In an attempt to improve the mechanical properties of the fiber reinforced Durez 10694 system, the resin content of the formulation was increased from 25 to 40 percent. It was hoped that the mechanical properties would improve and that the material would remain trowelable or castable.

Test specimens of tensile and compression were fabricated, using the 40 percent Durez 10694 resin with 5 percent carbon fiber and 55 percent graphite powder (GL-1012). In addition, a TU-379 nozzle was fabricated using the above formulation (Mix No. 671).

An increase in tensile and compressive strength was realized when the resin content was raised from 25 to 40 percent. The test results are shown on Table XV. It may be noted that the cure pressure on the 40 percent resin mix was only 30 psi ($0.21 \times 10^6 \text{ N/m}^2$), while the 25 percent resin content specimens were cured at 200 psi ($1.38 \times 10^6 \text{ N/m}^2$) pressure. It was believed that additional structural strength could be attained with the 40 percent resin mix if the sample had been cured at 200 psi ($1.38 \times 10^6 \text{ N/m}^2$). This was confirmed during the Filler Additive Study in Task 03.

Results of the static test firing of the 40 percent Durez 10694 chopped carbon TU-379 nozzle (Figure 69) showed that the throat erosion rate was good (4.46 mil/sec or $11.33 \text{ m} \times 10^{-5}$) and exhibited a firm char and had no radial cracking. Figure 70 shows the sectioned nozzle. The 25 percent Durez 10694 with 75 percent GL-1012 graphite powder and no fiber reinforcement showed a throat erosion rate of 6.2 to 6.9 mils/sec ($5.7 \text{ to } 16.5 \text{ m} \times 10^{-5}$). Refer to Table X.

It was noted that during the cure of the 40 percent Durez 10694 chopped carbon (Motor No. 671) nozzle, there was an excessive flow of resin. In order to reduce the flow, it is recommended that the carbon fiber be increased to 10 percent. The additional fiber should absorb the excess resin and reduce the excessive flow during cure.

TABLE XV. - LCCM DEVELOPMENT, PHYSICAL PROPERTIES FIBER REINFORCEMENT STUDY,
DUREZ 10694, 25 AND 40 PERCENT RESIN

Resin	Percent Resin	Filler	Percent	Cure Conditions	Mix No.	Tensile		Density	Compressive	
						(psi)	Strength (N/m ² x 10 ⁶)	(g/cc)	(psi)	Strength (N/m ² x 10 ⁶)
Durez 10694	40	Chopped Carbon Fiber	5	*	659	2,075	14.3	1.39	8,540	58.9
						1,840	12.7	1.38	8,640	59.6
						1,925	13.3	1.40	7,780	53.7
						1,945	13.4	1.38	8,333	57.5
Durez 10694	40	Chopped Carbon Fiber	10	*	663	1,800	12.4	1.34	10,760	74.2
						1,792	12.4	1.33	9,100	62.7
						1,796	12.4	1.35	10,540	72.7
						1,796	12.4	1.33	10,133	69.9
Durez 10694	25	Chopped Carbon Fiber	5	*	635	1,765	12.2		7,080	48.9
						1,760	12.2		6,620	45.7
						2,120	14.6		7,310	50.4
						1,881	13.0	1.38	7,000	48.3
Durez 10694	25	None		**		1,770	12.2	1.47	7,175	49.5
						2,720	18.8	1.45	7,230	49.9
						2,480	17.1	1.47	6,120	42.2
						2,323	16.0	1.46	6,841	47.2

*30 psi (0.207 x 10⁶ N/m²) at 300° F (422° K)

**200 psi (1.38 x 10⁶ N/m²) at 300° F (422° K)

Durez 10694

5% Chopped Carbon Fiber
(0.25 in. , 0.64 cm)

Station No.	Initial		Erosion		Material Loss		Char Depth		Erosion Rate	
	(in.)	(m)	(in.)	(m x 10 ⁻²)	(in.)	(m x 10 ⁻⁴)	(in.)	(m x 10 ⁻²)	(mils/sec)	(m/sec x 10 ⁻⁵)
22	0.92	0.0233	0.93	2.36	+	--	0.67	1.70	--	--
21	0.96	0.0243	0.94	2.38	0.02	5.0	--	--	--	--
20	0.99	0.0251	1.00	2.54	+	--	0.70	1.77	--	--
19	1.04	0.0264	1.04	2.64	0.00	0	--	--	--	--
18	1.08	0.0274	1.08	2.74	0.00	0	--	--	--	--
17	1.11	0.0281	1.12	2.84	+	--	--	--	--	--
16	1.14	0.0284	1.14	2.89	0.00	0	--	--	--	--
15	1.18	0.0299	1.17	2.97	0.01	2.5	0.73	1.86	0.56	1.42
14	1.21	0.0307	1.18	2.99	0.03	7.6	--	--	1.67	4.24
13	1.24	0.0314	1.17	2.97	0.07	17.78	--	--	3.90	9.9
12	1.24	0.0314	1.17	2.97	0.07	17.78	--	--	3.90	9.9
11	1.24	0.0314	1.16	2.94	0.08	20.32	--	--	4.46	11.33
10	1.23	0.0312	1.16	2.94	0.07	17.78	0.77	1.95	3.90	9.9
9	1.22	0.0309	1.15	2.92	0.07	17.78	--	--	3.90	9.9
8	1.22	0.0309	1.14	2.89	0.08	20.32	--	--	4.46	11.33
7	1.21	0.0307	1.13	2.87	0.08	20.32	--	--	4.46	11.33
6	1.20	0.0304	1.12	2.84	0.08	20.32	--	--	4.46	11.33
5	1.19	0.0300	1.11	2.82	0.08	20.32	0.74	1.88	4.46	11.33
4	1.18	0.0299	1.10	2.79	0.08	20.32	--	--	4.46	11.33
3	1.12	0.0284	1.06	2.69	0.06	15.2	--	--	3.34	8.48
2	1.04	0.0264	1.01	2.56	0.03	7.6	--	--	1.67	4.24
1	0.95	0.0241	0.94	2.38	0.01	2.5	--	--	0.59	1.5
0	0.87	0.0220	0.84	2.12	0.03	7.6	0.46	1.16	1.67	4.24

Legend		Motor Parameters	
—————	Prefiring Surface	Burn Time	17.92 sec
-----	Postfiring Surface	Web Pressure	230 psi (1.59 x 10 ⁶ N/m ²)
-----	Char Depth	Throat Diameter	0.34 in. (0.86 cm)

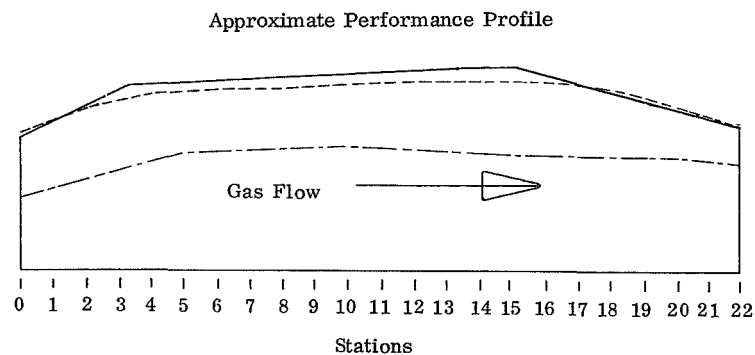


Figure 69. - TU-379 Nozzle Data, Durez 10694, Motor No. 671

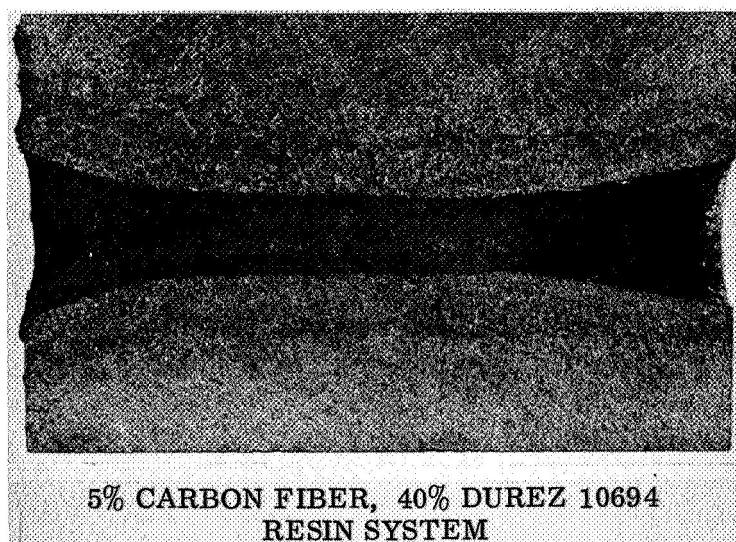


Figure 70. - TU-379 Nozzle Section

TASK 03 - FILLER ADDITIVE STUDIES

The objective of this phase of the development program was to lower the thermal conductivity and decrease density, while maintaining the existing erosion resistance and mechanical properties of the basic LCCM formulations developed in Task 01.

This study was accomplished by combining various types of inert filler materials in the candidate LCCM formulations and subjecting the composites to physical and thermal property tests. The filler materials to be evaluated included ground cork, phenolic and glass microballoons, wood flour, and a liquid elastomer.

The ground cork was supplied by Armstrong Cork Company. It had a bulk density of approximately 7 lb/ft³ (112.14 kg/m³) and a size range approximately the same as sand.

The phenolic microballoons were a Union Carbide BJO-0930 product, with a bulk density of 6.5 lb/ft³ (104.13 kg/m³) and a size range from 0.0002 to 0.005 in. (0.0005 to 0.0127 cm) diameter.

The glass microspheres were Emerson Cumming's 1W-101, with a bulk density of 14 lb/ft³ (224.28 kg/m³) and a size range of 0.0004 to 0.01 in. (0.001 to 0.0254 cm) diameter.

The wood flour was supplied by Hooker Chemical, with a bulk density of 19 lb/ft³ (304.38 kg/m³). It is a finely ground powder.

The elastomer was Hitco 158 (a liquid NBR rubber), which cures after solvent evaporation and the application of heat.

Study effort included optimizing filler concentration to provide the best possible combination of mechanical and thermal properties and erosion resistance. Table XVI outlines the test matrix and filler additives evaluated. The material performance and the evaluations of filler studies involving the three LCCM formulations are discussed below. Formulations are identified with the resin system used in the LCCM's.

Monsanto RI-4009 Resin

The various filler variations outlined in Table XVI were mixed and cured into 12 by 12 by 0.5 in. (30.48 by 30.48 by 1.27 cm) panels. The cork, wood flour, and microballoons mixed into the LCCM formulation quite easily. However, the NBR elastomer presented problems. It caused the mixture to become gummy and prevented a uniform dispersement of all the ingredients.

TABLE XVI. - LCCM DEVELOPMENT, FILLER ADDITIVES TEST MATRIX

Resin	Percent Resin	Basic Formulation			Percent Graphite	Filler Additives*				
		Cure Temperature (°F)	Cure Temperature (°K)	Cure Pressure (psi)		Elastomer Hitco 158 (%)	Cork (%)	Wood Filler (%)	Phenolic Microballoons (%)	Glass Microballoons (%)
RL-4009	40	300	422	1,000	6.9	3	3	3	3	3
					GL-1012	10	10	10	10	10
						15	15	15	15	15
Durez 10694	25					30	30	30	30	30
		300	422	200	1.38	3	3	3	3	3
					GL-1012	10	10	10	10	10
Durez 10694	25					15	15	15	15	15
		300	422	200	1.38	--	3	3	3	--
					75 200 mesh or 74 micron (max)	--	15	15	15	--

Physical property tests**

Tensile strength	(3)
Compressive strength	(3)
Density	(3)
Modulus	(3)
Hardness	(1)

*Evaluated in all three resin systems.

**Applicable to resin and filler combinations that appeared most promising from torch test.

An attempt was made to mix the elastomer with cork, drive off the solvent, grind it to powder or granules, and then introduce it into the LCCM formulation. This also proved unsuccessful, and the Hitco 158 elastomer was dropped as a candidate filler material.

The panels containing the other fillers were subjected to a torch test using an oxyacetylene torch operating in excess of 5,000° F (3,033° K) for 60 seconds. When the panels cooled, they were cut in half and examined. The various panels were compared and photographed. The photos are shown in Figures 71 thru 74. Severe spalling and cracking was noted in all the panels containing glass microspheres. Glass microspheres were dropped as a candidate filler material, and no further testing of the panels with that filler were conducted for mechanical, physical, and thermal properties.

The remaining test panels fabricated from the basic graphite particle/Monsanto RI-4009 resin system formulation with filler additives (cork, phenolic microballoons, and wood flour) were machined into test specimens and tested for tensile strength, density, compressive strength, hardness, and thermal conductivity. Tables XVII, XVIII, and XIX contain the complete test data. The performance data and a brief evaluation of the three above mentioned filler systems are discussed below.

Cork Filler. - The data in Table XVII show approximately a 15 percent reduction in tensile strength when the cork filler is increased from 3 to 10 percent. As the cork filler continues to increase to 30 percent, there is no further reduction in tensile and compressive strengths. The thermal conductivity decreases by 65 percent as the filler content is increased from 3 to 30 percent; however, the char hardness resulting from the torch test is rather low at the higher levels of filler. The density at 3.0 percent cork filler is 1.64 g/cc compared to 1.42 g/cc with 30.0 percent cork.

Phenolic Microballoons. - The data shown in Table XVIII show a slight decrease in tensile strength with increasing filler content and an 11 percent increase in compressive strength only with the filler content of microballoons at 30 percent. Thermal conductivity is decreased 68 percent by increasing the phenolic microballoon percentage, and the char hardness becomes lower, decreasing from a Shore D of 52 with 3 percent filler to 25 with 30 percent filler.

Wood Filler. - Tensile and compressive strengths are greatly improved by addition of the wood flour filler into the basic formulation. Table XIX shows an increase of 57 percent in tensile strength and a 42 percent increase in compressive strength at 30 percent filler.

The wood filler is actually a flour, and it is felt that the increase in tensile strength is due to the small particle size. Earlier studies of tensile strength/particle size relationship showed that physical properties increase with the smaller graphite particles.

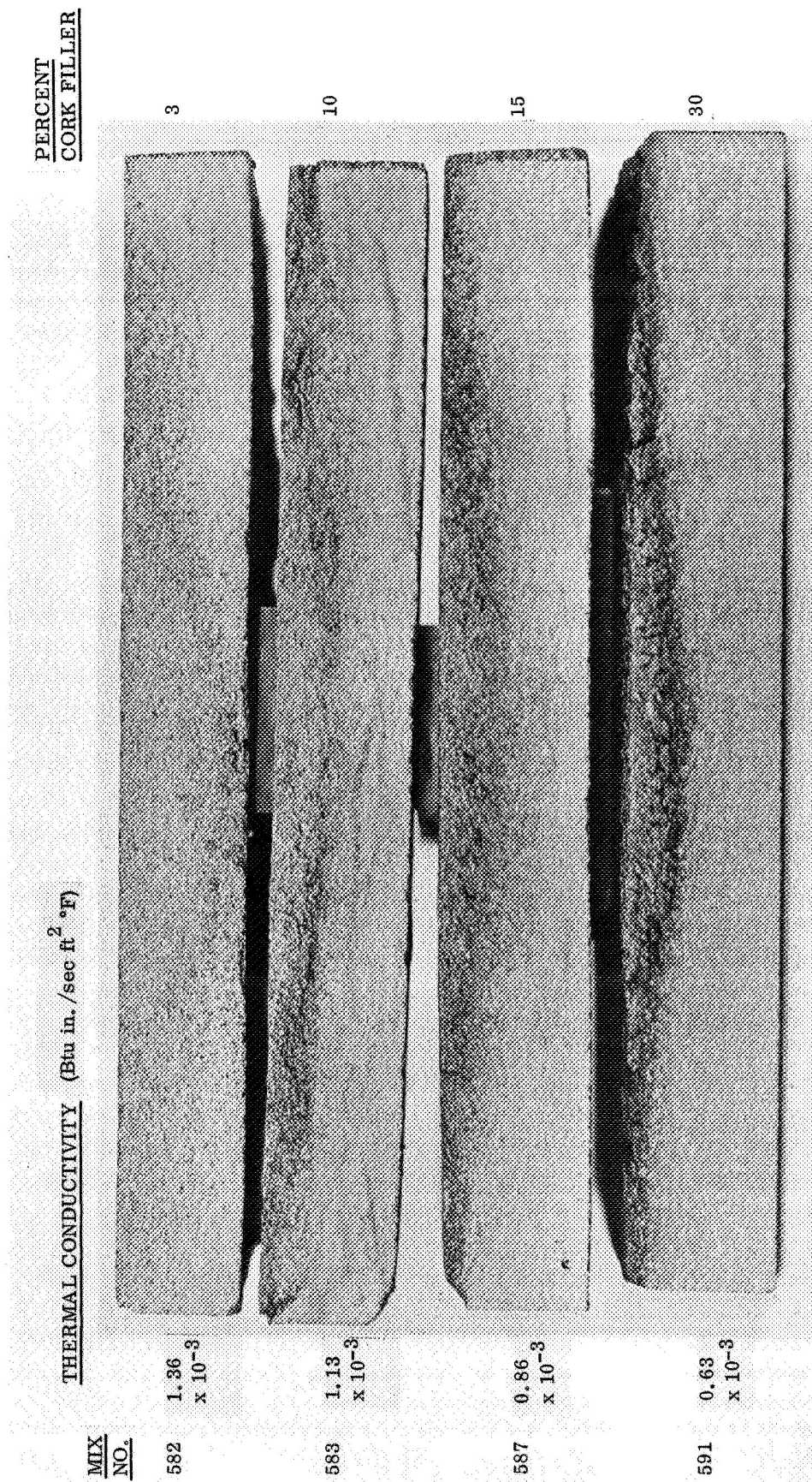


Figure 71. - Test Panels, Cork Filler, RI-4009 Resin

PERCENT PHENOLIC
MICROBALLOON FILLER

THERMAL CONDUCTIVITY (Btu in. /sec ft² °F)

MIX
NO.

576 1.29
x 10⁻³

579 1.23
x 10⁻³

584 0.99
x 10⁻³

588 0.59
x 10⁻³

3

10

15

30

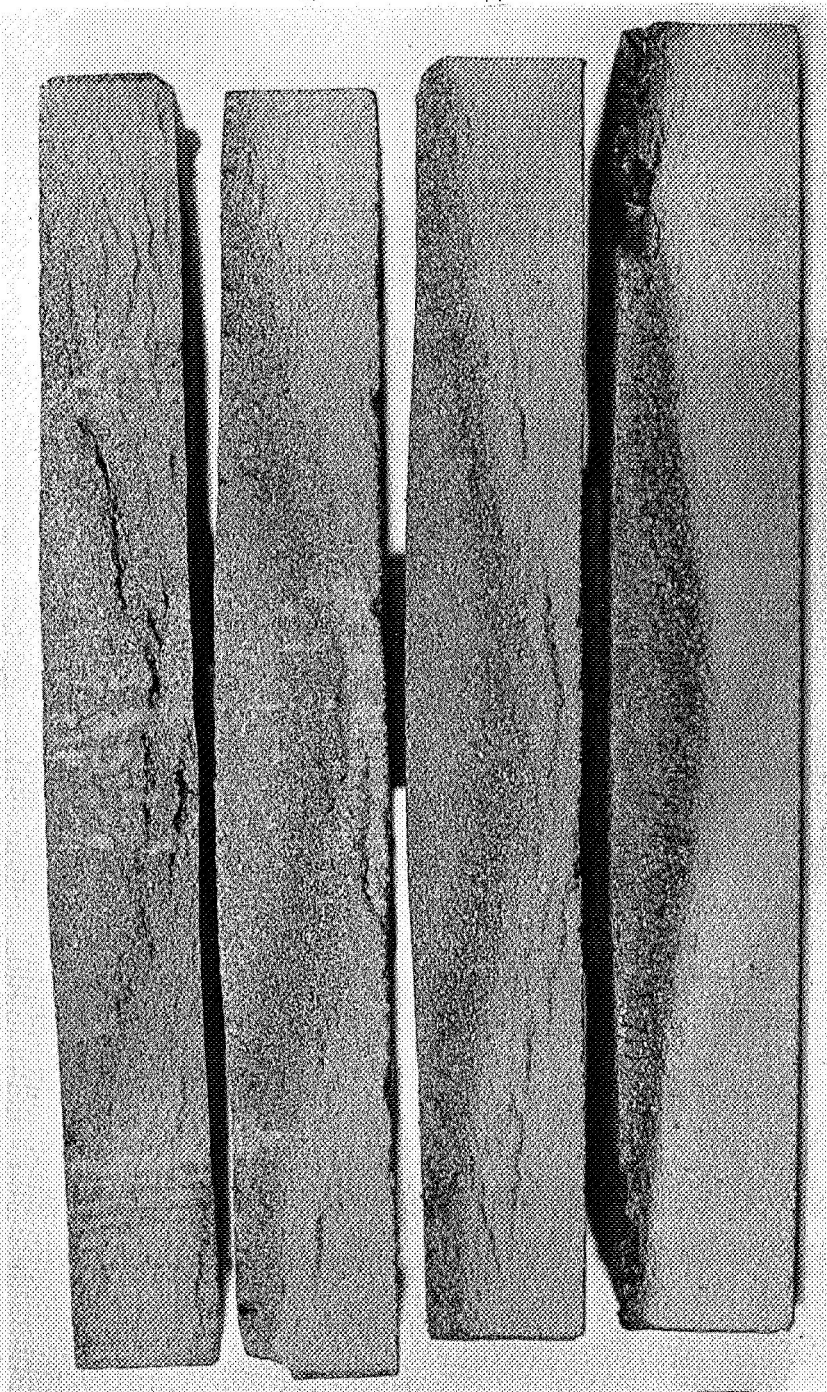


Figure 72. - Test Panels, Phenolic Microballoon Filler, RI-4009 Resin

PERCENT
WOOD FILLER

THERMAL CONDUCTIVITY (Btu in./sec ft.² °F)



Figure 73. - Test Panels, Wood Filler, RI-4009 Resin

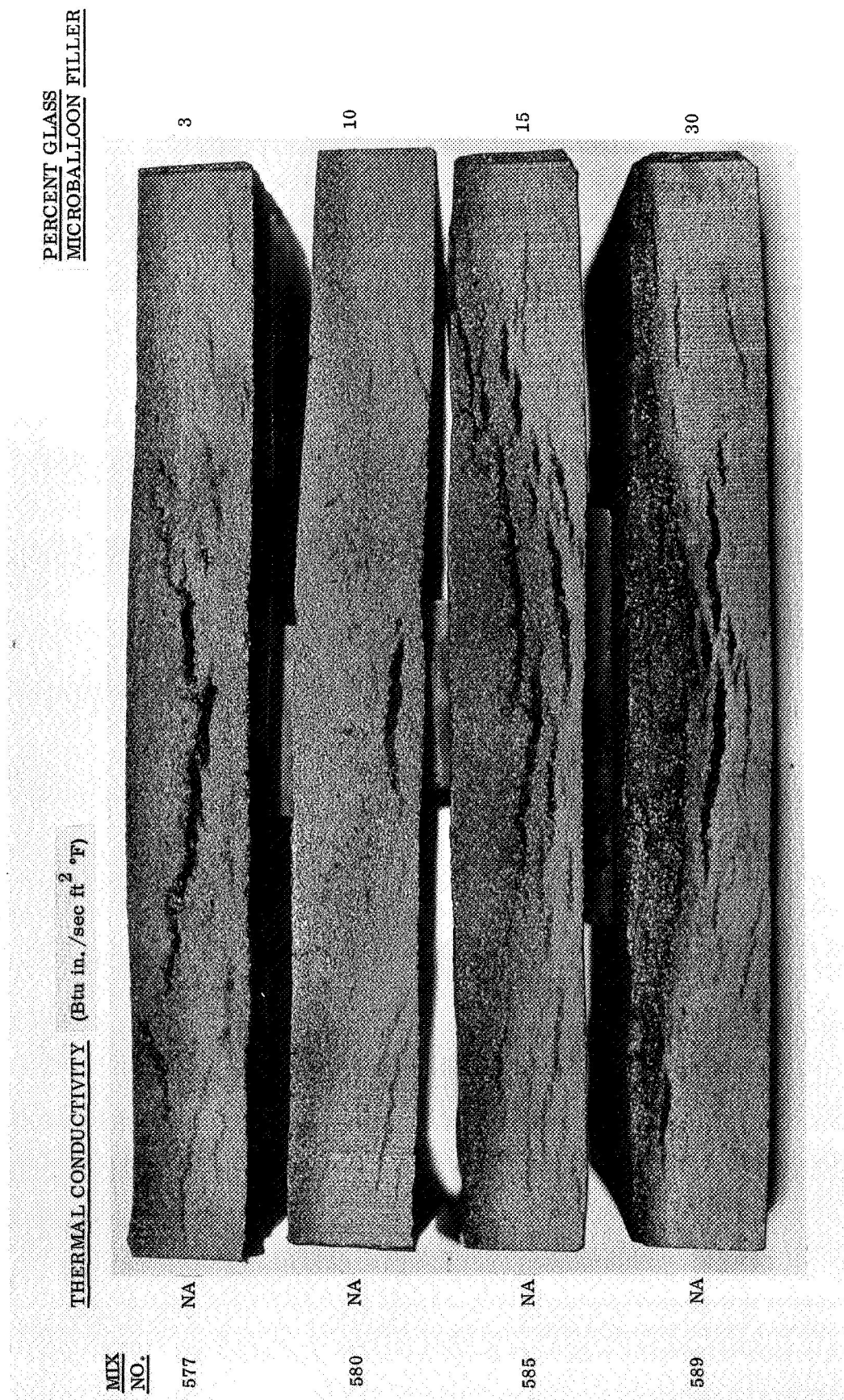


Figure 74. - Test Panels, Glass Microballoon Filler, RI-4009 Resin

TABLE XVII. - LCCM DEVELOPMENT, FILLER ADDITIVE STUDY, CORK PHYSICAL PROPERTIES

Resin	Percent Resin	Percent Cork	Cure Conditions	Mix No.	Tensile Strength		Density (g/cc)	Compressive Strength		Hardness Shore D		Thermal Conductivity	
					(psi)	(N/m ² x 10 ⁶)		(psi)	(N/m ² x 10 ⁶)	Virgin	Char	(Btu in./sec ft ² °F)	(oule/m sec °K x 10 ⁻²)
Monsanto RI-4009	40	3		582	3,645	25.1	1.64	14,200	98.0	79	47	1.26 x 10 ⁻³	64.75
					3,975	27.4	1.64	14,200	98.0			1.46 x 10 ⁻³	75.75
					3,910	27.0	1.64	13,950	96.3			--	--
					3,843	26.5	1.64	14,100	97.3			1.36 x 10 ⁻³	70.25
Monsanto RI-4009	40	10		583	3,145	21.7	1.55	12,975	89.5	82	37	1.09 x 10 ⁻³	56.55
					3,680	26.4	1.59	12,775	88.1			1.19 x 10 ⁻³	61.74
					3,360	23.2	1.58	12,825	88.5			--	--
					3,395	23.4	1.57	12,858	88.7			1.14 x 10 ⁻³	59.15
Monsanto RI-4009	40	15	(6.9 x 10 ⁶ N/m ² at 422°K) 300°F at 1,000 psi	587	3,440	23.7	1.53	12,450	85.9	80	41	0.92 x 10 ⁻³	47.73
					3,495	24.1	1.54	12,300	84.9			0.83 x 10 ⁻³	43.06
					3,180	21.9	1.46	12,450	85.9			--	--
					3,371	23.3	1.51	12,400	85.6			0.88 x 10 ⁻³	45.40
Monsanto RI-4009	40	30	300°F at 1,000 psi	591	3,160	21.8	1.41	12,775	88.2	77	18	0.66 x 10 ⁻³	34.24
					3,430	23.7	1.40	10,750	74.2			0.59 x 10 ⁻³	30.61
					3,535	24.4	1.45	10,675	73.7			--	--
					3,308	22.8	1.42	11,400	78.7			0.63 x 10 ⁻³	32.43
Base													
(Without filler)					4,376	30.17	1.65	17,000	117.21	84	--	1.88 x 10 ⁻³	97.54

TABLE XVIII. - LCCM DEVELOPMENT, FILLER ADDITIVE STUDY, PHENOLIC MICROBALLOON PHYSICAL PROPERTIES

Resin	Percent Resin	Percent Phenolic Microballoons	Cure Conditions	Mix No.	Tensile Strength (psi)		Density (g/cc)		Compressive Strength (psi)		Hardness Shore D		Thermal Conductivity (Btu in./sec ft ² °F)		Thermal Conductivity (joule/m sec °K x 10 ⁻²)	
					(psi)	(N/m ² x 10 ⁶)	(g/cc)	(N/m ² x 10 ⁶)	(psi)	(N/m ² x 10 ⁶)	Virgin	Char	(Btu in./sec ft ² °F)	(joule/m sec °K x 10 ⁻²)	(Btu in./sec ft ² °F)	(joule/m sec °K x 10 ⁻²)
Monsanto RI-4009	40	3		576	4,680	32.3	1.63	105.1	15,225	105.1	83	52	1.36 x 10 ⁻³	70.56	1.36 x 10 ⁻³	70.56
					4,365	30.1	1.63	110.7	16,050	110.7			1.23 x 10 ⁻³	63.82	1.23 x 10 ⁻³	63.82
					3,825	26.4	1.63	111.1	16,100	111.1						
					4,266	29.4	1.63	108.9	15,791	108.9					1.30 x 10 ⁻³	67.19
Monsanto RI-4009	40	10		579	4,520	31.2	1.55	114.7	16,625	114.7	82	47	1.23 x 10 ⁻³	63.82	1.23 x 10 ⁻³	63.82
					4,650	32.1	1.55	106.9	15,500	106.9			1.23 x 10 ⁻³	63.82		
					3,865	26.7	1.53									
					4,345	30.0	1.54	110.8	16,062	110.8					1.23 x 10 ⁻³	63.82
Monsanto RI-4009	40	15		584	4,180	28.8	1.52	122.4	17,750	122.4	80	45	0.99 x 10 ⁻³	51.36	0.99 x 10 ⁻³	51.36
					3,950	27.3	1.54	114.7	16,625	114.7			0.96 x 10 ⁻³	49.81	0.96 x 10 ⁻³	49.81
					4,250	29.3	1.54	112.5	16,300	112.5						
					4,126	28.5	1.54	116.5	16,891	116.5					0.98 x 10 ⁻³	50.59
Monsanto RI-4009	40	30		588	4,420	30.5	1.37	118.3	17,150	118.3	86	25	0.59 x 10 ⁻³	30.61	0.59 x 10 ⁻³	30.61
					3,835	26.5	1.38	119.0	17,250	119.0			0.59 x 10 ⁻³	30.61		
					3,870	26.7	1.39	124.2	18,000	124.2						
					4,041	27.9	1.38	120.5	17,466	120.5					0.59 x 10 ⁻³	30.61
Base (Without filler)				557	4,140	28.6	--	116.1	16,825	116.1	84	N/A				
					4,740	32.7	--	117.5	17,025	117.5						
				530	4,250	29.3	--	118.3	17,150	118.3						
					4,376	30.2	1.65	117.3	17,000	117.3					1.88 x 10 ⁻³	97.54

Note: Percent GL-1012 graphite = 100 - (percent filler + percent resin).

