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# **REFRACTORY COMPOSITE MATERIALS** for SPACECRAFT THRUST CHAMBERS

# FINAL REPORT for THE PERIOD 23 JULY 1968 to 22 FEBRUARY 1970

CONTRACT: NAS7-555 MOD-3 & 4 Subcontract 700149 (Raytheon)

PROJECT 5033

# REFRACTORY COMPOSITE MATERIALS for SPACECRAFT THRUST CHAMBERS

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> CONTRACT: NAS7-555 MOD-3 & 4 SUBCONTRACT 700149 (RAYTHEON)

> > PROJECT 5033

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#### FOREWORD

This report covers Phase III and Phase IV work performed by The Marquardt Company under Prime Contract NAS7-555, Modifications 3 and 4, and by the Research Division of the Raytheon Company under Marquardt Subcontract 700149, during the period 23 July 1968 to 22 February 1970. This program was a continuation of work reported in Marquardt Report 6142 on Phase I and Phase II investigations of pyrolytic refractory composites.

The work was sponsored by NASA-OART, Washington, D.C. The NASA Technical Manager was Mr. William Tyler (JPL).

The Program Manager, Mr. C. D. Coulbert (Marquardt), was responsible for the overall program direction and supervision. Dr. J. S. Waugh (Raytheon) was responsible for the technical supervision of the Raytheon subcontract. Mr. J. G. Campbell (Marquardt) was responsible for all the theoretical thermal, stress, thermodynamic and chemical analyses.

Mr. M. S. Wilson (Marquardt) performed heat transfer and thermodynamic analyses and oxidation testing. Mr. M. L. Haas (Marquardt) performed stress analysis and established analysis methods for multilamina composites.

Report S-989

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# TABLE of CONTENTS

SECTION	TITLE	PAGE
I	SUMMARY	1
Π	INTRODUCTION	2
ш	PYROREFRACTORY ALLOY FABRICATION UNDER RAYTHEON SUBCONTRACT	5
	<ul> <li>A. Multilamina Thrust Chambers</li> <li>B. Pyrocarbide/Carbon Felt Composites</li> <li>C. Monolithic Deposits</li> </ul>	5 9 11
IV	MATERIALS CHARACTERIZATION	12
	<ul> <li>A. Pyrolytic Graphite/Carbitex Composites</li> <li>B. Thornel Reinforced Pyrolytic Graphite</li> <li>C. Vitreous Carbon</li> <li>D. Oxidation Tests</li> <li>E. Copper Impregnated POCO Graphite</li> </ul>	12 14 18 19 20
V	THRUST CHAMBER DESIGN and FABRICATION	21
	<ul> <li>A. Thrust Chamber Designs</li> <li>B. Thrust Chamber Fabrication and Evaluation</li> <li>C. Metal-Carbon Bonding</li> </ul>	22 23 26
VI	TEST FIRING WITH FLOX/METHANE	30
	<ul> <li>A. Test Period I</li> <li>B. Test Period II</li> <li>C. Test Period III</li> </ul>	31 32 34
vп	STRESS ANALYSIS	36
	A. Stress Analysis Program	37
VIII	THERMAL and THERMOCHEMICAL ANALYSIS	37
	<ul> <li>A. Heat Transfer Analysis</li> <li>B. Thermochemical Analysis</li> <li>C. Thrust Chamber Concepts</li> </ul>	38 40 44
IX	CONCLUSIONS	48
X	REFERENCES	49
6.000 6740 Film	DISTRIBUTION	151

### Report S-989

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# LIST of ILLUSTRATIONS

FIGURE	TITLE			
1	Free Standing Pyrolytic Chamber History	81		
2	Layouts of Structural Thrust Chambers for Pyrobond Fabrication Studies	82		
3	Raytheon Multilamina Chambers	83		
4	Chamber MRS-50	84		
5	Schematic Diagram of Filament Orientation in a Carbitex Thrust Chamber	85		
6	Dimensions of Pyrolytic Graphite/Carbitex Composite Test Cones	86		
7	Pyrolytic Graphite/Carbitex Test Cones	87		
8	Fixture for Pressurization of Test Cones	88		
9	Variation of Circumferential Modulus of Elasticity of Carbitex with Wrap Angle	89		
10	Variation of Axial Modulus of Elasticity of Carbitex with Wrap Angle	90		
11	Variation of Axial Strength of Carbitex Cones with Wrap Angle	91		
12	Dimensions of PG/Thornel Test Sample Spool	92		
13	PG/Thornel Test Sample Spools	93		
14	PG/Thornel NOL Ring Specimens for Tensile Tests	94		
15	Results of Tensile Tests of PG/Thornel NOL Ring Specimens	95		
16	Microstructure of PG/Thornel Ring Specimen 1C	96		
17	Microstructure of PG/Thornel Ring Specimen 2C	97		
18	Microstructure of PG/Thornel Ring Specimen 3C	98		
19	Microstructure of PG/Thornel Ring Specimen 4C	99		
20	Photomicrographs of Representative Sections of PG Infiltrated Thornel, Spools Nos. 1 and 2	100		
21	Photomicrographs of Representative Sections of PG Infiltrated Thornel, Spools Nos. 3 and 4	101		
22	PG/Thornel/PG Chamber	102		
23	PG Infiltrated Thornel-50 Carbon Yarn	103		
24	PG/Thornel/PG Tensile Test Apparatus	104		
25	PG/Thornel/PG Tensile Test Rings after Failure	105		

# List of Illustrations - (Continued)

Figure	Title			
26	Carbon Materials Erosion Rate - Oxyacetylene Flame in Air	106		
27	Loss of Copper from AXF-5QC	107		
28	Copper Impregnated Graphite After Cyclic Heating	108		
29	Composite Chamber, FLOX/LPG	109		
30	POCO Graphite Heat Sink Thrust Chambers	110		
31	Uncooled Copper Adapter Section for Testing Coated Tantalum-Tungsten Chambers	111		
32	Copper Impregnated POCO Chamber Segment	112		
33	100 Pound Thrust Carbitex 713 Chambers	113		
34	PG/Carbitex Thrust Chamber with RPG Nozzle Extension	114		
35	X-Ray Photograph of PG/Carbitex Thrust Chamber with RPG Nozzle Extension	115		
36	100 Pound Thrust FLOX/LPG Engine Assembly	116		
37	PG/Carbitex 100 Pound Thrust Sea Level Chamber with Coating on the Inside Only	117		
38	PG Liners Used for PG/Thornel Composite Altitude Thrust Chambers	118		
39	Thornel Wound Sea Level Chamber before PG Infiltration	119		
40	Thornel Wound Altitude Chamber before PG Infiltration	120		
41	Filament Winding Setup for PG/Thornel Thrust Chamber	121		
42	Electrodeposited Nickel on RPG/PG Cylinders before Machining	122		
43	Electrodeposited Nickel on RPG/PG Cylinder after Machining	123		
44	Test Specimen Used for Thermal Cycling Test of Bond of Electrodeposited Nickel on RPG	124		
45	Setup for Thermal Cycling Test of Electrodeposited Nickel on RPG	125		
46	Typical Cycles of Thermal Cycling Test of Electrodeposited Nickel on RPG	126		
47	Photomicrographs of Plasma Sprayed Nickel - RPG Interface	127		
48	Final Configuration of the 100 Pound Thrust FLOX/Methane Injector, S/N 002	128		
49	Final Configuration, S/N 002 Injector	129		
50	100 Pound Thrust FLOX/Methane Engine	130		
51	Erosion Pattern of PG Chamber S/N 006 after Test Run 45	131		

4

# THE Harquardt VAN NUYS, CALIFORNIA

# List of Illustrations - (Continued)

Figure	Title	Page
52	Carbon Deposition in Sea Level PG Streak Chamber after 20 Seconds Firing	132
53	Water Flow Test of a Modified Injector Showing Film Spreading in a Lucite Chamber, $\Delta P = 80 PSI$	133
54	Spray Pattern from Cold Flow Tests of the 100 Pound Thrust FLOX/Methane Injector with 18 Fuel Film Holes	134
55	Layout of 100 Pound Thrust PG/Carbitex Altitude Chamber with Nozzle Extension	135
56	PG/Carbitex Chamber with RPG Nozzle Extension after 322 Seconds Firing	136
57	HfC Coated Tantalum-Tungsten Chamber	137
58	Predicted Transient Temperature Distribution in a ZrC Composite Fired with FLOX/Methane	138
59	Inner Surface Stresses in a ZrC Composite Chamber Fired with FLOX/Methane	139
60	Outer Surface Stresses in a ZrC Composite Chamber Fired with FLOX/Methane	140
61	1K Chamber Geometry for Thermal Analysis	141
62	Combustion Temperature of $OF_2/B_2H_6$	142
63	Chamber Heating Rate	143
64	Sensible Heat Capacity - Vs - Supply Temperature	144
65	B <sub>2</sub> H <sub>6</sub> Cooling Capacity	145
66	Enthalpy of Gaseous Diborane	146
67	Equilibrium Chamber Wall Temperature	147
68	Combustion Products of $OF_2/B_2H_6$	148
69	Heat Transfer Mechanisms in an Endothermically Cooled Pyrolytic Graphite Nozzle	149
70	Composite Materials Chamber	150

THE Alarquardt van NUYS, CALIFORNIA

TABLE

# LIST OF TABLES

<u>FABLE</u>	TITLE	PAGE
I	Explanatory Notes for Figure 1	51
п	Thrust Chamber Process Data	53
ш	Thrust Chamber Physical Property Data	54
IV	Thickness Profile	55
v	Process Data for Felt Impregnation	59
VI	Average Physical Properties	59
VII	Local Density of Run No. MQF-5	59
VШ	Flexure Strength	60
IX	Process Data – Monolithic Deposits	60
х	Results of NOL Ring Tensile Tests of PG/Thornel - 50	61
XI	Summary of the Test Period II Injector Evaluation Firings	62
хп	Summary of Erosion of Three Free Standing PG Chambers and One POCO Graphite Chamber	64
XIII	Test Period II – Summary of Ambient Thrust Chamber Firing Tests in Magic Mountain Cell M-2	65
XIV	Summary of Average Thrust Chamber Throat Erosion Data, Test Period II, December 1968 to February 1969	70
XV	Extended Range Film Temperature Data, Phase II Tests	71
XVI	Test Period III – 100 Pound Thrust FLOX/Methane Engine Test Data	72
XVII	Summary of Stresses in ZrC/PG Composite Thrust Chambers	74
XVIII	Throat Heating Rates - $OF_2/B_2H_6$	75
XIX	Combustion Products of $OF_2/B_2H_6$	76
XX	Graphite Equilibrium with $OF_2/B_2^H_6$	77
XXI	$B_{A}C$ Equilibrium with $OF_{A}/B_{A}H_{c}$	78
XXII	$\tilde{\operatorname{Boron}}$ Nitride Equilibrium with $\operatorname{OF}_2/\operatorname{B_2H}_6$	79
XXIII	Tungsten Equilibrium with $OF_2^{/B}B_2^{H}6$	80

THE Jarquardt VAN NUYS, CALIFORNIA

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#### REFRACTORY COMPOSITE MATERIALS FOR SPACECRAFT THRUST CHAMBERS

#### SECTION I SUMMARY

Pyrolytic refractory materials using vapor deposition techniques and combined in composites with new carbon fiber materials have demonstrated a unique potential for high temperature structural service in a severe rocket thrust chamber environment. A family of these pyrolytically bonded (Pyrobond) materials has been formulated which combines the erosion resistance of a pyrolytic refractory thrust chamber inner wall with the high strength and fabrication flexibility of a carbon fiber composite structure.

These material systems were evaluated theoretically and experimentally in structural and hot firing tests with FLOX/methane propellants. A total of nine different 100-lb thrust refractory composite chambers, plus a hafnium carbide coated tantalum-10 tungsten chamber, seven POCO heavy wall graphite chambers, and six free standing pyrolytic graphite chambers were test fired with FLOX/methane propellants at 100 psia. A total of 2800 seconds of hot firing was accumulated during three test periods. Combustion efficiencies between 89 and 98 percent were attained as a function of chamber L\* values between 10 inches and 18 inches. During these tests, both chamber erosion and carbon deposition were evaluated locally in the chamber from the injector to the nozzle throat as a function of fuel film flow, wall material, O/F, L\*, and duty cycle.

Two of the most promising structural materials evaluated were the PG/Carb-I-tex and PG/Thornel composites. The PG/Carb-I-tex system has been incorporated into thrust chamber designs ranging from 25 to 3000 pounds thrust for evaluation in at least four fluorinated propellant combinations. Test specimens of the PG/Thornel composites have demonstrated tensile strengths in the 50,000 to 100,000 psi range for different filament orientations.

New strength and modulus data were obtained on Carbitex filament wound components and are presented as a function of fabrication parameters. Axial strength of Carbitex is from 400 to 2000 psi, depending on wrap angle.

In studies of forming metal-to-carbon joints with carbon fiber composites, both plasma spraying and electrodeposition of nickel appear to be promising techniques for attachment of carbon composites to metal injector components.

Thermochemical and heat transfer studies were made to define the severity and composition of the thrust chamber environment in a 1000-lb thrust  $OF_2/B_2H_6$  rocket engine. Heat transfer analyses were made of heating rates with  $OF_2/B_2H_6$  and compared with the results of other investigators. The chemical composition of the combustion gases



were used in a thermodynamic equilibrium analysis using various chamber wall materials including graphite, boron carbide, boron nitride and tungsten to evaluate chemical compatibility as a function of temperature.

On the basis of these thermochemical studies, it was concluded that tungsten is the least reactive material with the combustion products of  $OF_2/B_2H_6$ . The feasibility of using graphite was found to be dependent on rates of reaction which must be determined by engine test firings, since heterogeneous reaction rates cannot be predicted theoretically. It was also concluded that boron nitride might have less chemical reaction with the combustion products of  $OF_2/B_2H_6$  than graphite up to a temperature of 4,000°R.

Deposition of an anisotropic form of pyrolytic boron nitride coating on a Carb-I-tex structural wall was not as successful as PG and pyrocarbide coatings due to the larger thermal expansion differences for the boron nitride. However, an isotropic form of pyrolytic boron nitride was successfully deposited on a graphite tube. This form of boron nitride may be of value as a chamber liner for  $OF_2/B_2H_6$ .

### SECTION II INTRODUCTION

This technical report covers work accomplished under Contract NAS7-555, Phases III and IV, on the development and evaluation of pyrolytic refractory thrust chamber materials during the nineteen-month period from August 1958 through February 1970. This program is a continuation of the investigation of improved pyrolytic thrust chamber materials conducted under NASA Contracts NAS7-54, NAS7-262 and NAS7-373 (References 1 through 4). Marquardt Report 6142 (Reference 5) covers the work on Contract NAS7-555 from March 1967 through July 1968 and presents the background and initial developments on the materials work presented in this report.

#### A. <u>Objective</u>

The basic objective of this continuing program has been to provide the necessary technology to design, fabricate and test spacecraft liquid rocket thrust chambers using advanced state-of-the-art pyrolytic refractory materials and composites. The scope of work undertaken in achieving this goal has included:

- 1. New Materials Research To develop new material concepts to achieve strength at high temperature, chemical compatibility and fabrication flexibility.
- 2. Material Characterization To evaluate experimentally, material properties and structural capabilities which are critical to the practical application of these materials in severe rocket thrust chamber environments.



3. Application Studies - To demonstrate by the design, analysis, fabrication and testing of complete thrust chamber assemblies, the feasibility of the material systems which appear the most promising and define problem areas, structural limitations and criteria for improved material and fabrication concepts.

#### B. Related Programs

In earlier work on pyrolytic refractories, thrust chambers were fabricated and tested with  $N_2O_4$ /hydrazine blend earth storable propellant combinations. During this current program, all test firings were conducted with FLOX/CH<sub>4</sub> propellants. However, analytical and design studies included application to both FLOX/CH<sub>4</sub> and OF<sub>2</sub>/B<sub>2</sub>H<sub>6</sub> propellant systems. During concurrent contracted programs at Marquardt (References 6, 7 and 8), pyrolytic refractory composite thrust chambers were designed and fabricated for testing with the following propellant combinations at the indicated thrust levels:

Air Force RPL	3000-1b	CLF <sub>3</sub> /BA1014 (hydrazine blend)
NASA-LeRC	100-1b	FLOX/methane
NASA-JPL	200-1b	$OF_2/B_2H_6$
Air Force RPL	25 <b>-</b> 1b	CLF <sub>5</sub> /Hydrazine blend

The material system used in each of the above thrust chamber designs was PG/ Carb-I-tex, which consists of a pyrolytic graphite chamber inner wall deposited on a structural shell of Carb-I-tex 713, a filament wound graphite yarn composite manufactured by the Carborundum Co. The initial development work on this material system at Marquardt under Phase I and Phase II of Contract NAS7-555 was reported in Reference 5.

The PG/Carb-I-tex system was one of several new thrust chamber fabrication concepts investigated during Phase I and Phase II of this program. Others which were conceived and included in the evaluation program were:

- 1. Multiple lamina pyrolytic carbide alloys including ternary and radially graded compositions.
- 2. High modulus carbon yarn wound over a free standing pyrolytic graphite thrust chamber and pyrolytically infiltrated and bonded.
- 3. Pyrolytic graphite infiltrated carbon felt combined with free standing pyrolytic graphite and filament wound reinforcing.
- 4. The use of vitreous carbon, stacked pyrocarbide washers, high density graphite impregnated with copper, and pyrolytic boron nitride materials in composite structures.



The technology for these newer materials had advanced sufficiently by the summer of 1968 that thrust chamber components could be fabricated and test fired with high-energy fluorinated propellants. A concurrent coordinated program to continue the development and test firing evaluation of the PG/Carb-I-tex system for a 100-lb thrust FLOX/LPG reaction control engine was supported by a contract with NASA-LeRC (NAS3-11215) (Reference 6).

A historical outline of the development of pyrolytic refractory thrust chambers at Marquardt, including PG, PG alloys, and composites is presented in Figure 1, which outlines the development of free standing thrust chambers of pyrolytic refractory materials and composites. Concurrent work under these same contracts included material development and property measurement as well as work on flat plate stock and washertype nozzle designs. Explanatory notes for Figure 1 are given in Table I.

Details of the 100-lb FLOX/LPG injector design and its test firing evaluation are presented in Reference 6, which covers the NASA-LeRC-supported engine program. Over 900 seconds of firing on FLOX/methane, with one injector, were achieved with no plugging or overheating during operation at C\* efficiencies between 89 to 98 percent of theoretical for chamber L\* values of 10 inches and 18 inches, respectively. During these tests with FLOX/methane, both chamber erosion and carbon deposition were evident and varied locally in the chamber from the injector to the nozzle throat as a function of fuel film flow, wall material, O/F, L\*, and duty cycle.

It was concluded that the injector design approach was successful and that with additional effort, the design could be optimized to provide high performance and minimum erosion and carbon deposition.

Thrust chambers of nine different material systems, including pyrolytic refractories, fiber composites, vitreous carbon, and refractory metals were designed and fabricated for structural evaluation and test firing with FLOX/methane propellants.

Fabrication studies, property measurement, and design analyses were continued to explore and evaluate newer material concepts for specific application to the  $OF_2/B_2H_6$  propellant combination.

The Raytheon Company, participating as a major subcontractor, under a continuing subcontract, fabricated multilamina pyrocarbide alloy thrust chambers and investigated fabrication techniques for producing pyrocarbide/carbon fiber composites and pyro-nitride/carbon fiber composites.