TABLE XIX. - LCCM DEVELOPMENT, FILLER ADDITIVE STUDY, WOOD FLOUR PHYSICAL PROPERTIES

Resin	Percent Resin	Percent Wood Flour	Cure Conditions	Mix No.	Tensile Strength		Density (g/cc)	Compressive Strength		Hardness Shore D		Thermal Conductivity	
					(psi)	(N/m ² x 10 ⁶)		(psi)	(N/m ² x 10 ⁶)	Virgin	Char	(Btu in./sec ft ² °F)	(joule/m sec °K x 10 ⁻²)
Monsanto RI-4009	40	3		578	3,530 3,880 3,395 3,601	24.3 26.8 23.4 24.8	1.65 1.65 1.65 1.65	16,100 14,700 16,025 15,608	111.1 101.4 110.6 107.6	80	50	1.32 x 10 ⁻³ 1.46 x 10 ⁻³ --- 1.39 x 10 ⁻³	68.49 75.75 --- 72.12
	40	10		581	4,565 4,835 4,585 4,661	31.5 33.3 31.6 32.1	1.61 1.60 1.61 1.61	-- 15,425 18,000 16,712	-- 106.3 124.2 115.2	84	49	1.19 x 10 ⁻³ 1.29 x 10 ⁻³ --- 1.24 x 10 ⁻³	61.74 66.93 --- 64.34
	40	15		586	5,210 5,020 5,380 5,203	35.9 34.6 37.1 35.9	1.58 1.58 1.57 1.58	18,000 18,300 18,280 18,193	124.2 126.3 126.1 125.4	84	49	1.06 x 10 ⁻³ 0.92 x 10 ⁻³ --- 0.99 x 10 ⁻³	55.00 47.73 --- 51.37
	40	30		590	5,975 6,125 4,920 5,673	41.2 42.2 33.9 39.1	1.50 1.50 1.50 1.50	22,150 22,350 21,850 22,116	152.8 154.2 150.8 152.5	84	30	0.69 x 10 ⁻³ 0.79 x 10 ⁻³ --- 0.74 x 10 ⁻³	35.80 40.99 --- 38.40
Base													
(Without filler)				449	4,140 4,740 4,250 4,376	28.5 32.7 29.3 30.2	-- -- -- 1.65	16,825 17,025 17,150 17,000	116.1 117.5 118.3 117.2	84	N/A	1.88 x 10 ⁻³	97.54

Note: Percent GL-1012 graphite = 100 - (percent filler + percent resin).

The wood flour samples appeared to have good char retention at the 15 percent filler level. A char hardness of 49 (Shore D) is recorded at 15 percent filler, which decreases to a hardness of 30 at the 30 percent level.

Thermal conductivity is decreased by 61 percent as the filler concentration is increased from 3 to 30 percent.

TU-379 Testing of Formulations Containing Fillers. - Based upon the test results shown in this study, the following filler formulations were evaluated in TU-379 material screening motors for the RI-4009 resin.

Cork Filler (Mix No. 582)

Monsanto RI-4009 resin	40.0 percent
Cork	3.0 percent
GL-1012 graphite	57.0 percent
Cure temperature	300° F (422° K)
Cure pressure	1,000 psi (6.9×10^6 N/m ²)

The above formulation is based upon: (1) the higher tensile and compressive strengths attained at the low filler level, (2) the char retention appears to be better at the lower level of filler.

Phenolic Microballoons (Mix No. 584)

Monsanto RI-4009 resin	40.0 percent
Microballoons	15.0 percent
GL-1012 graphite	45.0 percent
Cure temperature	300° F (422° K)
Cure pressure	1,000 psi (6.9×10^6 N/m ²)

This selection is based upon: (1) the thermal conductivity appears to be lower at the 15 percent filler level, and (2) the char hardness remains practically unchanged at this filler concentration. Tensile strength is about the same with or without filler.

Wood Flour

(Three Formulations Were Evaluated)

Monsanto RI-4009 resin (%)	40.0	40.0	40.0
Wood flour (%)	10.0	15.0	20.0
GL-1012 graphite (%)	50.0	45.0	40.0
Cure temperature	300° F (422° K)		
Cure pressure	1,000 psi (6.9×10^6 N/m ²)		

The above formulations are based upon: (1) good structural strength maintained with 10 and 15 percent filler, (2) no change in char hardness noted until after the 15 percent level, and (3) lower thermal conductivity properties. Although no

physical data were available, the 20 percent level was evaluated in a TU-379 nozzle as both thermal conductivity and char hardness change significantly between the 15 and 30 percent level.

Test results of the TU-379 nozzles fabricated with the various fillers are shown in Figures 75 thru 79. Figures 80 and 81 show photographs of the sectioned postfired nozzles.

It appears that as the wood filler content increases, the erosion rate also increases. However, the erosion sections as shown in Figure 80 are clean and uniform in appearance. A slight swelling in the aft exit cone occurred in all five of the TU-379 nozzles. All nozzles show some radial cracking. However, the cracking appeared to be the most severe for the phenolic microballoon nozzle (Figure 81).

The wood filler at the 10 percent level and the cork filler (3 percent) showed the least amount of material loss of any of the filled formulations.

It was decided to characterize the 10 percent wood filler/RI-4009 formulation because of the good mechanical properties, good erosion resistance, good char, and lower thermal conductivity.

Durez 10694 Phenolic Resin

The various filler variations outlined in Table XVI were mixed and cured into 12 by 12 by 0.5 in. (30.48 by 30.48 by 1.27 cm) panels. In addition, two graphite particle sizes were evaluated: GL-1012 325 micron (average) and 74 micron (maximum).

The test panels were torch tested on the flat side at 5,000° F (3,035° K) for 60 seconds. However, only three or four panels were tested by this method because it was observed during the initial part of the torch test that excessive spalling and cracking occurred. Therefore, in order to obtain a better comparison of the various fillers in the Durez 10694 resin system formulations, the test was repeated using a lower flame temperature of approximately 2,000° F (1,366° K). At this temperature, the test panels visibly appeared unaffected by the torch test, with the exception of the panels containing mixes incorporating the glass microballoon filler. Severe spalling and cracking were found to be a common condition among test panels produced from mixes containing glass microballoons, regardless of basic formulation variations of filler and graphite particle size.

Photographs of the panels produced from the Durez 10694 resin system formulation were not taken since the panels were unaffected by the torch test at the lower flame temperature. The additional test panels (except those containing the glass microballoons) were prepared for tensile and compressive tests.

RI-4009 Resin
10% Wood Filler

Station No.	Contour				Material Loss		Char Depth		Erosion Rate	
	Initial		Erosion		(in.)	(m x 10 ⁻⁴)	(in.)	(m x 10 ⁻²)	(mils/sec)	(m/sec x 10 ⁻⁵)
	(in.)	(m)	(in.)	(m x 10 ⁻²)						
22	0.92	0.0233	0.94	2.38	+	--	0.76	1.93	--	--
21	0.96	0.0243	0.99	2.51	+	--	0.78	1.98	--	--
20	0.99	0.0251	1.02	2.59	+	--	0.79	2.0	--	--
19	1.04	0.0264	1.06	2.69	+	--	0.79	2.0	--	--
18	1.08	0.0274	1.12	2.84	+	--	0.82	2.08	--	--
17	1.11	0.0281	1.14	2.89	+	--	0.84	2.13	--	--
16	1.14	0.0284	1.18	2.99	+	--	0.80	2.03	--	--
15	1.18	0.0299	1.18	2.99	0	--	0.80	2.03	--	--
14	1.21	0.0307	1.19	3.02	0.02	5.08	0.81	2.05	1.18	2.99
13	1.24	0.0314	1.19	3.02	0.05	12.70	0.81	2.05	2.95	7.49
12	1.24	0.0314	1.18	2.99	0.06	15.24	0.81	2.05	3.55	9.02
11	1.24	0.0314	1.18	2.99	0.06	15.24	0.82	2.08	3.55	9.02
10	1.23	0.0312	1.17	2.97	0.06	15.24	0.84	2.13	3.55	9.02
9	1.22	0.0309	1.17	2.97	0.05	12.70	0.83	2.10	2.95	7.49
8	1.22	0.0309	1.16	2.94	0.06	15.24	0.81	2.05	3.55	9.02
7	1.21	0.0307	1.15	2.92	0.06	15.24	0.80	2.03	3.55	9.02
6	1.20	0.0304	1.15	2.92	0.05	12.70	0.79	2.0	2.95	7.49
5	1.19	0.0300	1.14	2.89	0.05	12.70	0.77	1.95	2.95	7.49
4	1.18	0.0299	1.13	2.87	0.05	12.70	0.77	1.95	2.95	7.49
3	1.12	0.0284	1.09	2.76	0.03	7.6	0.72	1.82	1.77	4.50
2	1.04	0.0264	1.03	2.62	0.01	2.5	0.70	1.77	0.59	1.50
1	0.95	0.0241	0.96	2.43	+	--	0.63	1.69	--	--
0	0.87	0.0220	0.88	2.23	+	--	0.53	1.35	--	--

Legend

—————	Prefiring Surface
-----	Postfiring Surface
-----	Char Depth

Motor Parameters

Burn Time	16.90 sec
Web Pressure	339 psi (2.34 x 10 ⁶ N/m ²)
Throat Diameter	0.34 in. (0.86 cm)

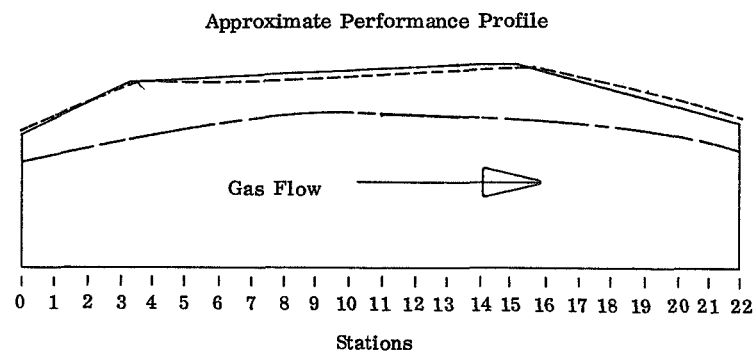


Figure 75. - TU-379 Nozzle Data, RI-4009 Resin, Motor No. 643

15% Wood Filler

Station No.	Contour				Material Loss		Char Depth		Erosion Rate	
	Initial (in.)	(m)	Erosion (in.)	(m x 10 ⁻²)	(in.)	(m x 10 ⁻⁴)	(in.)	(m x 10 ⁻²)	(mils/sec)	(m/sec x 10 ⁻⁵)
22	0.92	0.0233	0.96	2.43	+	--	0.75	1.90	N/A	N/A
21	0.96	0.0243	0.98	2.48	+	--	0.76	1.93		
20	0.99	0.0251	1.03	2.62	+	--	0.80	2.03		
19	1.04	0.0264	1.08	2.74	+	--	0.81	2.05		
18	1.08	0.0274	1.10	2.79	+	--	0.81	2.05		
17	1.11	0.0281	1.12	2.84	+	--	0.82	2.08		
16	1.14	0.0284	1.16	2.94	+	--	0.83	2.10		
15	1.18	0.0299	1.17	2.97	0.01	2.54	0.84	2.13		
14	1.21	0.0307	1.15	2.92	0.06	15.24	0.84	2.13		
13	1.24	0.0314	1.15	2.92	0.09	22.86	0.83	2.10		
12	1.24	0.0314	1.13	2.87	0.11	27.94	0.84	2.13		
11	1.24	0.0314	1.12	2.84	0.12	30.48	0.83	2.10		
10	1.23	0.0312	1.12	2.84	0.11	27.94	0.82	2.08		
9	1.22	0.0309	1.11	2.81	0.11	27.94	0.82	2.08		
8	1.22	0.0309	1.10	2.79	0.12	30.48	0.81	2.05		
7	1.21	0.0307	1.10	2.79	0.11	27.94	0.81	2.05		
6	1.20	0.0304	1.11	2.81	0.09	22.86	0.79	2.01		
5	1.19	0.0300	1.11	2.81	0.08	20.32	0.77	1.95		
4	1.18	0.0299	1.10	2.79	0.08	20.32	0.76	1.93		
3	1.12	0.0284	1.07	2.71	0.05	12.70	0.72	1.82		
2	1.04	0.0264	1.02	2.59	0.02	5.08	0.67	1.70		
1	0.95	0.0241	0.95	2.41	0	0	0.52	1.31		
0	0.87	0.0220	0.85	2.15	0.02	5.08	0.40	1.01		

Legend		Motor Parameters	
—————	Prefiring Surface	Burn Time	N/A
-----	Postfiring Surface	Web Pressure	N/A
-----	Char Depth	Throat Diameter	0.34 in. (0.86 cm)

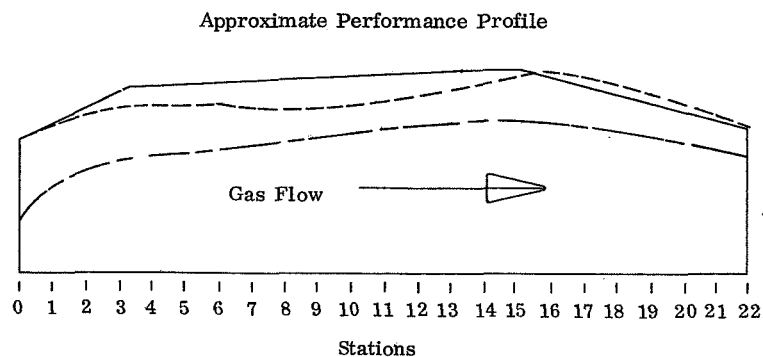


Figure 76. - TU-379 Nozzle Data, RI-4009 Resin, Motor No. 644

RI-4009 Resin
20% Wood Filler

Station No.	Contour				Material Loss		Char Depth		Erosion Rate	
	Initial (in.)	(m)	Erosion (in.)	(m x 10 ⁻²)	(in.)	(m x 10 ⁻⁴)	(in.)	(m x 10 ⁻²)	(mils/sec)	(m/sec x 10 ⁻⁵)
22	0.92	0.0233	0.94	2.38	+	--	0.69	1.75	--	--
21	0.96	0.0243	0.99	2.51	+	--	0.73	1.85	--	--
20	0.99	0.0251	1.02	2.59	+	--	0.73	1.85	--	--
19	1.04	0.0264	1.04	2.64	0	--	0.76	1.93	--	--
18	1.08	0.0274	1.09	2.76	+	--	0.81	2.05	--	--
17	1.11	0.0281	1.12	2.84	+	--	0.83	2.10	--	--
16	1.14	0.0284	1.16	2.94	+	--	0.84	2.13	--	--
15	1.18	0.0299	1.18	2.99	0	--	0.83	2.10	--	--
14	1.21	0.0307	1.17	2.97	0.05	12.70	0.82	2.08	2.42	6.15
13	1.24	0.0314	1.15	2.92	0.09	22.86	0.82	2.08	4.36	11.07
12	1.24	0.0314	1.15	2.92	0.09	22.86	0.83	2.10	4.36	11.07
11	1.24	0.0314	1.15	2.92	0.09	22.86	0.84	2.13	4.36	11.07
10	1.23	0.0312	1.13	2.87	0.10	25.4	0.85	2.15	4.85	12.32
9	1.22	0.0309	1.14	2.89	0.08	20.32	0.85	2.15	3.88	9.85
8	1.22	0.0309	1.14	2.89	0.08	20.32	0.85	2.15	3.88	9.85
7	1.21	0.0307	1.13	2.87	0.08	20.32	0.84	2.13	3.88	9.85
6	1.20	0.0304	1.13	2.87	0.07	17.78	0.83	2.10	3.39	8.61
5	1.19	0.0300	1.12	2.84	0.07	17.78	0.82	2.08	3.39	8.61
4	1.18	0.0299	1.11	2.81	0.07	17.78	0.80	2.03	3.39	8.61
3	1.12	0.0284	1.06	2.69	0.06	15.24	0.80	2.03	2.91	7.39
2	1.04	0.0264	0.99	2.51	0.05	12.70	0.73	1.85	2.42	6.15
1	0.95	0.0241	0.94	2.38	0.01	2.54	0.66	1.67	0.48	1.22
0	0.87	0.0220	0.89	2.25	+	--	0.58	1.47	--	--

Legend		Motor Parameters	
—————	Prefiring Surface	Burn Time	20.6 sec
-----	Postfiring Surface	Web Pressure	256 psi (1.77 x 10 ⁶ N/m ²)
-----	Char Depth	Throat Diameter	0.34 in. (0.86 cm)

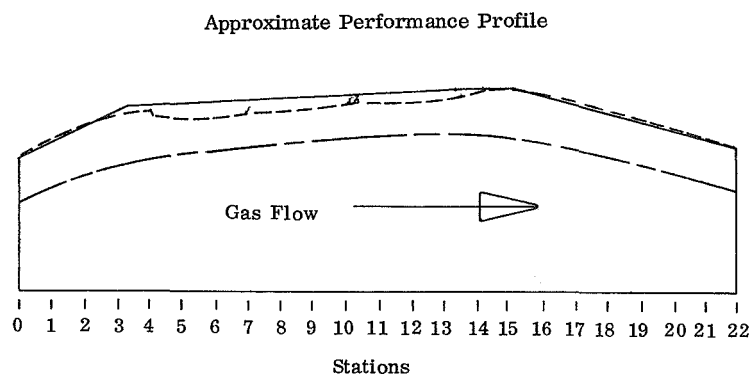


Figure 77. - TU-379 Nozzle Data, RI-4009 Resin, Motor No. 645

RI-4009 Resin

3% Cork Filler

Station No.	Contour				Material Loss		Char Depth		Erosion Rate	
	Initial		Erosion		(in.)	(m x 10 ⁻⁴)	(in.)	(m x 10 ⁻²)	(mils/sec)	(m/sec x 10 ⁻⁵)
22	0.92	0.0233	0.94	2.38	+	--	0.71	1.80	--	--
21	0.96	0.0243	0.99	2.51	+	--	0.71	1.80	--	--
20	0.99	0.0251	1.02	2.59	+	--	0.75	1.90	--	--
19	1.04	0.0264	1.06	2.69	+	--	0.78	1.98	--	--
18	1.08	0.0274	1.09	2.76	+	--	0.81	2.05	--	--
17	1.11	0.0281	1.13	2.87	+	--	0.81	2.05	--	--
16	1.14	0.0284	1.16	2.94	+	--	0.80	2.03	--	--
15	1.18	0.0299	1.19	3.02	+	--	0.79	2.01	--	--
14	1.21	0.0307	1.19	3.02	0.02	5.08	0.77	1.95	1.26	3.2
13	1.24	0.0314	1.18	2.99	0.06	15.24	0.74	1.87	3.79	9.63
12	1.24	0.0314	1.18	2.99	0.06	15.24	0.74	1.87	3.79	9.63
11	1.24	0.0314	1.18	2.99	0.06	15.24	0.74	1.87	3.79	9.63
10	1.23	0.0312	1.18	2.99	0.05	12.70	0.73	1.85	3.16	8.03
9	1.22	0.0309	1.18	2.99	0.04	10.16	0.72	1.82	2.53	6.43
8	1.22	0.0309	1.17	2.97	0.05	12.70	0.72	1.82	3.16	8.03
7	1.21	0.0307	1.17	2.97	0.04	10.16	0.71	1.80	2.53	6.43
6	1.20	0.0304	1.17	2.97	0.03	7.62	0.72	1.82	2.53	6.43
5	1.19	0.0300	1.16	2.94	0.03	7.62	0.72	1.82	1.89	4.8
4	1.18	0.0299	1.15	2.92	0.03	7.62	0.71	1.80	1.89	4.8
3	1.12	0.0284	1.12	2.84	0	0	0.69	1.73	--	--
2	1.04	0.0264	1.04	2.67	0	0	0.59	1.49	--	--
1	0.95	0.0241	0.97	2.46	+	--	0.51	1.29	--	--
0	0.87	0.0220	0.85	2.15	0.02	5.08	0.42	1.07	1.26	3.2

Legend

————— Prefiring Surface
 - - - - - Postfiring Surface
 - - - - - Char Depth

Motor Parameters

Burn Time 15.8 sec
 Web Pressure 360 psi ($2.48 \times 10^6 \text{ N/m}^2$)
 Throat Diameter 0.34 in. (0.86 cm)

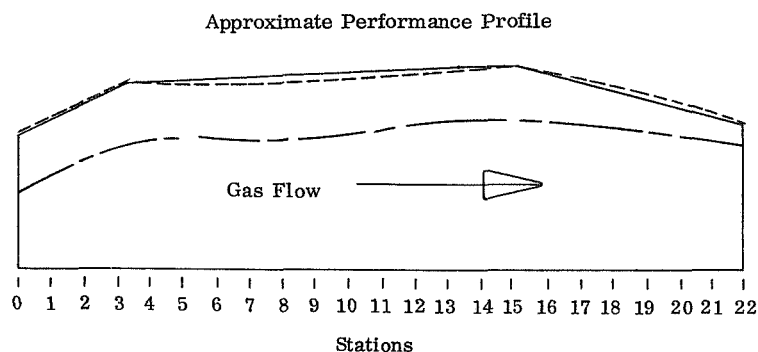


Figure 78. - TU-379 Nozzle Data, RI-4009 Resin, Motor No. 646

RI-4009 Resin

15% Phenolic Microballoon Filler

Station No.	Contour		Erosion		Material Loss		Char Depth		Erosion Rate	
	Initial (in.)	(m)	(in.)	(m x 10 ⁻²)	(in.)	(m x 10 ⁻⁴)	(in.)	(m x 10 ⁻²)	(mils/sec)	(m/sec x 10 ⁻⁵)
22	0.92	0.0233	0.98	2.48	+	--	0.71	1.80	--	--
21	0.96	0.0243	1.02	2.59	+	--	0.76	1.93	--	--
20	0.99	0.0251	1.04	2.67	+	--	0.78	1.98	--	--
19	1.04	0.0264	1.07	2.71	+	--	0.79	2.01	--	--
18	1.08	0.0274	1.11	2.81	+	--	0.80	2.03	--	--
17	1.11	0.0281	1.14	2.89	+	--	0.79	2.01	--	--
16	1.14	0.0284	1.15	2.92	+	--	0.80	2.03	--	--
15	1.18	0.0299	1.14	2.89	0.04	10.16	0.81	2.05	2.29	5.81
14	1.21	0.0307	1.14	2.89	0.07	17.78	0.84	2.13	4.02	10.21
13	1.24	0.0314	1.14	2.89	0.10	25.4	0.85	2.15	5.74	14.58
12	1.24	0.0314	1.13	2.87	0.11	27.94	0.82	2.08	6.32	16.05
11	1.24	0.0314	1.13	2.87	0.11	27.94	0.80	2.03	6.32	16.05
10	1.23	0.0312	1.10	2.79	0.13	33.02	0.78	1.98	7.47	18.97
9	1.22	0.0309	1.08	2.74	0.14	35.52	0.78	1.98	8.04	20.42
8	1.22	0.0309	1.10	2.79	0.12	30.48	0.78	1.98	6.89	17.50
7	1.21	0.0307	1.10	2.79	0.11	27.94	0.78	1.98	6.32	16.05
6	1.20	0.0304	1.12	2.84	0.08	20.32	0.75	1.90	4.59	11.66
5	1.19	0.0300	1.12	2.84	0.07	17.78	0.75	1.90	4.02	10.21
4	1.18	0.0299	1.10	2.79	0.08	20.32	0.71	1.80	4.59	11.66
3	1.12	0.0284	1.08	2.74	0.04	10.16	0.70	1.77	2.29	5.81
2	1.04	0.0264	1.02	2.59	0.02	5.08	0.63	1.69	1.14	2.89
1	0.95	0.0241	0.94	2.38	0.01	2.54	0.54	1.37	0.57	1.45
0	0.87	0.0220	0.89	2.25	+	--	0.47	1.19	--	--

Legend

————— Prefiring Surface
 - - - - - Postfiring Surface
 - - - - - Char Depth

Motor Parameters

Burn Time 17.4 sec
 Web Pressure 300 psi ($2.07 \times 10^6 \text{ N/m}^2$)
 Throat Diameter 0.34 in. (0.86 cm)

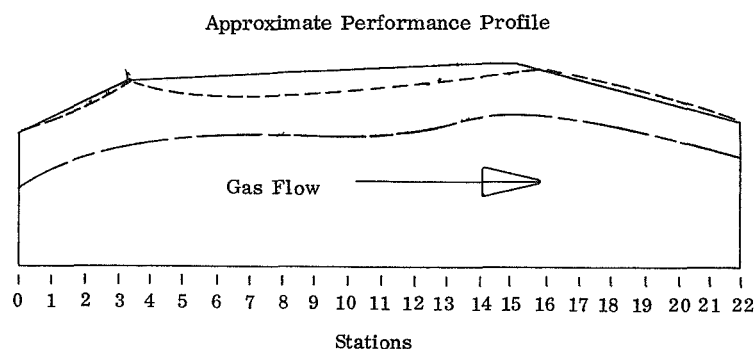
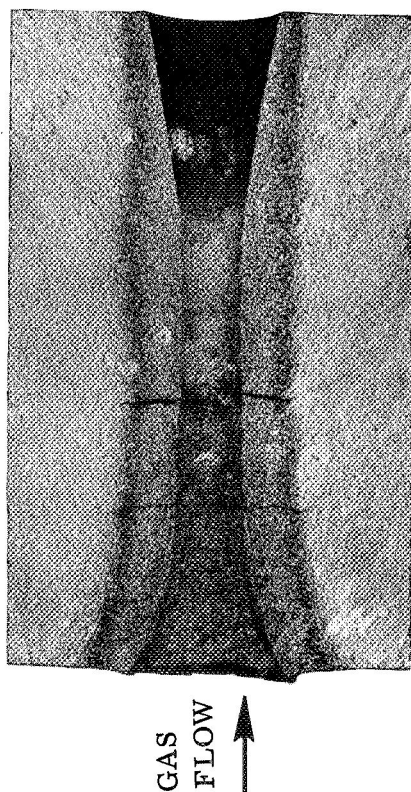
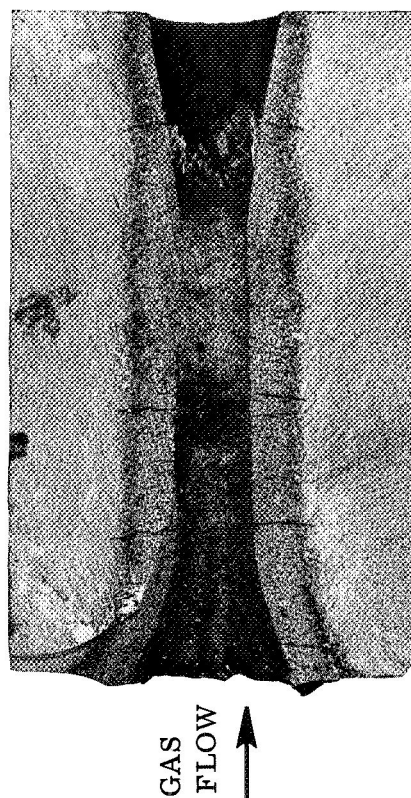


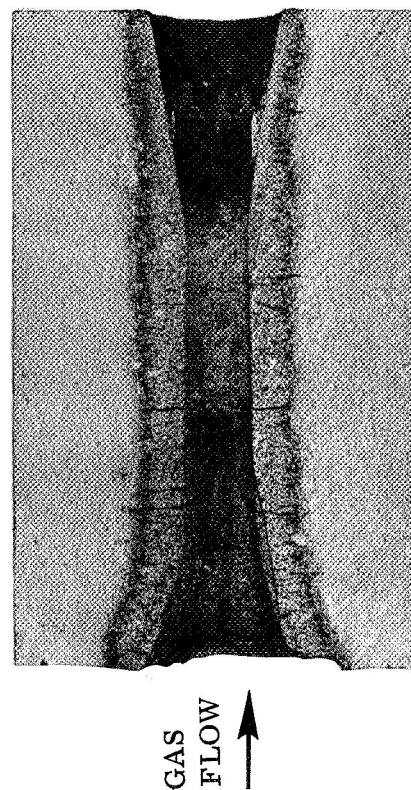
Figure 79. - TU-379 Nozzle Data, RI-4009 Resin, Motor No. 647



10% Wood Filler

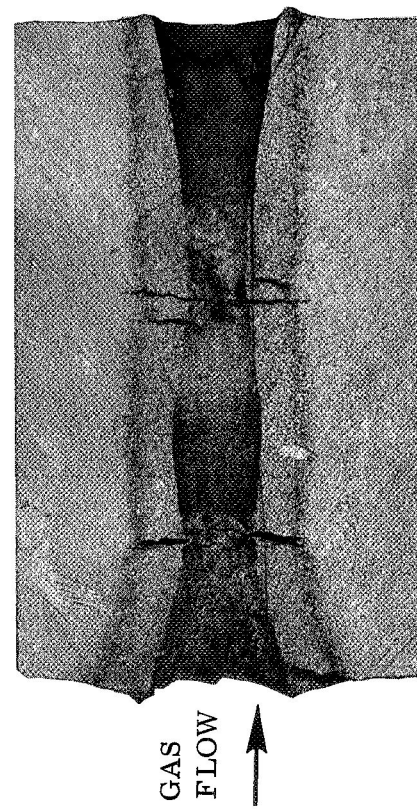


15% Wood Filler



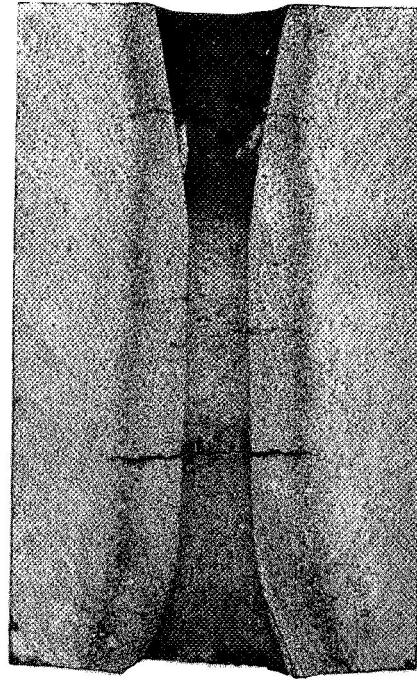
20% Wood Filler

Figure 80. - Wood Filler (10, 15, and 20 Percent)/RI-4009 Resin



GAS
FLOW
↑

15% Phenolic Microballoon



GAS
FLOW
↑

3% Cork

Figure 81. - Microballoon and Cork Fillers/RI-4009 Resin

The Hitco 158 elastomer was found to be incompatible with the Durez 10694 resin; as a result no test samples were fabricated.