Report S-989



## SECTION III PYROREFRACTORY ALLOY FABRICATION UNDER RAYTHEON SUBCONTRACT

#### A. <u>Multilamina Thrust Chambers</u>

#### 1. Previous Chamber Experience

Seven 100-lb thrust, thick ( $\approx 0.25$ -in.) walled, laminated chambers with graded ZrC-PG composition were fabricated and proof tested during Phase II of this program (reported in Reference 5). The highest internal pressure withstood by this group of chambers was 650 psi. Four of the chambers failed during internal pressure testing due to tensile cracking in the inner lamina. Inasmuch as achieving the potential strength of the thick walled chamber at low initial temperatures during rocket ignition depends upon uniform transfer of the pressure loads to the outer shells, the inner lamina must be tight or even in compression if possible. Apparently this was not the case, so several alternative fabrication variations were evaluated during Phase III and Phase IV of the contract which are documented in this report.

Three interacting anisotropic physical phenomena occur in the material which vary in degree with chemical composition, temperature, and duration of elevated temperature. These are the anisotropic thermal expansion, compressive creep, and crystal lattice transformation (or growth). Initial simplified analysis indicated that it would be desirable to grade the chemical composition of the laminae from a higher ZrC content on the outside to pure PG on the inside, so that the laminae would tend to tighten during cool down to room temperature from the 1850°C (3400°F) deposition temperature.

In order to assess the effect of growth and creep on a composite cylinder, a ZrC-PG graded composition multilamina cylinder made of five laminae was annealed for two hours at 2500°C. The laminae appeared to be loosened at cool down.

The nominal wall thickness of these multiple lamina chambers was 0.25-inch. This thickness had been selected primarily to evaluate the quality of material which could be achieved in the multiple step deposition process, and fabricate a chamber capable of withstanding ignition overpressures above 3000 psi. Subsequent analytical stress studies (presented later in this report) indicated excessive external transient thermal stresses would be experienced for a 0.25-inch wall thickness during firing. Reducing the maximum wall thickness to 0.125-inch reduced the maximum thermal stresses in a 2-inch diameter cylinder by a factor of three, while maintaining a potential burst strength above 2000 psi if tight laminae could be achieved.

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#### 2. <u>New Chamber Design</u>

Raytheon undertook the fabrication of additional 100-lb thrust chambers to improve the maximum burst pressure attainable and provide pyrocarbide alloy thrust chambers for evaluation of erosion in FLOX/LPG test firings. The typical thrust chamber configurations are shown in Figure 2. The design parameters to be covered included:

- a) Wall thickness of 0.125 (ZrC-PG) with three laminae and 0.050 (HFC-PG) for single wall.
- b) Alloy composition ZrC-PG (25, 12 and 0 wt % Zr) with constant radial composition.
- c) Post run anneal or heat treatment.
- d) Use of male and female mandrels for deposition.
- e) Exit expansion cones of 1.5:1 and 12:1.
- 3. Results of Chamber Deposition Runs

Four of the chambers fabricated by Raytheon and delivered to Marquardt are shown in Figure 3. Eighteen furnace deposition runs were made to fabricate thrust chambers for structural and test firing evaluation. The furnace process conditions, physical data and dimensional data are presented in Tables II, III and IV. Descriptions of the furnace runs and the chambers obtained from each run are given in the following paragraphs. The thrust chamber numbering sequence for the Phase III and Phase IV furnace deposition runs was MRS-50 through MRS-67.

Chamber MRS-50 (Figure 4) was deposited in a similar fashion to the earlier sea level chambers with the exception that the composition of ZrC-PG was held constant during the run rather than being graded to pure PG on the inner lamina. The purpose of this change was to try to bind the inner laminae tightly to the outer layers so that the entire shell would be load bearing, but this was not accomplished. MRS-51 was a pure PG deposition designed for the same purpose as MRS-50, to obtain a tightly bound multilamina chamber. An exit plug was used in this run to divert the gases after passing from the throat to improve the deposit in the expansion cone. Unfortunately, the plug caused turbulence in the gas stream and nodules on the inner lamina that ran the full length of the chamber. Both chambers were shipped to Marquardt.

The ZrC composition of MRS-50 was uniform at both ends, but the thickness profiles for both chambers showed thinning toward the exit cone. The deposition conditions were changed for the following runs in order to: 1) improve the structure of the deposit in the exit cone area and 2) decrease the thickness gradient throughout the length of the chamber.

While waiting for additional graphite female mandrels to be fabricated, two male mandrel deposits were made to evaluate the feasibility of making the altitude chambers by the external deposition technique. MRS-52 was a PG deposit and MRS-53 a ZrC-PG deposit. The mandrels were machined from single pieces of CS grade graphite into a shape that approximated a finished thrust chamber. The pyrolytic deposit was removed from the mandrel for examination by slicing it along the axis, yielding two halves of a chamber. The thicknesses for both MRS-52 and MRS-53 are listed in Table IV. MRS-53 was a repeat of -52, except that the deposit was ZrC-PG. In both runs, the deposit was smooth and fairly uniform in thickness. Upon removal from the mandrel, the deposit closed on itself indicating that the outside was in compression.

Chamber MRS-54 was the first deposit using the newly received female mandrels. A plug was used in the expansion cone to improve the deposit thickness in that region. The running conditions were identical (Table II) to those used for previously deposited (MRS-50, -53) chambers. The chamber exit plugged after 2-1/2 hours because the deposit completely filled the region where the throat and the plug tip met. Undoubtedly, the plug was too close to the throat; therefore, for MRS-55, the plug tip was raised above the throat. This deposit ran for the full term, but after removal of the mandrel and plug, it was apparent that again the plug constricted the gas flow and the inside of the chamber was coated with a rough deposit. The profile was also poor (Table IV).

For Run MRS-56, changes were needed to improve the profile and improve the deposit in the expansion cone region. The plug was raised again, the pressure was lowered to 7.5 mm Hg from 10.0 mm Hg, and the temperature was lowered to 1825°C from 1850°C. This procedure resulted in a sound chamber which was very smooth over the inside surface. The wall thickness profile was thinner than desired, varying from 0.118-inch at the injector end to 0.040-inch at the exit.

Chambers MRS-57 through -62 were deposited in the same way, that is, a diffuser plug was positioned in the expansion cone area. The pressure was lowered for this series to 6.0 mm Hg from 7.5 mm Hg for MRS-56. This change eliminated the choking that occurred in the throat area in previous chambers. Chambers MRS-57 and -58 were identical depositions except for time adjustments to obtain a more desirable thickness.

Chambers MRS-59 and -60 were 0.050-inch thick single wall chambers and were identical depositions except for run time adjustments. Both of these chambers were HfC-PG depositions to achieve 25 percent Hf. Mandrel removal was a problem because of the thinner chamber wall thicknesses, and both chambers cracked during removal. A redesign of the mandrel allowing for easier removal was undertaken. MRS-61 and -62 were ZrC composition graded, that is, the chlorine flow was varied from 0.15 lpm to 0.05 lpm. The 0.15 lpm flow was maintained during the first laminate, a flow of 0.10 lpm  $Cl_2$  was maintained during the middle laminate, and 0.05 lpm  $Cl_2$  was maintained during the first laminate. The depositions were ideal, being free of nodules and other deleterious

AN NUYS CALIFORNIA

effects, but both of the chambers cracked while the mandrels were being removed. The graded structure apparently either changed: 1) the growth at temperature or 2) the average expansion coefficient, so that when the mandrels were removed, cracking occurred in the expansion cone area. A redesign of the mandrel may help to correct this problem for graded structures. MRS-62, however, had sufficient sound material remaining after mandrel removal to be used as a sea level chamber. MRS-56, -57 and -58 were altitude chambers with 12:1 expansion cones (Figure 3).

The metal content in these chambers was considerably lower than that reported for MRS-50, even though the only significant process change was a reduction of pressure from 10 mm Hg to 6 mm Hg. A reduction of this magnitude was totally unexpected. Apparently the relative rates of decomposition of the methane and metal tetrachloride changed with pressure, resulting in reduced metal content in the deposition. The precise explanation for this result is not clear.

MRS -63 was deposited under conditions similar to those used in deposition of the previous chambers, but with a slightly higher chlorine flow to yield a higher zirconium content chamber. The deposition was successful, but the chamber cracked during mandrel removal because of isolated sticking between the deposit and the mandrel. This is an unusual, but not unprecedented occurrence, and no particular reason has been singled out as the cause of this problem.

MRS-64 was a repeat of run MRS-58. During the last portion of the run (the final third), the gas inlet tube plugged due to a malfunction in the retort system used to generate the  $\text{ZrCl}_4$ . The chamber deposition was thin and the inside layer, which was only about 10 mils thick, buckled and rendered the chamber useless. MRS-65 was deposited using identical conditions as those for MRS-64 and a sound chamber was obtained. After deposition temperature while still on the mandrel. The reason for this operation was to promote "growth" and thereby tighten the three laminae against each other. Visual observation of the chamber indicates that this did not happen. The growth-anisotropic stress relationship is sufficiently complex to make predictions of this kind rather difficult.

Chamber MRS-66 was deposited as a 0.050 wall thickness HfC-PG altitude chamber. The deposition was good and the chamber was sound but cracked during mandrel removal. Further mandrel modification to provide a weaker mandrel would solve this problem, but available time was too short to try another mandrel design.

MRS-67 was deposited utilizing identical operating conditions (Table II) as for MRS-58. A one-inch section was cut from the bottom of the chamber (excess material), and this section was heat treated after removing the mandrel at the deposition temperature (1825°C) in argon in order to tighten the laminae. Previous heat treating experiments have been: 1) conducted at temperatures far above the deposition temperature and significant

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graphitization occurred, or 2) at the deposition temperature with the sample restricted within the mandrel, resulting in growth stresses sufficiently high to buckle the inner lamina. The present experiment was designed to allow the inner lamina to grow, unrestricted by the mandrel, and thereby tighten against the outer laminae. After the heating cycle, the laminae were found to have loosened.

During the final thrust chamber fabrication study in which 18 chamber deposition runs were completed, there were seven thrust chambers shipped to Marquardt (MRS-50, 51, 56, 57, 58, 62 and 65). Chamber MRS-58 was received in time to be test fired with FLOX/methane during the January 1969 test period reported in Section VI.

#### 4. Current State-of-the-Art

Based on the results of these chamber deposition studies, it was concluded

- that:
- a) Multilamina thrust chambers of controlled geometry and thickness can be fabricated reproducibly and with good internal surface finish.
- b) Production controls and fabrication techniques need to be improved by further development and design studies.
- c) Tightening of the laminae of thick pyrorefractory cylinders was not achieved by any of the techniques tried and thus the burst pressure potential of this material system was not realized.

#### B. Pyrocarbide/Carbon Felt Composites

Composites made of carbon felts infiltrated with pyrolytic graphite have demonstrated ultimate flexure strengths of 10,000 psi using rayon precursor and 19,000 psi using a wool precursor, with compressive strengths as high as 65,000 psi (Reference 9). Tensile strengths for the pyrocarbide materials (ZrC-PG and HfC-PG) measured for ealier Raytheon materials (Reference 4) exceeded 40,000 psi and flexure strengths above 100,000 psi were measured. Since these values were more than double the values measured for pure PG, it seemed reasonable to expect an appreciable increase in carbon fiber composite strength if the felts could be similarly infiltrated with the pyrocarbides. Additional potential advantages over PG/Carbon felt and pure ZrC-PG materials would be fabrication flexibility, hardness, and oxidation resistance.

Initial exploratory furnace deposition runs were conducted by Raytheon as reported in the Phase I and Phase II report (Reference 5). These initial runs were attempts to obtain uniform impregnation of the felt without sealing off the surface. The feasibility of the material system was demonstrated, but the flow and process conditions required to achieve a uniform high density composite needed further investigation.

Ten furnace deposition runs were made during the Phase IV program to further define the impregnation process parameters for the uniform densification of 3/4-inch thick carbonized rayon felt with the ZrC-PG alloy.

The furnace setup for run MQF-1 was a square box design, inside dimensions of approximately  $1-1/2 \ge 1-1/2 \ge 10$  in. Two of the sides of the box were made of 3/4-inch thick carbonized rayon felt. The incoming gases were made to pass through a preheat chamber so that by the time the gases were in the felt chamber, they were hot enough to crack and react with the substrate. Deposition conditions (Table V) similar to those used to successfully deposit pyrolytic carbon in felt structures, were used as starting points. The run plugged after 15 hours in the preheat chamber, indicating that the enhancement of gas phase reaction in the pyrocarbide system, even at such a low temperature as  $1200^{\circ}$ C, is greater than was anticipated.

The setup for run MQF-2 was of similar design as MQF-1, except that the preheat chamber was eliminated, the temperature lowered to 1100°C, and the chlorine flow was reduced to 0.05 lpm. All these changes were designed to reduce the rate of cracking of the gases so that the carbide would be deposited in the interior of the felt. After 35 hours of deposition the run plugged and examination of the box after cool down indicated a surface coating of sooty material on the felt approximately 10 mils thick. This rate of deposition was about 50 times greater than that observed for the pyrocarbon system deposted under similar conditions.

For run MQF-3, the box dimensions were the same. The pressure was reduced to 10 mm Hg, the rationale being that the lower pressure would reduce gas phase reaction by reducing the number of gas molecules available for collision prior to contact with the fiber surfaces. The run duration was 40 hours. The material was found to be sooty and not usable.

Larger boxes than those for the first three runs were made for the furnace setup for the next three runs. The sides were  $3 \times 10 \times 3/4$ -inch, but in all other respects the setup was the same.

Deposition MQF-4 was run for 20 hours, MQF-5 for 40 hours and MQF-6 for 28 hours, although it was scheduled to be run for 60 hours. The increased deposition temperature (1500°C) increased the efficiency of the process over those runs using the lower temperatures, whereas the reduced pressure of MQF-6 improved, at least from visual appearances, the homogeneity of the deposits throughout the thickness of the felt. MQF-6 did not run full term. The gas inlet tube plugged for indeterminate reasons.

The average bulk and toluene densities of material from runs MQF-4, -5, -6, -7, and -10 are presented in Table VI. The variation in densities is due both to degree of buildup and to metal composition of the deposit.

The local density data from MQF-5 given in Table VII show some interesting information. The density gradient at the top is greater than the gradient at the bottom because: 1) the gas is coldest at the top and is the most rich, and 2) the bottom exhaust arrangement allows the gas to deposit on the outside as well as the inside walls. The increase of the toluene density from botton to top shows that the carbide phase is preferentially deposited, a fact observed many times before in this program (during cylinder and chamber depositions).

The carbide infiltration runs (MQF-7 through -10) were deposited utilizing the methods described before as the  $\Delta T - \Delta P$  techniques. The sample consisted of a felt pad formed into a closed shape such that the gases were required to pass through the fiber network. In addition, a temperature gradient was imposed across the thickness of the felt, similar to the method developed by the Sandia Corp. (Reference 10). The gases enter the network of fibers, decompose and deposit carbide-pyrolytic carbon onto the hot side of the felt. As material builds up on the surface of the fibers the temperature gradient moves in toward the cold side of the felt and deposits additional material. This process is self limiting in that once the hot side of the felt becomes filled with material, no additional gas flow is permitted and surface coating of the inside surface occurs. However, this technique is relatively rapid and the heavily coated side of the felt can be trimmed from the remainder and used for the measurements intended.

MQF-8 and -9 were not successful runs in that both terminated early due to malfunctions of equipment.

MQF -4, -5 and -6, deposited by a different technique, were made by simply passing gases over the felt with no pressure or temperature differential imposed with only surface diffusion operating to densify the felt. Their compositions were considerably lower than the later runs mainly because of lower chlorine to methane ratios.

Table VIII presents flexural strength data measured on the felt infiltration runs. A comparison of the room temperature flexure data between MQF-6 and MQF-10 shows the effect of carbide content on flexural strength (~4000 psi for 3% Zr vs. 5700 psi for 27% Zr), and the increased test temperature shows the increase in strength observed in other carbon systems (i.e., PG). All of the flexure tests were conducted with the load parallel to the ab plane. Since the rayon precursor is not isotropic, loading in the direction perpendicular to the ab plane would probably yield lower values of flexure strength.

#### C. Monolithic Deposits

Monolithic deposits (MQP-106, -107 and BN-794) were made on the inside of Carbitex mandrels, the purpose of the runs being to determine the compatibility of the deposited material with the substrate. Process conditions are given in Table IX.

MQP-106 was a standard ZrC-PG run, MQP-107 an anisotropic pyrolytic boron nitride run, and BN-794 an isotropic pyrolytic boron nitride run made under proprietary conditions. The ZrC-PG deposit appeared to adhere well to the mandrel, whereas neither of the boron nitride materials were compatible with the carbitex. Raytheon has successfully deposited isotropic BN on the inside of a commercial graphite tube.

#### SECTION IV MATERIALS CHARACTERIZATION

#### A. Pyrolytic Graphite/Carbitex Composites

In order to design composite thrust chambers of PG/Carb-I-tex and to calculate the axial and circumferential stress distributions, it was necessary that values of axial and circumferential elastic moduli for Carb-I-tex be known. In pressure tests of Carb-I-tex 713 chambers, the mode of failure was a gross axial tensile failure of the wall. The approach to improving the axial strength was to decrease the wrap angle (Figure 5) of the filament winding.

In order to obtain data on strength and elastic properties applicable to chamber design as a function of wrap angle, twelve Carb-I-tex 713 cones were purchased from Carborundum Co. The design of the cones is shown in Figure 6. Six of the cones were filament wound with a 60° wrap angle and six with a 75° wrap angle. The cones are shown in Figure 7, with strain gages installed.

The following properties of Carb-I-tex 713 in the filament wound axisymmetric form were determined using both the 60° and 75° cones as structural test specimens:

- 1. Modulus of elasticity in the circumferential direction.
- 2. Modulus of elasticity in the axial direction.
- 3. Ultimate tensile strength in the axial direction.

The cones were held at the large end and internally pressurized, with the small end sealed (Figure 8). This setup created maximum stresses in the large end of the cone. At an internal pressure of 600 psig, the axial stress 0.6-inch from the large end was 1880 psi, perhaps exceeding the axial strength. The circumferential stress was only 5500 psi, well below the expected hoop strength of the Carb-I-tex. Therefore, it was expected that the axial strength determined in this cone under biaxial pressure loading would be more representative of the loading in a thrust chamber than that obtained from loading of a flat test specimen.

The wrap angle of the cones actually varied with diameter because of the type of filament winding machine used. The wrap angle varies as follows:

 $\frac{\text{Diameter}}{\tan \alpha} = \text{Constant}$ 

where

 $\alpha$  = Wrap angle as defined in Figure 5.

It was possible to break the cones more than once at successively smaller diameters, thus obtaining more data on the variation of axial strength with wrap angle.

Six of the twelve Carb-I-tex cones obtained were used in the uncoated condition to measure axial and circumferential properties. The remaining six cones were coated with PG for the measurement of composite properties.

The Carb-I-tex cones and tubes were fitted with strain gages and pressurized to obtain modulus and strength data as a function of the wrap angle. Figure 9 shows circum-ferential modulus values from cone data and from tube No. 1. The circumferential modulus varies from  $2 \times 10^6$  to  $5 \times 10^6$  psi. The axial modulus (Figure 10) follows the opposite trend and is considerably lower than the circumferential modulus, ranging from 0.9 x  $10^6$  to  $.2 \times 10^6$  psi. One data point was obtained by compressing tube No. 2 (from Phase II, Ref. 5) axially on a Baldwin loading machine.

Figure 11 shows axial tensile strength as a function of wrap angle as obtained from cones pressure stressed to destruction. Most cones were tested to failure twice, once near the large end and then near the small end by remounting the remainder of the cone. All cones failed axially with the exceptions of Cone No. 1-60, which had a hole blown in the side and Cone No. 2-60, which could not be broken because of excessive leakage. Some cones with high leakage rates were broken when a very thin plastic liner was used inside as a seal. The axial strength was much lower than the circumferential strength. Several  $60^{\circ}$  cones showed circumferential stresses as high as 9600 psi when failing axially. The circumferential strength of  $75^{\circ}$  cones would be expected to be higher.

The surfaces of the Carb-I-tex cones were not as good quality as those of previous Carb-I-tex tubes (2-inch diameter) and 100-pound thrust chambers (Reference 5). Numerous rough areas with graphite filament breakage existed on both the ID and OD. Polishing techniques were used to alleviate this condition. Part of the problem seems to be small voids in the Carb-I-tex. A smooth Carb-I-tex surface is desired to produce a good quality PG coating with minimum size and number of nodules. Other observations made during inspection of the cones were as follows:

- 1. The  $75^{\circ}$  wrap angle cones were smoother inside and outside than the  $60^{\circ}$  cones.
- 2. The delaminations in the Carb-I-tex wall were common at the small end of the  $60^{\circ}$  cones.
- 3. None of the cones were completely smooth or free from small rough areas.

Cone Wrap Angle (Minimum) (degrees)	Inside PG Coating (in.)	Outside PG Coating (in.)	
60	0.025	None	
75	0.025	None	
60	0.050	None	
75	0.050	None	
60	0.025	0.025	
75	0.025	0.025	

Six of the Carb-I-tex cones were coated with PG according to the following schedule:

The resulting tubes had good quality, well bonded coatings both inside and outside. Surface flaws in the Carb-I-tex are still apparent in the PG surface after coating. The cones are still available for further testing of the effect of these flaws on pressure failure modes.

#### B. Thornel Reinforced Pyrolytic Carbon

#### 1. Description of Test Specimens

Testing was completed on the Thornel-50 wound, pyrolytic carbon infiltrated, NOL-type composite ring specimens which were prepared and partially tested during the course of the Phase II program (Reference 5). The specimens were prepared by winding the Thornel yarn around four foam carbon, spool shaped mandrels - each divided into four sections as shown in Figure 12. Two ring specimens were obtained from each section. The spools, after winding and pyrolytic carbon infiltration, are shown in Figure 13, and examples of test rings are shown in Figure 14. Spool No. 1 was filament wound without additives or special treatments to the filaments before PG infiltration. Chopped carbon fibers were added to Spool No. 2 during the winding process to form a network of fibers to fill the space between the graphite yarn bundles. The objective of this addition was to improve the shear strength of the composite. The same objective was pursued in preparing Spool No. 3 by causing frayed fibers of the Thornel yarn itself to bridge the space between adjacent yarn or yarn layers. To accomplish this, the yarn was lightly abraded with a sharp tool during winding. Spool No. 4 was wound with the addition of carbon black and furfural binder to fill the potential pores and also for the purpose of improving the composite shear strength. The subsequent PG infiltration process provided inter-filament bonding within the yarn for effective load transfer.

#### 2. Discussion of Test Results

The results of the tensile test of the rings using the NOL Split-D technique are shown in Table X and plotted in Figure 15 as a function of specimen thickness. Because of the effect of bending moment on the test section, the thinnest ring would be expected to produce the highest apparent tensile strength, provided the materials are uniform and without major defects. Without a prior knowledge of the magnitude of this bending moment, its actual effect cannot be accurately assessed. The effect depends not only on the magnitude of the maximum strain which the composite undergoes, but also on other factors such as its modulus and the mismatch between the diameter of the Split-D fixture and the inner diameter of the specimen.

If the cross section of the test region of the specimen contains major pores or other defects, a defect of a given size would have the largest influence on the thinnest specimen. In the macrographs and micrographs of these specimens, it was noted that many lacked uniformity and denseness in packing. Figures 16 through 19 show the Section C from each spool as examples of the cross sectional microstructure. The influence of porosity and other defects on the mechanical properties can be seen in the degree of data scatter for the thin specimens (Section A) in Figure 15.

The 10X photomacrographs show the full cross section of the specimen. The 50X photomicrographs show an enlarged section of the specimen under polarized light conditions to bring out the highly anisotropic pyrolytic graphite (white areas). Much of the thick pyrolytic graphite on the outer surface was machined off during the preparation of these specimens. The presence of the chopped fiber or the frayed fiber effect in the windings of Spools Nos. 2 and 3, respectively, are not readily evident in the photomicrograph (Figures 17 and 18), except for a few individual PG-coated fibers in the void area. However, the carbon black with furfural binder added during the winding of Spool No. 4 is clearly seen as the smoother gray areas (Figure 19). In Section D of each spool, the

Thornel filaments were wound over pyrolyzed felt. Although the felt layer was designed to be 0.20-inch thick with a Thornel wrapping of 0.050-inch, it was noted in the 10X micrographs (not shown) that some variation in these values was obtained in the actual windings. On the average, there was approximately 0.10-inch of felt, with approximately 0.10-inch of Thornel. Since the underlying felt/PG material contributes little to the total strength, the actual PG/Thornel strength is much larger than the indicated strength.

The 750X photomicrographs (Figures 20 and 21) of representative sections of each spool show the details of pyrolytic coating over each fiber within the yarn, and in some cases, regions of more massive depositions around the yarns. The pyrolytic graphite deposition around each filament in Spool No. 1 (Figure 20A) was uniform (approximately  $0.05 \ge 10^{-3}$ -in.) with no preferentially high deposition rate over the outer fiber of the yarn. In Spools Nos. 2 and 3 (Figures 20B and 21A, respectively), however, the coating rates were higher. Each fiber was coated with approximately  $0.10 \times 10^{-3}$  to  $0.20 \times 10^{-3}$  in. of PG, whereas heavy coatings occurred around the periphery of yarn bundle adjacent to the voids (which constitute the primary passageway for infiltrants). The absence of the corresponding heavy coating in the large voids of Spool No. 1 is not adequately explained. It would appear that the chopped fibers introduced in Spool No. 2 and the frayed yarns in Spool No. 3 served to open up the primary passageways to allow free flow of infiltrants to permit the heavier deposits. In Spool No. 4 (Figure 21B), however, the PG coating of the fiber was less than in the others, indicating the effect of the carbon black and furfural binder in closing up the primary passages. Although not readily evident in the polarized light photomicrograph, the stray outer strands of the yarn are tightly embedded in the pyrolyzed, furfural-bonded carbon black. Because of the blocked primary passages, the coating rates of the inner fibers are reduced, and longer time would be required to infiltrate for adequate inter-filament bonding.

On the basis of very limited data, the following observations are noted:

- 1. The addition of the chopped carbon fibers into the Thornel windings was effective in increasing the strength of PG impregnated Thornel reinforced composite, probably by partially filling the void between yarn with PG-bonded chopped fibers, thereby increasing the overall interlaminar shear strength.
- 2. The abrasion of filament while winding the spool did not improve the properties despite good PG infiltration; rather, the properties degraded, probably from the direct damage incurred by the filament during the knife edge abrasion. The admonitation to handle the filaments with care should be well heeded.

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- 3. The beneficial effect of the furfural and carbon black additions was not clearly evident. These additions can be expected to provide improved shear strength of the composite by bridging the space between yarns. On the other hand, the presence of the filler material can prevent the thorough penetration by the infiltrant, or decrease its rate. The inter-filament bonding within the yarn would require a longer processing time.
- 4. Improved winding techniques under conditions of constant tension should provide better filament packing. Judicious choice of impregnants and impregnation techniques can lead to improvements in high strength, high modulus graphite, filament wound structures.

As noted in the microstructure of Spool No. 4 specimens with the carbon black/furfural impregnant, some of the outer filaments of the graphite yarn were completely encased in the pyrolyzed impregnant. If means were devised to impregnate thoroughly into the inner regions of the yarn, it may result in a produce superior to the PG infiltrated structure. Currently, all resin materials undergo considerable shrinkage ( $\approx 20\%$  or more) requiring re-impregnation to obtain reasonably low porosities. The use of PBI (polybenzimidazole) resin with high char yield is an attempt to accomplish this purpose. The shrinkage of PBI during carbonization is claimed to be approximately 10%. Preliminary studies conducted by the Narmco Research and Development Division of the Whittaker Corporation showed carbonized PBI resin impregnated, thornel reinforced, uniaxial composite laminates to be stronger than corresponding PG infiltrated materials. Additional work is required to substantiate these early observations.

Better testing techniques which do not impose bending moments are available in measuring the circumferential tensile strength of ring specimens. A common technique involves the hydraulic pressurization of an axially restrained internal bladder. A method using an axial compression of a solid rubber cylinder to provide radial expansion inside the ring specimen has recently been described (Reference 11).

3. Filament Wound Thrust Chamber Tensile Tests

Three composite thrust chambers utilizing PG infiltrated high modulus carbon fiber were fabricated for structural and test firing evaluation.

The PG/Thornel/PG chambers (Figure 22) consists of free standing PG chambers with .050 walls which were filament wound with Thornel-50 to a 0.125 wrap thickness at an  $80^{\circ}$  lead angle. The chambers were then infiltrated with PG during a 96-hour furnace run. The free standing PG liners had been previously pressure checked

to 200 psig before filament winding. During the trimming and finishing of one of the composite chambers, the PG liner cracked and would not hold pressure. Therefore, the PG liner was removed leaving a shell of the Thornel-50/PG composite. The structure of the Thornel 50/PG composite is shown in Figure 23. The cylindrical chamber section of this part was cut into three NOL tensile rings. A set of "D" blocks (Figure 24) was made and the three NOL rings were pulled at room temperature. The tensile strength results are tabulated below for the broken specimens shown in Figure 25.

Sample	I.D. In.	Wall Thi Max. In.	ckness Min. In.	Height In.	Area In. <sup>2</sup>	Failure Load Lb.	Aver. Stress psi
1	1.53	. 100	• 092	. 758	. 145	8400	57,700
2	1.53	.109	<b>.</b> 102	.761	. 161	7420	46,200
3	1.53	.096	• 093	.760	.144	7720	53,800

#### NOL Ring Test Results

The above level of available strength in the chamber wall would provide a chamber pressure capability of over 7,000 psi if the cold start pressure stresses could be transferred to the composite before an inner liner failure. As the PG chamber liner heats due to combustion heating, the inner liner does transfer the load and, in fact, the PG liner goes into compression.

The carbon composite material consisting of high strength carbon filaments and PG bonding appears to have excellent potential for high strength at high temperature. However, there are several fabrication parameters subject to variation for tailoring the material to a specific application. The carbon yarn Young's modulus may be varied from 1 to  $100 \times 10^6$  psi with a corresponding increase in tensile strength. A number of other different types of high modulus carbon fibers are also being developed. At the present time, some of these PG/fiber material concepts are being developed for other applications by Super Temp Co.

#### C. <u>Vitreous Carbon</u>

Compared to the better known forms of carbon, vitreous carbon possesses properties which are desirable for structural applications. It is isomorphous, relatively strong and stiff, has good thermal shock resistance, and its glassy structure is stable (does not graphitize) to over 3000°C. It is also very hard and it should exhibit good erosion resistance. However, it also has a serious weakness in its sensitivity to mechanical shock, and a crack once started propagates easily and rapidly. It is therefore important to protect a vitreous carbon structure from impact-type loads and from high stress concentrations. Vitreous carbon as produced by Beckwith Carbon was investigated for application as thrust chamber liners.

Initial investigations involved the use of available cylindrical crucible shapes for the combustion chamber section and washer-type inserts for the throat section. A principal, nonrecurring expense for such items is the fabrication of a metal transfer mold. Once this item is amortized, the cost of vitreous carbon liners would be significantly less than the cost of pyrolytic graphite liners.

In preparation for the use of vitreous carbon parts in a thrust chamber, preliminary machining tests were conducted at Marquardt. The hardness and brittleness of vitreous carbon precludes the use of carbide cutting tools as used in lathe operations. Diamond tools were effective in cutoff, core drilling, or grinding operations. Procedures generally used for ceramic materials are useful on vitreous carbon. Vitreous carbon is sufficiently conductive that EDM (electrical discharge machining) processes are very effective, although time consuming. Brass and carbon electrodes were used; both suffered erosion, but the metal electrode resulted in a better finish. Because this technique is very slow, it should be used for intricate shape cuts where the more rapid, diamond cutoff, drilling, or grinding operations are not practical. Ultrasonic machining techniques should also prove to be well suited to vitreous carbon materials.

The brittle fracture characteristic of vitreous carbon by which a crack in the material propagates readily through the whole piece could possibly be overcome by introducing fibrous carbon reinforcements. This possibility was explored in cooperation with Beckwith Carbon Corporation. However, due to several furnace run malfunctions, the results were inconclusive. Two approaches were being evaluated. The first was to pyrolyze a phenolic/carbon fiber composite through the same furnace cycle used in vitrifying a normal phenolic molded component. Due to the 20 percent shrinkage in the phenolic during processing, it would be expected that voids would develop in the composite, while the normal vitreous carbon is impervious and free of all porosity. An alternate approach would be to formulate a resin/rayon composite in which the pyrolyzation shrinkages would be matched for each material. The furnace runs on the phenolic carbon fiber samples developed operating problems before satisfactory material was obtained and this limited effort was terminated.

#### D. Oxidation Tests

An oxyacetylene torch test was performed to determine the relative oxidation resistance of a number of advanced carbon materials. Samples of each material were supported on alumina blocks and exposed to the oxyacetylene flame. Throughout the heating time, the maximum surface temperature was monitored with an optical



pyrometer. The exposure time was set for each material to give significant erosion without burnthrough. Some samples were limited to 2-1/2-minute runs, and others were run 8 minutes. When more than one sample of each material was available, test runs were made at different surface temperatures by adjusting the torch-to-sample distance.

The materials evaluated included:

- 1. Pyrolytic graphite (as a reference)
- 2. POCO graphite (AXF-5Q)
- 3. Copper impregnated POCO (AXF-5QC)
- 4. Vitreous carbon (Beckwith)
- 5. Fluidized bed pyrolytic carbon (isotropic)
- 6. PG/Thornel composite

Figure 26 shows erosion rate as a function of surface temperature for the materials tested. The small sample of graphite coated with isotropic pyrolytic carbon burned through during the test so that an exact erosion rate could not be determined. It can be stated, however, that the erosion resistance of this material was not greater than anisotropic PG.

Of the materials tested, POCO graphite (AXF-5Q) showed the least erosion resistance. Copper impregnated POCO (AXF-5QC) gave nearly identical results. Both materials eroded about six times faster than PG. The erosion rate of PG impregnated Thornel was about twice that of PG. Vitreous carbon eroded at a rate about three times that of PG. The vitreous carbon sample cracked in two after 30 seconds of heating, but the test was continued with one piece without additional cracking.

The oxidation rates of vitreous carbon in oxygen, argon, and  $CO_2$  gas mixtures at temperatures up to  $3000^{\circ}$ K have been compared to the rates for pyrolytic and molded graphite in Reference 12. The oxidation rates of vitreous carbon and PG were similar and they are somewhat lower than those for molded graphite. At a surface temperature of  $3000^{\circ}$ K (  $4900^{\circ}$ F), vitreous carbon had an oxidation rate in carbon dioxide less than one-half that of PG.

#### E. Copper Impregnated POCO Graphite

Copper impregnated POCO-AXF-5Q graphite is being evaluated as a potential chamber lining material. The potential advantages of the material include:

- 1. Strength equal to or better than POCO-AXF-5Q graphite
- 2. Double the thermal conductivity at temperatures to 3500°F
- 3. Capable of structural operation to the temperature limits of graphite



- 4. Reduced brittleness and increased resistance to thermal stress failure
- 5. Improved heat sink and conduction cooling capability
- 6. Potential for combining with a PG/fiber composite for operation at high temperature.

Two other characteristics which have been evaluated in tests were oxidation resistance in an oxidizing flame and effect on carbon deposition in a FLOX/LPG test chamber. In both tests the performance of the copper impregnated graphite was essentially the same as the pure graphite.

Another characteristic evaluated was the mechanism of copper loss during high temperature heating. In one series of tests, samples of Cu/POCO were heated in a vacuum furnace to nominal temperatures of  $2300^{\circ}$ ,  $3000^{\circ}$  and  $3400^{\circ}$ F for times of 10 and 20 minutes. The results are shown in Figure 27. In a second series of tests, a sample of Cu/POCO was heated to  $3300^{\circ}$ F four times. During the first heat up cycle, molten copper exuded from the sample surface and the total weight loss was 31.3 percent. During subsequent heating cycles the weight loss of copper examined visually (Figure 28). After each cycle the copper boundary within the sample was well defined indicating that the loss of copper is from an internal effective copper surface. After the first heating cycle the molten copper is exuded from the pores of the graphite by the difference in thermal expansion. On subsequent cycles, no molten copper is seen on the surface and the copper is apparently lost by evaporation.

A similar heating test was conducted with Cu/POCO sample in which a 1/2-inch diameter tantalum tube was inserted. After heating to  $3000^{\circ}$ F three times, it was found that the interface gap between the tube and graphite remained filled with copper while the copper loss boundary was again seen at the outside of the sample (Figure 28).

#### SECTION V THRUST CHAMBER DESIGN and FABRICATION

The results of preliminary design studies and fabrication experiments demonstrated the feasibility of fabricating complete lightweight, all carbon, composite thrust chambers with the potential of very high burst pressures and high operating temperatures. At the same time, a number of design variables remained uncertain for the various composite structures. These unknowns included:

1. The values of thermal conductivity, thermal expansion, elastic modulus and creep rates of the PG/carbon fiber composites as a function of process parameters and operating temperatures.



- 2. The residual stresses and nature of the bonds in the various composite structures.
- 3. The optimum choice of thickness, carbon fiber modulus, wrap orientation, etc. to achieve the maximum rocket chamber strength at ignition heat up and steady state conditions.

These design unknowns are all amenable to experimental and analytical determination in time, but it was decided that a screening program to evaluate fabricability, structural soundness and erosion resistance of the chamber liners in a single environment would be worthwhile at the present time.

A. Thrust Chamber Designs

The chamber design concepts described below were incorporated into chamber designs for fabrication, pressure testing and test firing with FLOX/methane propellants. Along with the advanced composite designs, additional materials and chambers also described below were fabricated to broaden the base of comparison and provide additional data on the operating characteristics of the 100-lb thrust FLOX/methane injectors used.

1. <u>Multilamina Pyrocarbide Chambers</u>

These chambers have been described in Section III-A.

2. Free Standing Thin Wall HFC-PG Chambers

Chamber fabrication described in Section III-A.

3. Free Standing Pyrolytic Graphite

Chambers with 0.050 walls were used both as streak test chambers to evaluate injector performance in short runs and as the liner for the PG/Thornel/PG chambers below.

4. PG/Thornel/PG Chambers

A high quality PG free standing liner filament wound with high modulus carbon yarn to a thickness 0.125 or 0.25 and then infiltrated with pyrolytic graphite to bond the carbon fibers and PG liner into a high strength composite, pressure tight thrust chamber.



#### 5. PG/Thornel/Resin Chambers

Same as above, except the Thornel wrapped chambers were PG/resin impregnated and pyrolyzed in multiple steps to achieve a pyrolyzed resin/Thornel composite over the PG liner.

#### 6. <u>Carb-I-tex 713 Chambers</u>

With and without PG coatings.

#### 7. <u>Vitreous Carbon Chamber Liner (Figure 29, bottom)</u>

- 8. Pyrocarbide Alloy (ZrC-PG) Stacked Washer Nozzle Insert (Figure 29, top)
- 9. High Density Isotropic Graphite (POCO) (Figure 30)

Used for long run evaluation of injector streaking and deposition plus graphite erosion characteristics.

#### 10. Refractory Metal Chambers (Figure 31)

Ta-10W chambers coated by IITRI (Reference 13) with the carburized Hf-10Ta-4, 5C-0.4S: slurry coating.

#### 11. High Density POCO Graphite Impregnated with Copper (Figure 32)

Cylindrical section inserted between injector and POCO graphite chamber.