Table XX presents data from formulations that utilized the small (74 micron, maximum) particle size graphite, whereas Tables XXI, XXII, and XXIII give data from formulations that utilized the larger (325 micron, average) particle size graphite.

As was the case with this resin system in the fiber reinforcement study, the filler property test data were disappointing. Tensile and compressive strengths were lower in value than materials formulated without filler. Increasing the filler content apparently does not increase physical properties as it does in the Monsanto RI-4009 resin/filler study. The graphite particle size appears to make little difference in structural strength on the various filler/Durez resin composites.

Due to the poor performance of these formulations containing the 25 percent resin, test panels were fabricated using a higher level of resin (40 percent) with only the wood filler. Those formulations containing the wood filler appeared to give higher tensile and compressive values than any of the other fillers. In addition, only the GL-1012 graphite was utilized, as material fabrication and processing is unconstrained with the use of the larger graphite particle.

Tensile and compression test specimens and two TU-379 nozzles were fabricated using 40 percent Durez 10694 resin with 5 percent wood flour and 55 percent graphite powder (GL-1012). One TU-379 nozzle was cured at 300° F (422° K) at 30 psi (2.07×10^5 N/m²). The second TU-379 nozzle was cured at 300° F (422° K) at 200 psi (1.38×10^6 N/m²).

The uncured material maintained good mixing and performing characteristics; i.e., all components appeared to be thoroughly mixed and wetted with resin, material was trowelable with a good "packing" consistency. In addition, the formulation did not flow when subjected to heat and pressure.

Static firing results showed the erosion rates on both TU-379 nozzles were good; the nozzles had firm char characteristics and displayed no radial cracking. Nozzle No. 673 cured at 200 psi (1.38×10^6 N/m²) demonstrated a slightly higher erosion rate, 6.09 mils/sec at station 13 (throat) (see Figure 82), than Nozzle No. 672 cured at 30 psi (2.07×10^5 N/m²), 5.41 mils/sec, at the same location, Figure 83. A photograph of the nozzle sections is shown in Figure 84. An increase in tensile and compressive strength was recorded when the resin content was increased from 25 to 40 percent. The maximum percent of filler that would allow the formulations to be trowelable or workable was 3.0 percent of wood flour with 25 percent resin and 5 percent wood flour with 40 percent resin. The test results are shown on page 138.

TABLE XX. - LCCM DEVELOPMENT, PHYSICAL PROPERTIES FILLER ADDITIVE STUDY, 74 MICRON PARTICLE SIZE

Resin	Percent Resin	Filler and Percent	Cure Conditions	Mix No.	Tensile Strength (psi)	Tensile Strength (N/m ² x 10 ⁶)	Density (g/cc)	Compressive Strength (psi)	Compressive Strength (N/m ² x 10 ⁶)	Hardness Shore D Virgith	Hardness Shore D Char
Durez 10694	25	Phenolic Microballoons 10		613	1,785 1,512 1,087 1,461	12.31 10.42 7.49 10.07	1.06 1.14 1.18 1.13	2,965 4,830 5,260 4,351	20.44 33.30 36.27 30.00	67	---
Durez 10694	25	15		614	1,242 1,432 1,495 1,389	8.56 9.87 10.31 9.58	1.08 0.85 1.03 0.98	3,710 1,505 3,330 2,848	25.58 10.38 22.96 19.64	67	---
Durez 10694	25	Wood Filler 10		615	1,358 1,894 2,022 1,758	9.36 13.06 13.94 12.12	1.39 1.42 1.27 1.36	5,725 6,000 3,580 5,101	39.47 41.37 24.68 35.17	71	---
Durez 10694	25	15	300°F at 200 psi (422°K at 1.38 x 10 ⁶ N/m ²)	616	2,480 2,705 1,795 2,326	17.10 18.65 12.38 16.04	1.22 1.30 1.41 1.31	3,000 4,100 6,720 4,606	20.68 28.27 46.33 31.76	75	---
Durez 10694	25	Cork 10		617	1,816 1,503 2,086 1,800	12.52 10.36 14.38 12.41	1.42 1.40 1.46 1.43	4,925 4,535 2,910 4,128	33.96 31.27 20.06 28.43	72	---
Durez 10694	25	15		618	2,038 1,343 1,442 1,607	14.05 9.26 9.94 11.08	1.42 1.41 1.40 1.41	4,920 4,760 4,520 4,733	33.92 32.32 31.16 32.63	73	---
Base											
Durez 10694	25	None		547	1,760 2,720 2,480 2,320	12.13 18.75 17.09 15.99	1.56 1.56 1.56 1.56	6,950 9,570 10,100 8,873	47.91 65.98 89.63 61.17	---	---

Note: Graphite particles less than 200 mesh.

*---Test data not available

TABLE XXI - LCCM DEVELOPMENT, PHYSICAL PROPERTIES FILLER ADDITIVE STUDY, PHENOLIC MICROBALLOON FILLER

Resin	Percent Resin	Filler and Percent	Cure Conditions	Mix No.	Tensile Strength		Density (g/cc)	Compressive Strength		Hardness Shore D	
					(psi)	(N/m ² x 10 ⁶)		(psi)	(N/m ² x 10 ⁶)	Virgin	Char
Durez 10694	25	Phenolic Microballoons 3	300°F at 200 psi (422°F at 1.38 x 10 ⁶ N/m ²)	594	1,375 1,985 1,795 1,718	9.48 13.69 12.38 11.8	1.42 1.42 1.40 1.41	6,900 6,880 6,820 6,866	47.57 47.44 47.02 47.34	75	52
Durez 10694	25	10		596	1,495 1,315 1,515 1,442	10.31 9.07 10.45 9.94	1.29 1.19 1.32 1.25	6,480 5,580 6,025 6,028	44.68 38.47 41.54 41.56	75	60
Durez 10694	25	15		600	1,182 1,232 1,077 1,163	8.15 8.49 7.43 8.02	1.02 1.17 1.10	4,760 5,240 5,000	32.82 36.13 34.47	70	40
Base											
Durez 10694	25	None		548	1,770 1,945 1,815 1,843	12.20 13.45 12.51 12.70	1.47 1.45 1.47 1.46	7,175 7,230	49.46 49.84	---	---

Note: Graphite particles are GL-1012 graphite.

*--Test data not available

TABLE XXII. - LCCM DEVELOPMENT, PHYSICAL PROPERTIES FILLER ADDITIVE STUDY, CORK FILLER

Resin	Percent Resin	Filler and Percent	Cure Conditions	Mix No.	Tensile Strength		Density (g/cc)	Compressive Strength		Hardness Shore D	
					(psi)	(N/m ² x 10 ⁶)		(psi)	(N/m ² x 10 ⁶)	Virgin	Char
Durez 10694	25	Cork 3	300°F at 200 psi (422°K at 1.88 x 10 ⁶ x N/m ²)	593	1,445 1,625 1,655 1,575	9.96 11.20 11.41 10.86	1.43 1.43 1.38 1.41	5,570 5,425 5,380 5,458	38.40 37.40 37.09 37.63	66	55
Durez 10694	25	10		599	1,545 1,513 1,440 1,500	10.65 10.43 9.93 10.34	1.28 1.31 1.07 1.22	3,590 4,225 3,365 3,726	24.75 29.13 23.20 25.69	65	30
Durez 10694	25	15		603	1,883 1,708 1,232 1,607	12.98 11.78 8.49 11.08	1.41 1.20 1.38 1.33	5,975 5,570 6,800 6,115	41.20 38.40 46.88 42.16	71	25
Base											
Durez 10694	25	None		548	{	1,770	12.20	1.47	7,175	49.46	---
						1,945	13.45	1.45	7,230	49.84	
						1,815 1,843	12.51 12.70	1.47 1.46	6,120 6,842	42.19 47.17	

Note: Graphite particles are GL-1012 graphite.

*---Test data not available

TABLE XXIII. - LCCM DEVELOPMENT, PHYSICAL PROPERTIES FILLER ADDITIVE STUDY, WOOD FILLER

Resin	Percent Resin	Filler and Percent	Cure Conditions	Mix No.	Tensile Strength		Density	Compressive Strength		Hardness Shore D	
					(psi)	(N/m ² x 10 ⁶)	(g/cc)	(psi)	(N/m ² x 10 ⁶)	Virgin	Char
Durez 10694	25	Wood Filler 3		595	1,445 1,625 1,655 1,575	10.0 11.2 11.4 10.9	1.56 1.55 1.53 1.55	8,720 8,680 6,925 8,108	60.2 59.9 47.8 55.9	75	50
Durez 10694	25	10		598	2,290 2,510 2,420 2,406	15.8 17.3 16.7 16.6	1.45 1.23 1.43 1.37	6,480 2,500 6,720 5,233	44.7 17.3 46.4 36.1	73	50
Durez 10694	25	15		602	1,683 827 1,477 1,329	11.6 5.7 10.2 9.2	1.38 1.22 1.36 1.32	5,540 4,660 2,910 4,370	38.3 32.2 20.1 30.2	65	35
<u>Base</u>											
Durez 10694	25	None			1,770	12.20	1.47	7,175	49.46	-- *	--
				548	1,945	13.45	1.45	7,230	49.84		
					1,815 1,843	12.51 12.70	1.47 1.46	6,120 6,842	42.19 47.17		

Note: Graphite particles are GL-1012 graphite.

*-- Test data not available

Durez 10694 Resin
 5% Wood Flour
 200 psi (6.9×10^6 N/m²)

Station No.	Contour		Erosion		Material Loss		Char Depth		Erosion Rate	
	Initial (in.)	(m)	(in.)	(m x 10 ⁻²)	(in.)	(m x 10 ⁻⁴)	(in.)	(m x 10 ⁻²)	(mils/sec)	(m/sec x 10 ⁻⁵)
22	0.92	0.0233	0.95	2.41	+	--	0.72	1.82	--	--
21	0.96	0.0243	0.98	2.48	+	--	--	--	--	--
20	0.99	0.0251	1.02	2.59	+	--	0.76	1.93	--	--
19	1.04	0.0264	1.06	2.69	+	--	--	--	--	--
18	1.08	0.0274	1.09	2.76	+	--	--	--	--	--
17	1.11	0.0281	1.12	2.84	+	--	--	--	--	--
16	1.14	0.0284	1.15	2.92	+	--	--	--	--	--
15	1.18	0.0299	1.18	2.99	0	--	0.86	2.18	0	--
14	1.21	0.0307	1.14	2.89	0.07	17.78	--	--	4.26	10.82
13	1.24	0.0314	1.14	2.89	0.10	25.4	--	--	6.09	15.47
12	1.24	0.0314	1.16	2.94	0.08	20.32	--	--	4.87	12.41
11	1.24	0.0314	1.17	2.97	0.07	17.78	--	--	4.26	10.82
10	1.23	0.0312	1.17	2.97	0.06	15.24	0.90	2.28	3.65	9.27
9	1.22	0.0309	1.16	2.94	0.07	17.78	--	--	4.26	10.82
8	1.22	0.0309	1.16	2.94	0.07	17.78	--	--	4.26	10.82
7	1.21	0.0307	1.17	2.97	0.04	10.16	--	--	2.43	6.17
6	1.20	0.0304	1.16	2.94	0.04	10.16	--	--	2.43	6.17
5	1.19	0.0300	1.18	2.99	0.01	2.54	0.83	2.10	0.61	1.55
4	1.18	0.0299	1.15	2.92	0.03	7.62	--	--	1.82	4.62
3	1.12	0.0284	1.09	2.76	0.03	7.62	--	--	1.82	4.62
2	1.04	0.0264	1.01	2.56	0.03	7.62	--	--	1.82	4.62
1	0.95	0.0241	0.92	2.33	0.03	7.62	--	--	1.82	4.62
0	0.87	0.0220	0.81	2.06	0.06	15.24	0.42	1.05	3.65	9.27

Legend		Motor Parameters	
—————	Prefiring Surface	Burn Time	16.40 sec
-----	Postfiring Surface	Web Pressure	
-----	Char Depth	Throat Diameter	0.34 in. (0.86 cm)

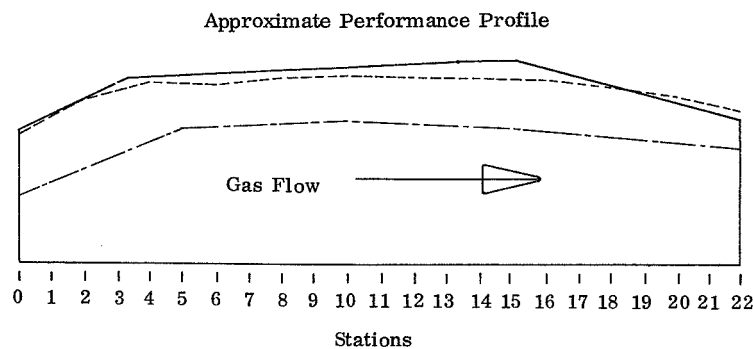


Figure 82. - TU-379 Nozzle Data, Durez 10694 Resin, Motor No. 673

Durez 10694 Resin
 5% Wood Flour
 30 psi (2.07×10^5 N/m²)

Station No.	Contour				Material Loss		Char Depth		Erosion Rate	
	Initial		Erosion		(in.)	(m x 10 ⁻⁴)	(in.)	(m x 10 ⁻²)	(mils/sec)	(m/sec x 10 ⁻⁵)
22	0.92	0.0233	0.92	2.33	0	0	0.70	1.77	--	--
21	0.96	0.0243	0.96	2.43	0	0	--	--	--	--
20	0.99	0.0251	1.00	2.54	+	--	0.74	1.87	--	--
19	1.04	0.0264	1.03	2.62	0.01	2.54	--	--	0.60	3.04
18	1.08	0.0274	1.06	2.69	0.02	5.08	--	--	1.20	6.09
17	1.11	0.0281	1.10	2.79	0.01	2.54	--	--	0.60	3.04
16	1.14	0.0284	1.13	2.87	0.01	2.54	--	--	0.60	3.04
15	1.18	0.0299	1.16	2.94	0.02	5.08	0.82	2.08	1.20	6.09
14	1.21	0.0307	1.13	2.87	0.08	20.32	--	--	4.80	12.19
13	1.24	0.0314	1.15	2.92	0.09	22.86	--	--	5.41	13.74
12	1.24	0.0314	1.17	2.97	0.07	17.78	--	--	4.21	10.69
11	1.24	0.0314	1.18	2.99	0.06	15.24	--	--	3.61	9.17
10	1.23	0.0312	1.15	2.92	0.08	20.32	0.84	2.13	4.81	12.22
9	1.22	0.0309	1.16	2.94	0.06	15.24	--	--	3.61	9.17
8	1.22	0.0309	1.16	2.94	0.06	15.24	--	--	3.61	9.17
7	1.21	0.0307	1.16	2.94	0.05	12.70	--	--	3.01	7.64
6	1.20	0.0304	1.15	2.92	0.05	12.70	--	--	3.01	7.64
5	1.19	0.0300	1.14	2.89	0.05	12.70	0.80	2.03	3.01	7.64
4	1.18	0.0299	1.12	2.84	0.06	15.24	--	--	3.61	9.17
3	1.12	0.0284	1.09	2.76	0.06	15.24	--	--	3.61	9.17
2	1.04	0.0264	0.99	2.51	0.05	12.70	--	--	3.01	7.64
1	0.95	0.0241	0.92	2.33	0.03	7.6	--	--	1.80	4.57
0	0.87	0.0220	0.82	2.08	0.05	12.70	0.52	1.31	3.01	7.64

Legend		Motor Parameters	
—————	Prefiring Surface	Burn Time	16.60 sec
-----	Postfiring Surface	Web Pressure	
-----	Char Depth	Throat Diameter	0.34 in. (0.86 cm)

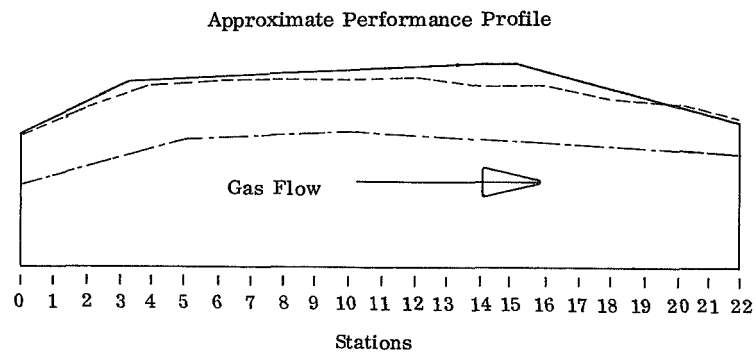
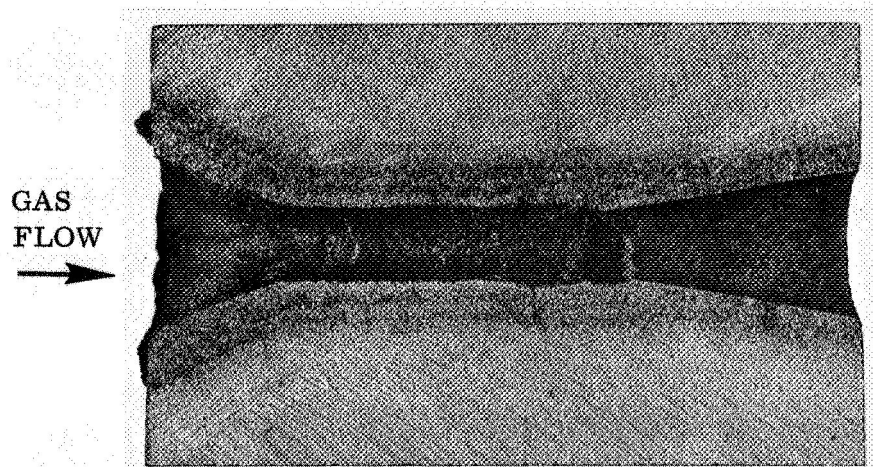
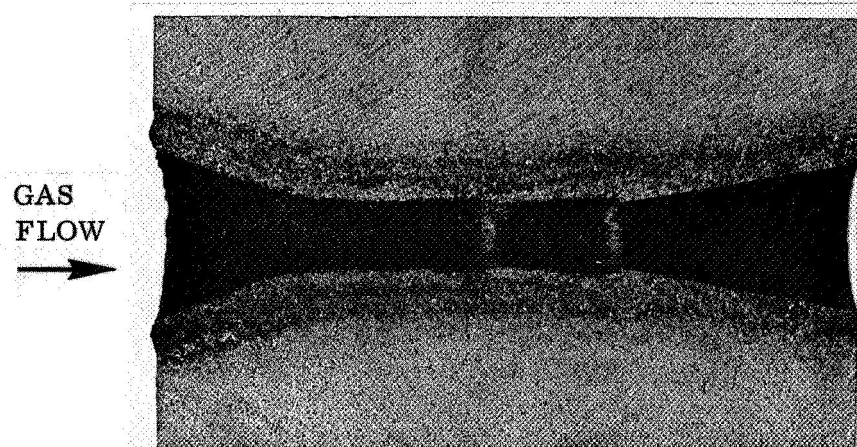


Figure 83. - TU-379 Nozzle Data, Durez 10694 Resin, Motor No. 672



5% WOOD FLOUR, 200 psi ($1.38 \text{ N/M}^2 \times 10^6$) 40% W/DUREZ
 10694 RESIN SYSTEM
 55% GL-1012 GRAPHITE POWDER



5% WOOD FLOUR, 30 psi ($0.207 \text{ N/M}^2 \times 10^6$) CURE, W/DUREZ
 55% GL-1012 GRAPHITE POWDER
 10694 RESIN SYSTEM

Figure 84. - TU-379 Nozzle Sections

Mix No.	Filler and Percent	Cure Conditions	Tensile Strength		Density (g/cc)	Compressive Strength	
			(psi)	(N/m ² x 10 ⁶)		(psi)	(N/m ² x 10 ⁶)
660	Wood Flour (5%)	300° F (422° K)	1,660	11.45	1.43	8,900	61.41
	Durez 10694 (40%)	30 psi (0.207 x 10 ⁶ N/m ²)	1,685	11.62	1.43	8,620	59.48
			<u>1,605</u>	<u>11.07</u>	<u>1.43</u>	<u>8,520</u>	<u>58.74</u>
			1,648	11.37	1.43	8,670	59.82
596	Wood Flour (3%)	300° F (422° K)	1,445	9.97	1.56	8,720	60.17
	Durez 10694 (25%)	200 psi (1.38 x 10 ⁶ N/m ²)	1,625	11.21	1.55	8,680	59.89
			<u>1,655</u>	<u>11.42</u>	<u>1.53</u>	<u>6,925</u>	<u>47.78</u>
			1,575	10.87	1.55	8,108	55.94

Special TU-379 Nozzle Test

A special TU-379 nozzle was fabricated using 10 percent wood flour, 10 percent chopped 0.25 in. (0.64 cm) carbon fiber, 40 percent RI-4009 resin, and 40 percent GL-1012 graphite powder. The mixture was cured at 1,000 psi (6.9 x 10⁶ N/m²) and 300° F (422° K). This nozzle was tested to determine the combined effects of the wood flour filler and carbon fiber reinforcement on the LCCM formulation. Each of the filler and reinforcement systems tested separately have merit. The wood flour tends to improve the physical properties (tensile and compressive strengths) and reduce thermal conductivity and density, whereas the carbon fiber tends to eliminate radial cracking and tested components retain a solid char.

Erosion performance results are shown in Figure 85. Figure 86 shows the sectioned TU-379 nozzle (Mix 665).

The nozzle maintained a hard char with some radial cracking. The erosion profile was not uniform in appearance as compared to nozzles tested separately with wood flour or carbon fibers. The Mix 665 test material was better than wood flour in the throat area but was not as good as carbon fiber reinforcement. However in the inlet area, the mixture was better than both wood flour or carbon fiber tested separately.

	Station 13				
	Throat Erosion Rate		Maximum Inlet Erosion		
	<u>(mils/sec)</u>	<u>(m x 10⁻⁵/sec)</u>	<u>(mils/sec)</u>	<u>(m x 10⁻⁵/sec)</u>	<u>Station</u>
10 percent carbon, 10 percent wood	2.25	5.70	3.38	8.55	7
10 percent carbon	1.24	3.14	4.35	11.00	10
10 percent wood	2.95	7.50	3.55	9.05	7-8, 10-12

RI-4009 Resin
10% Wood Flour
10% Chopped Carbon Fiber

Station No.	Contour				Material Loss		Char Depth		Erosion Rate	
	Initial		Erosion		(in.)	(m x 10 ⁻⁴)	(in.)	(m x 10 ⁻²)	(mils/sec)	(m/sec x 10 ⁻⁵)
22	0.92	0.0233	0.94	2.38	+	--	0.57	1.44	--	--
21	0.96	0.0243	1.00	2.54	+	--	--	--	--	--
20	0.99	0.0251	1.03	2.62	+	--	0.65	1.67	--	--
19	1.04	0.0264	1.07	2.71	+	--	--	--	--	--
18	1.08	0.0274	1.09	2.76	+	--	--	--	--	--
17	1.11	0.0281	1.12	2.84	+	--	--	--	--	--
16	1.14	0.0284	1.16	2.94	+	--	--	--	--	--
15	1.18	0.0299	1.19	3.02	+	--	0.69	1.75	--	--
14	1.21	0.0307	1.20	3.04	0.01	2.54	--	--	0.56	1.42
13	1.24	0.0314	1.20	3.04	0.04	10.16	--	--	2.25	5.71
12	1.24	0.0314	1.19	3.02	0.05	12.70	--	--	2.82	7.14
11	1.24	0.0314	1.20	3.04	0.04	10.16	--	--	2.25	5.71
10	1.23	0.0312	1.18	2.99	0.05	12.70	0.71	1.80	2.82	7.14
9	1.22	0.0309	1.17	2.97	0.05	12.70	--	--	2.82	7.14
8	1.22	0.0309	1.17	2.97	0.05	12.70	--	--	2.82	7.14
7	1.21	0.0307	1.15	2.92	0.06	15.24	--	--	3.38	8.58
6	1.20	0.0304	1.15	2.92	0.05	12.70	--	--	2.82	7.14
5	1.19	0.0300	1.14	2.89	0.05	12.70	0.76	1.92	2.82	7.14
4	1.18	0.0299	1.12	2.84	0.06	15.24	--	--	3.38	8.58
3	1.12	0.0284	1.10	2.78	0.02	5.08	--	--	1.13	2.87
2	1.04	0.0264	1.06	2.69	+	--	--	--	--	--
1	0.95	0.0241	0.98	2.48	+	--	--	--	--	--
0	0.87	0.0220	0.98	2.48	+	--	0.49	1.25	--	--

Legend

———— Prefiring Surface
----- Postfiring Surface
----- Char Depth

Motor Parameters

Burn Time 17.7 sec
Web Pressure 250 psi ($1.73 \times 10^6 \text{ N/m}^2$)
Throat Diameter 0.34 in. (0.86 cm)

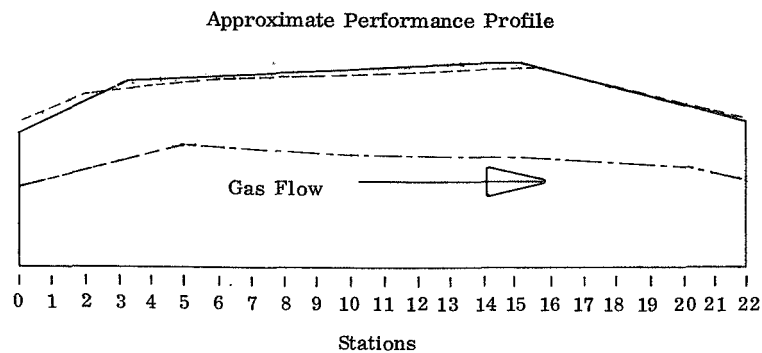


Figure 85. - TU-379 Nozzle Data, RI-4009 Resin, Motor No. 665

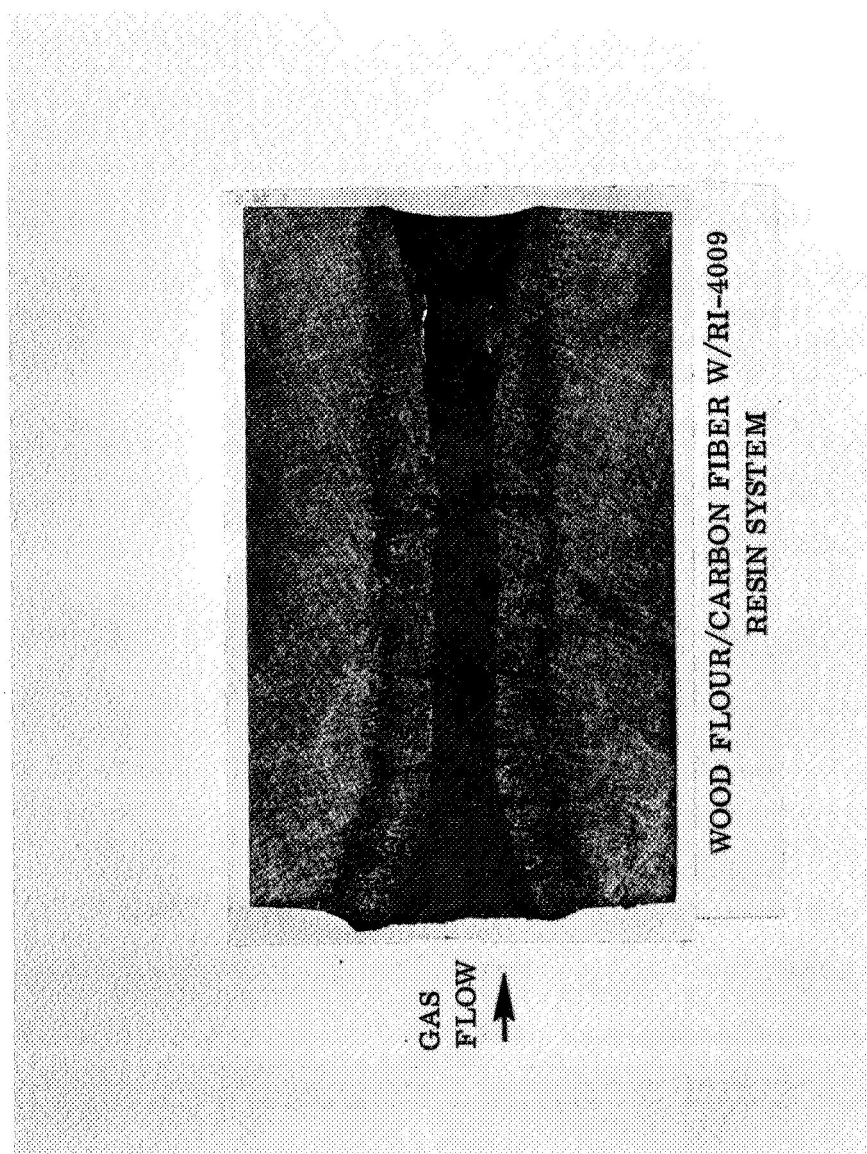


Figure 86. - TU-379 Nozzle Section (Special Test)

FORMULATION OPTIMIZATION MATERIAL SELECTIONS

Based on the results of the formulation optimization studies, four materials were selected for additional erosion testing and final characterization. These materials are indicated below and have been assigned an internal Thiokol UF designation as shown.

<u>UF No.</u>	<u>Material</u>	<u>Percent by Weight</u>
UF-1161	GL-1012 graphite	60.0
	RI-4009 Monsanto resin	40.0
UF-1162	GL-1012 graphite	50.0
	RI-4009 Monsanto resin	40.0
	Wood flour	10.0
UF-1163	GL-1012 graphite	55.0
	Wood flour	5.0
	Durez 10694 resin	40.0
UF-1164	GL-1012 graphite	50.0
	Chopped carbon fiber (0.25 in., 0.64 cm)	10.0
	Durez 10694 resin	40.0

TU-622 MATERIAL EROSION AND CHAR TESTING

Four graphite powder phenolic materials (UF-1161, UF-1162, UF-1163, and UF-1164) were rocket nozzle tested as ablative liners on the Thiokol TU-622 motor, (throat diameter 1.74 in., 4.42 cm) to compare to previously tested T-2610, T-2626, and T-4120 materials.