All of the above design concepts were carried through the design stage and to various stages of fabrication, structural evaluation and test firing as described in the following sections.

- B. Thrust Chamber Fabrication and Evaluation
  - 1. <u>PG/Carb-I-tex Chambers</u>

a) Three 100-lb thrust size Carb-I-tex 713 chambers (Figure 33) were received from the Carborundum Co. for coating with PG. These chambers were to have 0.25-inch Carb-I-tex walls, but the thicknesses of the delivered shells varied from 0.19 to 0.25-inch. Because the shells were thin, the outside surfaces were not machined smooth except at the attach end. The inside surfaces had been machined using a standard tool bit which left several rough places where fibers had been torn loose.

The three Carb-I-tex chambers were proof pressure tested with nitrogen to 200 psig without failure. However, the chambers leaked gas through the walls, indicating a degree of porosity.

Previous experience showed that the quality of the PG coating depended directly on the surface finish of the PG mandrel substrate. All surface blemishes and machining marks are picked up and propagated through the PG coating, often resulting in large nodules. These, in turn, might become points of initiation of failure.

One Carb-I-tex chamber was sent with its existing finish to Super Temp Co. to have a 12:1 nozzle extension added. The 12:1 nozzle exit cone was fabricated of RPG and attached to the Carb-I-tex by a threaded joint. The Carb-I-tex chamber and nozzle were then coated inside and outside with a 0.040-inch layer of PG. The chamber as received from Super Temp is shown in Figure 34. An X-ray photograph of the chamber (Figure 35), as well as visual examination, revealed that the outer layer of PG separated from both the RPG and the Carb-I-tex. An axial crack occurred in the outside PG on the exit cone. In finish machining the chamber, most of the loose PG on the exit cone was removed. The finished chamber is shown in Figure 36 attached to the injector and bipropellant valve.

The remaining two Carb-I-tex chambers were used for surface finishing studies. The surface finishes on the Carb-I-tex chambers and test cones were not as good as on the previously received tubes. There were numerous rough spots which appeared to be voids in the material. In some cases, loose fiber ends were exposed.

A number of methods of grinding and polishing was attempted to eliminate the rough spots. A very sharp tool bit at 300 rpm was tried, but the finish was not improved. Hand finishing with fine (600 grit) emery paper gave a reasonably good finish in some cases, but rough areas were uncovered on other samples by the sanding. As with other finishing techniques, it was found advantageous to sand in the same direction in which the Carb-I-tex was wrapped to prevent the edge of the paper from picking up fiber ends.

The inside surfaces of the Carb-I-tex cones were finished by lapping using an anodized aluminum tapered plug. A good finish was obtained on 75<sup>°</sup> cones, but the  $60^{\circ}$  cones still had rough areas. A reasonably good PG coating was obtained on the cones without large nodules, but rough areas still existed, especially on the  $60^{\circ}$  cones.

Grinding wheels were tried in an attempt to get a good finish on the Carb-I-tex tubes. A 1-1/2-inch Cratex rubber wheel turning at 5000 rpm was tried with fair results, but other rough areas were uncovered. A 60 grit Al $_2O_3$  wheel was tried and also a 46 grit open wheel. Neither gave good results.

The best surface finish was obtained using diamond tools. A single diamond cutting tool gave a reasonably good finish, but best results were obtained with a small diamond grinding wheel (about 0.5-inch in diameter) mounted on a balance quill. A surface free from serious rough areas was obtained. The insides of two Carb-I-tex 100-lb thrust chambers were finished in this way using a template to guide the wheel. A surface grinding speed of 40 ft. per second was used. Equally good results were obtained with two tubes ground with the same wheel at 6 ft. per second.

One of the Carb-I-tex 100-lb thrust chambers was finished for firing without a PG coating and the other chamber was coated with a 0.04-inch layer of PG on the inside only (Figure 37).

#### 2. PG/Thornel Composite Chambers

Two pyrobond thrust chamber fabrication concepts were evaluated using a free standing PG chamber over-wrapped with Thornel and bonded. These chambers consist of thin (0.050-inch wall), pyrolytic graphite liners wrapped with multilayer helical windings of high modulus graphite yarns with matrices of either infiltrated pyrolytic carbon or impregnated and pyrolyzed PBI resin. The high modulus reinforcements used for these chambers were Thornel-50 (Union Carbide). The impregnant resin was PBI (Polybenzimidazole).

The PG liners for the altitude thrust chambers are shown in Figure 38. The Thornel-wound sea level and altitude chamber structures are shown in Figures 39 and 40, respectively, before PG infiltration. Two-piece graphite inserts which extended beyond the liner ends were placed into the PG liners in order to provide a cylindrical end section to prevent slippage during winding of the yarn down the conical section of the chamber liner. These extensions were machined off after the completion of the infiltration and impregnation and pyrolyzation process. A steel bolt through the axis served to hold the graphite mandrel in place and to provide a chucking medium for the lathe. The filament winding setup at Narmco-Whittaker is shown in Figure 41 showing the chamber in the lathe and the Thornel feed bobbin with the lathe in the background. On removing the bolts before PG infiltration at Super Temp, one of the filament-wound chambers, the altitude chamber, broke in the chamber region. The broken sections were placed in the infiltration furnace along with the two sea level chambers for production of material test samples.

Although it was intended to leave the stainless steel bolts in the three filamentwound chambers for PBI impregnation during the impregnation-pyrolyzation treatments at Narmco, the bolts were removed after the experience at Super Temp. No difficulties were encountered in the removal of the bolts from these chambers and the impregnationpyrolyzation cycles were completed without difficulty.



#### 3. Vitreous Carbon Thrust Chambers

Three vitreous carbon crucibles (1.5-in. dia. x 6.0-in. length x 0.09-in. wall) were purchased for use as thrust chamber liners to be encased in a POCO graphite, heavy walled chamber. Prior to use, one of the crucibles was machined by grinding to obtain a true outer surface that was round and uniformly tapered. The closed end of the crucible was removed by grinding. During grinding of the outer crucible surface, cracks developed in the wall at the ground surface. Since the material is isotropic and the heating and cooling cycles were very gradual, the existence of residual stresses would not be expected. One possibility was the stresses due to mounting during machining. Also considered was the heating effect of the grinding operation. This heat should have been small due to the light cuts taken and the ease of carbon removal.

The characteristic glass-like propagation of the wall crack during machining demonstrates the need to overcome this property limitation by the use of some reinforcement technique.

The two remaining crucibles on hand were also broken during attempts to fill the crucibles with a potting material to facilitate mounting the part while grinding. A low temperature metal alloy was used to fill the crucible in one case and a plastic was used in the other. The reason for these failures was not clear. However, this experience with cracking of the vitreous carbon while grinding, mounting, heating and potting point out the brittleness of vitreous carbon above normal commercial graphite and the pyrolytic graphites.

#### 4. Stacked Pyrocarbide Washer Nozzle

A chamber of graphite, steel and reinforced plastic was designed (Figure 29) to hold exit nozzle inserts for test firing evaluation. Two sets of ZrC-PG washers were fabricated with a 0.868 throat diameter by Raytheon during Phase II. One nozzle set was test fired with  $N_2O_4$ /MMH (Reference 5). The second set was available for evaluation with FLOX/Methane. Fabrication of the chamber was postponed because it could not have been completed in time for the January 1969 time period. Therefore, a set of exit nozzle insert washers is available for test evaluation.

#### C. <u>Metal-Carbon Bonding</u>

All current carbon composite and PG thrust chamber attachment designs employ a clamping action to seal the carbon to the metal interface. Work done by Solar (Contract NAS 7-100) demonstrated that PG could be brazed to Ta-10W alloys. However, the mechanical integrity of such a joint was poor under thermal cycling conditions due to

thermal expansion stresses. However, in a thrust chamber design using a carbon fiber composite with a PG inner wall, the possible techniques of bonding metal to the carbon fiber composite were re-examined. Three techniques have been studied in exploratory experiments, namely, brazing, electroforming and plasma spraying. The objective of these initial studies was to form a metal-to-carbon joint for operation and thermal cycling at temperatures below  $2000^{\circ}$ F such as those allowable at an injector/chamber interface.

#### 1. Brazing

A study at Marquardt was conducted to determine the practicality of brazing PG/Carbon felt (RPG) to itself and to 321 stainless steel and also of brazing Carb-I-tex to itself and to stainless steel. An attempt was made to braze the following materials using small samples:

Materials	Braze Alloy
RPG-RPG	Coast 53
RPG-RPG	Palniro 7
RPG-Cres 321	Coast 53
PRG-Cres 321	Palniro 7
Carbitex-Cres 321	Coast 53
Carbitex-Cres 321	Palniro 7

The composition of Coast 53 braze alloy is 83.5Ni-6.5Cr-3B-4.5Si-2.5Fe. The composition of Palniro 7 is 70Au-22Ni-8Pa. The braze material was applied as a paste using Nicrobraz cement as the binder. Brazing was done in a vacuum at  $1975^{\circ}$ F.

In no case was a good braze achieved. On the RPG-RPG samples, neither alloy would wet the material; the braze material did not flow to the interface. The results for the RPG-Cres 321 samples were the same except that the steel was uniformly wetted. There was no bond to the RPG.

Likewise, no success was achieved in brazing Carbitex to Cres 321. The Carbitex and the steel were wetted by both alloys (more uniformly by Coast 53), but the joints failed in cooling. A thin layer of Carbitex was found adhering to the steel sample. This failure is due to the difference in thermal expansion between the two materials. The coefficient of thermal expansion of Carbitex is  $1.2 \times 10^{-60} F^{-1}$ , that of Cres,  $9.3 \times 10^{-60} F^{-1}$ . This problem is less severe for RPG which had a coefficient of thermal expansion of  $4.5 \times 10^{-60} F^{-1}$ .
Future work may include investigation of brazing metal infiltrated or metal coated RPG. It is hoped that infiltration of an appropriate material will cause the RPG to be wetted by the braze alloy and enable a good bond to be made. Several techniques of metal infiltration of RPG have been discussed with Super Temp and could be evaluated in the future.

### 2. <u>Electroformed Nickel</u>

Two short cylindrical specimens of RPG/PG composite were used as a substrate for electrodeposition of a thick layer of pure nickel. The appearance of the nickel deposit prior to machining is shown in Figure 42. After machining, a 50X photomicrograph of the nickel RPG interface (Figure 43) shows the penetration of the nickel into the RPG.

### a. Thermal Cycling Test

To evaluate the structural integrity of the joint, a thermal cycling test was run to define the nature of the metal bond failure. The geometry of the test specimen is shown in Figure 44 and the experimental heating setup is shown in Figure 45.

Two thermocouples located as shown in Figure 44 were used to monitor the temperatures. Both thermocouples were imbedded in the RPG, the inner one being near the PG interface and the outer one near the nickel interface.

In order to simulate thrust chamber performance, the specimen was heated from the inside diameter portion by inductively heating the ends of a cylindrical graphite susceptor which passed through the ring specimen. The heating was conducted under a moderate vacuum of about  $5 \times 10^{-2}$  torr (50 micron Hg).

In an initial coil configuration, the split coil was placed too close to the specimen, causing direct susceptance by the specimen. The specimen heated more rapidly on the outside and caused heating beyond the temperature intended for the Ni-RPG interface, especially at the position opposite the side with the thermocouple.

The setup was modified by using a longer susceptor and placing the two coil sections farther apart. Figure 45 was taken during the high temperature phase of the heating cycle.

With this test configuration, thermal cycling tests were programmed for 5 cycles, each to nominal temperatures of  $1000^{\circ}$ ,  $1500^{\circ}$  and  $2000^{\circ}$ F as monitored by the inner thermocouple. The low temperature portion of each cycle was determined by practical time considerations of thermal decay of the system. The actual typical cycles are shown in

Figure 46. The upper temperature for the last series of cycling tests (intended to be  $2000^{\circ}$ F) was not achieved, due to the indirect heating method for the heater-specimen geometry used, in which the thermal losses by radiation were excessive. At the peak of these cycles, the susceptor temperature within the induction coils was ~2320°F and the temperature in the region of the specimen was ~2025°F, as determined by optical pyrometry.

### b. Discussion of Results

The temperature experienced by the nickel-RPG bond interface was very closely that indicated by the outer thermocouples. From the typical temperature cycles (Figure 46), it is noted that the bond interface reached temperatures of approximately  $800^{\circ}$ ,  $1200^{\circ}$  and  $1400^{\circ}$ F during the three cycling series.

Under the conditions noted, that of three series of thermal cycling tests to the temperatures shown in Figure 46, the following observations are noted:

- (1) The inner PG ring remained intact.
- (2) Although most of the interface between the nickel and RPG remained apparently well bonded, a hair line crack developed in the RPG near the nickel interface.
- (3) The nickel surface appeared to have small blisters, probably due to the vaporization of occluded electrolyte material. This blistering effect began during the second cycling series. A slight distortion of the nickel band was also observable without the use of measuring devices.
- (4) Considerable outgassing occurred during the initial phase of the test leaving white deposits on the glass bell jar wall. It is suspected that most of the gaseous evolution took place in the RPG, possibly that of the electrolyte entrapped during the electroforming process.

The physical bond between electroformed nickel and RPG is sufficiently stable to permit nickel to be used as an intermediate bonding medium for graphite structures under moderate steady or cyclic temperature conditions for a limited but undefined period of time. The most severe conditions encountered by the Ni-RPG interface during the final cyclic series was five 25 minute cycles between 700° and 1400°F with a hold



period of approximately 6 minutes at the maximum temperature in each cycle. Further testing is required to define the various time-temperature cycle conditions under which this system is stable. Electroforming conditions should be selected to minimize occlusion of electrolytes in the nickel, or entrapment in the graphitic structure. Preliminary thermal treatments which eliminate such entrapped electrolyte slowly may be beneficial to the stability of nickel-graphite structures.

### c. Plasma Sprayed Nickel

Two short cylindrical specimens of RPG were supplied to the Wall Colmonoy Corporation of Santa Fe Springs, California, to have a layer of nickel deposited on the RPG by plasma spraying. After deposition of approximately a 0.010-inch layer of nickel, a tensile failure occurred in the outer layer of RPG and the band of nickel and a layer of RPG opened up due to the buildup of tensile residual stress in the nickel layer. The layer of nickel was still well bonded to the RPG and the nickel also exhibited fair ductility in bending the nickel layer over a 1/4-inch diameter rod. The nickel apparently wetted the carbon fibers without causing local failures. The interface is shown in Figure 47.

This type of joint may work much better on a material such as Carb-I-tex. The nickel layer would serve as an intermediate layer to which other metal parts could be joined by brazing or welding.

### SECTION VI TEST FIRING WITH FLOX/METHANE

Test firings with FLOX/methane during the Phase III and Phase IV programs were conducted during three periods in Marquardt's Magic Mountain Cell M-2. These three test periods were:

- I. August 2 September 9, 1968 47 runs
- II. December 28, 1968 February 11, 1969 30 runs
- III. July 9 July 25, 1969 27 runs

These test periods were cooperative test programs in support of two NASA programs (NAS3-11215 and NAS7-555) in order to achieve reduced hardware and test costs.

The main test objective of the NAS3-11215 testing was the demonstration of the maximum firing durability of the PG/Carb-I-tex thrust chamber with a 12:1 exit nozzle. The main objectives of the NAS7-555 testing were the determination of the structural and erosion resistance capabilities of a variety of advanced materials and fabrication concepts with the development of chamber design criteria and guides to improved materials and fabrication techniques.

### A. <u>Test Period I</u>

During the test period I, the primary objective was the evaluation of the FLOX/methane 100-lb thrust injector designed under Contract NAS3-11215. Two injectors of the same design were fabricated, one under NAS3-11215 and one under NAS7-555.

The injector design utilized like doublet injection elements and fuel film boundary layer injection. The original injector was described in physical detail in Reference 6. The final configuration is shown in Figure 48 and the photograph of Figure 49. The injector and copper heat sink chamber are shown in Figure 50.

Evaluation tests of the injector were conducted at the Marquardt Magic Mountain Cell M-2 sea level test site utilizing both copper heat sink chambers for combustion performance evaluation and graphite chambers for thermal and erosion characteristics. A listing of all test firings and a summary of results are tabulated in Table XI.

These test results indicated that the prime injector requirements of efficiency and durability were satisfied since C\* efficiency in excess of 95 percent was consistently demonstrated with smooth ignition and combustion. The requirement of film protection was not completely met, since excessive local erosion of the free standing PG chambers occurred at the plane of the injector face. Quantitative erosion data for three free standing PG chambers and one POCO graphite chamber are shown in Table XII. It is to be noted that the POCO chamber did not show evidence of upstream erosion or carbon deposition encountered by the free standing PG chambers, due to lower wall temperatures resulting from the much higher heat sink capacity of the POCO chamber. The PG chamber erosion pattern (Figure 51) and carbon deposition (Figure 52) were correlated with the regions which were covered by the fuel film jets. The erosion is also correlated with regions of relatively high oxidizer concentration as influenced by the injector spray fan pattern and combustion reactions. This possibility had been anticipated with provisions for modifying the injector to increase the boundary layer film protection at minimal compromise of C\* performance.

The two existing injectors were modified prior to the next test period, which included long duration chamber tests at altitude conditions. The injector modification consisted of adding an additional ring of fuel film injection elements consisting of 12 equally spaced, 0.005 inch diameter jets impinging on the chamber wall in the plane of the injector face. The existing original set of 6 jets impinging at a plane approximately 3/4-inch from the injector face remained, since the film produced by them provided adequate protection to the downstream chamber and throat region. A plastic chamber section was used during cold flow injector tests to evaluate the film protection coverage and spreading pattern of the additional holes (Figure 53). The spray pattern during water flow tests of the modified injector is shown in Figure 54.

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### B. <u>Test Period II</u>

The objective of test period II was to evaluate the structural and erosion characteristics of widely diverse refractory materials and composites in a common FLOX/methane combination environment during long duration runs (60 - 1800 seconds). The fabrication of more than 30 thrust chambers was undertaken between test period I and II. A number of chambers experienced fabrication problems and were either completed too late for the January 1969 tests or were not available for testing due to structural problems as discussed in the previous section. Twelve thrust chambers were used during Period II with an additional ten thrust chambers fired during test period III in July 1969.

The test period II runs are summarized in Table XIII. Briefly, the test items and firing durations were as follows:

- 1. Heat sink copper two chambers, No. 1, 8 runs, 45 seconds No. 2, 3 runs, 141 seconds
- 2. Free standing PG three chambers, 3 runs, 58 seconds
- 3. POCO graphite one chamber 5 runs, 5 runs, 194 seconds
- 4. Multilamina pyrocarbide (MRS 58) one chamber 1 run, 60 seconds
- 5. PG/Carb-I-tex (sea level) one chamber 1 run, 40 seconds
- 6. PG/Carb-I-tex (12:1 exit) one chamber 6 runs, 322 seconds.

Essentially, all of the 860 seconds of run time listed above was accumulated with one injector with no indication of injector plugging or overheating during operation.

Initial test firings during test period II with the injector modified by the additional fuel film jets showed wall erosion near the injector face. Accordingly, the final configuration also included the addition of a short deflection ring welded to the face of the injector just outside the circumference of the oxidizer doublets. This modification, shown in Figures 48 and 49, was very effective in reducing local erosion near the injector face. No overheating of the ring as shown was experienced.

The PG/Carb-I-tex chamber (SL-3) with an L\* of 18 inches, which was fired for 322 seconds, had been modified by attaching a 12:1 exit nozzle extension of a PG/carbon felt composite (RPG). The joint was formed by threading the exit nozzle and chamber. The inside and outside of the assembly were coated with a 0.040-inch layer of PG (Figure 55).