The sectioned T-2610 and T-4120 TU-622 nozzle billets are shown in Figures 87 and 88 and reported in "Development of Low Cost Ablative Nozzles," NASA-CR-72641. The UF-1161, UF-1162, UF-1163, and UF-1164 graphite powder phenolic sectioned billets are shown in Figure 89. The UF compounds exhibited more uniform density and fewer axial cracks, but showed more radial cracks, especially the UF-1161 and UF-1162.

The four TU-622 motor pressure traces are shown in Figures 90 thru 93. The average web pressure varied between 372 and 480 psia, with the web time varying between 31.5 and 36.6 seconds.

GAS FLOW →

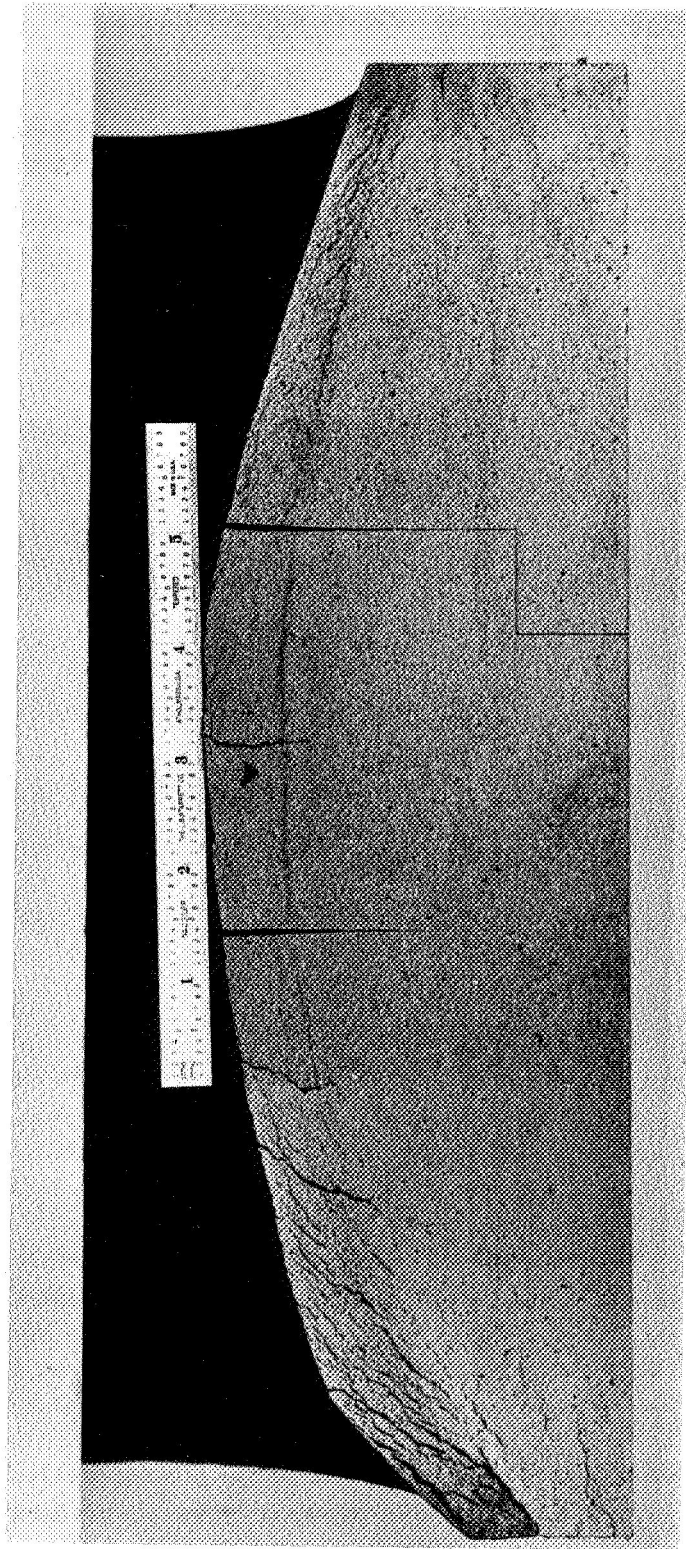


Figure 87. -Sectioned TU-622 Test Nozzle, LCCM-2610 (Graphite Phenolic)

GAS FLOW →

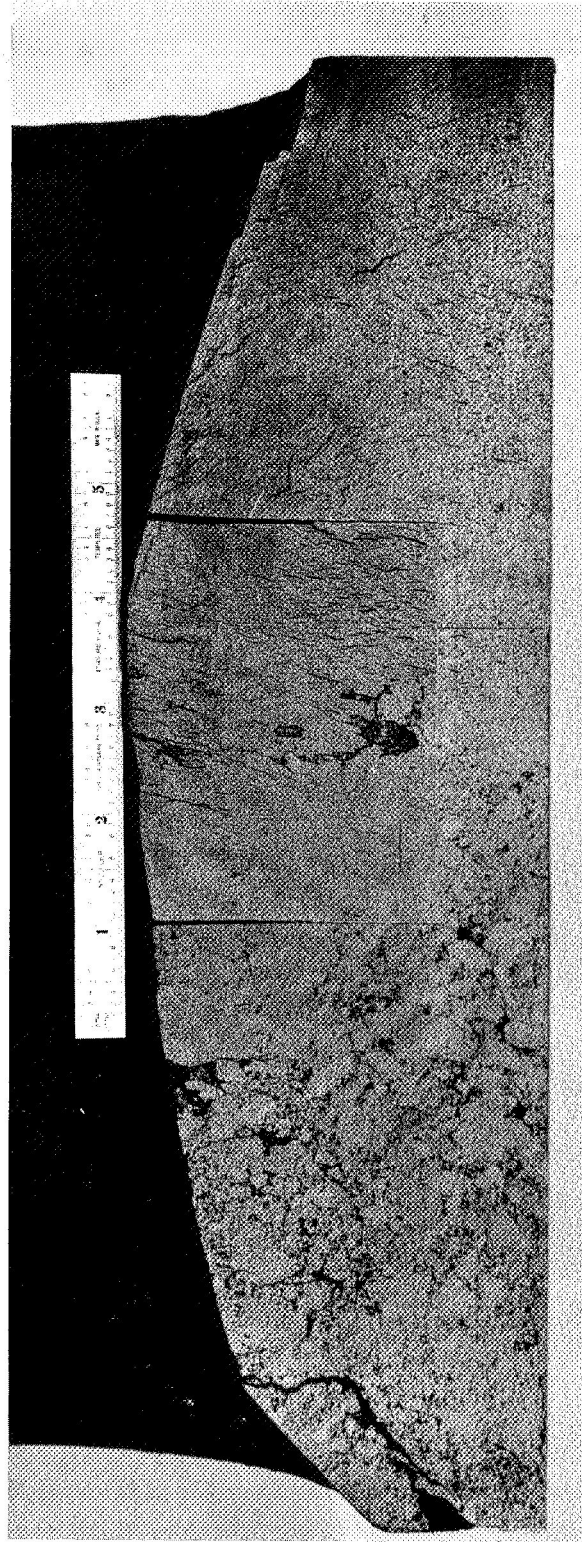
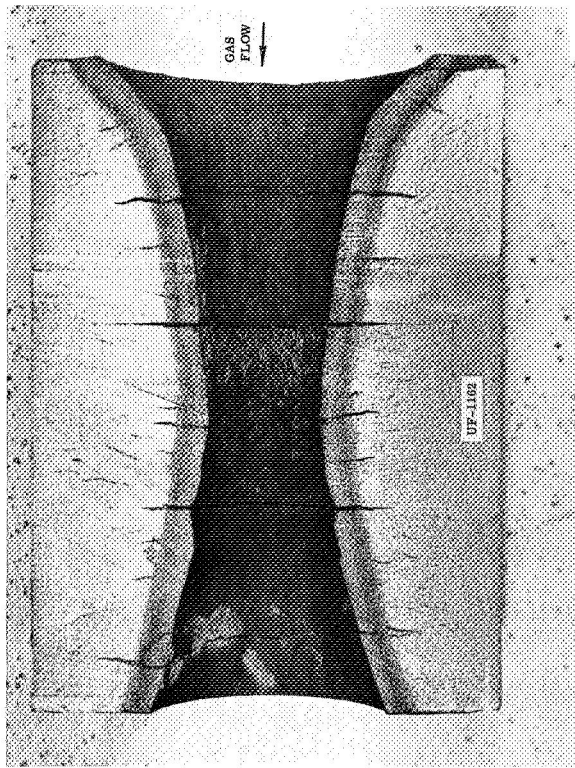
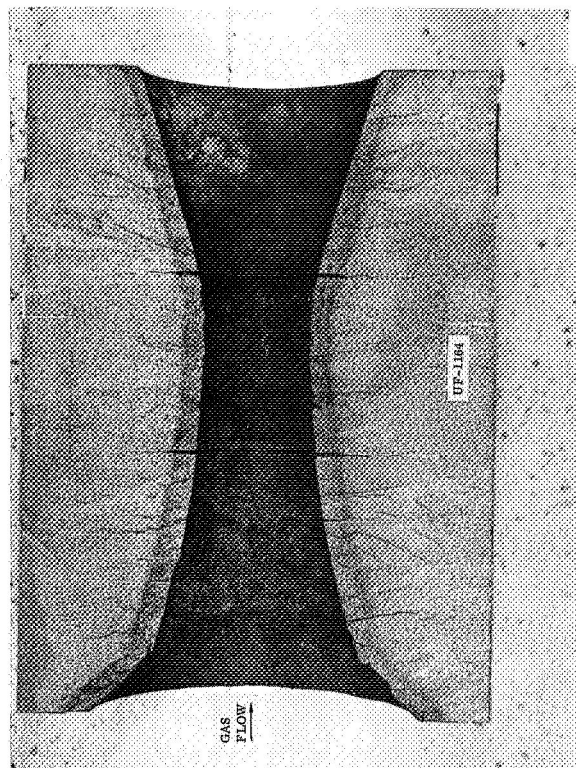


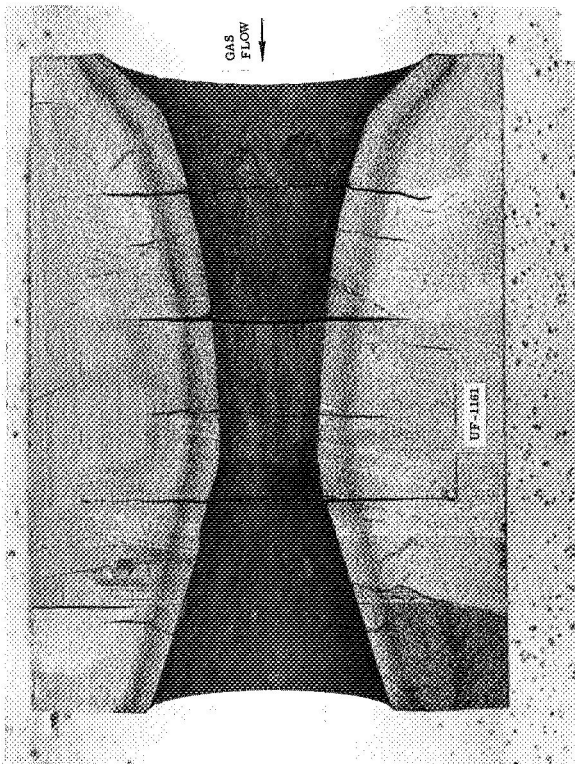
Figure 88. -Sectioned TU -622 Test Nozzle, LCCM-4120 (Graphite Phenolic)



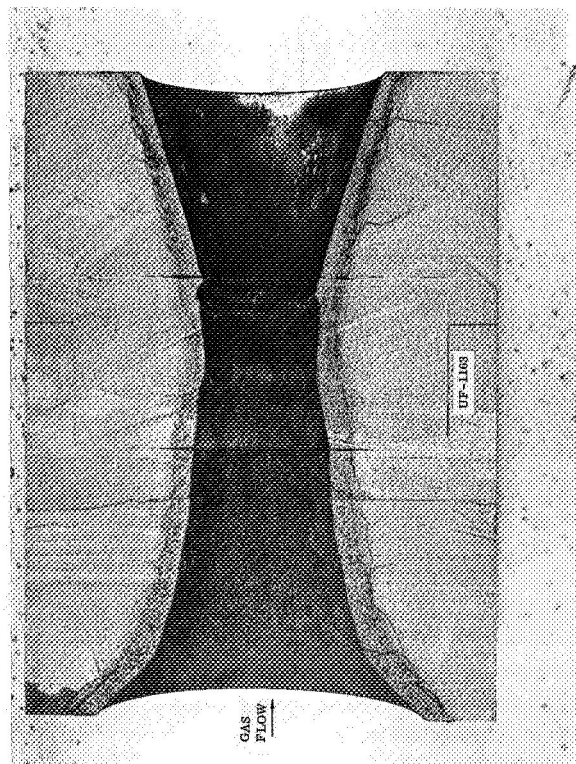
UF-1162



UF-1164



UF-1161



UF-1163

Figure 89. -Sectioned TU -622 Test Nozzle, UF-1161, UF-1162, UF-1163, and UF-1164

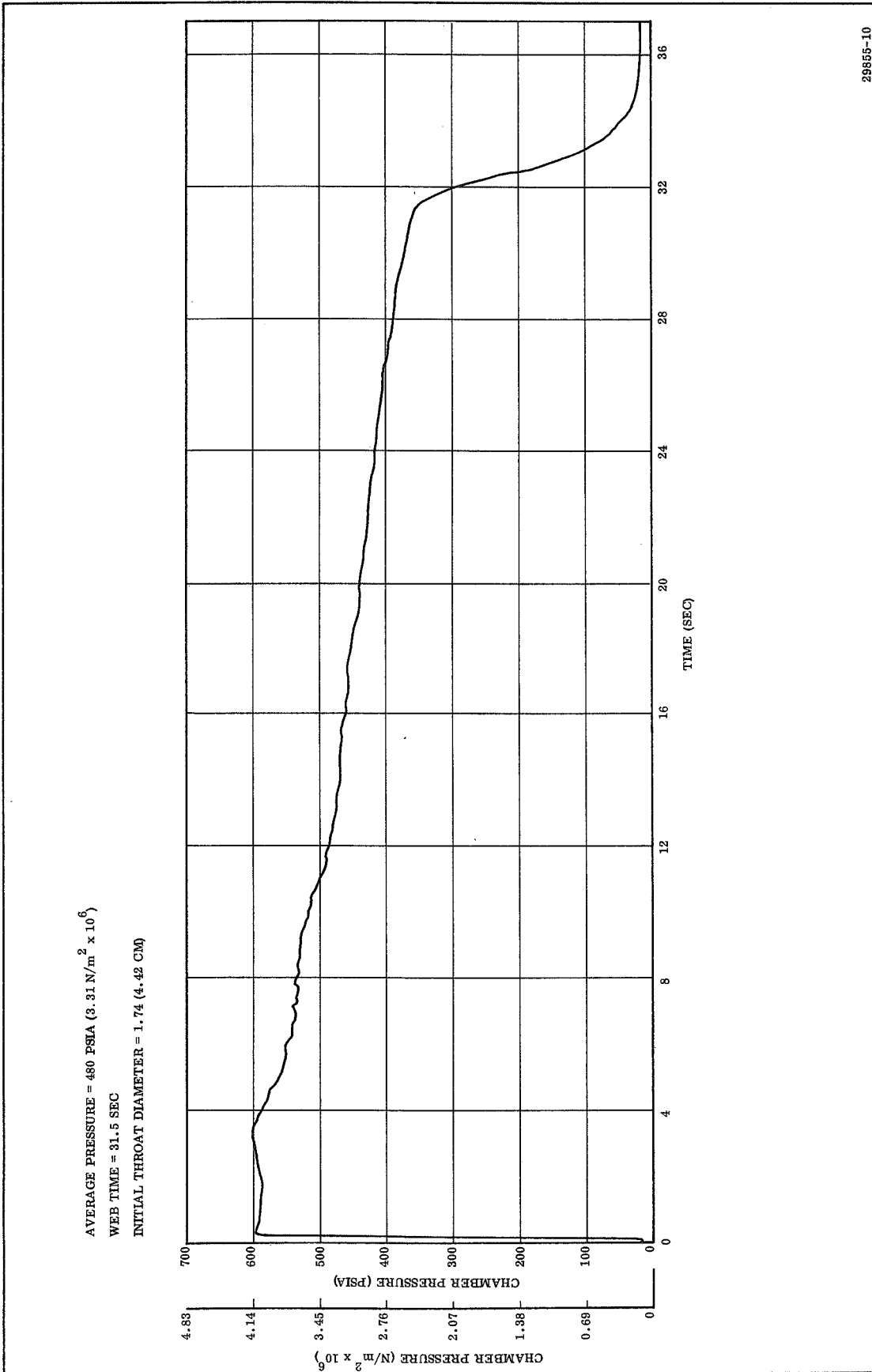
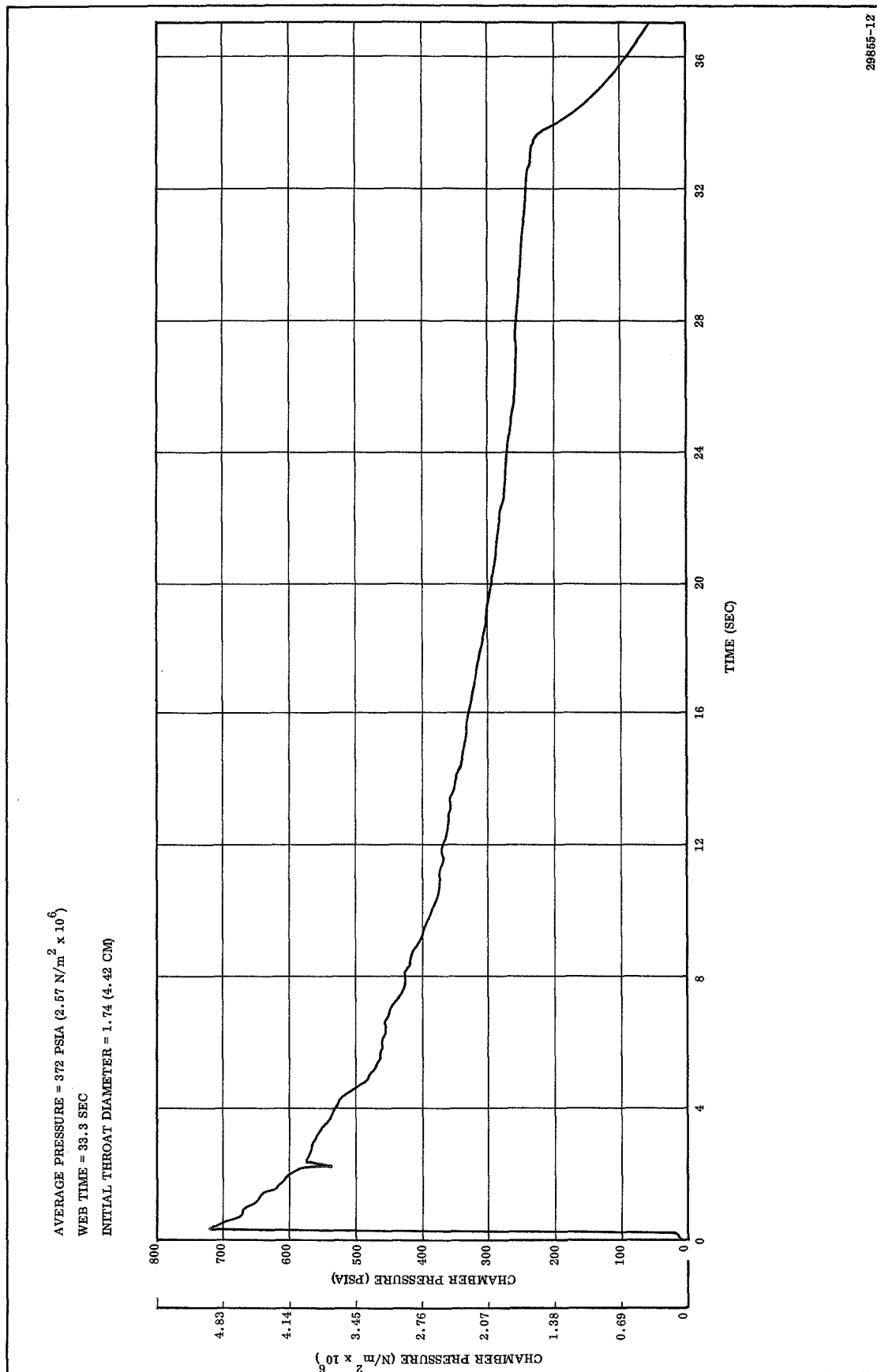


Figure 90.- UF-1161 Pressure Trace

29855-10



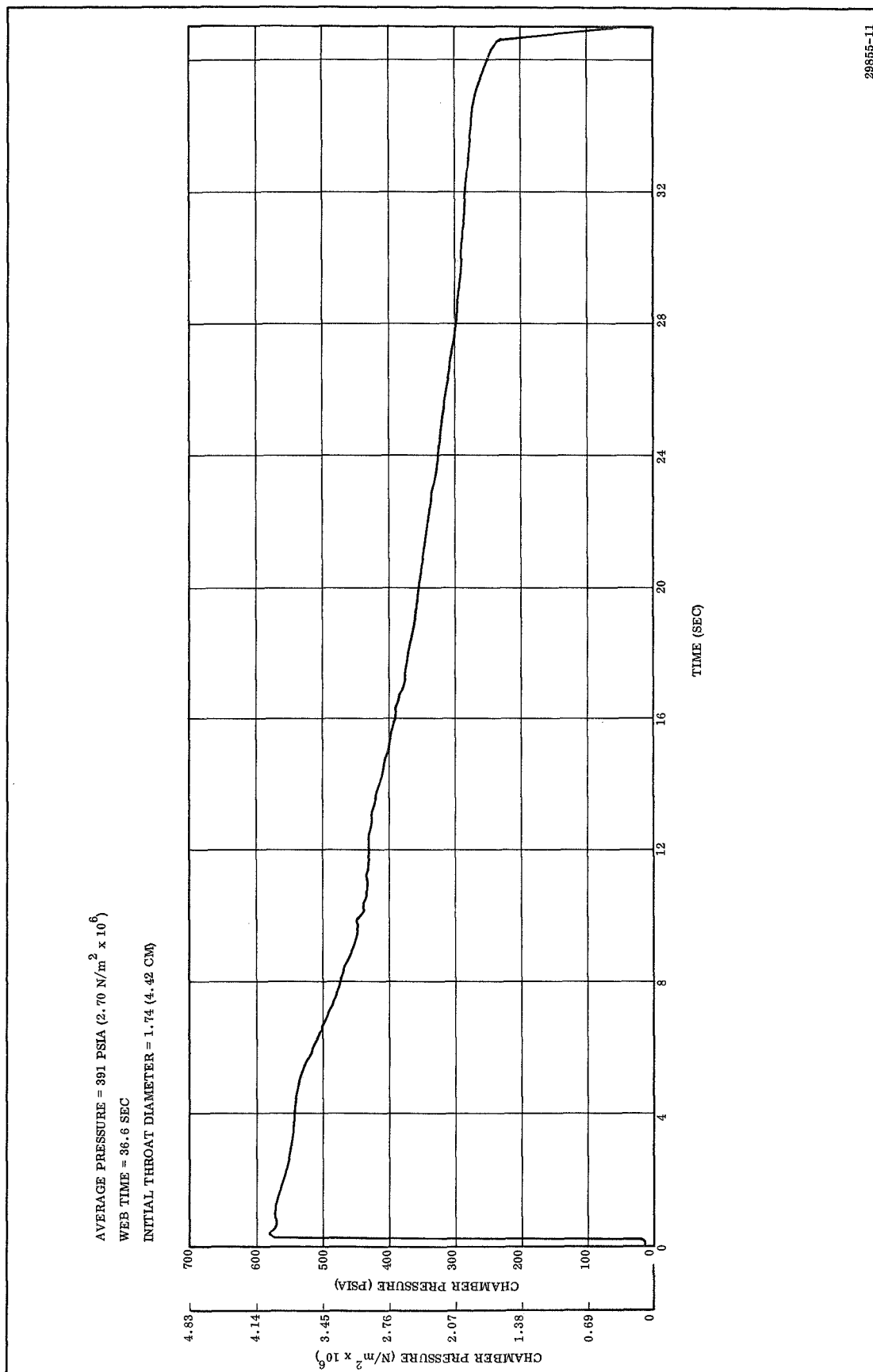


Figure 92. - UF-1163 Pressure Trace

29855-11

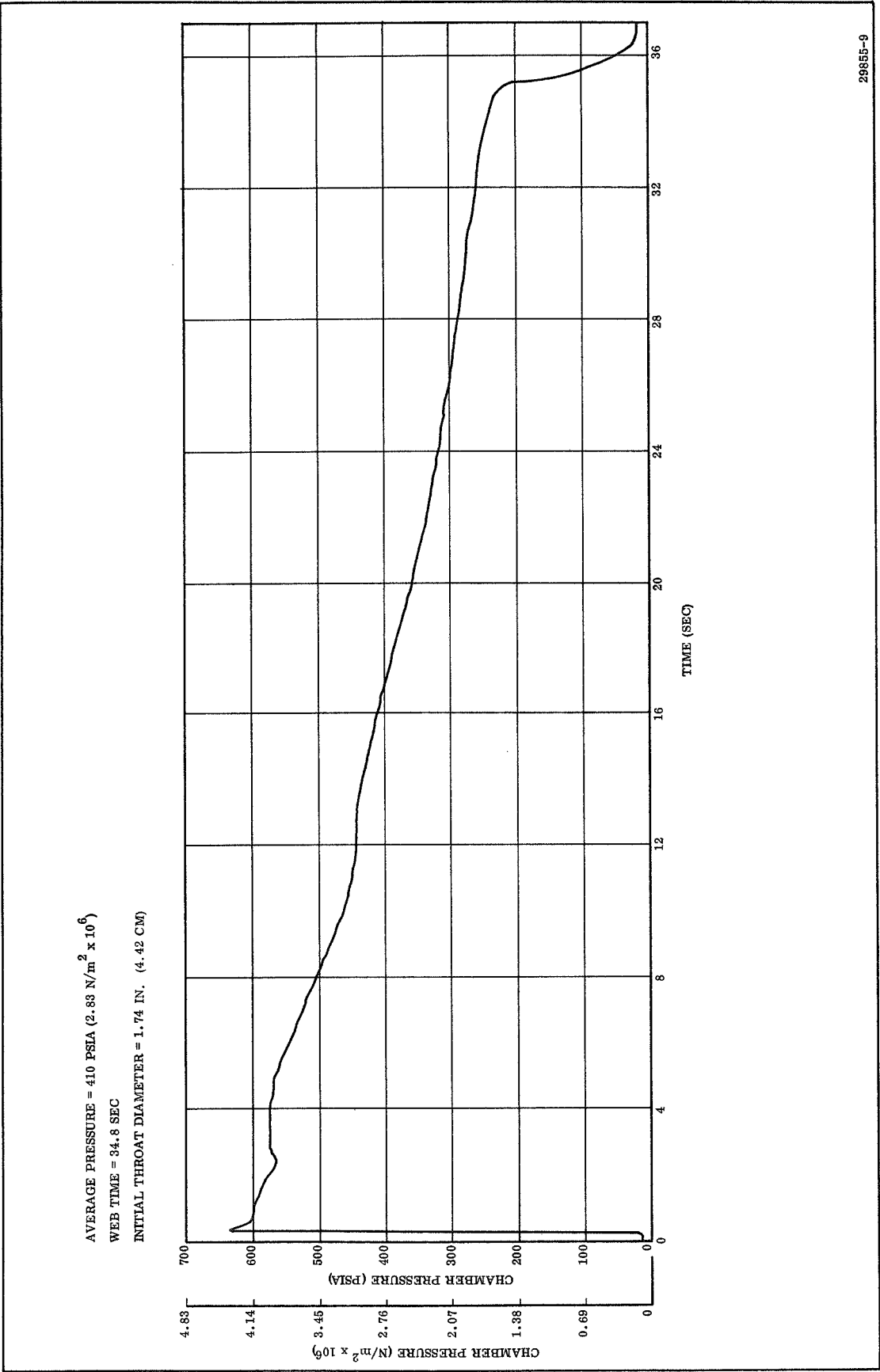


Figure 93. - UF-1164 Pressure Trace

A comparison between the old and new graphite powder phenolic materials for erosion and char is shown in Table XXIV. At each axial station along the ablative billet, the corrected erosion and char rates are shown. Generally for erosion, the new UF-1161 material was better than the old counterpart, T-2610, while UF-1162, UF-1163 and UF-1164 were inferior to the previously developed T-2610 and T-4120 materials. A discussion of the relative performance of the LCCM materials in particular areas of the TU-622 nozzle is presented in the following paragraphs.

At the nozzle inlet area the nine liner materials were rated as shown in Table XXIX on page 184 for large diameter nozzles. The best material was UF-1164, followed by UF-1163 (both graphite powder phenolics) and 4C-1686 carbon cloth in third place. UF-1164 was thus rated superior to the current T-2610/2626 used in nozzle inlet areas.

For the nozzle throat area, the nine liner materials were given a weight rating for large diameter nozzles as shown in Table XXX on page 185. The best material was the 4C-1686 carbon cloth polyphenylene followed by silica cloth SP-8030 and UF-1161 graphite powder phenolic in second and third place. Two other graphite powder phenolics, UF-1164 and T-2610, were rated close to the UF-1161 graphite powder. Two graphite powder phenolic materials, T-4120 and T-2610, missed one rating but were shown with a nine characteristic rating, and supplied a minimum/maximum rating for the missing tenth characteristic. It is interesting to note that the newly developed UF-1161 was rated superior to the current T-2610 graphite phenolic previously used in throat area applications.

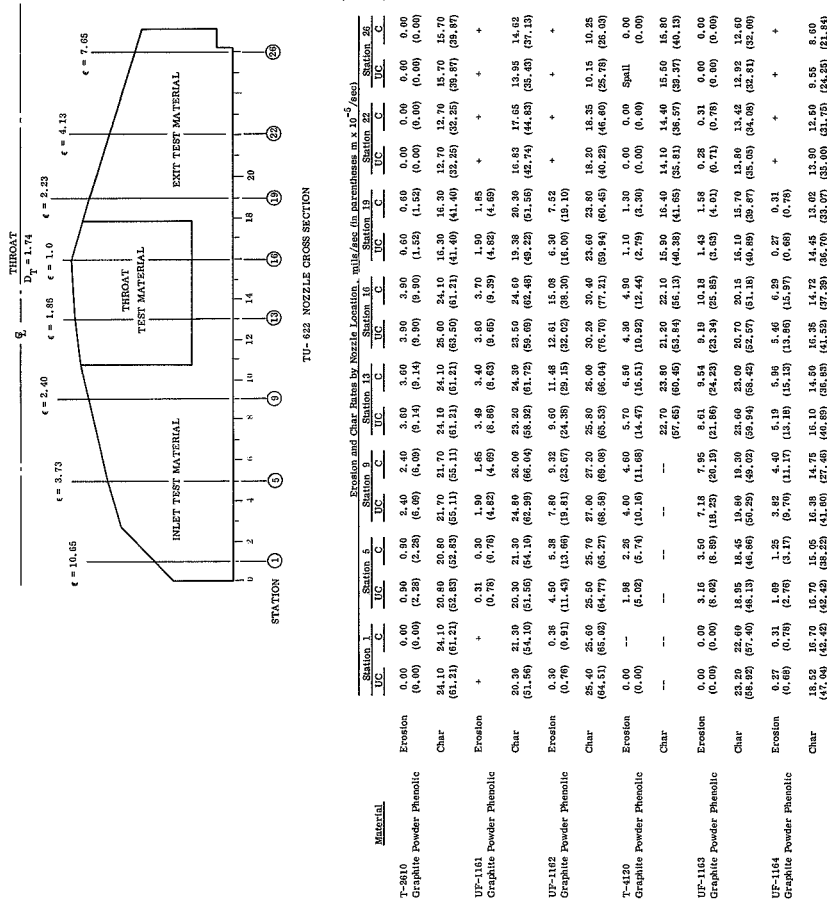
For the nozzle exit area, the nine liner materials were rated as shown in Table XXXI on page 187 for large diameter nozzles. The best material was the carbon cloth phenolic followed by UF-1163 and UF-1164 (graphite powder phenolics) in second and third place. Two other materials, SP-8030 (silica cloth) and T-2626/T-2610 (graphite powder), were rated close enough for favorable mention. It is interesting to note that the new graphite powders UF-1163 and UF-1164 were rated higher than the current T-2610 graphite powder.

Erosion

Inlet-Throat Erosion. - Figure 94 shows the erosion rate performance of the materials as TU-622 inlet-throat nozzle liners subjected to identical motor conditions and plotted vs the one dimensional gas heat transfer coefficient (h/cp), gas blowing parameter (β), and wall material density (ρ).

The seven graphite powder phenolic materials erosion performance is bracketed by the silica cloth preimpregnated SP-8030-96 on the high side and the carbon cloth preimpregnated 4C-1686 on the low side.

TABLE XXIV. - TU-622 MATERIAL PERFORMANCE



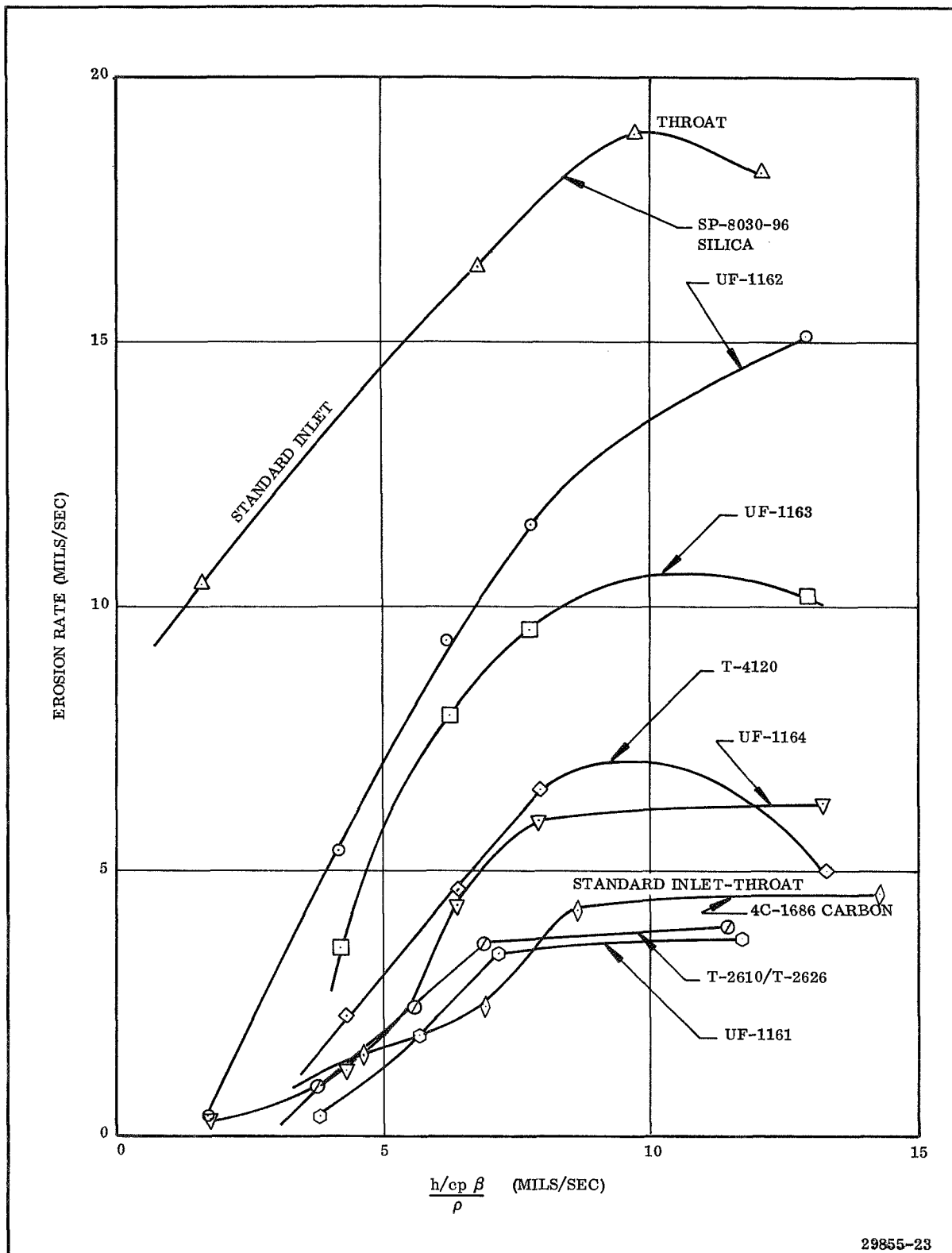


Figure 94. - TU-622 Nozzle Liner Material Erosion Rate Performance, Inlet-Throat

Since the liner design thickness is a function of the erosion rate, the lower the erosion rate the thinner the liner design. For erosion rate performance on large nozzles exceeding 15.00 in. (38.10 cm) diameter throats, all the graphite powder phenolic materials would be acceptable. However, the best graphite powder materials are UF-1161, UF-1164, T-2610, T-2626, and T-4120.

The ranking of the materials on the inlet-throat area for the lowest erosion rate performance is:

1. 4C-1686 (carbon); UF-1161, T-2610, T-2626 (graphite powders).
2. UF-1164 (graphite powder).
3. T-4120 (graphite powder).

Throat-Exit Erosion. - The erosion rate performance of the nine TU-622 nozzle liner materials in the throat-exit area vs h/cp , β , and ρ is shown in Figure 95.

The seven graphite powder phenolic materials are bracketed by SP-8030-96 silica phenolic on the high side and 4C-1686 carbon polyphenylene on the low side (the same as the inlet-throat).

With the liner design thickness a function of the erosion rate, the 4C-1686 would provide the thinnest liner design. For large nozzles with $D_T > 15.00$ in. (38.10 cm), all the graphite powder materials could be used; however, T-2626, T-2610, T-4120, UF-1163, and UF-1164 would be the most likely candidate materials.

The ranking of the materials on the throat-exit area for the lowest erosion rate performance is:

1. 4C-1686 (carbon cloth).
2. T-2610, T-2626, UF-1164 (graphite powders).
3. T-4120, UF-1161 (graphite powders).

Char

Inlet-Throat Char. - The char rate performance of the nine TU-622 nozzle liner materials in the inlet-throat area subjected to identical motor conditions is shown in Figure 96 and plotted vs h/cp , β , and ρ .

Five of the graphite powder phenolic materials (UF-1162, UF-1161, T-2610, T-2626, and T-4120) exhibited higher char rates than the carbon polyphenylene 4C-1686 and silica phenolic SP-8030-96, while two graphite powder materials (UF-1163 and UF-1164) performed as well as the standard liner materials.

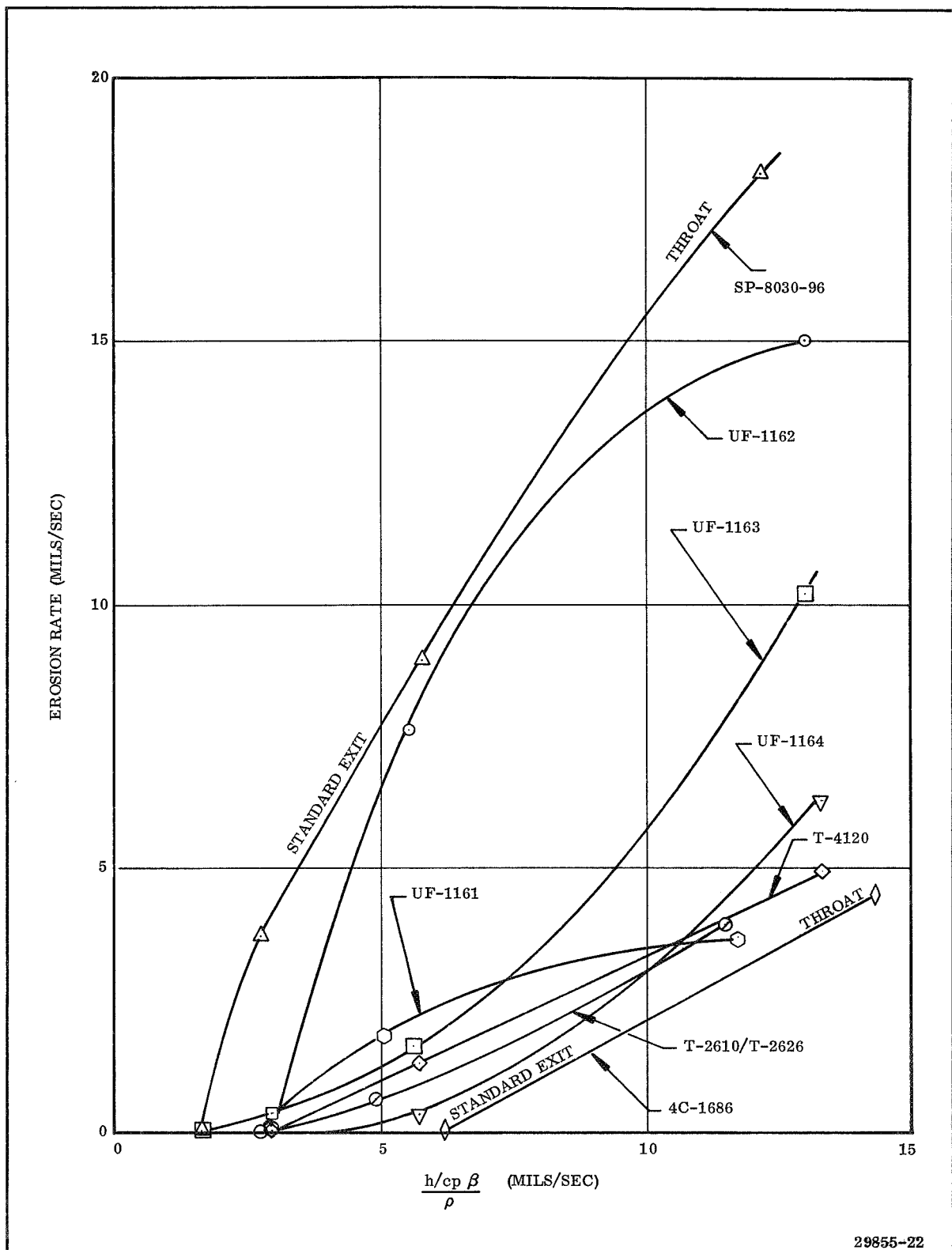


Figure 95. - TU-622 Nozzle Liner Material Erosion Rate Performance, Throat-Exit

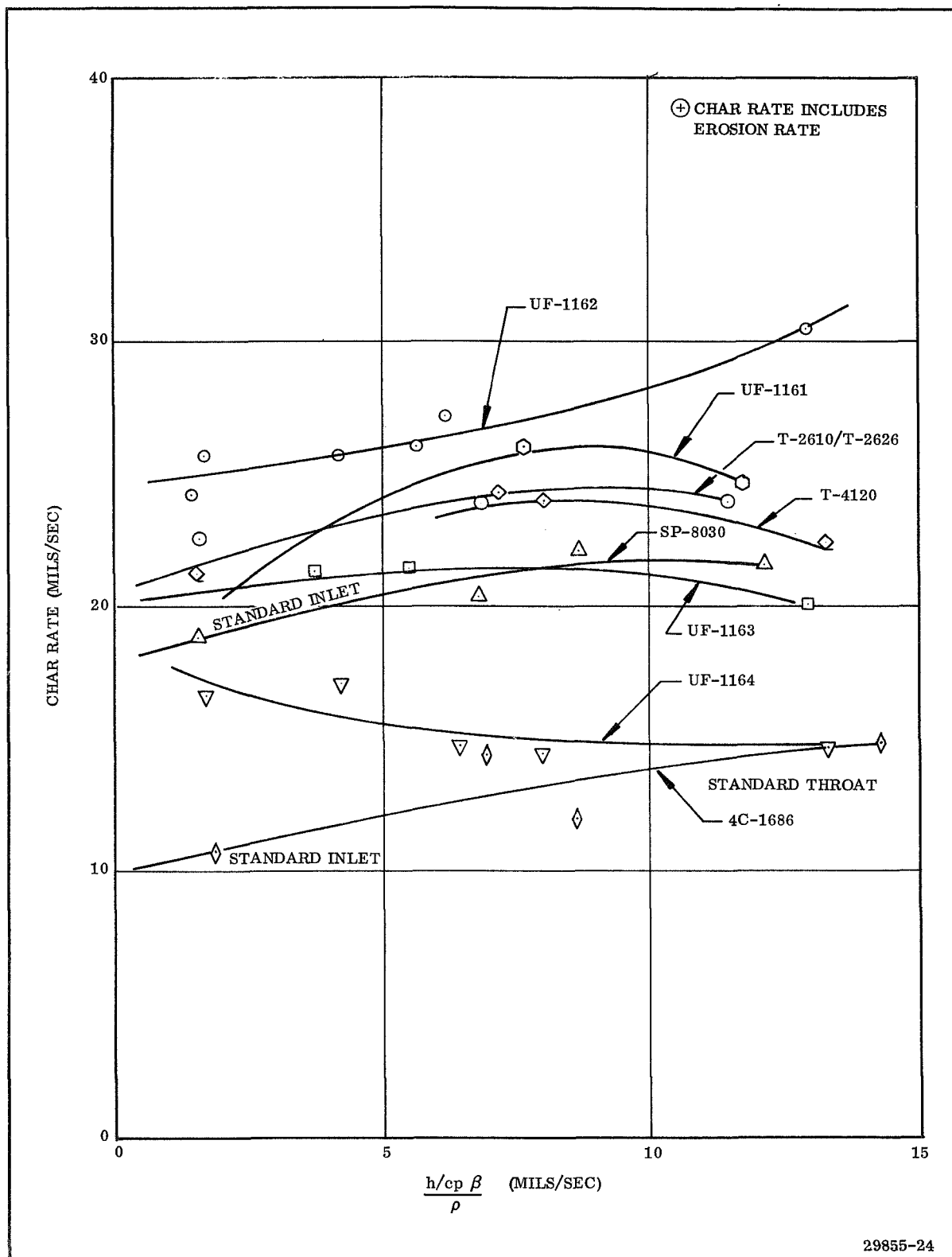


Figure 96. - Nozzle Liner Material Char Rate Performance, Inlet-Throat

The liner design thickness is proportional to char rate; thus the lowest char rate provides the lowest liner design thickness. For liner materials in large nozzles with $D_T > 15.00$ in. (38.10 cm) at the inlet and throat, the UF-1163 and UF-1164 would provide the best low cost material performance.

The ranking of the materials on the inlet-throat area for the lowest char rate performance is:

1. 4C-1686 (carbon cloth).
2. UF-1164 (graphite powder).
3. SP-8030 (silica phenolic), UF-1163 (graphite powder).

Throat-Exit Char. - The char rate performance of the nine TU-622 nozzle liner materials in the throat-exit area is shown in Figure 97 vs the h/cp , β , and ρ .

At the exit all seven of the graphite powder phenolic materials exhibited higher char rates than did the SP-8030-96 silica phenolic and the 4C-1686 carbon polyphenylene. Locally at the throat, UF-1163, UF-1164, and T-4120 perform as well as the standard liner materials.

Since the liner design thickness is proportional to char rate, the thinnest liner material shows the least char rate. The ranking of the materials in the throat-exit area for lowest char rate performance is:

1. 4C-1686 (carbon cloth).
2. UF-1164 (graphite powder).
3. SP-8030 (silica phenolic), UF-1163 (graphite powder).

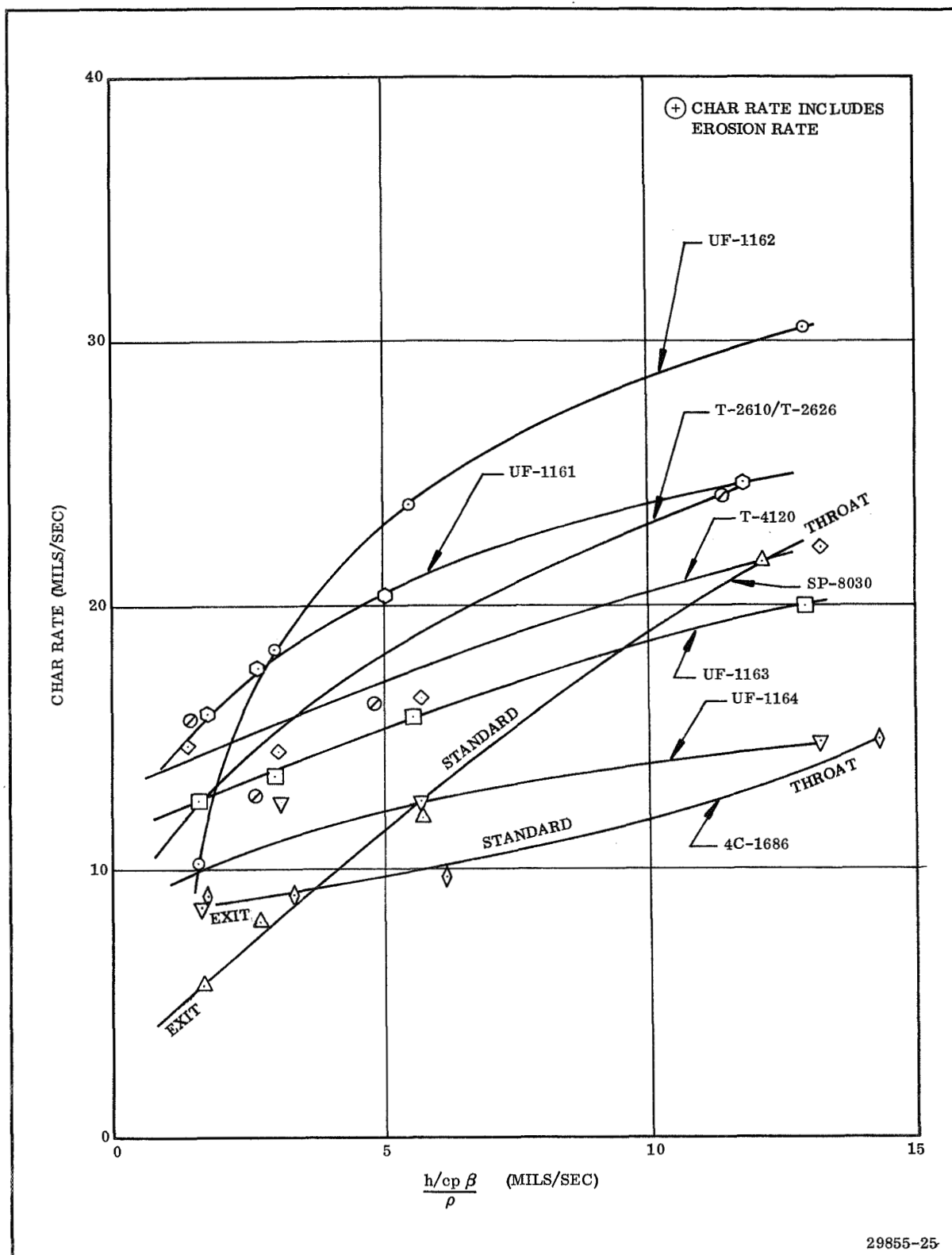


Figure 97. - Nozzle Liner Material Char Rate
Performance, Throat-Exit

MATERIAL CHARACTERIZATION AND COMPARISON

Four selected materials were characterized by tests for mechanical and thermal properties. The results of this testing are shown in Table XXV.

To properly compare these formulations, it is necessary to go back to the original Thiokol LCCM formulations, T-4120, T-2610, and T-2626.

Physical property data and other characteristics associated with the UF formulations and the baseline materials (T-2610, T-4120, and T-2626) are listed in Table XXV for comparative purposes.

Based on data collected to date, the high pressure cured materials, UF-1161 and UF-1162, show considerable improvement over the similar high pressure cured baseline formulations (T-2610 and T-2626):

1. Tensile strengths of UF-1161 and UF-1162 (4,865 psi, $33.57 \text{ N/m}^2 \times 10^6$) have been increased by 53 percent over the baseline materials. T-2610 and T-2626 have tensile strengths of 3,185 psi ($21.98 \text{ N/m}^2 \times 10^6$).
2. Compressive strengths have been increased 41 percent. UF-1161, UF-1162 at 17,095 psi ($117.96 \text{ N/m}^2 \times 10^6$) was compared to the T-2610, T-2626 at 12,108 psi ($83.55 \text{ N/m}^2 \times 10^6$).
3. The new formulations have lower densities (UF-1161 = 1.67 g/cc and UF-1162 = 1.61 g/cc) as compared to 1.70 g/cc for T-2610 and 1.88 g/cc for T-2626 (or an average density decrease of 8.35 percent).
4. Thermal conductivity for the UF-1161 and UF-1162 decreased by 6 percent from the thermal conductivity of the T-2626 and T-2610 baseline materials.
5. The UF-1161 erosion rate of 3.70 mils/sec ($9.4 \text{ m} \times 10^{-5}/\text{sec}$) at the throat compares favorably with the T-2610 erosion rate of 3.90 mils/sec ($9.9 \text{ m} \times 10^{-5}/\text{sec}$) for a 5 percent decrease.

TABLE XXV. - COMPARISON OF UF MATERIALS AND ORIGINAL THICKOL FORMULATIONS

Material	Percent Resin	Fiber and Resin	Cure Conditions	Mix No.	Tensile Strength		Density g/cc	Compressive Strength		Tensile Modulus		Compressive Modulus		Thermal Conductivity at 300° F (422° K)		Flexural Strength		Shear Strength	TL-022 Corrected Erosion/Char Rates						
					psi	(N/m ² × 10 ⁶)		psi	(N/m ² × 10 ⁶)	psi	(N/m ² × 10 ⁶)	psi	(N/m ² × 10 ⁶)	psi	(N/m ² × 10 ⁶)	mm x 10 ⁻³ /sec	mm x 10 ⁻³ /sec		mm x 10 ⁻³ /sec	mm x 10 ⁻³ /sec					
UF-1151	Monsanto R1-1008	None	1,000 psi at 232° F	655	4,720	32.5	1.67	15,700	108.24	1.59	10.96	3.98	27.44	75.75	5,475	37.74	3,589	25.71	1.65/26.00	4.70/65.0	3.70/24.60	1.86/20.30	4.66/31.5		
					4,520	31.5	1.67	15,400	106.17	1.58	10.99	3.96	27.30	5,085	35.05	5,085	35.05	5,085	35.05	5,085	35.05	5,085	35.05	5,085	35.05
					4,380	30.5	1.61	15,650	107.51	1.51	10.42	3.98	27.40	5,215	36.76	5,215	36.76	5,215	36.76	5,215	36.76	5,215	36.76	5,215	36.76
					4,380	30.5	1.61	15,650	107.51	1.51	10.42	3.98	27.40	5,215	36.76	5,215	36.76	5,215	36.76	5,215	36.76	5,215	36.76	5,215	36.76
UF-1152	Monsanto R1-1008	Wood Floor	1,000 psi at 232° F	670	5,300	36.5	1.61	18,700	128.34	1.53	10.54	4.92	33.82	55.00	6,435	44.78	5,025	34.94	9.32/37.20	22.7/68.10	15.08/30.40	38.2/77.1	7.45/23.30	19.2/60.5	
					5,300	36.5	1.61	18,700	128.34	1.53	10.54	4.92	33.82	55.00	6,435	44.78	5,025	34.94	9.32/37.20	22.7/68.10	15.08/30.40	38.2/77.1	7.45/23.30	19.2/60.5	
					5,300	36.5	1.61	18,700	128.34	1.53	10.54	4.92	33.82	55.00	6,435	44.78	5,025	34.94	9.32/37.20	22.7/68.10	15.08/30.40	38.2/77.1	7.45/23.30	19.2/60.5	
					5,300	36.5	1.61	18,700	128.34	1.53	10.54	4.92	33.82	55.00	6,435	44.78	5,025	34.94	9.32/37.20	22.7/68.10	15.08/30.40	38.2/77.1	7.45/23.30	19.2/60.5	
UF-1153	Durez 10694	Wood Floor	200 psi at 232° F	657	1,320	9.1	1.57	8,825	61.53	0.98	6.76	7.07	48.74	75.75	4,410	30.40	2,925	20.15	7.95/13.30	20.2/48.8	10.18/26.15	25.8/51.0	1.56/15.70	4.0/46.0	
					1,320	9.1	1.57	8,825	61.53	0.98	6.76	7.07	48.74	75.75	4,410	30.40	2,925	20.15	7.95/13.30	20.2/48.8	10.18/26.15	25.8/51.0	1.56/15.70	4.0/46.0	
					1,320	9.1	1.57	8,825	61.53	0.98	6.76	7.07	48.74	75.75	4,410	30.40	2,925	20.15	7.95/13.30	20.2/48.8	10.18/26.15	25.8/51.0	1.56/15.70	4.0/46.0	
					1,320	9.1	1.57	8,825	61.53	0.98	6.76	7.07	48.74	75.75	4,410	30.40	2,925	20.15	7.95/13.30	20.2/48.8	10.18/26.15	25.8/51.0	1.56/15.70	4.0/46.0	
UF-1154	Durez 10694	CCF 1/4 in.	200 psi at 232° F	658	1,540	10.7	1.53	10,800	69.84	0.76	5.54	6.03	41.36	69.01	4,110	28.28	3,405	23.47	4.40/14.75	11.2/37.4	8.29/14.72	16.0/37.2	0.31/13.02	0.776/23.0	
					1,540	10.7	1.53	10,800	69.84	0.76	5.54	6.03	41.36	69.01	4,110	28.28	3,405	23.47	4.40/14.75	11.2/37.4	8.29/14.72	16.0/37.2	0.31/13.02	0.776/23.0	
					1,540	10.7	1.53	10,800	69.84	0.76	5.54	6.03	41.36	69.01	4,110	28.28	3,405	23.47	4.40/14.75	11.2/37.4	8.29/14.72	16.0/37.2	0.31/13.02	0.776/23.0	
					1,540	10.7	1.53	10,800	69.84	0.76	5.54	6.03	41.36	69.01	4,110	28.28	3,405	23.47	4.40/14.75	11.2/37.4	8.29/14.72	16.0/37.2	0.31/13.02	0.776/23.0	
T-5010	Monsanto SC-1008	None	Rising 170° F to 5 hr	1	2,326	16.19	1.70	11,616	82.22	1.62	10.73	6.04	41.94	84.05	4,026	27.75	3,288	22.65	2.40/21.70	6.1/55.0	3.90/24.10	8.9/61.0	0.69/18.30	0.153/41.4	
					2,326	16.19	1.70	11,616	82.22	1.62	10.73	6.04	41.94	84.05	4,026	27.75	3,288	22.65	2.40/21.70	6.1/55.0	3.90/24.10	8.9/61.0	0.69/18.30	0.153/41.4	
					2,326	16.19	1.70	11,616	82.22	1.62	10.73	6.04	41.94	84.05	4,026	27.75	3,288	22.65	2.40/21.70	6.1/55.0	3.90/24.10	8.9/61.0	0.69/18.30	0.153/41.4	
					2,326	16.19	1.70	11,616	82.22	1.62	10.73	6.04	41.94	84.05	4,026	27.75	3,288	22.65	2.40/21.70	6.1/55.0	3.90/24.10	8.9/61.0	0.69/18.30	0.153/41.4	
T-5026	Durez 23362	None	1,000 psi at 232° F	3,445	23,77	1.88	12,390	84.87	1.68	10.73	6.04	41.94	84.05	4,026	27.75	3,288	22.65	2.40/21.70	6.1/55.0	3.90/24.10	8.9/61.0	0.69/18.30	0.153/41.4		
					23,77	1.88	12,390	84.87	1.68	10.73	6.04	41.94	84.05	4,026	27.75	3,288	22.65	2.40/21.70	6.1/55.0	3.90/24.10	8.9/61.0	0.69/18.30	0.153/41.4		
					23,77	1.88	12,390	84.87	1.68	10.73	6.04	41.94	84.05	4,026	27.75	3,288	22.65	2.40/21.70	6.1/55.0	3.90/24.10	8.9/61.0	0.69/18.30	0.153/41.4		
					23,77	1.88	12,390	84.87	1.68	10.73	6.04	41.94	84.05	4,026	27.75	3,288	22.65	2.40/21.70	6.1/55.0	3.90/24.10	8.9/61.0	0.69/18.30	0.153/41.4		
T-4120	Durez 10694	None	1,000 psi at 232° F	2,326	16.0	1.59	8,185	56.47	1.59	10.73	6.04	41.94	84.05	4,026	27.75	3,288	22.65	2.40/21.70	6.1/55.0	3.90/24.10	8.9/61.0	0.69/18.30	0.153/41.4		
					16.0	1.59	8,185	56.47	1.59	10.73	6.04	41.94	84.05	4,026	27.75	3,288	22.65	2.40/21.70	6.1/55.0	3.90/24.10	8.9/61.0	0.69/18.30	0.153/41.4		
					16.0	1.59	8,185	56.47	1.59	10.73	6.04	41.94	84.05	4,026	27.75	3,288	22.65	2.40/21.70	6.1/55.0	3.90/24.10	8.9/61.0	0.69/18.30	0.153/41.4		
					16.0	1.59	8,185	56.47	1.59	10.73	6.04	41.94	84.05	4,026	27.75	3,288	22.65	2.40/21.70	6.1/55.0	3.90/24.10	8.9/61.0	0.69/18.30	0.153/41.4		

In comparing the low pressure cured formulations, UF-1163 and UF-1164, with the baseline formulation (T-4120), the characteristics of this material family reflected a different pattern. The tensile strengths of UF-1163 and UF-1164 decreased by 25 and 8 percent, respectively, when compared to the tensile strength of the baseline T-4120 formulation.