During the first 60 second firing run with the above chamber at altitude conditions using the no-flow ejector, the exit nozzle extension apparently failed early in the run. The mode of failure of the exit cone is not known since the parts of the cone were ejected sideways into the altitude bell rather than downstream. There was no sign of burning or erosion on the cone pieces. One possible explanation is that a lateral deflection of the chamber assembly against the diffuser inlet occurred during the start transient and cracked the exit cone.

After the first firing, the chamber was removed from the cell and examined. There was very little throat erosion. There was carbon deposit buildup within the chamber and throat. There was some minor local erosion at the injector end of the chamber. There did appear to be several hairline cracks in the PG inner wall. The appearance of the chamb er was similar to that of the first PG/Carb-I-tex chamber (SL-1) (Figure 56) which had been fired for 40 seconds.

The first PG/Carb-I-tex chamber (Figure 56) (which also developed hairline cracks) was pressurized after the 40 second firing run. At 50 psi there was leakage through the wall at the throat. It should be noted that all the uncoated 100-lb Carb-I-tex chambers leaked gas at all pressures when they were pressurized to 100 psig prior to coating.

Test firing of PG/Carb-I-tex chamber SL-3 with FLOX/methane was continued for five more runs. The maximum firing durations were limited by the pressure drop increases across the FLOX line filter. It is postulated that this was due to solid crystals of HF, since after each run, when the filter was allowed to heat up, the blockage disappeared. It was the objective of this firing series to run until some definite failure occurred in the chamber. At the beginning of the sixth run, after successful runs of 60, 47, 60, 71 and 68 seconds, there was an apparent burn through. Examination of the chamber after the run showed failure due to gradual erosion through the PG coating on the wall ahead of the throat and then a more rapid burn through in the Carb-I-tex. The PG inner wall in the throat region showed nearly uniform circumferential erosion. At local areas, where a very thin coating of PG remained, the PG/Carb-I-tex bond remained intact. There was no tendency for the PG to separate from the inner wall of the Carb-I-tex. The hairline cracks did not initiate any local erosion or failure (Figure 56).

The estimated PG liner erosion rate over the total burn time was about 0.12 mil/second at the pressure, temperatures and mixture ratios involved.

Erosion rates for the free standing PG chambers, POCO graphite, and copper chambers are presented in Table XIV. Throat erosion rates in the multilamina (MRS-58) chamber which had an inner lamina of ZrC/PG alloy (12 wt. % Zr) were similar in appearance and magnitude (0.19 mil/sec) to PG materials. A local erosion streak in the combustion chamber also indicated that the erosion characteristics of the alloy were similar to PG.

Structurally the ZrC-PG alloy chamber was similar to a three laminae PG chamber fired with  $F_2$ /BA1014 propellants during the Air Force PG chamber program (Reference 14). The maximum exterior wall temperature reached at the outside surface of the multi-lamina chamber was  $3150^{\circ}$ F as shown in Table XV. This table summarizes maximum wall temperatures measured using the calibrated extended-range photographic film technique. The estimated interior wall temperature based on heat transfer analyses was close to  $5000^{\circ}$ F.

### C. <u>Test Period III</u>

The objective of test period III was to continue the test firing evaluation of advanced chamber material systems not available during the period II tests. Twenty-seven test firing runs were completed with ten chambers between 9 July 1969 and 25 July 1969. The test firing summary is shown in Table XVI. The ten chambers fired included:

- 1. POCO graphite (three)
- 2. PG/Thornel/Resin Composite (two)
- 3. PG/Thornel/PG Composite
- 4. PG/Carb-I-tex
- 5. Carb-I-tex (uncoated)
- 6. HfC coated tantalum-tungsten
- 7. Copper impregnated POCO graphite

From these test firings, data were obtained on erosion rates, carbon deposition and structural integrity of the different material and fabrication concepts. The results are discussed below:

### 1. POCO Graphite

Three POCO graphite chambers were test fired for durations up to 60 seconds. Despite relatively slow heating time because of the heat sink effect, the throat erosion was as much as 0.32 mils/sec (Run 15). The POCO oxidized on the outer surface during the longer runs, and it was noticed during post fire proof tests that the porosity was increased by the firings.

### 2. <u>PG/Thornel/Resin Composite</u>

Of the two PG/Thornel/Resin composite chambers tested, good erosion data was obtained on Run 9 of 82 seconds duration. The erosion rate (Table XVI) was 0.14 mils/sec, which is a typical value for pyrolytic graphite. The outer wrap was depleted by oxidation from the atmosphere during the test firings, with the residue having the appearance of loose Thornel. It was concluded that the Thornel/Resin reinforcement did not offer any advantages over free standing pyrolytic for the test conditions involved.

### 3. PG/Thornel/PG Composite

The PG/Thornel/PG chamber was trimmed and pressure checked to 200 psig. However, in mounting the chamber on the injector with the tapered seal attachment, the clamping load caused a crack in the PG liner at the attach flare. To allow the test firing to proceed, a sealing compound was used to fill the crack in the liner flare and the test firing was accomplished satisfactorily. During the 65 seconds of firing a small hole was eroded in the side of the chamber through both the PG liner and Thornel overwrap. No crack or further failure was initiated by the burn through.

4. <u>PG/Carb-I-tex</u>

Chamber SL-1, fired previously for 40 seconds during Test Period II, was fired for 40 and 105 seconds during Runs 11 and 12, respectively.

The chamber pressure varied erratically during Run 12 because of carbon buildup in the throat which periodically was ejected. The overall throat erosion rate was 0.15 mils/sec. Post test examination of the chamber revealed heavy carbon deposits about two inches downstream of the injector face, and thin carbon deposits in several portions of the throat. The rest of the combustion chamber and contraction region was free of carbon deposits. The PG had been smoothly eroded away in a 90<sup>°</sup> segment of the contraction region, but no evidence of leakage through the Carb-I-tex was found. There were about eight hairline cracks in the throat PG coating, which did not cause any further damage to the Carb-I-tex wrap.

### 5. <u>Carb-I-tex (Uncoated)</u>

An uncoated Carb-I-tex chamber was given four test firings. During the longest run of 61 seconds (Run 19), the throat erosion was 0.41 mils/sec. The chamber was in good condition after the tests except for the throat erosion.

### 6. <u>HfC Coated Tungsten</u>

A tantalum-10 tungsten chamber coated with hafnium carbide by IITRI was test fired for 10 seconds during Run 27 using the test installation shown in Figure 31. The chamber is shown in Figure 57. The coating was completely removed from the throat during the test firing.

### 7. Copper Impregnated POCO Graphite

The two-inch long cylindrical section of copper impregnated POCO graphite was clamped between the copper adapter and POCO chamber as shown in Figure 32. Looking at the inside of the chamber after Runs 22, 23 and 24, a total of 60 seconds of firing, the film cooling impingement patterns were clearly visible. The heating pattern was clearly visible by the boundary of copper melting and depletion and by the oxidized areas on the outside of the POCO and the copper impregnated (Cu/POCO) sections. Inside the chamber, carbon deposit streaks built up in line and downstream of the six inside fuel film jets. There was no difference noted between deposits on the Cu/POCO and POCO chamber sections. In regions of no deposit the outside wall temperatures were coolest in line with the six inside fuel film jets.

Based on this limited testing and previous heating tests with the Cu-POCO, it would be concluded that the chemical and structural performance of the Cu/POCO would be the same as straight POCO graphite with the possible advantage of higher thermal conductivity and heat capacity.

### SECTION VII STRESS ANALYSIS

A stress analysis of a multilamina ZrC/PG thrust chamber was made to evaluate the possible advantages of this type of structure. The analysis was made for the conditions of a 100 lb thrust engine using  $FLOX/CH_4$  at a chamber pressure of 100 psia, a mixture ratio of 4.0, a C\* efficiency of 95%, and a combustion gas temperature of 6345<sup>o</sup>F.

The chamber wall consisted of six laminae of 0.040 inch thickness each, for a total wall thickness of 0.24 inch. Data for the thermal conductivity of ZrC in PG are very limited but are of the same order as pure PG. Therefore, the thermal conductivity was assumed to be identical to that of PG.

The specific heat was also assumed to be the same as for PG. Reference 4 data was used to obtain the density of ZrC/PG.

The transient temperatures in the combustion chamber wall (ID = 1.4 inches) for a 300 second firing are plotted in Figure 58. The outer chamber wall was assumed to be radiating to space with an emissivity of 0.8.

Several distributions of alloy composition were assumed. The first distribution studied consisted of 0% Zr content in the inner lamina, varying linearly to 30% Zr (by weight) in the outer lamina. The Zr content in each lamina was assumed constant, and increased between laminae by 6% increments.

A transient stress analysis was made of the cylindrical sections of the thrust chamber. The results are shown in Figures 59 and 60, which present the maximum stresses at about 300 seconds into a run at both surfaces.

The maximum stress on the inside surface (pure PG) is shown in Figure 59 to be about 11,000 psi in the axial direction. The maximum stress on the outside surface (30% ZrC) is shown in Figure 60 to be about 28,000 psi in the circumferential direction.

Alternate distributions of ZrC concentration (30% inside/0% outside; 30% constant; 0% inside/12% middle/0% outside; etc.) did not significantly reduce the stresses for the same basic chamber configuration. However, for the same ZrC distribution (0% inside, 30% outside), a large reduction in stresses is achieved by reducing the wall thickness to 0.12 or by increasing the chamber diameter to 2.8 inches. These comparisons are also shown in Table XVII.

### A. Stress Analysis Program

The Marquardt non-homogenous, anisotropic stress analysis computer program was used to calculate stress profiles for the engine configuration being studied. This program calculates stresses and radial displacements in a cylindrical tube of constant wall thickness. The mathematical model assumes an axially symmetric temperature distribution and incorporates internal pressure and residual stresses. The program was originally written for studies on pyrolytic graphite, which is a transversely isotropic material. The program can handle end conditions of complete axial restraint, finite end loads, or free ends; and it also takes into account growth (permanent deformation), creep, and temperature dependent material properties, together with differing material compositions. This is done by dividing the cylinder into a specified number of concentric cylindrical shell elements and considering the material properties at the mid-point of each element. The material properties are assumed constant for each element, and a composite stress distribution is obtained by determining the unknowns in each element and matching corresponding quantities on the boundaries. The calculations involve the solution of a set of (2N+1) linear equations, where N is the number of cylindrical shell elements. Another feature of the program is the so-called post run analysis, which corresponds to a cooling down of the cylinder under no external loads. The resulting final stresses in this case are due to the anisotropic residual stresses and to any growth during the loaded case.

In addition to this basic program, a version of the program was modified to handle chambers with delaminations. This was done by requiring that a positive (or tensile) radial stress could not exist between two delaminations. If the original calculations predicted such a situation, the stresses were redistributed so that this situation did not exist.

### SECTION VIII THERMAL and THERMOCHEMICAL ANALYSES

Thermochemical and heat transfer studies were made to define the severity and composition of the thrust chamber environment in a 1000 lb. thrust  $OF_2/B_2H_6$  rocket engine. Heat transfer analyses were made of heating rates with  $OF_2/B_2H_6$  and compared with the results of other investigators. The chemical composition of the combustion gases were used in a thermodynamic equilibrium analysis to evaluate chemical compatibility of various chamber wall materials including graphite, boron carbide, boron nitride, and tungsten as a function of temperature.

### A. <u>Heat Transfer Analysis</u>

Heat transfer analysis was done to predict the wall temperatures for several chamber design concepts. All analysis was done for a thrust chamber producing a space rated thrust of 1000 lbs. at a chamber pressure of 100 psia and a mixture ratio of 3.0, using a throat diameter of 2.6 inches.

The thrust chamber contours used in the materials selection are shown in Figure 61. Contraction ratios of 2.0 and 6.0 were used, with L\* values of 16 and 46, respectively. The portion of the chamber more than two inches downstream of the throat was not considered in calculating heat loads.

The combustion temperature of  $OF_2/B_2H_6$  at 100 psia is shown in Figure 62 over a range of mixture ratios. At a mixture ratio of 3.0 and a C\* efficiency of 95 percent, the combustion temperature is about 6000°F, which was used in the calculations of chamber heat loads.

The convective heating of the thrust chamber wall was calculated from the Bartz equation modified by the reference enthalpy method. The heat transfer coefficient and heat flux at the throat for a wall temperature of  $1000^{\circ}$ F is compared in Table XVIII with experimental data from several sources. It can be seen that the throat heat flux from all sources is fairly uniform, approximately 5 BTU/in<sup>2</sup> sec., regardless of the contraction ratio or type of propellant stratification used.

The total heating rate to isothermal chambers at various wall temperatures is shown in Figure 63. The total heating rate is somewhat greater for the contraction ratio of 2.0 than for the contraction ratio of 6.0. Turbulent convective heating without any film cooling was assumed throughout the chamber. An attempt to induce laminar heating rates at the throat of a 1K chamber with a contraction ratio of 8:1 is reported in Reference 15. However, the throat heat flux, shown in Table XVIII was still about 5.4 BTU/in<sup>2</sup> sec.

The cooling capacity by sensible heat absorption of various liquid propellants is shown in Figure 64. The amount of sensible heat which can be absorbed in the liquid phase depends on the supply temperature at which the propellant is provided. The maximum possible amount of cooling corresponds to supply just above the freezing point, with subsequent heating to the boiling point at the chamber pressure. Actual cooling systems would have to operate over a somewhat narrower temperature range to maintain liquid propellant throughout the system. The maximum possible sensible heat absorption of liquid  $B_2H_6$ is 140 BTU/lb., compared to 260 BTU/lb. for MMH.

The cooling capacity of diborane is indicated in Figure 65, which presents the potential heat absorption rate of fuel cooling as a function of the percent of the total fuel flow (0.68 lb/sec) which is used for cooling. If all of the diborane could be heated from the freezing point and vaporized, about 250 BTU/sec. could be absorbed. Additional heat could be absorbed by the vaporized diborane if the cooling system could transfer heat into the diborane gas. The enthalpy-temperature relationship for gaseous diborane (Figure 66) shows that gaseous diborane could absorb 740 BTU/lb. by being heated to  $1000^{\circ}$ F, which is approximately the limit temperature to avoid decomposition (Reference 15).

The temperature to which an isothermal 1K chamber (contraction ratio of 6:1) could theoretically be cooled by the diborane are shown in Figure 67. If all of the diborane were used for chamber cooling, and only the sensible heat capacity of the liquid were used, the isothermal wall temperature could not be reduced below  $4400^{\circ}$  F. This condition (the line labled QSENS(LIQ) in Figure 67 defines the theoretical limit of regenerative cooling with a liquid.

If regenerative cooling could use the heat of vaporization of the diborane, the lower line in Figure 67 could theoretically be achieved. However, the isothermal wall temperature is still too high for conventional types of regeneratively cooled structures. In order to achieve wall temperatures below the lower curve, the cooling of gaseous diborane would have to be utilized, perhaps by gaseous film cooling or by gaseous regenerative cooling. Both of these methods would be difficult to implement.

The principal conclusion to be drawn from the heat transfer analysis is that the cooling capacity of the diborane for the 1K engine is very limited, even for the optimistic and idealized heat balances used in this analysis.

### B. Thermochemical Analysis

Thermochemical calculations were performed to evaluate the suitability of a number of materials when exposed to the combustion products of  $OF_2/B_2H_6$ . Those materials evaluated were graphite, boron carbide, boron nitride and tungsten.

The analysis consisted of calculating the composition of mixtures of the combustion species in thermodynamic equilibrium with a large amount of the candidate wall material at temperatures from  $1000^{\circ}$ R to  $7000^{\circ}$ R.

The weight ratio of initial wall material to the combustion products was usually assumed to be 20:1. Any reduction in the initial amount of the wall material may indicate the potential for chemical reactions between the wall and the combustion species. However, it is impossible to estimate the rate of such reactions unless experimental data are available describing the kinetics of the reactions. Such kinetics data is not presently available for most of the combustion species and wall materials studied.

### 1. <u>Chamber Environment</u>

The principal gaseous combustion species of  $OF_2/B_2H_6$  for a mixture ratio of 3.0 and a chamber pressure of 100 psia are plotted in Figure 68 for temperatures from 1000°R to 7000°R. The principal gaseous species above 3000°R are  $H_2$ , BOF, H and HF. A more precise compliation of combustion products is given in Table XIX which is normalized to include solid species such as  $B_2O_3$ . Therefore, the mole fraction of gaseous species at low temperatures is somewhat smaller in Table XIX than in Figure 68.

Below  $3000^{\circ}$ R, the principal gaseous species are H<sub>2</sub> and BF<sub>3</sub>. The combustion products of OF<sub>2</sub>/B<sub>2</sub>H<sub>6</sub> also produce solid B<sub>2</sub>O<sub>3</sub> at low temperatures. Boric oxide (B<sub>2</sub>O<sub>3</sub>) is a glassy substance which melts at approximately 650°F, forming a viscous liquid with a boiling point of about 4000°F. This implies that B<sub>2</sub>O<sub>3</sub> will deposit or condense on portions of the chamber wall which are below 4000°F.

Another factor to consider in evaluating the chamber environment is the possibility that the  $B_2H_6$  will be used for film cooling of the chamber wall. In that case, the mixture ratio adjacent to the wall would be much lower than the overall value of 3.0 assumed in the analytical study. The use of  $B_2H_6$  film cooling would result in decomposition of the  $B_2H_6$ , creating a boundary zone with a large amount of  $H_2$  and boron. The boron might deposit on the chamber wall. This effect was found in the experimental measurements in Reference 15, which reported that about 90% of the gaseous species adjacent to the wall of chamber film cooled with  $B_2H_6$ . Near the throat, the effect of the decomposition of  $B_2H_6$  film coolant had diminished so that only 31% of the solid wall deposits was boron, while 61% was  $B_2O_3$ .

2. Graphite

### a. Compatibility with Combustion Species

The equilibrium composition of a mixture of the combustion products of  $OF_2/B_2H_6$  and a 20:1 weight addition of graphite is presented in Table XX. Reactions are indicated by the presence of carbon bearing reaction products. Therefore, it can be seen that reactions occur at all temperatures. Methane is the predominant product at temperatures below 4000°R, while carbon monoxide and acetylene become more prevalent above  $4000^{\circ}R$ .

b. <u>Compatibility with B</u>203-

A calculation of the free energy change for the reaction

$$7C + 2B_2O_3 = 6CO + B_4C$$

shows that the reaction will begin at about 3500°R.

c. <u>Compatibility with Boron</u>

Graphite is predicted on the basis of free energy change to react with boron at all temperatures. Again, as in the reactions with the combustion gases, the rate of the reaction cannot be predicted.

d. <u>Compatibility with H</u>2\_

Graphite is predicted on the basis of thermodynamic equilibrium to react with hydrogen at all temperatures. There is some data on the reaction rates between hydrogen and graphite (Reference 16), which becomes appreciable at about 3000<sup>O</sup>R.

### 3. Boron Carbide

### a. Compatibility with Combustion Species

The equilibrium composition of a mixture of the combustion products of  $OF_2/B_2H_6$  and a 20:1 weight addition of boron carbide (B<sub>4</sub>C) is given in Table XXI. The behavior of B<sub>4</sub>C is similar to that of graphite, in that some amount of carbon bearing reaction products are predicted at all temperatures. Boron carbide would be inert to attack by boron wall deposits, but would react with  $B_2O_3$ .

4. Boron Nitride

### a. Compatibility with Combustion Species

The composition of equilibrium mixtures of boron nitride with  $OF_2/B_2H_6$ is given in Table XXII. It is not possible to determine potential reaction by looking for the presence of boron bearing species, because there are many boron species present in the nominal combustion products. However, reactions or decomposition of BN may be indicated by the presence of nitrogen bearing species. Examination of the equilibrium compositions, listed in Table XXII, reveals some very small amounts of NH<sub>3</sub> and N<sub>2</sub> at 1000<sup>o</sup>R. The amount of these species is at a constant value at 2000<sup>o</sup>R and 3000<sup>o</sup>R, and the amount of solid BN is greater at these temperatures than at 1000<sup>o</sup>R. This behavior probably indicates insignificant reactions at these tempe ratures. The presence of small amounts of N<sub>2</sub> at 4000<sup>o</sup>R and 5000<sup>o</sup>R may indicate slight reactions, vaporization or dissociation.

b. <u>Compatibility with  $B_2O_3$ </u>

Boron nitride was predicted to react with  $B_2O_3$  at  $4300^{\circ}F$  (4750 $^{\circ}R$ ) according to the equation:

$$B_2O_3 + BN = 3B0 + 1/2N_2$$

c. Compatibility with Boron

Boron nitride would be inert to boron wall deposits.

d. Compatibility with Hydrogen

Boron nitride would react with hydrogen at temperatures above about  $2800^{\circ}$ F, but the rates are not known since heterogeneous reaction rates cannot be predicted theoretically.

### 5. Tungsten

### a. Compatibility with Combustion Species

The compatibility of tungsten with the products of  $OF_2/B_2H_6$  is very good, as shown by the equilibrium composition at 6500 <sup>O</sup>R shown in Table XXIII. Only small amounts of WF or tungsten oxides are predicted, implying little reaction. Tungsten borides were not included in this calculation because of the lack of thermodynamic properties, which also precluded calculating the compatibility of tungsten with boron or  $B_2O_3$ .

### b. Compatibility with Hydrogen

Tungsten does not react with hydrogen at any temperature.

### 6. Summary

The results of the thermochemical analysis lead to the following conclusions:

a. Of the materials evaluated, tungsten is the least reactive with the combustion products of  $OF_2/B_2H_6$ . Tungsten has the disadvantages of high density (19.3 gm/cc) and marginal ductility, despite its high melting point (6120°F) and resistance to chemical erosion.

b. The feasibility of using graphite as the thrust chamber material for  $OF_2/B_2H_6$  must be evaluated by engine test firings, since the rates of predicted reactions are unknown. Graphite has the advantage of low density (1.5 - 2.2 gm/cc) and a very high sublimation temperature (6600°F). Toughness or effective ductility of graphite is attainable with composite graphitic structures such as Carb-I-tex.

c. Boron nitride will probably have somewhat less reaction with the combustion products of  $OF_2/B_2H_6$  than graphite up to a temperature of 4000°R.

d. Boron nitride may have advantageous properties for use in intermediate temperature regions, compatible with its sublimation temperature ( $4500 - 5400^{\circ}$ F) adjacent to the injector, due to its inertness in contact with boron and B<sub>2</sub>O<sub>2</sub> wall deposits.

e. Boron carbide ( $B_4C$ ) is probably similar to graphite in its chemical reactions with the combustion products of  $OF_2/B_2H_6$ , and is limited to lower temperatures because of its melting point of 4260°F. Brittle behavior of  $B_4C$  would also be of concern.

### C. Thrust Chamber Concepts

Various types of thrust chamber construction and cooling were evaluated in order to devise thrust chamber concepts to meet the following requirements:

Propellants:	$OF_2/B_2H_6$
Thrust (space):	1000 lbs
Chamber pressure:	100 psia
Mixture ratio:	3.0
C* efficiency:	95 percent
Firing duration:	1000 seconds

### 1. Graphite - Film Cooled

The feasibility of using graphite thrust chambers must be evaluated by test firings, since the preceding section indicates possible chemical erosion, but does not answer the question of reaction rate. Reaction with the combustion gases could be excessive for wall temperatures as low as  $3000^{\circ}$  F. It is not possible to keep the wall temperature below  $3000^{\circ}$  F by film cooling with reasonable amounts of film cooling or propellant stratification. For example, the analysis of Reference 15 shows that 40 percent of the propellant would be required in stratified flow (mixture ratio = 1.0) along the wall of a 1K chamber to reduce the boundary layer temperature at the throat to  $5000^{\circ}$  R ( $4540^{\circ}$  F). The performance loss expected for even larger amounts of boundary layer cooling required to obtain a  $3000^{\circ}$  F boundary layer temperature would be unacceptable.

### 2. Conduction Cooling

Conduction cooling is achieved by conducting heat from the throat through a high thermal conductivity chamber wall to a low wall temperature region near the injector. The low wall temperature is obtained by film cooling from the injector, ordinarily by a liquid fuel. The effectiveness of conduction cooling is limited by the tendency of the liquid fuel to boil off of the wall as the wall temperature exceeds the boiling point of the fuel. The cooling effectiveness of the gaseous film downstream of the liquid film is also low because of mixing with the combustion gas core.

### 3. Pyrolytic Graphite/Carb-I-tex Composite

A composite graphitic thrust chamber made by depositing pyrolytic graphite on Carb-I-tex has been tested at the 3K thrust level with CIF<sub>5</sub>/Hydrazine blend with excellent results (Reference 17). Chambers of this type will be tested with  $OF_2/B_2H_6$  by JPL.

A pyrolytic graphite/Carb-I-tex chamber has characteristics of very light weight, an impervious wall which permits good sealing at the injector attachment, and the lowest chemical erosion rates of any type of graphite material. The thickness of pyrolytic graphite which can be produced in this configuration is limited to a thickness/radius ratio of approximately 0.10. The inside wall temperature at the throat will approximately equal the local boundary layer temperature within 60 seconds and therefore the erosion rates by the products of  $OF_2/B_2H_6$  may be excessively high.

### 4. Edge Oriented Pyrolytic Graphite

A thrust chamber made of stacked washers of pyrolytic graphite would have the greatest durability and lowest erosion of any graphitic material.

The restart capability and low erosion of such a nozzle designed and fabricated by Marquardt was recently demonstrated by the AFRPL (Reference 18) during test firings with CIF<sub>3</sub>/Hydrazine blend. Pyrolytic graphite in the edge oriented configuration has very high thermal conductivity in the radial direction, so that the inside wall temperature of the 1K nozzle would require about 300 seconds to reach steady state temperatures.

The allowable erosion would probably not be limited by the thickness of the pyrolytic graphite, unlike the case of the pyrolytic graphite coating on Carb-I-tex. Nonetheless, the chemical erosion rates might be great enough to degrade performance. The weight of an edge oriented pyrolytic graphite nozzle of the size shown in Figure 61, a contraction ratio of 2.0, and an outside diameter of 8 inches, would be 32 pounds, an acceptable weight for a 1K engine.

### 5. <u>Regenerative Cooling</u>

The results of the heat transfer analysis (Figure 67) show that the inside wall temperature of a 1K thrust chamber could not be cooled below  $4500^{\circ}$ F by regenerative cooling with liquid  $B_2H_6$ . Therefore, some composite design would be required which would use high temperature coatings or a high temperature inner wall with the proper thermal conductivity to transfer heat to a regeneratively cooled outer shell. Many problems arise with such concepts, including thermal stresses within the inner wall, heat storage within the wall, and selection of an inner wall material which would be resistant to chemical erosion.

### 6. <u>Ablatives</u>

Graphite phenolic ablatives would be subject to the same types of chemical attack by the combustion products of  $OF_2/B_2H_6$  as graphite. Silica phenolic ablatives would probably also react rapidly with combustion species. Ablatives do not appear attractive for this application.

### 7. Radiation Cooling

A radiation cooled thrust chamber must have a thin, high conductivity, impervious wall. The chamber material must be resistant to thermal cyclic fatigue.

Tungsten might be considered for a radiation cooled thrust chamber. However, its resistance to thermal cycling and shock loading is questionable.

Wire wound tungsten has been tested with some success as a throat insert (Reference 19).

Its structural integrity as a free standing chamber is not known.

### 8. Transpiration Cooling

Transpiration cooling with liquid B<sub>2</sub>H<sub>6</sub> would be difficult to achieve because of decomposition of the diborane. Furthermore, transpiration cooling with any liquid has the problem of controlling the liquid flow rate. Film cooling of the chamber wall from many closely spaced injection slots would avoid some of the disadvantages of transpiration cooling. However, neither scheme is particularly attractive.

### 9. Endothermic Cooling

Endothermic cooling of a rocket nozzle has been demonstrated recently at the 3000 pound thrust level by test firings at the AFRPL. The endothermic nozzles were fabricated by Marquardt using pyrolytic graphite washers, although the cooling concept is applicable to many other types of nozzle materials. Test results are presented in Reference 18.

The principal heat transfer mechanisms operative in an endothermically cooled nozzle are shown schematically in Figure 69. A material which will absorb heat by endothermic phase changes is packed into the coolant storage cavities within the pyrolytic graphite washers. The endothermic coolant ideally has gaseous reaction products which pass through exit slots and into the boundary layer, providing a film cooling effect.

The ultimate effect of the endothermic coolant is to keep the inside wall temperature at a lower temperature than it would be without the endothermic cooling. If the nozzle is cooler, chemical erosion of the nozzle by the combustion gas will be retarded or eliminated.

A preliminary analysis was made of the applicability of endothermic cooling to the 1K  $OF_2/B_2H_6$  engine. The results of the analysis were that pyrolytic graphite

washers could be cooled to  $3500^{\circ}$ F in the combustion chamber. Maximum throat temperatures using NH<sub>4</sub>Cl or Si<sub>3</sub>N<sub>4</sub> coolants would be 4200°F and 4500°F, respectively. About 21 pounds of Si<sub>3</sub>N<sub>4</sub> or 67 pounds of NH<sub>4</sub>Cl would be consumed during a 1000 second firing.

The chemical erosion rates of pyrolytic graphite at the temperatures mentioned might be acceptable, although this could not be predicted without test firing data.

The preliminary design results were based on heat loads undiminsihed by film cooling. Hence, engine performance would not be penalized by the cooling system, unlike the situation encountered with film cooled engines.

### 10. Pyrolytic Boron Nitride

Boron nitride appears to be less reactive in the combustion products of  $OF_2/B_2H_6$ than graphite to at least 4000°R. Furthermore, it may be completely nonreactive with selected species such as  $B_2O_3$  and boron which will be present in very large amounts along the chamber walls which are film cooled by  $B_2H_6$ . Boron nitride can be made as a pyrolytic deposit by Raytheon Co. with isotropic physical properties and in much greater thicknesses than anisotropic boron nitride or anisotropic pyrolytic graphite. However, it is unlikely that a combustion chamber could be made entirely of BN without excessive amounts of film cooling because of its relatively low sublimation temperature (5400°F or less). Boron nitride might be valuable in constructing a composite thrust chamber, however. The boron nitride could be used in the chamber section adjacent to the injector which could be film cooled without excessive amounts of cooling. The boron nitride would be inert to deposits of boron or boron oxide in this region.

### 11. Composite Chamber

The results of the thermochemical and heat transfer analysis lead to the conclusion that a composite chamber design of the type shown in Figure 70 would be applicable to  $OF_2/B_2H_6$ . The boron nitride chamber liner would be kept below 4000°F with  $B_2H_6$  film cooling.

Pyrolytic boron nitride appears to be the best form for this application. The tungsten throat would be kept below  $5500^{\circ}$ F by film cooling. The expansion nozzle could be made of a composite graphite, since the chemical erosion rates (proportional to static pressure) would be low in this region.



### SECTION IX CONCLUSIONS

- 1. Fabrication techniques have been demonstrated for several different pyrolytic refractory composite thrust chamber components including:
  - a. PG vapor deposited on Carb-I-tex.
  - b. PG infiltrated carbon felt.
  - c. PG infiltrated high modulus carbon fiber (Thornel).
  - d. Multilamina pyrocarbide alloys.
  - e. Pyrocarbide infiltrated carbon felt.
  - f. Free standing PG filament wound with high modulus carbon fiber (Thornel) and infiltrated.
- 2. New material concepts which were explored for application to rocket thrust chambers, but require additional development, include:
  - a. Vitreous carbon reinforced with carbon fiber.
  - b. Copper impregnated isotropic graphite as a conduction cooled liner in a PG/Thornel structural shell.
  - c. Silicon nitride infiltrated carbon felt.
  - d. Metal/carbon joints formed by plasma spraying or electroforming nickel on carbon fiber composites.
- 3. Test firing of PG lined thrust chambers with FLOX/methane demonstrated the structural soundness of these pyrolytic refractory systems.
- 4. Pyrolytic graphite erosion rates in FLOX/methane were small but finite and sensitive to injector design and fuel film coolant distribution.
- 5. Thermal stress analytical studies provide an approach to optimizing wall thickness to handle cold, transient heating and steady state stress distribution in free standing composite thrust chambers.
- 6. Thermochemical analysis of materials for the  $OF_2/B_2H_6$  propulsion system have indicated that two materials which may be more suitable for thrust chamber walls than graphite are tungsten and boron nitride. Both of these may be made by chemical vapor deposition and possibly incorporated into composite structures.

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### SECTION X REFERENCES

- 1. Marquardt Report 5907, "Pyrolytic Graphite Rocket Thrust Chamber Development", Contract NAS 7-54, 31 July 1962.
- 2. Marquardt Report 5992, "Pyrolytic Refractory Thrust Chamber Development", Contract NAS 7-54, 20 May 1963.
- 3. Marquardt Report 6068, 'Refractory Thrust Chambers for Spacecraft Engines'', Contract NAS 7-262, 22 March 1965.
- 4. Marquardt Report 6115, "Pyrolytic Refractory Materials for Spacecraft Thrust Chambers", Contract NAS 7-373, 15 December 1966.
- 5. Marquardt Report 6142, "Pyrolytic Refractory Materials for Spacecraft Thrust Chambers", Contract NAS 7-555, 4 October 1968.
- 6. Marquardt Report 6147, 'Space Storable Thrustor Investigation'', Contract NAS 3-11215, 11 June 1969.
- 7. Contract No. 952290, California Institute of Technology, Jet Propulsion Laboratory.
- 8. Marquardt Report 6149, "Test and Evaluation of a Passively Cooled Thrust Chamber for Fluorinated Propellants (U)", Contract FO4611-68-C-0037, 19 December 1969, CONFIDENTIAL.
- Leeds, D.H., Bauer, D.W., et al, "Orthogonally Isotropic Wool Base Versus Anisotropic Rayon Base Carbon and Graphite Composites", presented at Ninth Biennial Conference on Carbon, Boston, June 1969, Super Temp Co.

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**References** (Continued)

- 10. Pierson, H.O., et al, "Effects of Deposition Temperature on Properties of Pyrolytic Carbon/Carbon Felt Composites", Sandia Laboratories, SC-DR-68-264, May 1968.
- Goble, G.G. and Campbell, F.S., "Tension and Compression Test Devices for Filament-Wound Composite Rings", presented at S.E.S.A. Meeting, San Francisco, California, 28 October - 1 November 1968.
- Lewis, J.C., "A Comparative Study of the Gaseous Oxidation of Vitreous Carbon and Various Graphites at 1500<sup>o</sup> to 3000<sup>o</sup>K", Report of the Plessy Co., Ltd., England, and the Beckwith Carbon Corp., Van Nuys, California, 1968.
- 13. IITRI-B6058-40, "Protective Coatings for Refractory Metals in Rocket Engines", Final Report, Contract No. NAS 7-431.
- Marquardt Report 6118, "Free Standing Pyrolytic Graphite Thrust Chambers for Space Operation and Attitude Control, Phase II: Small Scale Testing (U)", Contract AF04(611)-10790, AD 382057, 29 May 1967, CONFIDENTIAL.
- 15. Aerojet-General Corporation Report 659-F, "FLOX-Diborane Technology-Boundary Reaction", NAS 7-659, September 1969.
- 16. Rogers and Sesonske, Los Alamos Scientific Laboratory Report LAMS-2896, VC-4, Chemistry, TID-4500 (19th Ed.), August 1962.
- 17. Marquardt Report 6149, "Test and Evaluation of a Passively Cooled Thrust Chamber for Fluorinated Propellants (U)", AFRPL-TR-69-232, December 1969, CONFIDENTIAL.
- 18. Marquardt Report 6150, "Test and Evaluation of Endothermically Cooled Pyrolytic Graphite Nozzles (U)", AFRPL-TR-69-233, December 1969, CONFIDENTIAL.
- 19. AFRPL-69-54, "High Chamber Pressure Test Firing of a Wire-Wound Tungsten Throat Insert Nozzle (Test Nozzle No. 5) (U)", March 1969, CONFIDENTIAL.

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### TABLE I

### EXPLANATORY NOTES FOR FIGURE II-1

Contract	Note	
NAS7-54	l.	Pure boron nitride chambers fired as throat inserts with
		$N_2O_4/Hydrazine$ blend. Erosion greater than PG.
NAS7-54	2.	Boron doped PG essentially the same erosion rate as PG in
		N <sub>2</sub> O <sub>4</sub> /Hydrazine blend, 0.2 mil/sec.
NAS7-54	3.	Pyroid was a 1/2-inch thick wall PG chamber with uncontrollable
		delaminations. Not considered a practical fabrication approach.
NAS7-54	4.	The first 40:1 exit PG chamber made. PG quality and wall
		thickness control very good.
NAS7-555	5.	Test firing with $N_0O_{l}/Hydrazine$ blend gave 1/3 PG erosion
		rate. One chamber fired 305 seconds.
NAS7-555	6.	Test fired 300 seconds at 100 psia plus 18 seconds at 200 psi.
	_	
(Ref. 14)	' <b>(</b> •	Fourteen chambers fired with $N_2O_4/HYZ$ , total run time 1022 sec.
. ,		Fourteen chambers fired with LF /BA1014, total run time 507 sec.
		Eleven chambers fired with 12/GH2, total run time 505 sec.
NAS7-555	8.	Wall thickness 0.25 inches, five laminae, 20 wt % Zr in outer
		laminae with pure PG inside. Proof pressure failure by crack
		in inner lamina.
NAS7-555	9.	Fired for 60 seconds with erosion at throat of 0.2 mil/sec.
Ref. 14	10.	Three laminae, 0.125 wall thickness, fired 137 seconds.
AFRPL(Ref. 14)	11.	Wall thickness $\approx$ 0.050-in., failures during vibration tests.
IR&D	12.	Filament wound carbitex 713 walls, 1/4-inch thickness, with
		several internal delaminations.
NAS7-555	13.	Carbon felt impregnated with PG, machined to final shape with
		0.10 wall, then recoated internally with 0.050 PG.
		Impregnated carbon felt cracked on cooling.

51

# TABLE I (Continued)

### EXPLANATORY NOTES FOR FIGURE II-1

Contract	Note	
AF04(611)-10790	14.	Four chambers fired total of 2000 seconds.
Ref. 17	15.	Fired 300 Sec.
NAS3-11215	16.	Maximum run time 322 seconds, throat erosion rate 0.12 mil/sec.
	17.	Design goal, 1800 sec. firing at 100 psia.
Ref. 7	18.	Scheduled for firing at 100 psia.
F04(611 <b>)-</b> 70C-000	719.	To be fired at 100 psia with $ClF_5/non-deposit fuel.$
NAS7-555	20.	PG impregnated carbon felt (RPG) machined to fit over free standing PG chamber and bonded pyrolytically.
NAS7-555	21.	Thornel-50 (carbon yarn) wound over PG chamber and bonded by PG infiltration. Composite tensile strength over 50,000 psi.
NAS7 <b>-</b> 555	22.	Thornel-50 (carbon yarn) wound over PG chamber and bonded by resin impregnation and pyrolyzed. Lower strength and higher external oxidation than 22 above.