The compressive strengths of the new low pressure materials, UF-1163 and UF-1164, are 13 and 24 percent higher, respectively, than the graphite powder phenolic, T-4120.

The densities for the UF-1163 and UF-1164 are 1 and 0.4 percent lower, respectively, than the T-4120 base material.

A large decrease in thermal conductivity is exhibited by a decrease of 50 and 55 percent for the UF-1163 and UF-1164 compared to the T-4120 base material.

The UF-1164 graphite powder phenolic throat erosion rate of 6.29 mils/sec ($15.91 \text{ m} \times 10^{-5}/\text{sec}$) compares satisfactorily with the base T-4120 graphite powder phenolic erosion rate of 4.90 mils/sec ($12.49 \text{ m} \times 10^{-5}/\text{sec}$) with only a 28 percent increase.

Generally, the UF-1161 and UF-1162 would be applied to the inlet-throat where higher mechanical properties are required, whereas the UF-1163 and UF-1164 would be applied to the exit cone and submerged liner where the lower thermal conductivity is required.

The coefficient of thermal expansion data is shown in Table XXVI. The high pressure cured materials, UF-1161 and UF-1162, increase in thermal expansion at about 600° to 800° F (590° to 700° K) and contract at about 800° to 1,000° F (700° to 810° K). In Table XXVI the positive numbers indicate an increase in length, and the minus sign indicates a decrease in length. However, the average data do not appear to fall below the initial specimen length during the test temperature range from 200° to 1,000° F (366° to 811° K). The abrupt change in expansion measurements for UF-1161 and UF-1162 (Figures 98 and 99) may be attributed to the superimposed changes in moisture content, degrees of polymerization of the binder, loss of material, or release of stresses.

The low pressure cured materials, UF-1163 and UF-1164, show a continuous decrease in the coefficient of thermal expansion (Figures 100 and 101) going from 200° to 1,000° F (366° to 811° K).

The major difference between the parallel and perpendicular test data appears to be the degree of change in expansion. The perpendicular to mold pressure samples (high pressure samples) show a larger expansion than those samples taken parallel to mold pressure. Figures 102 thru 105 show the perpendicular to mold pressure data.

TABLE XXVI. - SUMMARY OF THERMAL EXPANSION DATA

Material	Temperature (°F) (°K)	Coefficient of Thermal Expansion			
		Parallel to Mold Pressure		Perpendicular to Mold Pressure	
		(in./in. - °F x 10 ⁻⁶)	(cm/cm - °K x 10 ⁻⁶)	(in./in. - °F x 10 ⁻⁶)	(cm/cm - °K x 10 ⁻⁶)
UF-1161	200	0	0	0	0
	400	3.8	6.84	14.0	25.20
	600	22.0	39.60	48.0	86.40
	800	8.0	14.40	40.0	72.00
	1,000	-2.0	3.60	7.0	12.60
UF-1162	200	8.0	14.40	0	0
	400	13.0	23.40	25.0	45.00
	600	15.0	27.00	43.0	77.40
	800	-7.0	12.60	38.0	68.40
	1,000	--	--	7.0	12.60
UF-1163	200	0	0	0	0
	400	-2.5	-4.50	2.5	4.50
	600	-4.0	-7.20	-2.0	-3.60
	800	-15.0	-27.00	-14.0	-25.20
	1,000	--	--	--	--
UF-1164	200	0	0	2.0	3.60
	400	2.0	3.60	4.8	8.64
	600	-3.5	-6.30	5.1	9.18
	800	-9.2	-16.56	-1.4	-2.52
	1,000	-17.0	-30.60	--	--

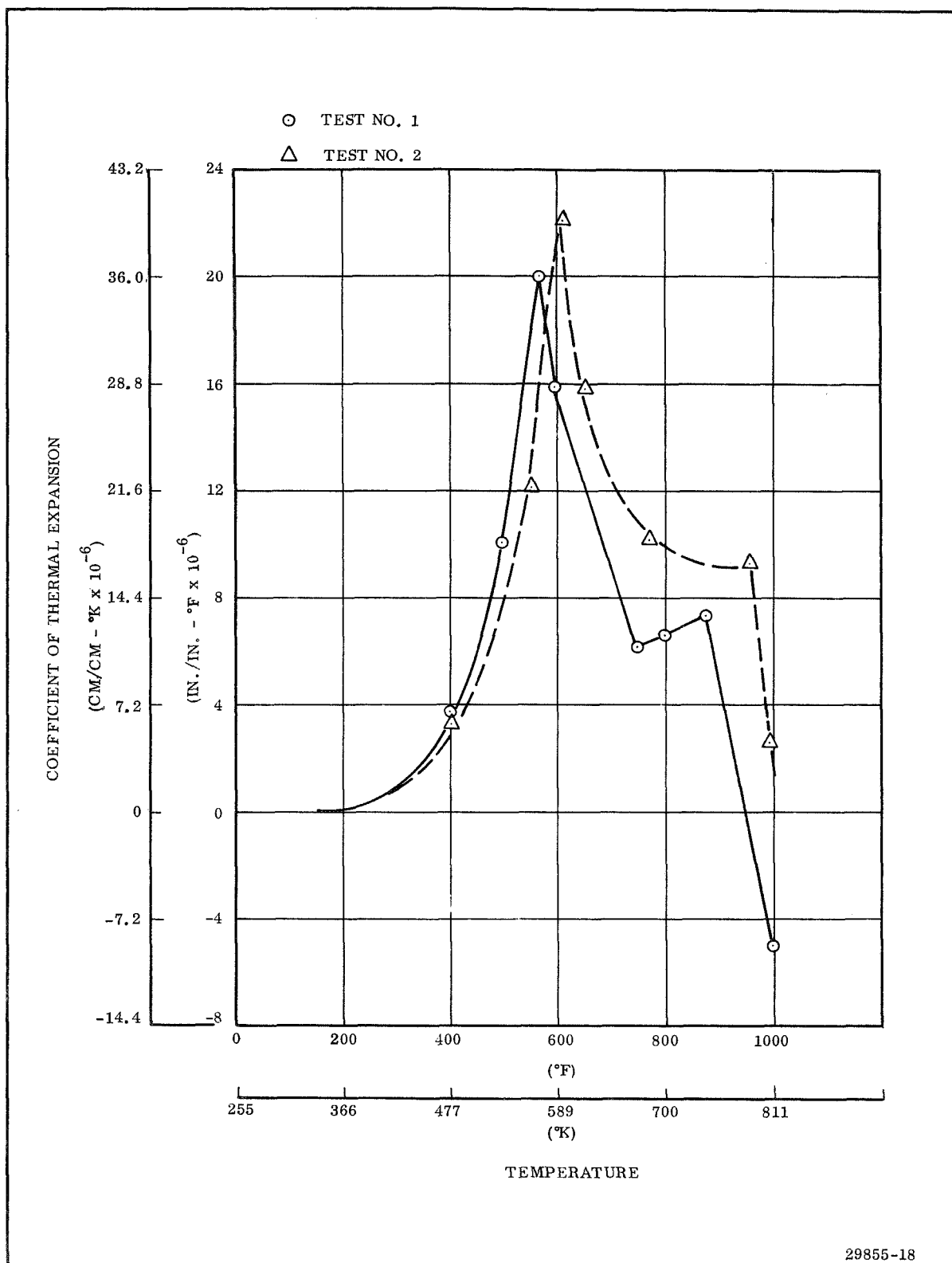


Figure 98. - UF-1161 (Parallel to Pressure) Coefficient of Thermal Expansion vs Temperature

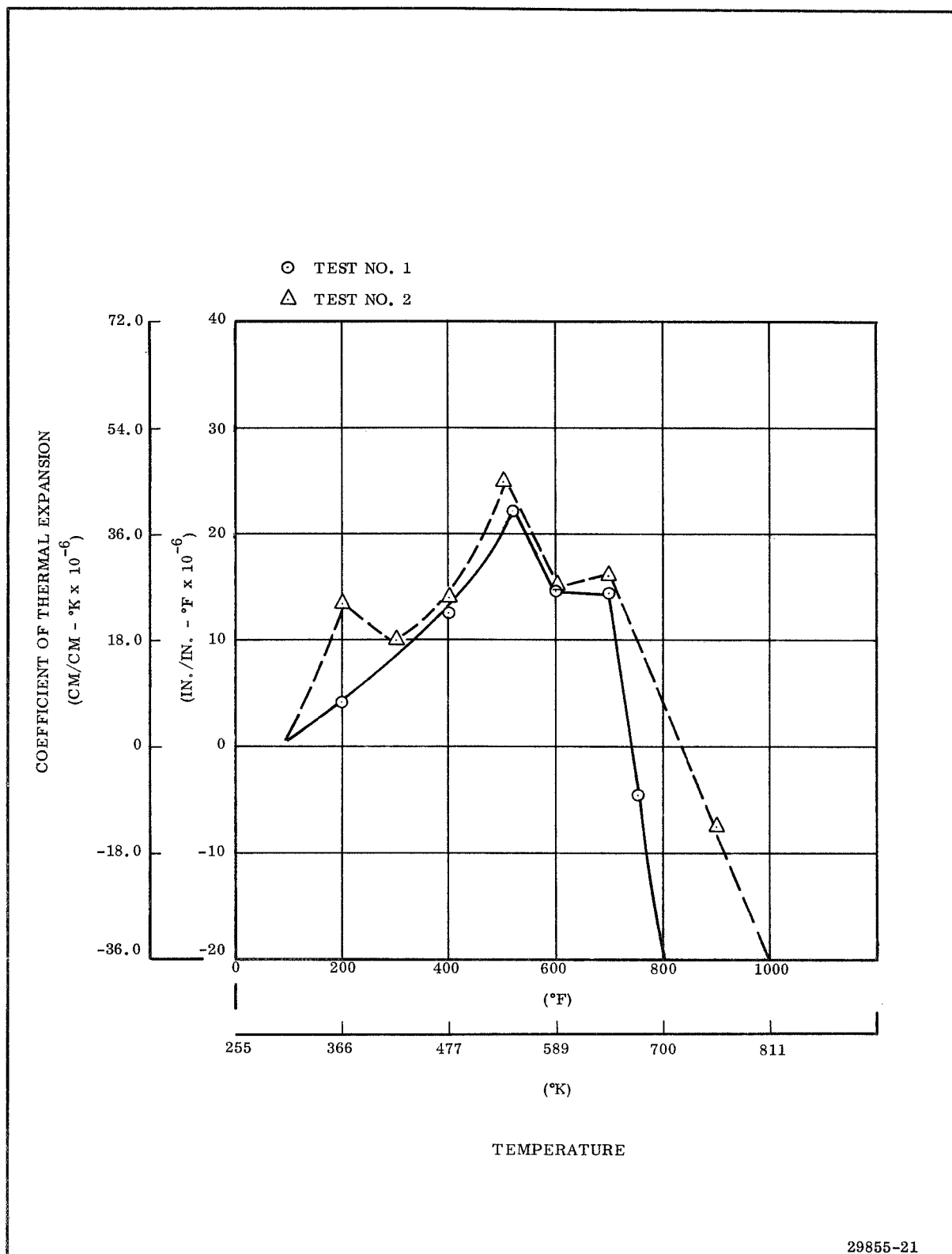


Figure 99. - UF-1162 (Parallel to Pressure) Coefficient of Thermal Expansion vs Temperature

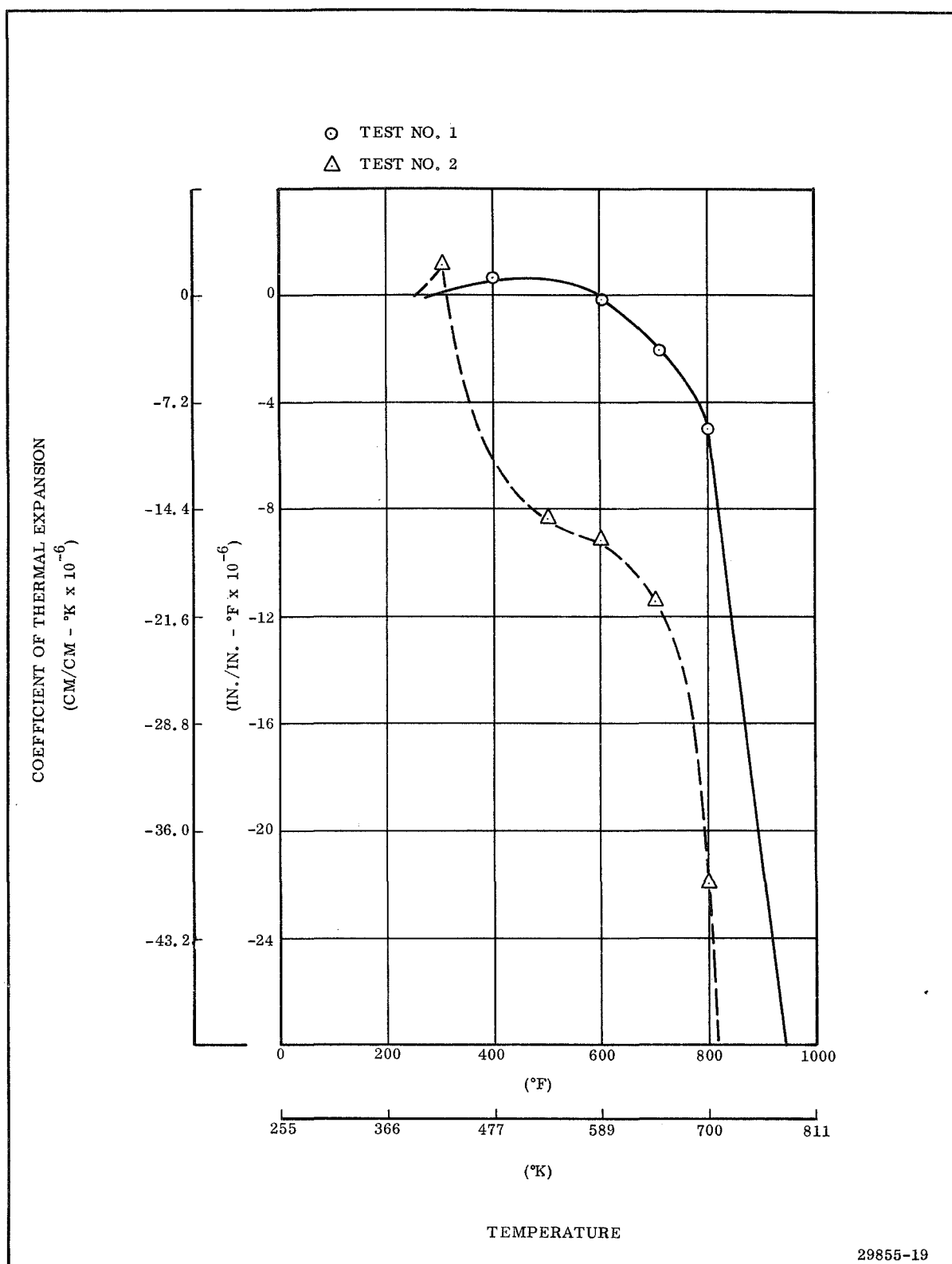
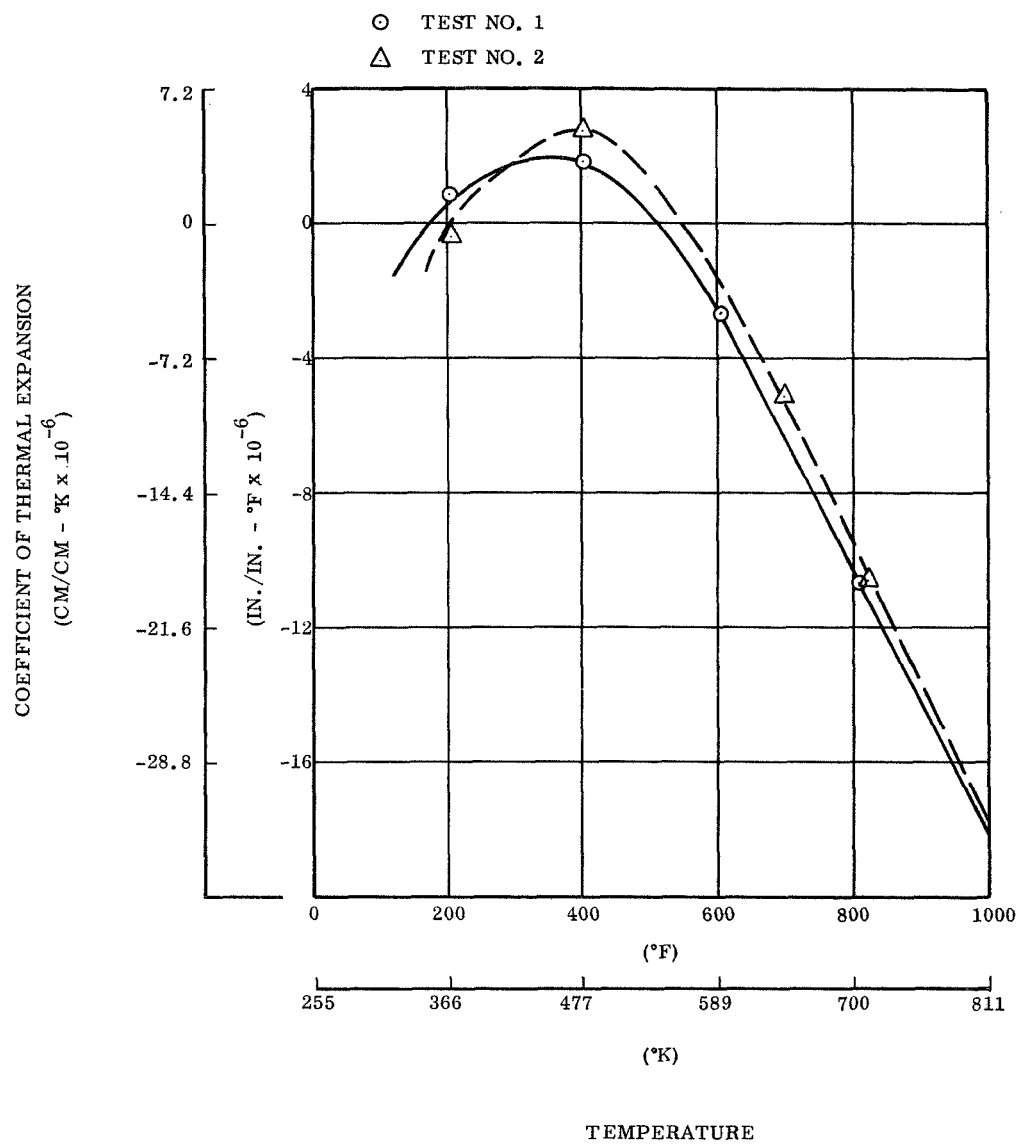
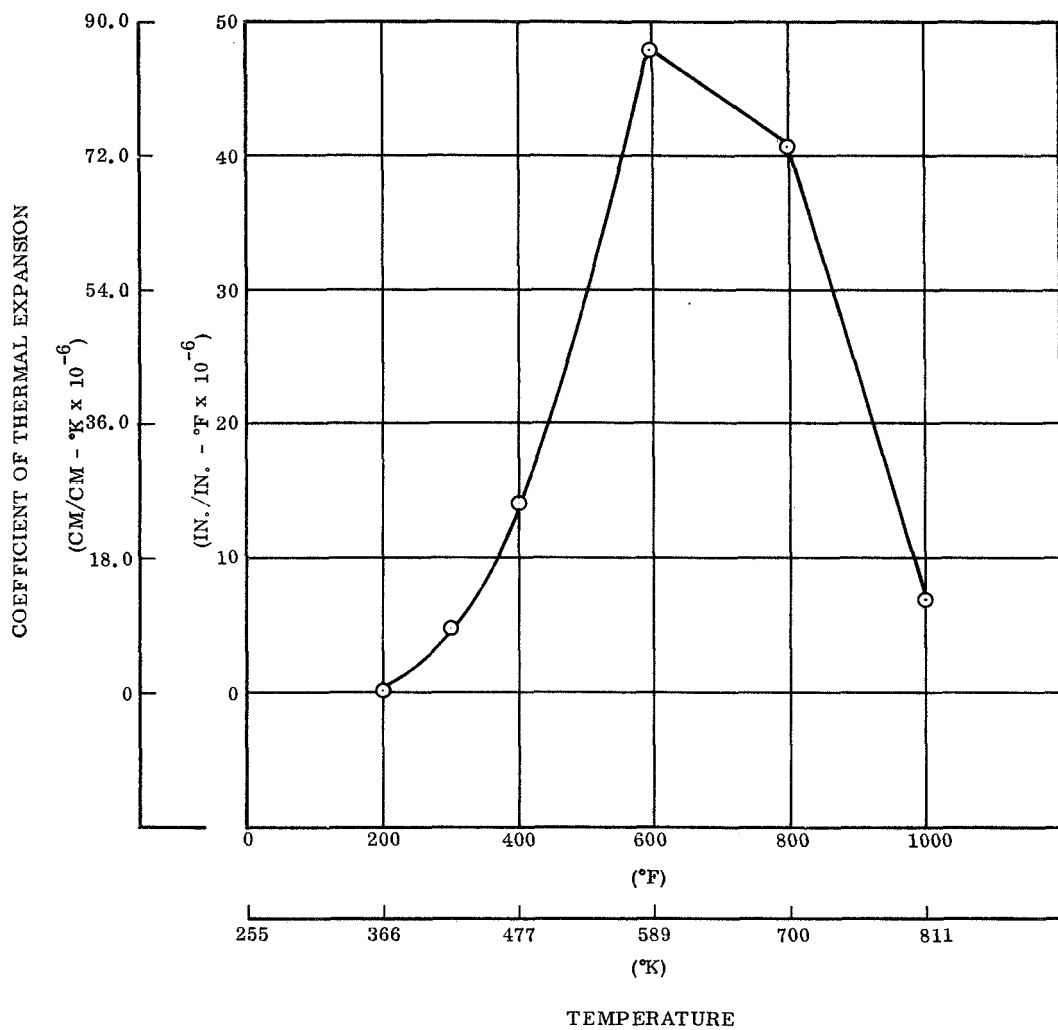


Figure 100.- UF-1163 (Parallel to Pressure) Coefficient of Thermal Expansion vs Temperature



29855-20

Figure 101. - UF-1164 (Parallel to Pressure) Coefficient of Thermal Expansion vs Temperature



29855-17

Figure 102. - UF-1161 (Perpendicular to Pressure) Coefficient of Thermal Expansion vs Temperature

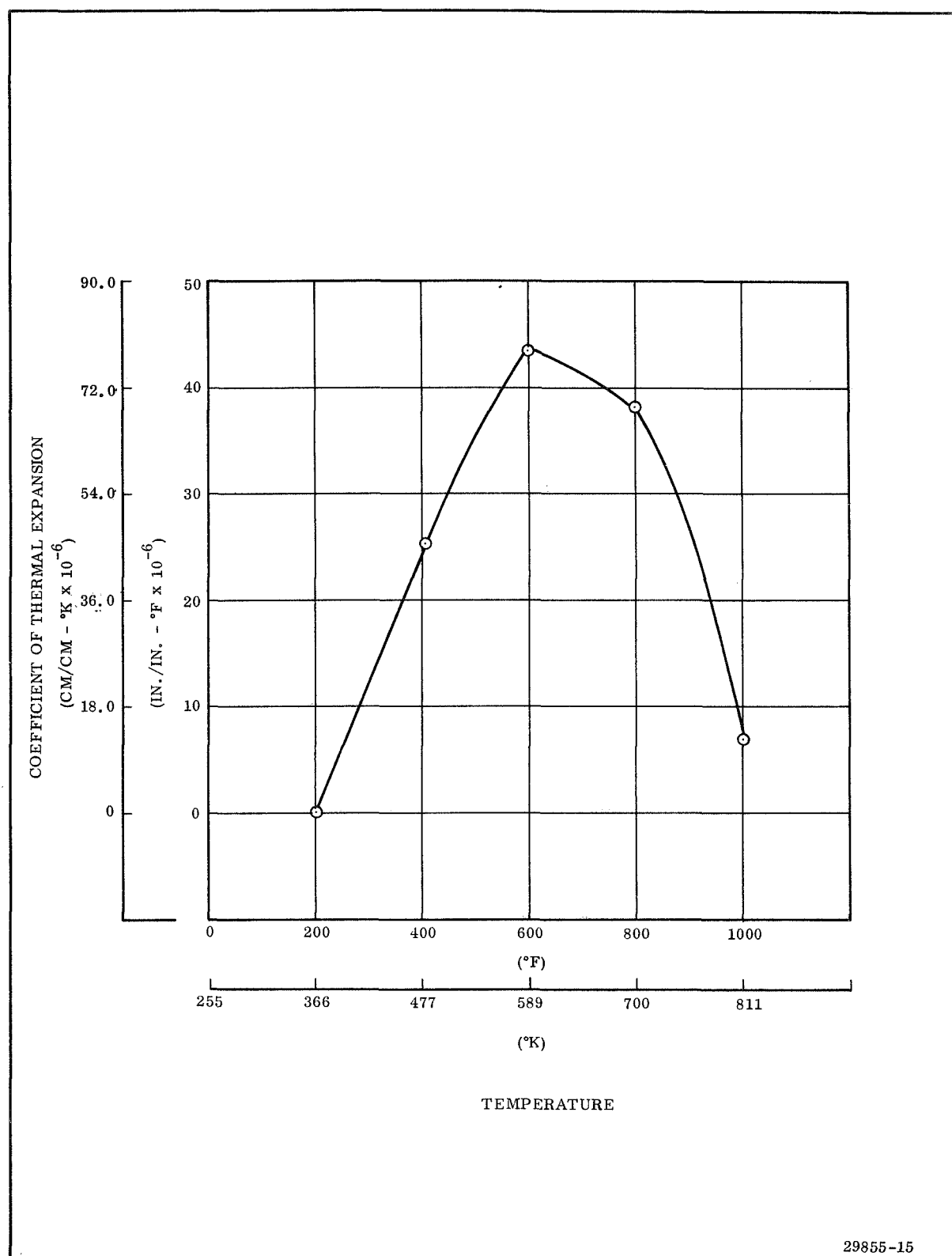
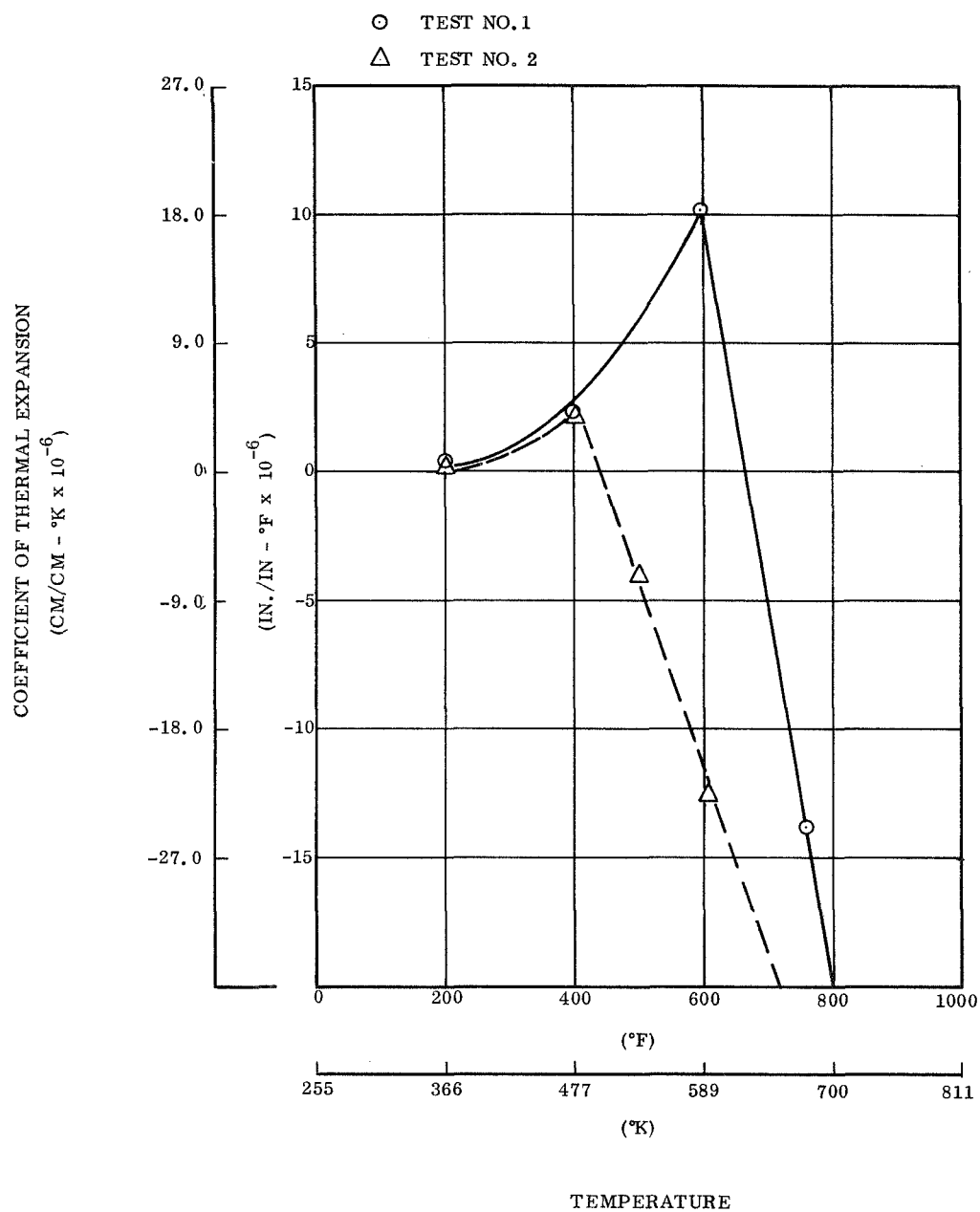
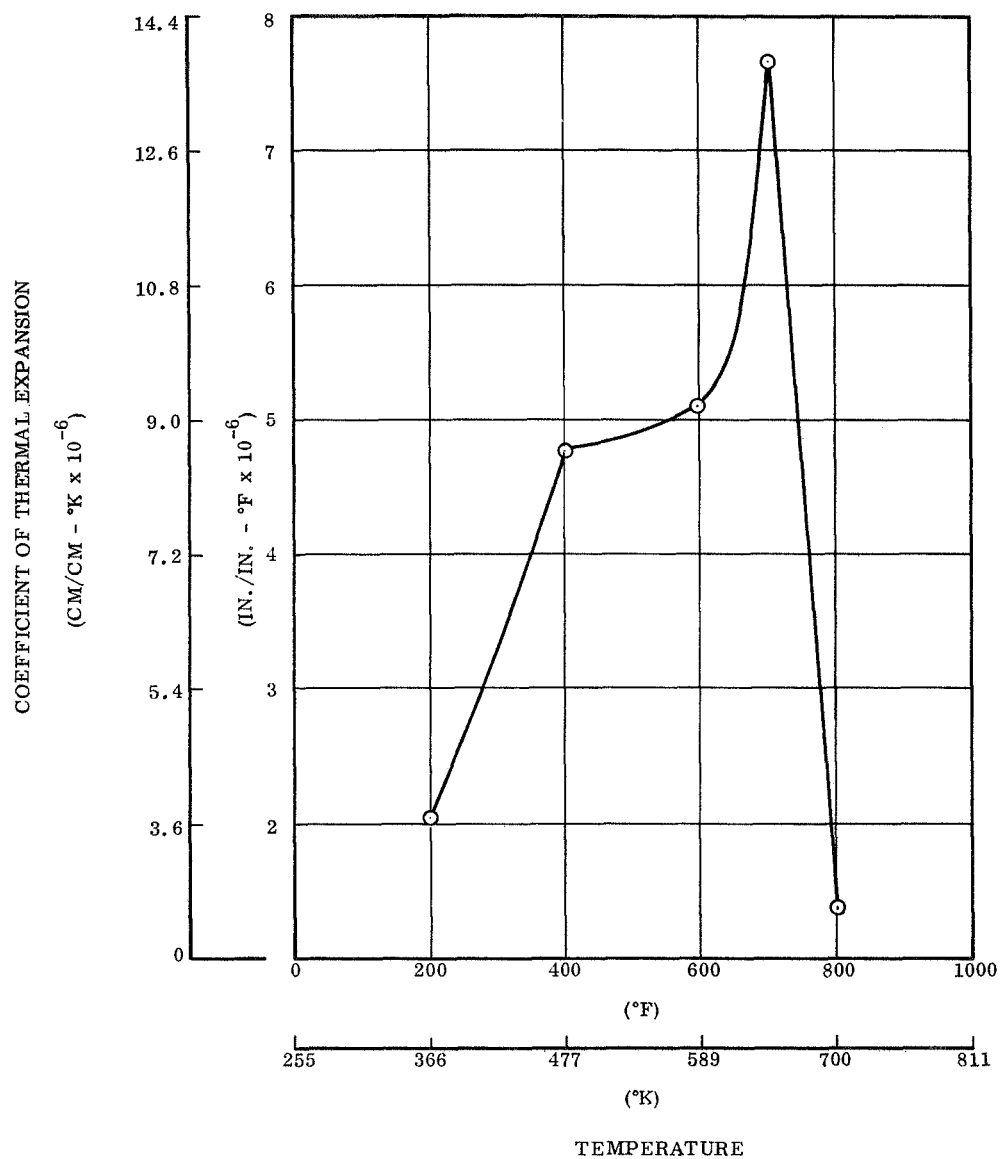