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### TABLE II

### THRUST CHAMBER PROCESS DATA

Run No.	Deposition Temperature	Pressure	Gas Flow Rates (lpm)		Low Rates Lpm)
	(°C)	(mm Hg)	CH <sub>14</sub>	A	Cl2
MRS-50	1850	10	2.4	0.5	0.15
MRS-51	1850	10	2.4		
MRS-52*	1850	10	3.0	treat scan	(an) with
MRS-53*	1850	10	3.0	0.5	0.2
MRS-54	1850	10	2.4	0.5	0.15
MRS-55	1850	10	2.4	0.5	0.15
MRS-56	1825	7.5	2.4	0.5	0.15
MRS-57	1825	6.0	2.4	0.5	0.15
MRS-58	1825	6.0	2.4	0.5	0.15
MRS-59*	1825	6.0	2.4	0.5	0.15
MRS-60*	1825	6.0	2.4	0.5	0.15
MRS-61†	1825	6.0	2.4	0.5	0.15 to 0.05
MRS-62†	1825	6.0	2.4	0.5	0.15 to 0.05
MRS-63	1825	6.0	2.4	0.5	0.20
MRS-64	1825	6.0	2.4	0.5	0.15
MRS-65	1825	6.0	2.4	0.5	0.15
MRS-66*	1825	6.0	2.4	0.5	0.15
MRS-67	1825	6.0	2.4	0.5	0.15

\*Male Mandrel deposit

<sup>†</sup>Radially graded composition

\*Hafnium alloy

Report S-989

## TABLE III

THRUST CHAMBER PHYSICAL PROPERTY DATA

	Run No.	Position (in.)	Mandrel	Composition (wt % Zr)
	MRS-50	0 12	ATJ ATJ	27.5 29.0
	MRS - 51	0 12	ATJ ATJ	O O PG
	MRS-56	Bottom Top	ATJ ATJ	11.6 14.5
	MRS-57	Bottom Top	ATJ ATJ	11.8 14.7
	MRS - 58	Bottom Top	ATJ ATJ	11.5 14.4
	MRS-62	Bottom Top	ATJ ATJ	8.0 10.0
	MRS-65	Bottom Top	ATJ ATJ	10.1 10.6
	MRS-67	Bottom Top	ATJ ATJ	12.1 15.8
ł		1	l	<b>{</b>

### TABLE IV

# THICKNESS PROFILES (Inches)

- \			,	
DISTANCE FROM INLET (BOTTOM) (Inches)	0°	90°	180°	270°
1	0.402	0.403	0.402	0.399
2	0.367	0.366	0.365	0.367
3	0.372	0.368	0,367	0.373
4	0.371	0.373	0.373	0.375
5	0.367	0.371	0.366	0.366
6	0.353	0.357	0.353	0,351
7 Threat	0.325	0.325	0.325	0.325
8 Throat	0.272	0,272	0.275	0.270
9	0.238	0.234	0.239	0.238
10	0.185	0.180	0.174	0.195
11	0.133	0.133	0.137	0.131
12 (Top)	0.113	0.114	0.114	0.110

### MRS-50 (Throat Diameter = 0.770 Inches)

O (Bottom)	0.260	0.265	0.260	0.285
l	0,295	0.320	0.300	0.275
2	0.350	0.310	0.350	0,325
3	0.330	0,280	0.345	0.345
<u>4</u> .	0.295	0.245	0.300	0.310
5	0.245	0.210	0,245	0.255
6	0.210	0.180	0.210	0.215
7 Throat	0.195	0.180	0,200	0,205
8	0.165	0.150	0.170	0.175
9	0.135	0.130	0.150	0.145
10	0.120	0.110	0,130	0,125
11	0.095	0,090	0.100	0.100

	MRS-52	MRS-53	MRS-55
Bottom	~0.035	~0.040	~0.130
Throat	~0.040	~0.045	~0.075
Тор	~0.035	~0.040	~0.030

# TABLE IV (Continued)

THICKNESS PROFILES (Inches <u>+</u>0.003)

MRS-56

DISTANCE FROM INLET (BOTTOM) (Inches)	0°	90°	180°	270°
O (Bottom)	0.118	0.118	0.115	0.115
1	0.113	0.113	0.108	0.110
2	0.108	0.106	0.104	0.106
3	0.107	0.106	0.106	0.107
4	0.107	0.105	0.104	0.106
5	0.106	0.105	0.103	0.106
6	0.098	0.098	0.098	0.098
7 Throat	0.096	0.097	0.097	0.098
8	0.093	0.096	0.095	0.093
9	0.078	0.086	0,083	0.075
10	0.056	0.065	0.061	0.050
11	0.040	0.042	0.042	0.038

MRS-57

	and the second state of th	****		
O (Bottom)	0.176	0.177	0,176	0.177
l	0.168	0.172	0.167	0.172
2	0.148	0.148	0.148	0.147
3	0.147	0.148	0.147	0.146
4	0.149	0.150	0.147	0.149
5	0.152	0.152	0.149	0.150
6	0.149	0.149	0.147	0.146
7 Throat	0.144	0.144	0.142	0.141
8	0.140	0.138	0.140	0.141
9	0.125			
10	0.094	0.085	0.111	0.112
11	0.066	0,059	0.072	0.083

# TABLE IV (Continued)

THICKNESS PROFILES

MRS-	-58
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DISTANCE FROM INLET (BOTTOM) (Inches)	0°	90 <b>°</b>	180°	270°
0 (Bottom)	0.190	0.183	0.178	0.181
l	0.173	0.171	0.165	0.171
2	0.156	0.150	0.150	0.152
3	0.154	0.151	0.150	0.148
4	0.154	0.151	0.151	0.150
5	0.155	0.153	0.151	0.150
6	0.151	0.150	0.149	0.147
7 Throat	0.148	0.146	0.143	0.145
8	0.143	0.146	0.143	0 <b>.1</b> 42
9	0.119	0.133	0.140	0.121
10	0.087	0.105	0.116	0.090
11	0.060	0,070	0.068	0,060

MRS-62

O (Bottom)	0.175	0.176	0.173	0.172
1	0.170	0.168	0.164	0.163
2	0.153	0,154	0.153	0.153
3	0.155	0.158	0.155	0.156
<u>14</u>	0.156	0.159	0.157	0.157
5	0.163	0.162	0.160	0.159
6	0.152	0.153	0.156	0.155
7 Throat	0.151	0,152	0.151	0.151
8	0.144	0.147	0.138	0.137
9	0.126	0.130	0.119	0.111

TABLE IV (	Continued)
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THICKNESS PROFILES (Inches <u>+</u>0.001)

MRS-65

DISTANCE FROM INLET (BOTTOM) (Inches)	0°	180°
O (Bottom)	0.208	0.208
l	0.175	0.179
2	0.156	0.158
3	0.156	0.158
4	0.155	0.158
5	0.155	0.158
6	0.150	0.150
7 Threat	0.144	0.146
8 Inroat	0.138	0.136
9	0.109	0.108
10	0.067	0.064
Тор	0.054	0.050

(Inches <u>+</u>0.002) MRS-67

DISTANCE FROM INLET (BOTTOM) (Inches)	٥°	90°	180°	270 <b>°</b>
O (Bottom)	0.171	0.170	0.169	0.169
l	0.150	0.152	0.152	0.152
2	0.147	0.149	0.148	0.146
3	0.148	0.147	0.148	0.148
<u></u>	0.148	0.148	0.148	0.148
5	0.145	0.146	0.145	0.145
6	0.139	0.138	0.136	0.139
7	0.137	0.138	0.136	0.137
8 8	0.134	0.137	0.134	0.136
9	0.130	0.131	0.129	0.132
10	0.101	0.103	0.102	0.105
Тор	0.077	0.080	0.078	0.078

	DEP.	DEP.	PRESSURE	GAS	S FLOW R	ATES (lp	m )
NON NO.	(°C)	(hrs)	(mm Hg)	CH)4	А	Cl <sub>2</sub>	H2
MQF-1	1200	999. auto	20.0	0.6	0.1	0.1	0.2
MQF-2	1100	88ad 0.68	20.0	0.6	0.1	0.05	0.2
MQF-3	1100	yan uni	10.0	0.6	0.1	0.05	0.2
MQF-4	1500	20	10.0	<b>1.</b> 0	0.5	0.05	talit mys
MQF-5	1500	40	10.0	2.0	0.5	0.05	tuna ayag
MQF-6	1500	28	5.0	2.0	0.5	0.05	-0013
MQF-7	1350	40	5.0	3.0	0.5	0.25	1.0
MQF-8	1350	12	5.0	1.0	0.25	0.10	<b>6</b> 0 64
MQF-9	1350		10.0	1.0	0.25	0.25	4403 may
MQF-10	1350	12	10.0	1.0	0.25	0.25	<b>6</b> 500

TABLE V PROCESS DATA FOR FELT IMPREGNATION

### TABLE VI

### AVERAGE PHYSICAL PROPERTIES

	DENSITY (gm/cc)		COMPOSITITON		
RON NO.	BULK	TOLUENE	(wt % Zr)	POSITION	
MQF-4	0.9	1.52	2.2	Middle	
MQF-5	1.07	1.67	4.1	Middle	
MQF-6	1.05	1.67	3.3	Middle	
MQF-7	1.12	1.80	24.1	Middle	
MQF-10	1.22	1.69	27.5	Middle	

TABLE	V	II
discription of the local distances of the	- 25	0011004

### LOCAL DENSITY OF RUN NO. MQF-5

	DENSITY (gm/cc)					
RUN NO.	BOTTOM POSITION		MIDDLE POSITION		TOP POSITION	
	BULK	TOLUENE	BULK	TOLUENE	BULK	TOLUENE
MQF-5 Inside Outside	1.13 1.10	1.42	1.12 1.02	1.67	1.23 0.86	1.99



### TABLE VIII

### FLEXURE STRENGTH

(4-point loading - 1 inch upper span, 2 inch lower span)

RUN NO.	TEMPERATURE (°C)	FLEXURE STRENGTH (psi)
MQF-6	20 20 20 20 20 20	3610 4440 3670 3700 4290 3670 3900 Average
MQF-10	20 20 20 20 20 20	5900 4690 7030 4740 6045 5700 Average
	1800 1800 1800 1800 1800	5715 8205 7300 4730 6485 6490 Average

### TABLE IX

RUN NO	DEP. DEP. PRESS		PRESSURE	GAS FLOW RATES (1p					pm)	
(°C) (hrs	(hrs)	(mm Hg)	He	CH <sub>4</sub>	А	NHZ	Cl2	BC13	H2	
MQP-106	1850	4	15.0	an ca	4.5	0.5	574 au	0.6		4.5
MQP-107	1650	10	1.0	7.0			1.2		0.8	
BN-794	1850	17	1000 <b>88</b> 8	*** ••						

### PROCESS DATA - MONOLITHIC DEPOSITS

# TABLE X

RESULTS OF NOL RING TENSILE TESTS OF PG/THORNEL-50 COMPOSITES

		Position and Thickness						
Spool No.	Description	A; 0.050 in.	B; 0.10 in.	C; 0.20 in.	D; 0.05 in. over 0.20 in felt			
		Te	Tensile Stress (Ksi); Two Specimens					
1	Standard composite	21.8 13.7	25.6 26.4	10.9 10.1	9.0 5.7			
2	Chopped carbon fibers added	48.2 33.6	9.2 26.5	15.9 14.9	9.4 9.1			
3	Yarn roughened during winding	7.6 22.2	10.8 15.8	8.2 11.1	8.9 5.9			
4	Furfural and carbon black added	14.5 24.7	22.8 15.9	19.0 15.9	13.0 8.6			

ТX	
TABLE	

# SUMMARY OF THE TEST PERIOD II INJECTOR EVALUATION FIRINGS

$ \begin{array}{ c c c c c c c c c c c c c c c c c c c$		······										
$ \begin{array}{ c c c c c c c c c c c c c c c c c c c$	Remarks		Facility shakedown and combustion performance. Instrumentation and propellant conditioning discrepancies resolved.	Chamber shattered, hard start	High response P <sub>c</sub> instrumentation installed to monitor effect of Mignition sequence and thermodynamic state.	Steady state run to evaluate effect of propellant thermodynamic state.	Ignition sequence and propellant thermodynamic state revised to achieve steady state transition.	Steady state run to evaluate effect of propellant thermodynamic state.	Steady state run to evaluate effect of propellant thermodynamic state. Oxidizer valve inadvertently closed during run.		Run cut prematurely, smooth start.	Chamber failed at ignition, hard start, marginal ignition.
$ \begin{array}{ c c c c c c c c c c c c c c c c c c c$		<sup>2</sup> *	68885889488	11		84		95	80	-		}
$ \begin{array}{ c c c c c c c c c c c c c c c c c c c$	2 1	z	N   262.4 258.0 213.5 213.5 213.5 2204.6 2214.0 			6.771		240.2	173.5	1	-	!
$ \begin{array}{ c c c c c c c c c c c c c c c c c c c$		16f	8889999988	11		04		54	39	1		1
$ \begin{array}{ c c c c c c c c c c c c c c c c c c c$		N/cm <sup>2</sup>	73.8 69.9 69.7 69.7 69.7 69.7 69.7 69.7 69.7	11		55.4		65.5	53.4	ł		ł
$ \begin{array}{ c c c c c c c c c c c c c c c c c c c$		psia	0228882020 0228886000	11	۸	80.4	on l y	95	77.4	1	lized	1
Run (1968)         Contriguration (1968)         Contriguration (1968)         Contriguration (1968)         Contriguration (1968)         Lt         Injector (1, cm)         Duration (1961)         Lt         Mun (1963)         Contriguration (1964)         Lt         Injector (1, cm)         Duration (1964)         Lt         Injector (1964)         Lt         Injector (1964)         Lt         Lt         Lt         Injector         Duration (10         Lt         Lt           1         2         Auge (Coper 18         45,72         No         001         10         1         -4           7         8         Auge (Coper 12         30.48         No         001         10         1         -4           8         8         Coper 12         30.48         No         001         10         1         -4           1         1         13         Auge         Coper 12         30.48         No         001         10         1         -4           1         1         1         30.48         No         001         19         -4         -4           1         1         30.48         No         001         19         -4         -4           1         1 <td< td=""><td></td><td>0/F</td><td>8.02 6.26 6.13 6.13 6.26 7.17 6.26 6.57 6.57 6.57</td><td>::</td><td>data</td><td>5.3</td><td>da ta</td><td>6.1</td><td>13.1</td><td>1</td><td>: stabi</td><td> </td></td<>		0/F	8.02 6.26 6.13 6.13 6.26 7.17 6.26 6.57 6.57 6.57	::	data	5.3	da ta	6.1	13.1	1	: stabi	
Run No. $\overline{\text{Lormingration}}$ $\overline{\text{Larmicr}}$ $\text{Larmic$		t data sec	*********	11	lgnition	5.3	Hgnition	5.8	6.1	1	Data not	1
$ \begin{array}{ c c c c c c c c c c c c c c c c c c c$		Category*		~~~	~~~~	1, 2	~~~~	1, 2	~	3	3	3
Run No.         Configuration           No.         (1968)         Configuration           1         2 Aug. 2 2 Aug. 6 Aug. 6 Aug. 6 Aug. 6 Aug. 6 Aug. 6 Aug. 7 6 Aug. 6 Aug. 7 6 Aug. 7 8 Aug. 7 9 048 7 7 7 8 8 001           11         13 Aug. 8 Aug. 7 9 Aug. 7 9 049 7 12         14 5.72 8 Aug. 7 8 Aug. 7 9 Aug. 7 8 Aug. 7		Duration	4400000000	lgn. 1gn.	0.000 2.00 2.00 2.00 2.00 2.00 2.00 2.0	10.0	0.0 0.5 0.5 0 0.5 0 0 5 0	6.0	20.0	lgn.	-3	lgn.
Run No.         Date         Configuration           No.         (1968)         In.         Kistler           No.         (1968)         In.         Ised           1         2 Aug.         Copper         Is         45.72         No           2         2 Aug.         Copper         Is         45.72         No           4         6 Aug.         Copper         Is         45.72         No           5         6 Aug.         Copper         Is         45.72         No           6         6 Aug.         Copper         Is         45.72         No           6         8         Aug.         Copper         Is         45.72         No           11         13 Aug.         Copper         Is         45.72         No           12         19 Aug.         Copper         12         30.48         No           13         19 Aug.         Copper         18         45.72         Yes           14         20 Aug.         Copper         45.72         Yes           15         20 Aug.         Copper         45.72         Yes           15         20 Aug.         Copper         45.72		Injector S/N	<u>8888888888888888888888888888888888888</u>	<u>8</u> 8	<u>55555</u>	001	<u>55555</u>	001	- 19	001	100	001
Run No.         Date (1968)         Configuration L*           No.         (1968)         L*           1         2 Aug. 2 Aug. 6 Aug. 6 Aug. 6 Aug. 6 Aug. 6 Aug. 7 6 Aug. 7 6 Aug. 7 8 Aug. 7 8 Aug. 7 8 Aug. 1 1         Copper 1 8 45.72 1 9 0.48 1 45.72 1 9 0.48 1 45.72 1 1 13 Aug. 8 Aug. 7 9 0.48 1 1 13 Aug. 7 0 0 0 1 18 45.72 1 45.72 2 2 2 Aug. 7 0 0 0 1 18 45.72 2 2 2 Aug. 7 0 0 0 1 18 45.72 2 2 2 Aug. 7 0 0 0 1 18 45.72 2 2 2 Aug. 7 0 0 0 1 18 45.72 2 2 2 Aug. 7 0 0 0 1 18 45.72 2 2 2 Aug. 7 0 0 0 1 18 45.72 2 2 2 Aug. 7 0 0 0 1 18 45.72 2 2 2 Aug. 7 0 0 0 1 18 45.72 2 2 2 Aug. 7 0 0 0 1 18 45.72 2 2 2 Aug. 7 0 0 0 1 18 45.72 2 2 2 Aug. 7 0 0 0 1 18 45.72 2 2 2 Aug. 7 0 0 0 1 18 45.72 2 2 2 Aug. 7 0 0 0 1 18 45.72 2 2 2 Aug. 7 0 0 0 1 18 45.72 2 2 2 0 0 0 1 18 4	ç	Kistler Used	₽₽₽₽₽₽₽₽₽ ₽₽₽₽₽₽₽₽₽₽₽₽₽	88	Yes Yes Yes Yes	No	Yes Yes Yes Yes	Ŷ	Ŷ	Ŷ	No	Ŷ
Run No.         Date (1968)         Confi in.           No.         (1968)         in.           1         2 Aug. 2 Aug. Copper 18         copper 18           5         6 Aug. 6 Aug. Copper 18         6 Aug. 6 Aug. Copper 18           6         6 Aug. Copper 12           7         8 Aug. Copper 12           8         8 Aug. Copper 12           8         8 Aug. Copper 12           10         8 Aug. Copper 12           8         8 Aug. Copper 12           11         13 Aug. PG 001         18           12         15 Aug. Copper 12           13         19 Aug. Copper 12           14         20 Aug. Copper 18           15         20 Aug. Copper 18           16         20 Aug. Copper 18           17         20 Aug. Copper 18           18         20 Aug. Copper 18           21         20 Aug. Copper 18           21         21 Aug. Copper 18           22         24 Qug. Copper 18           23         22 Aug. Copper 18           24         22 Aug. Copper 18           25         24 Qug. Copper 18           26         22 Aug. Copper 18           27         22 Aug. Copper 18	guratio	* 5	45.72 30.448 40.448 30.4487 30.44787 30.44777 30.4477777777777777777777777777777777777	45.72	45.72 45.72 45.72 45.72 45.72	45.72	45.72 45.72 45.72 45.72 45.72	45.72	45.72	45.72	45.72	45.72
Run No.         Date (1968)         Chamber (1968)           No.         (1968)         Copper           2         Aug.         Copper           3         6         Aug.         Copper           4         6         Aug.         Copper           5         6         Aug.         Copper           6         6         Aug.         Copper           7         8         Aug.         Copper           6         6         Aug.         Copper           11         13         Aug.         Copper           12         15         Aug.         Copper           13         19         Aug.         Copper           14         20         Aug.         Copper           13         19         Aug.         Copper           15         20         Aug.         Copper           15         2         Aug.         Copper           21         2         Aug.         Copper           22         Aug.         Copper         2           23         2         4ug.         Copper           23         2         4ug.         Copper	onfi		22228888882222	81 18 18	<u>8</u> 8 8 8 8 8 8	18	<u>8</u> 8 8 8 8 8	18	18	18	8	18
Run No.         Date (1968)           1         2 Aug.           2         2 Aug.           3         6 Aug.           6         8 Aug.           6         8 Aug.           7         8 Aug.           8         8 Aug.           9         8 Aug.           9         8 Aug.           10         8 Aug.           13         19 Aug.           14         20 Aug.           15         2 Aug.           16         2 Aug.           17         20 Aug.           18         20 Aug.           21         22 Aug.           23         22 Aug.           23         22 Aug.           25         22 Aug.           26         26 Aug.           27         29 Aug.           27         29 Aug.           28         29 Aug.           27         29 Aug.           28         29 Aug.           27         29 Aug.           28         29 Aug.		Chamber	Copper Copper Copper Copper Copper Copper Copper Copper	PG 001 P0C0	Copper Copper Copper Copper	Copper	Copper Copper Copper Copper	Copper	PG 002	PG 005	PG 007	PG 007
R Not		Date (1968)	2 Aug. 6 Aug. 6 Aug. 6 Aug. 6 Aug. 8 Aug. 8 Aug. 8 Aug. 8 Aug.	13 Aug. 15 Aug.	19 Aug. 20 Aug. 20 Aug. 20 Aug. 20 Aug.	20 Aug.	21 Aug. 22 Aug. 22 Aug. 22 Aug. 22 Aug.	22 Aug.	22 Aug.	26 Aug.	29 Aug.	29 Aug.
		Run No.	- 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4	12	17 55 F 33	18	21 22 23 23 23 23	24	25	26	27	28

SUMMARY OF THE TEST PERIOD II INJECTOR EVALUATION FIRINGS . .....