Figure 103. - UF-1162 (Perpendicular to Pressure) Coefficient of Thermal Expansion vs Temperature



29855-14

Figure 104. - UF-1163 (Perpendicular to Pressure) Coefficient of Thermal Expansion vs Temperature



29855-16

Figure 105. - UF-1164 (Perpendicular to Pressure) Coefficient of Thermal Expansion vs Temperature

A feeling for what the new LCCM formulations are and what they will do can best be accomplished by an evaluation of individual material characteristics and a tradeoff study or comparison against the original LCCM's: T-4120, T-2610, T-2626, SP-8030 silica, and 4C-1686 carbon cloth insulators. The raw material composition of these materials is shown in Table XXVII.

The new LCCM's use a higher resin content (40 percent) and make use of some fillers and reinforcements when compared with the original LCCM and standard silica and carbon cloth insulations.

To characterize all the liner materials for the tradeoff study, the following material properties are required.

1. Specific gravity
2. Raw material cost
3. Erosion rate
4. Char rate (encloses erosion rate)
5. Thermal conductivity
6. Ultimate compression strength
7. Ultimate tensile strength
8. Coefficient of thermal expansion (CTE)
9. Structural integrity
10. Ease of fabrication

These properties determine the liner material thickness, weight, and cost for preliminary production and demonstration rocket motor nozzles. The LCCM's T-2610 and T-2626 are used interchangeably, dependent on material property availability.

A carbon phenolic MX-4926 is substituted for the 4C-1686 carbon cloth poly-phenylene and MX-2600 silica for SP-8030 silica for evaluating the (CTE) properties in direct comparison to the graphite powder phenolic and silica cloth phenolic because no (CTE) properties were available for 4C1686 and SP-8030 in two planes, with and against ply.

Specific Gravity

The specific gravity of the nine liner materials is shown in Table XXVII. The new and the original LCCM formulations fall within the range of the standard

TABLE XXVII. - RAW MATERIAL COMPOSITIONS

<u>Standard Materials</u>	<u>Resin Manufacturer</u>	<u>Percent Resin</u>	<u>Percent Cloth Reinforcement</u>	<u>Percent Filler</u>	<u>Specific Gravity</u>
Carbon cloth polyphenylene (4C-1686)	N/A	33	0	0	1.40
Silica cloth phenolic (SP-8030)	Evercoat 201	32	0	0	1.60
<u>Original LCCM materials</u>					
T-2610	Monsanto SC-1008	25	75	0	1.80
T-2626	Durez 23362	25	75	0	1.77
T-4120	Durez 10694	25	75	0	1.50
<u>New LCCM materials</u>					
UF-1161	Monsanto RI-4009	40	60	0	1.67
UF-1162	Monsanto RI-4009	40	50	10 (wood flour)	1.61
UF-1163	Durez 10694	40	55	5 (wood flour)	1.57
UF-1164	Durez 10694	40	50	10 (carbon fiber)	1.53

carbon and silica cloth materials (specific gravity 1.4 to 1.6), except for T-2610 and T-2626 which have higher specific gravities (1.77 and 1.80) and UF-1161 with specific gravity of 1.67. None of the graphite powder materials is as light as carbon cloth phenolic, but four are lighter or equivalent to the silica cloth phenolic.

The ranking of the materials by the lowest density is:

1. 4C-1686 (carbon cloth polyphenylene)
2. T-4120 (original LCCM)
3. UF-1164 (new LCCM)

Raw Material Cost

The raw material cost per pound is shown in Figure 106 for the liner materials. The new and existing LCCM compounds all have lower costs than the standard carbon and silica cloth reinforced prepregs.

The new UF-1164 compound is higher in cost than the original T-4120.

In addition, the processing costs to form, cure, and shape the raw materials to the final net billet, also favor the LCCM's over the cloth reinforced prepregs as shown in Figure 106 (\$10/lb plus scrap compared to \$35/lb plus scrap).

The ranking of the materials by the lowest raw material cost is:

1. T-2610/T-2626 (graphite powder)
2. UF-1161 (graphite powder)
3. T-4120 and UF-1162 (graphite powder)

Erosion Rate and Char Rate

These properties are discussed in the section: TU-622 Material Erosion and Char Testing.

Thermal Conductivity

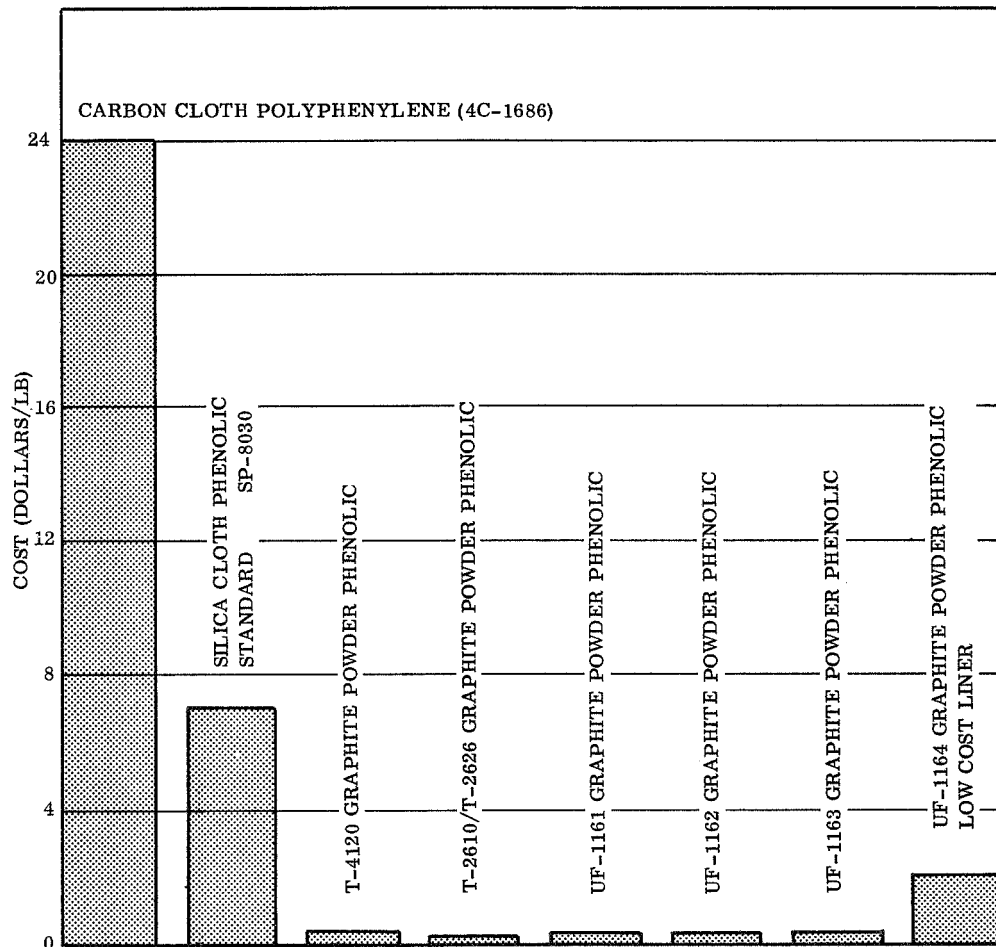
The thermal conductivity of the graphite powder and two standard carbon and silica cloth liners is shown in Figure 107.

PROCESSING COSTS*

CARBON AND SILICA CLOTH $\approx$ \$35/LB PLUS SCRAP

GRAPHITE POWDER $\approx$ \$10/LB PLUS SCRAP

*TOTAL COSTS INCLUDE PROCESSING AND
RAW MATERIAL



29855-13

Figure 106. - Raw Material Cost/Lb (1,000 Lb Quantities)

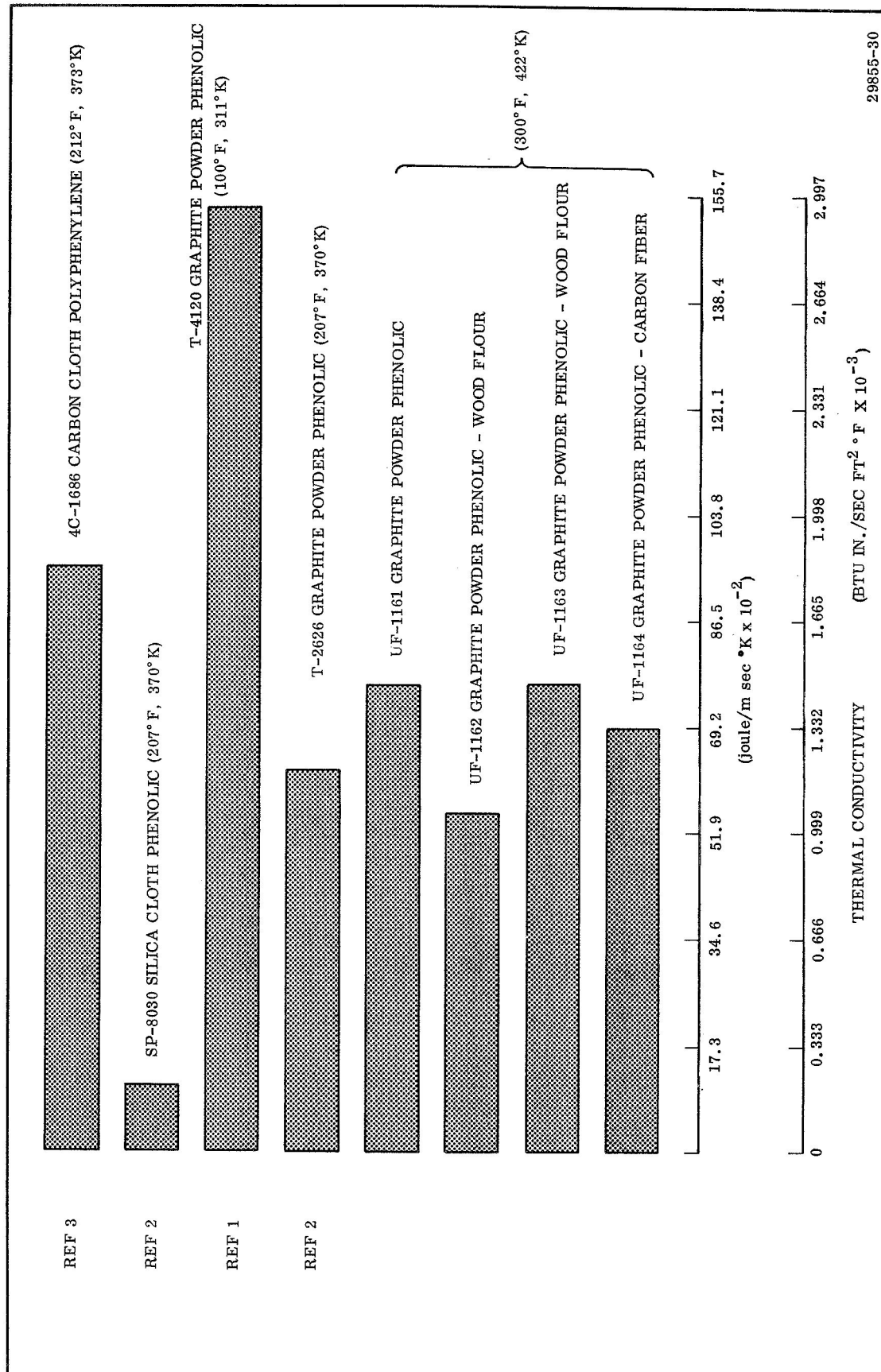


Figure 107. - Liner Material Thermal Conductivity

Five graphite powder phenolic materials (UF-1161, UF-1162, UF-1163, UF-1164, and T-2626) are bracketed by the 4C-1686 carbon polyphenylene on the high side and silica phenolic SP-8030 on the low side. The seventh graphite powder, T-4120, has higher thermal conductivity than the carbon and would be acceptable only with a backup insulation liner to maintain a room temperature metal structural shell.

Since the liner design thickness is proportional to thermal conductivity, the thinnest graphite powder phenolic liner would be UF-1162 with the lowest thermal conductivity.

The ranking of the materials for the lowest thermal conductivity is:

1. SP-8030 (silica)
2. UF-1162 (graphite powder)
3. T-2626 (graphite powder)

Ultimate Compressive Strength

The ultimate compressive strength and Young's modulus in compression for the six graphite powder phenolic and the two carbon and silica liners are shown in Figure 108.

Only two graphite powder phenolic materials (UF-1162 and UF-1161) equaled or exceeded the minimum 4C-1686 (carbon) liner ultimate compressive strength of 13,000 psi. However, the Young's modulus for all the graphite powder phenolics exceeded the normal Young's modulus of $2 \text{ to } 3 \times 10^6 \text{ psi}$ ($13.8 \text{ to } 20.7 \text{ N/m}^2 \times 10^9$) for typical silica and carbon cloth materials. For the specific silica (SP-8030) and carbon (4C-1686) cloth prepreg materials, no Young's compression modulus data were available from Thiokol laboratory test specimens.

Since the liner design thickness is proportional to $1/\text{ultimate compressive strength}$, the thinnest graphite powder phenolic liner would be UF-1162 with the highest ultimate compressive strength. A more complete thermal stress analysis considering the modulus, the Poisson's ratio, and coefficient of thermal expansion would be required for final verification of the material selection and liner thickness requirement.

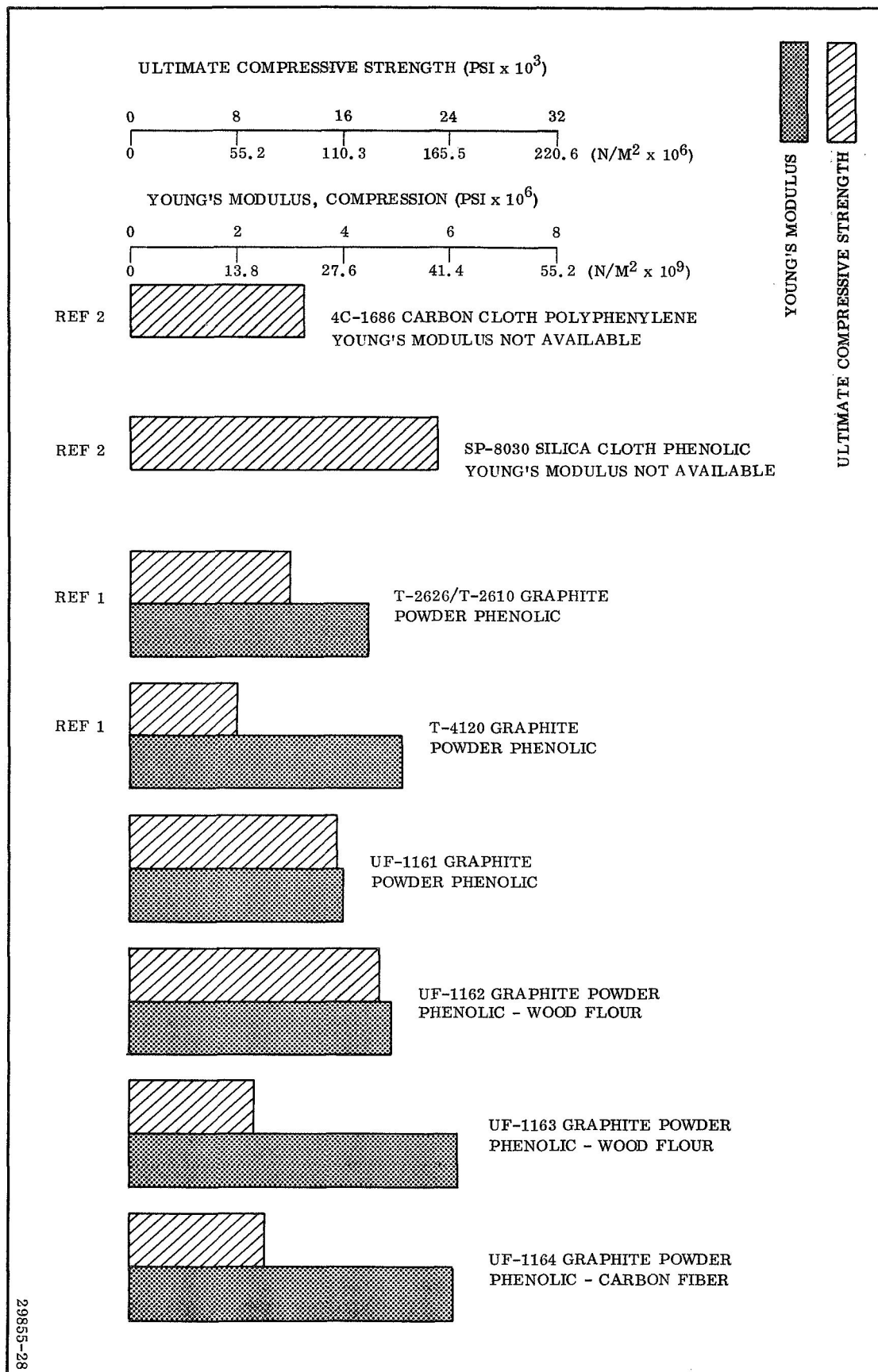


Figure 108. - Liner Material Ultimate Compressive Strength and Young's Modulus at Room Temperature (With Grain or With Ply)

The ranking of the materials for the highest ultimate compressive strength is:

1. SP-8030 (silica cloth)
2. UF-1162 (graphite powder)
3. UF-1161 (graphite powder)

Ultimate Tensile Strength

The ultimate tensile strength and Young's modulus in tension for the graphite powder phenolics and the two carbon and silica cloth liners is shown in Figure 109.

The graphite powder phenolic materials did not meet the minimum silica phenolic ultimate tensile strength of 6,300 psi ($43.47 \text{ N/m}^2 \times 10^6$). In addition, the graphite powder phenolic materials did not meet the carbon liner minimum Young's modulus of 1.85×10^6 psi ($12.7 \text{ N/m}^2 \times 10^9$).

Since the liner design thickness is proportional to $1/\text{ultimate tensile strength}$, the graphite powder liner thickness would be larger than the carbon and silica cloth liners. A more complete thermal stress analysis considering Young's modulus, Poisson's ratio, and coefficient of thermal expansion would be required for final verification of the best graphite phenolic material and its liner thickness.

The ranking of the materials for the highest ultimate tensile strength is:

1. 4C-1686 (carbon)
2. SP-8030 (silica)
3. UF-1161 (graphite powder)
4. UF-1162 (graphite powder)

Coefficient of Thermal Expansion

The coefficient of thermal expansions (CTE), with and against grain or ply, for the eight liner materials is shown in Figure 110.

Ideally, the best liner material shows low and uniform CTE in all planes. For the carbon cloth liner, the maximum CTE is $9.0 \text{ in./in.} \cdot ^\circ\text{F} \times 10^{-6}$ ($10.3 \text{ m/m} \cdot ^\circ\text{K} \times 10^{-6}$) in one plane and $-0.5 \text{ in./in.} \cdot ^\circ\text{F} \times 10^{-6}$ ($-0.6 \text{ m/m} \cdot ^\circ\text{K} \times 10^{-6}$) in the other plane with a maximum change of CTE equal to $9.5 \text{ in./in.} \cdot ^\circ\text{F} \times 10^{-6}$.

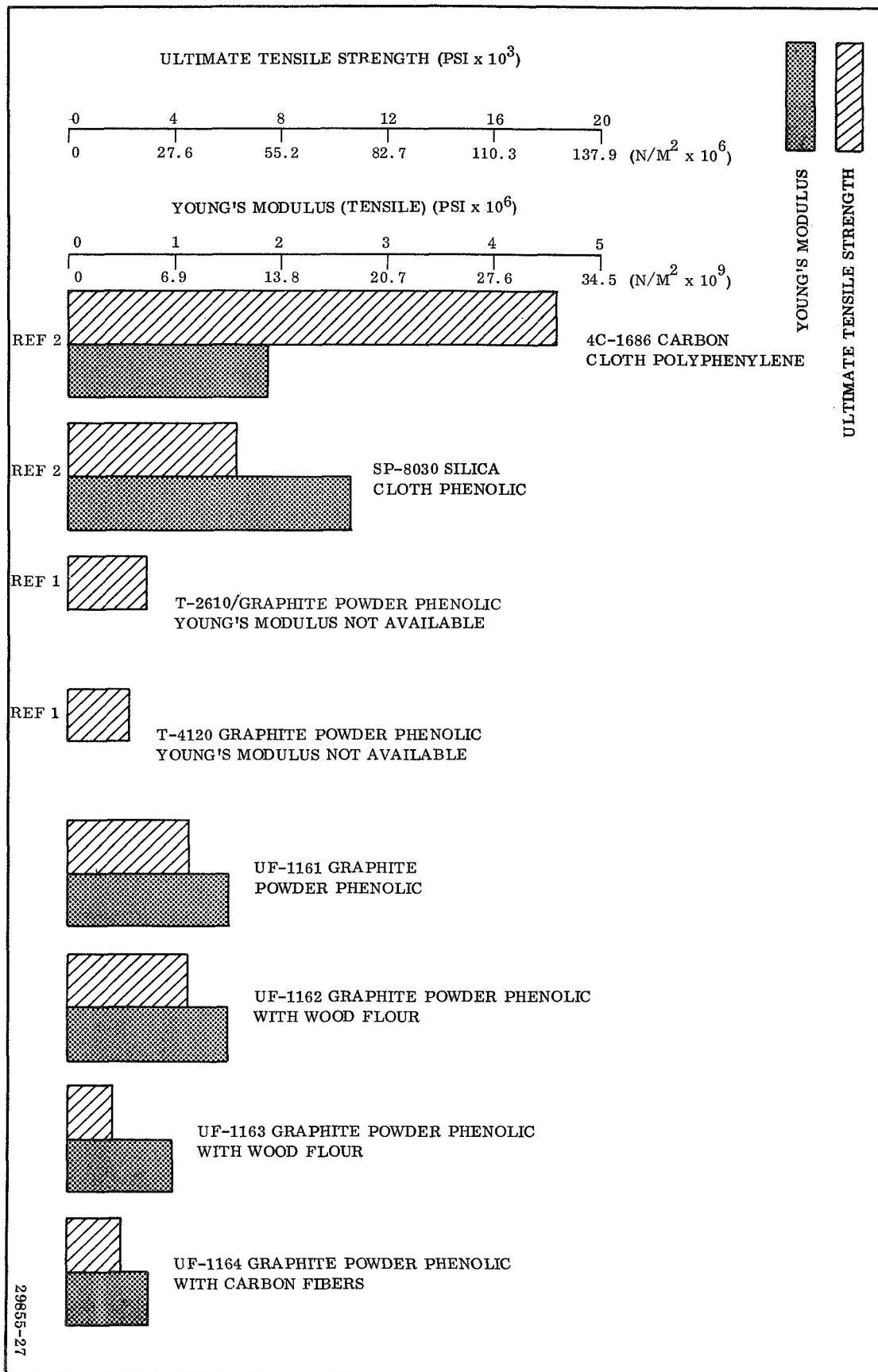


Figure 109. - Liner Material Ultimate Tensile Strength and Young's Modulus at Room Temperature (With Grain or With Ply)

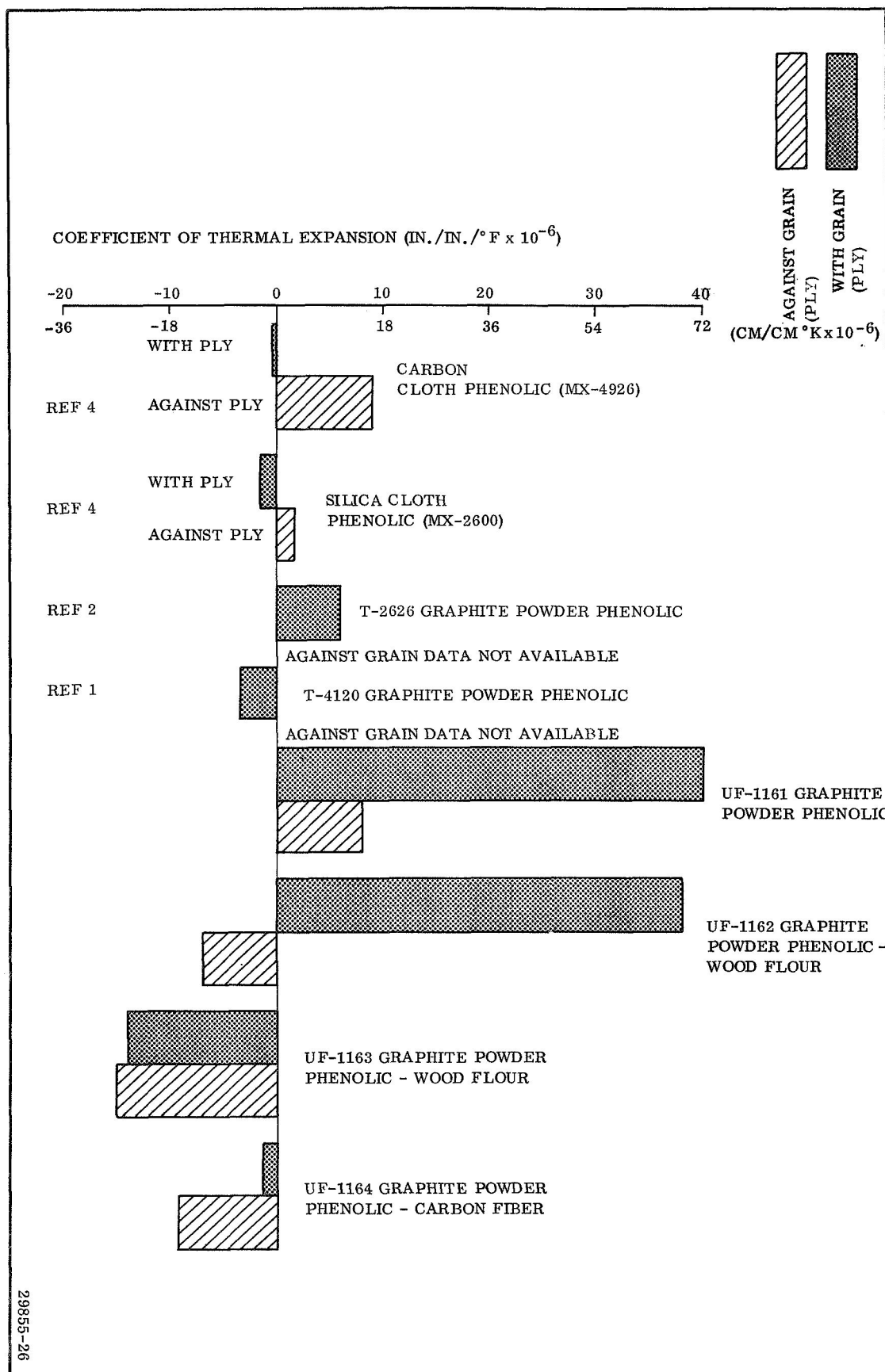


Figure 110. - Liner Material Coefficient of Thermal Expansion, With Grain and Against Grain or With Ply and Against Ply (at 800°F, 700°K)

($10.9 \text{ m/m-}^\circ\text{K} \times 10^{-6}$) between planes (with grain vs against grain). The only graphite powder phenolic material to equal the carbon liner performance with a low and uniform CTE is UF-1164 with a maximum CTE equal to $-9.0 \text{ in./in.-}^\circ\text{F} \times 10^{-6}$ ($-10.3 \text{ m/m-}^\circ\text{K} \times 10^{-6}$) in one plane and a maximum change of CTE equal to $7.8 \text{ in./in.-}^\circ\text{F} \times 10^{-6}$ ($8.9 \text{ m/m-}^\circ\text{K} \times 10^{-6}$) between planes. The T-2610, T-2626, and T-4120 materials are not mentioned since only one plane CTE data are available.

Since the liner design thickness and length are proportional to maximum CTE (one plane) and maximum CTE (between planes) the UF-1164 would be suitable for a long, standard thickness liner application. The UF graphite powder phenolic materials are compared to the carbon phenolic liner below.

	One Plane				Between Planes	
	Maximum at 800°F (700°K) CTE		Minimum at 800°F (700°K) CTE		Maximum at 800°F (700°K) CTE	
	(in./in.-°F x 10 ⁻⁶)	(cm/cm-°K x 10 ⁻⁶)	(in./in.-°F x 10 ⁻⁶)	(cm/cm-°K x 10 ⁻⁶)	(in./in.-°F x 10 ⁻⁶)	(cm/cm-°K x 10 ⁻⁶)
Carbon						
Phenolic	9.0	16.2	-0.5	-0.9	9.5	17.1
UF-1163	-15.0	-27.0	-14.0	-25.2	1.0	1.8
UF-1162	38.0	68.4	-7.0	-12.6	45.0	81.0
UF-1161	40.0	72.0	8.0	14.40	32.0	57.6
UF-1164	-9.0	-16.2	-1.2	-2.16	7.8	14.0

The UF-1163 would also be acceptable for liner design application as thick, long rings or cones, but the UF-1162 and UF-1163 could be best used as thick, short ring washers like bulk graphite and pyrolytic graphite.

The ranking of the liner materials for the lowest differential CTE between planes is:

1. UF-1163 (graphite powder)
2. SP-8030 (silica phenolic)
3. UF-1164 (graphite powder)

Structural Integrity and Ease of Fabrication

The structural integrity and ease of fabrication index for the nine standard and low cost ablative materials are shown in Figure 111. The T-2610 and T-2626 are assumed equal for this evaluation.

For structural integrity, the graphite powder phenolics all rated good to very good, while the carbon and silica cloth liners all rated excellent. Since the liner thickness is proportional to 1/structural integrity, the graphite powder phenolics would need a thicker ablative liner due to its lower structural integrity.

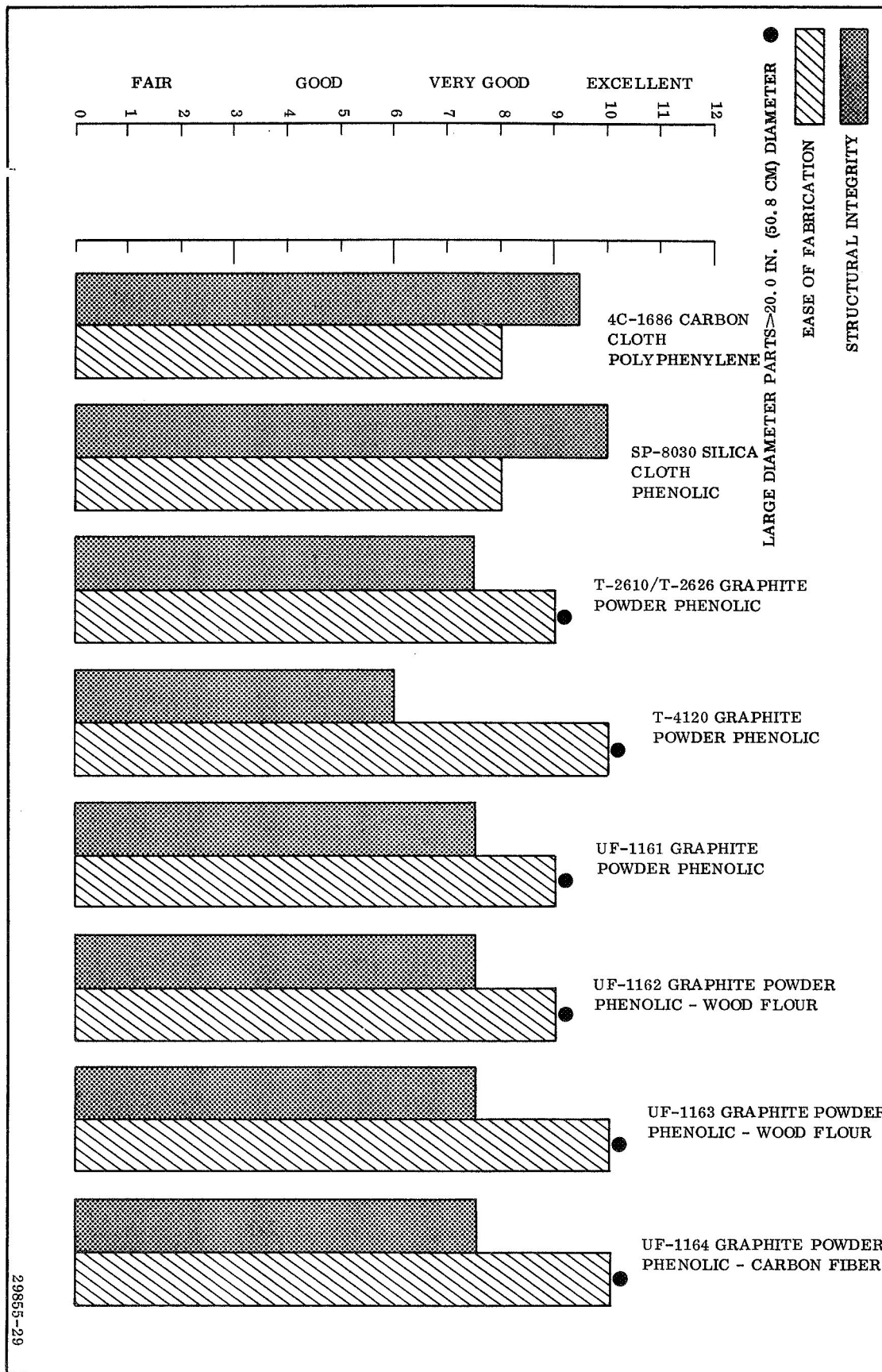


Figure 111. - TU-622 Liner Material Structural Integrity and Ease of Fabrication

In ease of fabrication, the graphite powder phenolics all rated excellent compared to the very good rating of the silica and carbon cloth liners. Since the liner cost is proportional to ease of fabrication, the graphite powder phenolics will exhibit a lower processing cost with mixing and autoclave press cure cycles than the standard materials with tape wrapping and hydroclave - autoclave cure cycles.

Further data is needed with graphite powder phenolics at diameters >20.00 in. (0.508 m) to evaluate structural integrity and ease of fabrication. Preliminary Minuteman Third Stage static firing diffuser tests with diameters of 30 to 37 in. (0.76 to 1.0 m) indicate that T-4120, UF-1163, and UF-1164 would be easier to fabricate than the T-4610, UF-1161, and UF-1162 materials at the larger diameters. This is a reverse of the ease of fabrication rating at the lower (<20.00 in. or 0.508 m) diameters where T-2610/T-2626, UF-1161, and UF-1162 are the best.

The ranking of the liner materials for the highest structural integrity and highest ease of fabrication are:

Structural Integrity

1. SP-8030 (silica)
2. 4C-1686 (carbon)
3. T-2610, UF-1161, UF-1162, UF-1163, UF-1164

Ease of Fabrication

1. T-4120, UF-1163 and UF-1164 (graphite powder)
2. UF-1161 and UF-1162 (graphite powder)
3. 4C-1686 (carbon) and SP-8030 (silica)

To evaluate and compare the nine liner materials, the ten material characteristics were used. Thus when evaluating each material for the ten characteristics, the best material received a maximum rating of 10, and the poorest material received a minimum rating of 4. The remaining materials received an intermediate rating number. The maximum sum of all rating numbers thus could not exceed 100.0.

To show the priority of some material characteristics over others for the three major areas of the nozzle liner, weighting factors were used as shown in Table XXVIII. Thus, in the inlet, low raw material cost, erosion rate, high structural integrity, and ease of fabrication were considered most important. For the throat, low erosion rate and high structural integrity were considered paramount. For each nozzle area, the total weighting factors must equal 10.

TABLE XXVIII. - WEIGHTING FACTORS

	<u>Inlet</u>	<u>Throat</u>	<u>Exit Cone</u>
1. Low specific gravity	1.00	0.50	1.50
2. Low raw material cost	1.50	0.50	1.50
3. Low erosion rate	1.50	2.00	1.00
4. Low char rate	1.00	1.00	1.00
5. Low thermal conductivity	0.50	1.00	0.50
6. High ultimate compressive strength	0.50	1.00	0.50
7. High ultimate tensile strength	0.50	1.00	0.50
8. Uniform coefficient of thermal expansion	0.50	0.50	0.50
9. High structural integrity	1.50	2.00	1.50
10. High ease of fabrication	<u>1.50</u>	<u>0.50</u>	<u>1.50</u>
Total	10.00	10.00	10.00

The use of the rating numbers and weighting factors to evaluate an inlet material is shown below:

<u>INLET MATERIAL I</u>			
<u>Material Characteristics</u>	<u>Rating Number</u>	<u>Weighting Factor</u>	<u>Weighted Rating Number</u>
A	10	1.0	10.0
B	4	1.5	6.0
C	5	1.5	7.5
D	7	1.00	7.0
E	6	0.50	3.0
F	4	0.50	2.0
G	10	0.50	5.0
H	8	0.50	4.0
I	7	1.50	10.5
J	<u>4</u>	<u>1.50</u>	<u>6.0</u>
Totals	65	10.00	56.0

Theoretically, the most perfect material for each nozzle area would receive a weighted rating number of 100.00.

Nozzle Inlet Area

For example in the nozzle inlet area, the nine liner materials were given weighted ratings as shown in Table XXIX for large diameter nozzles, 20.0 in. (0.508 m) diameter or larger.

All the graphite powder phenolic materials were rated as comparable to the carbon and silica cloth liners. Two graphite phenolic materials, T-2610/T-2626 and T-4120, missed one rating but were shown with nine characteristic ratings, plus a minimum and maximum rating for the missing tenth characteristic.

The best material was the graphite powder UF-1164. The second and third best materials were UF-1163 graphite powder and 4C-1686 carbon cloth. It is interesting to note that the newly developed UF-1163 and UF-1164 were rated superior to the current T-4120, T-2610, and T-2626 graphite powder.

Nozzle Throat Area

For the nozzle throat area, the nine liner materials were weight rated as shown in Table XXX for large diameter nozzles.

The best material was the standard carbon cloth phenolic (4C-1686), followed by standard silica cloth SP-8030 and UF-1161 graphite powder phenolic in second and third place. Two other graphite powder phenolics, UF-1164 and T-2610, were rated close to the UF-1161 graphite powder.

TABLE XXIX. - INLET MATERIAL SELECTION

Material Selection Parameter	Weighting Factor	4C-1686 Carbon Cloth	SP-8030 Silica Cloth	T-2626/T-2610 Graphite Powder Phenolic	T-4120 Graphite Powder Phenolic	UF-1161 Graphite Powder Phenolic	UF-1162 Graphite Phenolic Powder Wood Flour	UF-1163 Graphite Phenolic Powder Wood Flour	UF-1164 Graphite Phenolic Powder Carbon Fibers
		<u>Initial</u> <u>Adjusted</u>	<u>Initial</u> <u>Adjusted</u>	<u>Initial</u> <u>Adjusted</u>	<u>Initial</u> <u>Adjusted</u>	<u>Initial</u> <u>Adjusted</u>	<u>Initial</u> <u>Adjusted</u>	<u>Initial</u> <u>Adjusted</u>	<u>Initial</u> <u>Adjusted</u>
1. Low specific gravity	1.00	10 10.0	7 7.0	4 4.0	8.5 8.5	8 8.0	7 7.0	6.5 6.5	6.0 6.0
2. Low raw material cost	1.50	4 6.0	6 9.0	10 15.0	10 15.0	10 15.0	10 15.0	10 15.0	9 13.5
3. Low erosion rate	1.50	10 15.0	4 6.0	10 15.0	9.5 13.8	10 15.0	7 10.5	8 12.0	9.5 13.8
4. Low char rate	1.00	10 10.0	6.5 6.5	5.5 5.5	5.5 5.5	4.5 4.5	4 4.0	6.5 6.5	9.0 9.0
5. Low thermal conductivity	0.50	6.5 3.3	10 5.0	8 4.0	4.0 2.0	7.5 3.8	8 4.0	7.5 3.8	7.5 3.8
6. High ultimate compressive strength	0.50	6 3.0	10 5.0	5.5 2.8	4 2.0	7.0 3.5	8 4.0	4 2.0	4.5 2.3
7. High ultimate tensile strength	0.50	10 5.0	5.5 2.8	4.5 2.3	4 2.0	5 2.3	5 2.5	4 2.0	4 2.0
8. Uniform coefficient of thermal expansion	0.50	9 4.5	10 5.0	N/A* N/A*	N/A* N/A*	6 3.0	4 2.0	10 5.0	9 4.5
9. High structural integrity	1.50	9 13.5	10 15.0	6 9.0	4 6.0	6 9.0	6 9.0	6 9.0	6 9.0
10. High ease of fabrication	1.50	4 6.0	4 6.0	7 10.5	10 15.0	7 10.5	7 10.5	10 15.0	10 15.0
		76.3	67.3	68.1	69.8	74.6	68.5	76.8	78.9
		(3)		+ $\begin{cases} 2.0 \text{ Min} \\ 5.0 \text{ Max} \end{cases}$	+ $\begin{cases} 2.0 \text{ Min} \\ 5.0 \text{ Max} \end{cases}$			(2)	(1)

*Data not available. The maximum rating of 10 and the minimum rating of 4 have been multiplied by the weighting factor to indicate possible values.

TABLE XXX. - THROAT MATERIAL SELECTION

Material Selection Parameter	Weighting Factor	4C-1686 Carbon Cloth		SP-8030 Silica Cloth		T-2626/T-2610* Graphite Powder Phenolic		T-4120 Graphite Powder Phenolic		UF-1161 Graphite Powder Phenolic		UF-1162 Graphite Powder Phenolic Wood Flour		UF-1163 Graphite Powder Phenolic Wood Flour		UF-1164 Graphite Powder Phenolic Carbon Fiber	
		Initial	Adjusted	Initial	Adjusted	Initial	Adjusted	Initial	Adjusted	Initial	Adjusted	Initial	Adjusted	Initial	Adjusted	Initial	Adjusted
1. Low Specific Gravity	0.50	10	5.0	7	3.5	4	2.0	8.5	4.3	8	4.0	7	3.5	6.5	3.3	6	3.0
2. Low Raw Material Cost	0.50	4	2.0	6	3.0	10	5.0	10	5.0	10	5.0	10	5.0	10	5.0	9	4.5
3. Low Erosion Rate	2.00	10	20.0	4	8.0	10	20.0	9	18.0	10	20.0	5.5	11.0	7	14.0	9	18.0
4. Low Char Rate	1.00	10	10.0	7	7.0	6.5	6.5	7	7.0	6.5	6.5	4.0	4.0	7	7.0	10	10.0
5. Low Thermal Conductivity	1.00	6.5	6.5	10	10.0	8	8.0	4	4.0	7.5	7.5	8	8.0	7.5	7.5	7.5	7.5
6. High Ultimate Compressive Strength	1.00	6	6.0	10	10.0	5.5	5.5	4	4.0	7	7.0	8	8.0	4	4.0	4.5	4.5
7. High Ultimate Tensile Strength	1.00	10	10.0	5.5	5.5	4.5	4.5	4	4.0	5	5.0	5	5.0	4	4.0	4	4.0
8. Uniform Coefficient of Thermal Expansion	0.90	9	4.5	10	5.0	N/A**	N/A**	N/A**	N/A**	6	3.0	4	2.0	10	5.0	9	4.5
9. High Structural Integrity	2.00	9	18.0	10	20.0	6	12.0	4	8.0	6	12.0	6	12.0	6	12.0	6	12.0
10. High Ease of Fabrication	0.50	4	2.0	4	2.0	7	3.5	10	5.0	7	3.5	7	3.5	10	5.0	10	5.0
			84.0	74.0	74.0	67.0	67.0	59.3	59.3	73.5	73.5	62.0	62.0	66.8	66.8	73.0	73.0
			(1)	(2)	(2)	+ { 2.0 Min 5.0 Max	+ { 2.0 Min 5.0 Max	+ { 2.0 Min 5.0 Max	+ { 2.0 Min 5.0 Max		(3)						

Note: Maximum possible value is 10

*T-2626 and T-2610 were considered as having the same properties.

**Data not available. Maximum value was 10, minimum value was 4, possible maximum and minimum values when multiplied by weighting factors were 5 and 2, respectively.

Two graphite powder phenolic materials (T-4120 and T-2610/T-2626) missed one rating but were shown with nine characteristic ratings plus a minimum and maximum rating for the missing tenth characteristic.

It is interesting to note that the newly developed UF-1161 was rated superior to the current T-2610 graphite phenolic previously used in throat area applications.

Nozzle Exit Area

For the nozzle exit area, the nine liner materials were rated as shown in Table XXXI for large diameter nozzles.

The best material was the standard carbon cloth phenolic (4C-1686), followed by UF-1163 and UF-1164 graphite powder phenolics in second and third place. Two other materials, silica cloth SP-8030 and T-2610 graphite powder, were rated close enough for honorable mention. It is interesting to note that the new graphite powders UF-1163 and UF-1164 were rated higher than the current T-2610 graphite powder.

Two graphite powder phenolic materials, T-2610/T-2626 and T-4120, missed one rating evaluation but were shown with a nine characteristic rating plus a minimum and maximum rating for the missing tenth characteristic.

TABLE XXXI. - EXIT MATERIAL SELECTION

Material Selection Parameter	Weighting Factor	4C-1686 Carbon Cloth		SP-8030 Silica Cloth		T-2626/T-2610* Graphite Powder Phenolic		T-4120 Graphite Powder Phenolic		UF-1161 Graphite Powder Phenolic		UF-1162 Graphite Powder Phenolic		UF-1163 Graphite Powder Phenolic		UF-1164 Graphite Powder Phenolic	
		Initial	Adjusted	Initial	Adjusted	Initial	Adjusted	Initial	Adjusted	Initial	Adjusted	Initial	Adjusted	Initial	Adjusted	Initial	Adjusted
1. Low Specific Gravity	1.50	10	15.0	7	10.5	4	2.0	8.5	4.3	8	4.0	7	3.5	6.5	3.3	6	3.0
2. Low Raw Material Cost	1.50	4	6.0	6	9.0	10	15.0	10	15.0	10	15.0	10	15.0	10	15.0	9	13.5
3. Low Erosion Rate	1.00	10	10.0	4	4.0	10	10.0	9	9.0	8.5	8.5	4.5	4.5	9.0	9.0	10	10.0
4. Low Char Rate	1.00	10	10.0	9	9.0	6.5	6.5	7	7.0	5.5	5.5	4	4.0	9	9.0	9	9.0
5. Low Thermal Conductivity	0.50	6.5	3.3	10	5.0	8	4.0	4	2.0	7.5	3.8	8	4.0	7.5	3.8	7.5	3.8
6. High Ultimate Compressive Strength	0.50	6	3.0	10	5.0	5.5	2.8	4	2.0	7	3.5	8	4.0	4	2.0	4.5	2.3
7. High Ultimate Tensile Strength	0.50	10	5.0	5.5	2.8	4.5	4.3	4	2.0	5	2.3	5	2.5	4	2.0	4	2.0
8. Uniform Coefficient of Thermal Expansion	0.50	9	4.5	10	5.0	N/A**	N/A**	N/A**	N/A**	6	3.0	4	2.0	10	5.0	9	4.5
9. High Structural Integrity	1.50	9	13.5	10	15.0	6	9.0	4	6.0	6	9.0	6	9.0	6	9.0	6	9.0
10. High Ease of Fabrication	1.50	4	6.0	4	6.0	7	10.5	10	15.0	7	10.5	7	10.5	10	15.0	10	15.0
			76.3		71.3		64.1 + { 2.0 Min 5.0 Max		62.3 + { 2.0 Min 5.0 Max		65.1		59.0		73.1		72.1
			(1)												(2)		(3)

Note: Maximum possible value is 10.

*T-2626 and T-2610 were considered as having the same properties.

**Data not available. Maximum value was 10, minimum value was 4, possible maximum and minimum values when multiplied by weighting factors were 5 and 2, respectively.

Material Summary

The best three materials for each of the ten material characteristics are listed below:

Material Characteristic	Materials							
	4C-1686 Carbon	SP-8030 Silica	T-2610/ T-2626	T-4120	UF- 1161	UF- 1162	UF- 1163	UF- 1164
Specific gravity	1	--	--	2	--	--	--	3
Raw material cost	--	--	1	3	2	3	--	--
Erosion rate								
Inlet-throat	1	--	1	3	1	--	--	2
Throat-exit	1	--	2	3	3	--	--	2
Char rate								
Inlet-throat	1	3	--	--	--	--	3	2
Throat-exit	1	3	--	--	--	--	3	2
Thermal conductivity	--	1	3	--	--	2	--	--
Ultimate compres- sive strength	--	1	--	--	3	2	--	--
Ultimate tensile strength	1	2	--	--	3	--	--	--
Coefficient of thermal expansion	--	2	N/A	N/A	--	--	1	3
Structural integrity	2	1	3	--	3	3	3	3
Ease of fabrication	3	3	2	1	2	2	1	1

The carbon and silica were the best materials, followed closely by the graphite powder phenolics: UF-1164, UF-1161, and T-2610/T-2626. The new graphite powder phenolics, UF-1161 and UF-1164, appear to be slightly better than the current T-2610/T-2626 and much better than the current T-4120.

For the three nozzle areas, the three best materials are listed below:

Nozzle Area	Materials							
	4C-1686 Carbon	SP-8030 Silica	T-2610/ T-2626	T-4120	UF- 1161	UF- 1162	UF- 1163	UF- 1164
Inlet	3	--	--	--	--	--	2	1
Throat	1	2	--	--	3	--	--	--
Exit	1	--	--	--	--	--	2	3

Normally, for the inlet and exit areas of large nozzles, a combination of carbon and silica cloth is used. In the throat area, carbon cloth is used exclusively.

Carbon cloth is the best material for two of the three nozzle areas, but the high cost of the raw material (\$24/lb) necessitates the use of alternate materials for large nozzles. At least one of the graphite powder phenolics is a first, second, or third choice and could be used if a higher risk and thicker liners are acceptable for the advantages of lower raw material and processing cost, ease of fabrication, and equivalent erosion rates.

Thus, graphite powders could be used to replace the carbon and silica cloth phenolic, resulting in an average 75 percent raw material cost savings, thicker liners, and a corresponding average nozzle weight increase of 30 percent. The best graphite powder for each nozzle area and its best shape application are listed below:

Inlet	UF-1164	Long cones
Throat	UF-1161	Short rings
Exit	UF-1163	Long cones

A graphite powder phenolic test application nozzle for future development effort is shown in Figure 112 with a flex seal. The UF-1164 was applied to the submerged liner and inlet, with UF-1161 in the throat and UF-1163 for the exit. Canvas phenolic is used to protect the flex bearing.

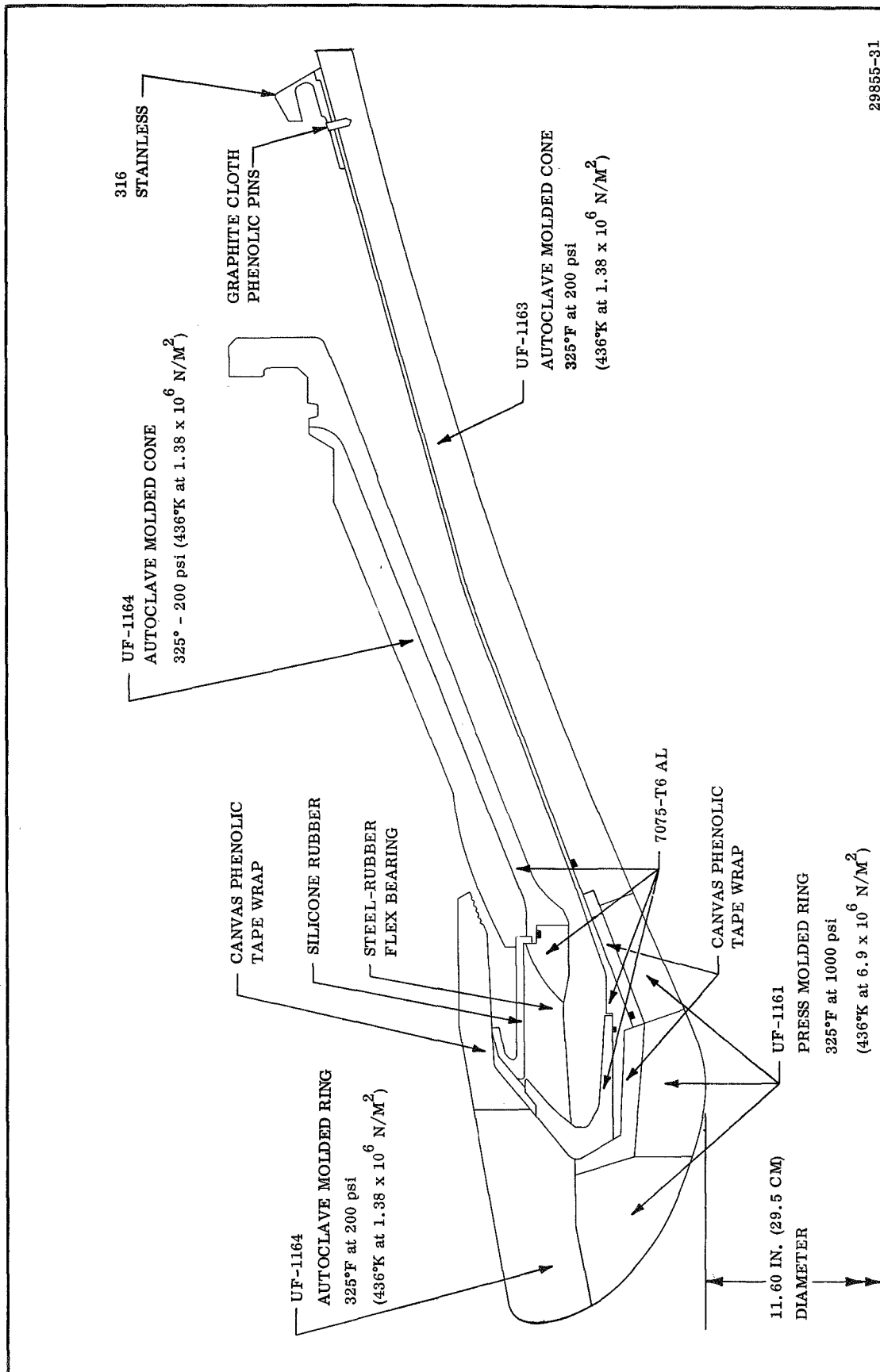


Figure 112. - Graphite Powder Phenolic Test Application Nozzle

CONCLUSIONS AND RECOMMENDATIONS

The conclusions and recommendations are listed by task followed by some general comments relative to the entire program.

Conclusions

Task 1. -

1. Monsanto RI-4009 was the best powdered phenolic resin and also the best two stage resin.
2. Durez 10694 was selected over the remaining phenolics and the polyphenylene resin because it produced molding compounds which could be cast or troweled without staging the material.

Task 2. -

1. The fibers were very difficult to mix with the phenolic graphite compounds.
2. In general, the fibers did increase the strength of the compounds and also lowered the thermal conductivity.
3. The 0.25 in. (0.64 cm) chopped carbon fiber provided the best physical properties improvements. However, some of the disadvantages outweighed the advantages for some resin-graphite formulations.

Task 3. -

1. The wood flour was the best of the filler materials from the standpoint of improving the physical strength and decreasing the thermal conductivity without impairing the erosion resistance of the compounds.
2. Increasing the percent of liquid resin Durez 10694 from 25 to 40 percent and adding wood flour as a filler produced an improved compound.

General Conclusions. -

1. The four formulations developed in this program, UF-1161 thru UF-1164, compare favorably with silica and carbon cloth prepregs in rocket nozzle components in several areas.
 - a. Specific gravity - carbon cloth is lowest.
 - b. Raw material cost - new UF compounds are lowest.
 - c. Throat-inlet erosion rate - UF compounds are lower than silica and carbon cloth.
 - d. Throat-exit erosion rate - carbon cloth is lowest.
 - e. Char rate throat-inlet - carbon cloth is lowest.
 - f. Char rate throat-exit - carbon cloth is lowest.
 - g. Thermal conductivity - silica cloth is lowest.
 - h. Compressive strength - silica cloth is highest.
 - i. Tensile strength - carbon cloth is highest.
 - j. Coefficient of thermal expansion - Silica cloth is lowest. Differential two plane CTE - UF compounds are lowest.
 - k. Structural integrity - silica is best.
 - l. Ease of fabrication - UF compounds are best.
2. The new UF formulations are improvements over the original low cost carbonaceous materials.

Recommendations

It is recommended that the following suggestions be considered to provide a higher degree of reliable data in a more realistic flight type design:

1. Test the standard carbon and silica cloth prepregs and the best of the new graphite powder phenolic compounds in a larger flight type nozzle on some standard test motor.

2. Evaluate the thermal and mechanical properties of carbon and silica cloth and the best new graphite powder phenolic compound in both virgin and charred conditions to higher test temperatures approximating chamber gas temperature.
3. Document, classify, and total the processing costs (raw material, forming, tooling, curing and shaping) for each of these materials: carbon and silica cloth, and the better graphite powder phenolic materials.

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