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	Remarks		GCH <sub>4</sub> system installed. Revised ignition sequence	Hard start Hard start Hard start	Soft start Soft start	Soft start	Hard start, ign. overpressure marginal	GF2 system installed. Revised ig-	HILION Sequence. SOLE Starts.	GF2 off prematurely. Steady state. Evaluate effect of	properiant thermoopmenic state. used transition from ignition transient to steady state.	Soft initial start. Mard restart. Fuel system start dynamics	Revised fuel start sequencesoft start [Thermal performance and erosion	Final data Pin hole in side of chamber	Thick wall graphite heat sink (POCO), minimum L*	Thin wall PG chamber, maximum L*		y weight)			
 	2 <sup>4</sup>	,	100							183	£	1	<u>5</u> 6	97.5	282	<u>76 00</u>		ture (b		_	
	2	z	1							200.2	5. 12	1	238.8 193.5	175.7	111	231.3 235.8 235.8		LO2 Mix		eight \$)	
		1bf	1							193	¥	1	53 43.5	39.5	111	222		1.5%		as (w	
		N/cm <sup>2</sup>	60							180	ن <i>۲</i>	1	63.7 60.3	55.4	51.3 52.7 52.0	65.1 86.5 86.5		LF2/1		ural g	87.5 11.6 0.5 0.4
		psia	87		yln			-	Á LU	168	4.00	ł.	92.4 87.4	80.4	74.4 76.4 75.4	94.4 102.4 96.4		82.5%		ed nati	ae gen
	0/F 5.6		5.6	n data c			n data c 6.5 6.1				:	3.8 5.1 6.2	6.2	4.0 3.7 3.6	3.5 3.45 5.22 (IDIZER:	(IDIZER:	FLOX	JEL:	Liquifi	Metha Ethan Propa Nitro	
	t data	sec	5		, Jignitio		_			ا مر د	/.0	1	2.5	19.0	3.3 13.4 20.6	4-5 8-6 16-0	8		Ē		
	Category∻		F	~ ~ ~		N -		~ ~	<b>7 7</b>		× •	~	ñ		٤	~				ice point	
	Duration	sec	5	0.5	1.76	5.3	8.0	0.5 1	 	4 6 6	y.y	lgn.	23		20	6				o performan	
	Injector S/M		002	002	002	005	802	002	002	888	700	002	002		002	002			stics	lants on to	
Ę	Kistler	Used	8	N NO NO	Yes	Yes	2	Yes	Yes	22	2	₹	£		¥	울			racter i	propel	
uratio		5	45.72	45.72 45.72	45.72 45.72	15.72	5.72	45.72	5.12	£2.72 £2.72	\$2. <b>/</b> 2	45.72	45.72		27.05	45.72		mance	on cha	m both	hite
onfig		in.	8	<u>8</u> 8 8	. <u>2</u> 22	<u></u>	<u>o</u> <u>co</u>	∞ :	<u>0</u> <u>00</u>	<u>@</u> @ 9	<u>e</u>	8	£		10.65	8		erfor	eros i	:) fro	; grap
	Chamber		Copper	Copper Copper	Copper	Copper	Copper	Copper	Copper	Copper	Copper	PG 003	PG 006		88	PG 004		ustion p	mal and	Time (sec	Pyrolytic
	DATE	(1968)	29 Åug.	29 Aug. 29 Aug. 30 Aug.	30 Aug.	30 Aug.	30 Aug.	4 Sept.	4 Sept.	4 Sept.	4 sept.	5 Sept.	5 Sept.		9 Sept.	9 Sept.	Category:	- Com	2. Ign 3. Thei	1 1 1 1	ي ا ا
	Run		29	825	124	582	8 E	8	<u>3</u> 3	33:	¥	1	ft2		9 <del>1</del>	47	*			Å t	<b>a.</b>
TABLE XII

SUMMARY OF EROSION OF THREE FREE STANDING PG CHAMBERS AND ONE POCO GRAPHITE CHAMBER

		[med])	Pug vod	Thiertor	e د د لا	PG CI	namber Mid_Chan	Erosion				+ aC s- ch		
		CDam	per and	I INJECTOI	race		MIQ-DIN	mber				Turoar		
	ې بې	Init	ial	Fina.l Thick	Wall mess	Init:	ial	Final	• •	[nitia]	·		Final	
Ē	ime	日	Wall Thick.	Between Film Orifices	In-Line Film Orifices	A	Wall Thick.	Wall Thick.	A	Ð	Wall Thick	Ð	Wall Thick.	Interna.l Erosion Rate
$\sim$	sec)	(în.)	(in.)	(in.)	(in.)	(in.)	(in.)	(in.)	(in.)	(in.)	(in.)	(in.)	(in.)	(in./sec)
<u>N</u>	<u>т</u> , т	1.396	0.066	0.029 to 0.041	0.050 to 0.053	1.396	0*066	0.042 to Deposit	0.814	0.931	0.057	0.928	0.043 to 0.052	0.00060 to 0.00016
H	6	1.396	0.066	<0.000 to 0.026	0.034 to 0.048	1.396	0*066	0.043 to Deposit	0.826	0.929	0.053	0.926	0.044 to Deposit	0.00040 to 0.0
н	9	1°407	0.062	0.047 to 0.050	0.056 to 0.062	1. <sup>407</sup>	0*005	0.041 to Deposit	0.827	0.931	0.052	0.925 to 0.928	0.038 to Deposit	0.00074 to 0.0
				2 - -		Ъ	oco chai	nber*						
		Initial ID (in.)		Fir II ir	1a.l ) 1. )							Throat	Interna Rate (in./se	l Erosion c)
FI	9.6	0.830		0.0	332 50 350								0.00005 to 0.001	

\* No measureable erosion at injector region

Report S-989

THE AIGUAIDT VAN NUYS, CALIFORNIA

TABLE XIII

TEST PERIOD II SUMMARY OF AMBLENT THRUST CHAMBER FIRING TESTS IN MAGIC MOUNTAIN CELL M-2

28 December 1968 to 11 February 1969, M-2 Test No. 6060

	Remarks	GF <sub>2</sub> /LCH <sub>4</sub> Ignition	GF2/LCH4 Ignition	FLOX flow decay due to tank near empty	FLOX flow decay due to tank near empty	Chamber shattered at ignition	Ambient purge prior to ignition GF <sub>2</sub> /LCH <sub>4</sub> Max. P <sub>c</sub> spike = 75 psia	Ambient purge prior to ignition GF2/LCH <sub>4</sub> Max. P <sub>C</sub> spike = 75 psia
	Run Objectives (Note 1)	(2), (4)	(2) , (4)	(2), (4)	(2), (4)	(3)	Ξ	Ξ
	лс (я)	92	33	8	8	1	1	1
	C* (ft/sec)	6340	6460	6620	6450	1		ł
iki oyaqı filitik shirtin u	P.c (psia)	98.3	100.4	88.4	<b>66.</b> 4	ł	ł	1
	0/F Ratio	5.07	4.98	3.55	2.48	ł	ł	1
	Duration (sec)	7.52	12.18	10.5	10.13	;	0.5	0.5
irat ion	l njector	X29401, S/N 001 18 weep holes (12 Hi Angle 6 Lo Angle)	X29401, S/N 001 18 weep holes (12 Hi Angle 6 Lo Angle)	X29401, S/N 001 18 weep holes (12 Hi Angle 6 Lo Angle)	X29401, S/N 001 18 weep holes (12 Hi Angle 6 Lo Angle)	X29401, S/N 001 18 weep holes (12 Hi Angle 6 Lo Angle)	X29401, S/N 001 18 weep holes (12 Hi Angle 6 Lo Angle)	X 29401, S/N 001 18 weep holes (12 Hi Angle 6 Lo Angle)
Confia	Chamber .	Cu L* = 18 in.	PG, SL-6	Cu L* = 18 in. Kistler P <sub>C</sub>	Cu L* = 18 in. Kistler P <sub>C</sub>			
	Date	28 Dec. 1968	28 Dec.	28 Dec.	28 Dec.	31 Dec.	6 Jan. 1969	6 Jan.
	5 ý	9 1	2	~		Ŵ	<b>.</b>	7

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TABLE XIII (Continued)

TEST PERIOD II SUMMARY OF AMBIENT THRUST CHAMBER FIRING TESTS IN MAGIC MOUNTAIN CELL M-2

28 December 1968 to 11 February 1969, M-2 Test No. 6060

A DE LA DESERVACIÓN D		Remarks	Max. P <sub>c</sub> spike = 100 psia	Data not stabilized	Erosion nil	Erosion ni l	Chamber shattered at ignition	GF <sub>2</sub> /GCH <sub>4</sub> Ignition procedure Upstream chamber erosion	Upstream erosion nil Erosion at mid-chamber in line with oxidizer doublets	Upstream erosion nil Erosion at mid-chamber in line with oxidizer doublets
	Run	Objectives (Note 1)	(1)	(1), (2)	(2), (3)	(2), (3)	(1), (3)	(3)	(3)	(3)
		п <sub>с</sub> (%)	l I	6	19	æ	}	95	63	6
		C* (ft/sec)	ļ	6100	6260	6000	U S	6550	0049	6080
a international de la constantion de la	I	P <sub>c</sub> (psia)	3	4.66	6.66	97.4	8	97.4	95.9	93.4
	0/F	Ratio	1	4.12	4.5	4.27	ł	4.18	4.56	3.9
		Duration (sec)	0.5	7.02	31.52	65.0	1	22.9	20.9	15.5
	uration	Injector	X29401, S/N 001 18 weep holes (12 Hi Angle 6 Lo Angle)	X29401, S/N 001 18 weep holes (12 Hi Angle 6 Lo Angle)	X29401, S/N 001 18 weep holes (12 Hi Angle 6 Lo Angle)	X29401, S/N 001 18 weep holes (12 Hi Angle 6 Lo Angle	X29401, S/N 001 18 weep holes (12 Hi Angle 6 Lo Angle)	X29401, S/N 001 18 weep holes (12 Hi Angle 6 Lo Angle)	X244401, S/N 001 12 weep holes (Hi Angle) Long splash ring	Same, with short splash ring
	Config	Chamber	Cu L* = 18 in. Kistler P <sub>c</sub>	Cu L* = 18 in. without Kistler	POCO No. 2 L* = 14 in.	POCO No. 2 L* = 14 in.	PG, SL~7 L* = 14 in.	PG, SL-11 L* = 14 in.	PG, SL-1 L* = 14 in.	PG, SL-9 L* = 14 in.
		Date	6 Jan.	6 Jan.	7 Jan.	7 Jan.	8 Jan.	8 Jan.	13 Jan.	14 Jan.
and the second sec	Run	No	ŵ	ۍ.	ć	• 	2	é	÷	15,

TABLE XIII (Continued)

TEST PERIOD II SUMMARY OF AMBLENT THRUST CHAMBER FIRING TESTS IN MAGIC MOUNTAIN CELL M-2

28 December 1968 to 11 February 1969. M-2 Test No. 6060

	Remarks	Hard start caused by defective Teflon seal at biprop blank plate	One axial streak in line with fuel doublet (Note 2)	Erosion ni l	Restart without engine disassembly after Run 18. Facility FLOX valve did not open.	Good run Throat dia. change 0.8232 to 0.8480 in.	Good run Throat dia. change 0.8480 to 0.8880 in.	Good run Erosion nil
	Run Objectives (Note 1)	ł	(3)	(3)	(3)	(9)	(9)	(3)
	η <sub>c</sub> (%)	ł	46	92.5	1	89.5	87	66
	C* (ft/sec)	1	0079	6300	}	6100	6000	6850
	P <sub>c</sub> (psia)	ł	106.9	100.9	32.4	96.4	6.46	101.4
	0/F Ratio	8	4.27	4.29	·l	4.10	4.43	4.42
	Duration (sec)	ł	ę	. 1.		63.7	67.5	36.7
uration	Injector	X24401, S/N 002 18 weep holes Short splash ring. Biprop valve modified with blank plate	X24401, S/N 001 with 18 weep holes Short splash ring	X24401, S/N 001 with 18 weep holes Short splash ring	X24401, S/N 001 with 18 weep holes Short splash ring	X24401, S/N 001 Short splash 18 weep	X24401, S/N 001 Short splash 18 weep	X24401, S/N 001 Short splash 18 weep
Confia	Chamber	PG, SL=2 L* = 14 in.	MRS-58 Pyrocarbide multilaminate L* = 14 in.	POCO No. 2 L* = 14 in.	POCO No. 2 L* = 14 in.	Cu L* = 12	Cu L* = 12	PG/Carbitex SL-1
	Date	15 Jan.	16 Jan.	23 Jan.	23 Jan.	1 Feb.	1 Feb.	1 Feb.
	Run Šo un	16.	500 2 2	<u>ي</u>		<b>50</b>	21°	22.

TABLE XIII (Continued)

## TEST PERIOD II SUMMARY OF AMBIENT THRUST CHAMBER FIRING TESTS IN MAGIC MOUNTAIN CELL M-2

28 December 1968 to 11 February 1969. M-2 Test No. 6060

CANCELLAND	***********************	Concernation and the second	Roccepterocometacione			COCCASE INVITATION	and the second	THE REAL PROPERTY AND INCOME.	AND DO
	Remarks	P <sub>c</sub> tap failure	Good run Cell transient ~20 sec	Good run Min. P <sub>cell</sub> = 3.12 psia Chamber bell failure at ~ 12 sec	Good run Throat erosion rate 0.221 mil/sec	un run	Good run Throat erosion rate 0.129 mil/sec	пл роод	Chamber failed at throat
Run	Objectives (Note 1)	(2)	(5)	(2)	(3)	(3)	(3)	(3)	(3)
	η <sub>c</sub> (%)	1	93	4.46	92.6	9.46	96.5	94.3	ł
	C* (ft/sec)	1	0049	6470	6350	6500	6690	6500	1
	P <sub>c</sub> (psia)	ł	101.9	107.4	97.4	96.4	98.4	92.4	ł
0/5	Ratio	8	4.27	4.22	4.18	4.23	4.67	4.50	ł
	Duration (sec)	10.7	10.2	59.8	47.5	59.5	71.3	68.2	15.7
ation	Injector	X24401, S/N 002 Biprop valve blank plate	X24401, S/N 001 Short splash 18 weep	X24401, S/N 001 Short splash 18 weep	X24401, S/N 001 Short splash 18 weep	X244401, S/N 001 Short splash 18 weep	X244401, S/N 001 Short splash 18 weep	X24401, S/N 001 Short splash 18 weep	X24401, S/N 001 Short splash 18 weep
Configu	Chamber	POCO No. 2	Cu Ch L* = 12 € = 12	PG/Carbitex SL-3 ¢ = 12	Same without nozzle expansion	Same without nozzle expansion	Same without nozzle expansion	Same without nozzle expansion	Same without nozzle expansion
	Date	4 Feb.	6 Feb.	7 Feb.	10 Feb.	10 Feb.	11 Feb.	11 Feb.	11 Feb.
	No.	23,	24.	25	26°	27.	28°	29.	30°
					A REAL PROPERTY AND A REAL	A CONTRACTOR OF A CONTRACTOR O			

TEST PERIOD II SUMMARY OF AMBIENT THRUST CHAMBER FIRING TESTS IN MAGIC MOUNTAIN CELL M-2

28 December 1968 to 11 February 1969, M-2 Test No. 6060

TOTAL ACCUMULATED RUN TIME:

213.4 sec	862.7 sec	852.0 sec	322.0 sec
		(Phase 11)	<pre>hase !!) (PG/Carbitex SL-3)</pre>
		l n j ec tor	Chamber (
		One	One
624	6457) 6267	5	5
hase	hase	Time	Time
Test P	Test P	Total	Total
<u>_</u>	2.	ŝ	4°

Notes:

- Run objectives: (1) = Ignition, (2) = Performance, (3) = Streaking and life, (4) = Facility check, (5) Altitude test, (6) Film conduction cooling investigation fices G
- Caused by misaligned film jet, which occurred when low angle weeper jets were reopened from 12 weeper configuration. 2°
- All data points taken at stabilized conditions 6 to 16 seconds from engine ignition ň
  - Oxidizer: FLOX 82.5% LF<sub>2</sub>/17.5% LO<sub>2</sub> (by weight) Fuel: Methane (LCH<sub>4</sub>) ŝ



## TABLE XIV

## Throat Throat Diameter Erosion Run Diameter Chamber Chamber Run After Duration Before Rate Number Type Number Run Run mil/sec in. in. sec (cm/sec) (cm)(cm)Copper, $L^* = 12$ in. 20 63.7 0.8232 0.8428 0.154 \_ ---= 30.48 cm (2.1407)(0.000391)(2.0909)Copper, $L^* = 12$ in. 0.8845 0.8428 21 67.5 0.309 ----= 30.48 cm (2.2466)(0.000785)(2.1407)180(1) 10, 11, 18 0.8188 0.8150 0 POCO 2 POCO Graphite (2.0798)(2.0701) (0)0.8278 SL 11 13 22.9 0.8258 0.044 PG Streak (2,0975)(2.1026)(0.000712)0.8345 0.8440 SL 1 14 20. 0.238 PG Streak (2.1428)(2.1196)(0.000605)0.8277 0.8365 PG Streak SL 9 15 15. 0.25 (0.000635) (2.1024)(2.1247)Multilamina MRS 58 60. 0.7907 0.8135 17 0.19 (0.000482) (2.0084)(2.0663)40. 0.8553 0.8572 0.024 PG/Carbitex Carbitex SL-1 22 (0.000061)(2.1724)(2.1773)0.8042 0.8333 PG/Carbitex/ Carbitex SL-3 25 59.8 0.243 RPG Altitude (2.0427)(2.1166)(0.000617) 107.(2) PG/Carbitex/ Carbitex SL-3 26, 27 0.8333 0.8609 0.129 RPG Altitude (2.1166)(2.1867)(0.000328) 155.2(3) 0.145(4) $0.906^{(4)}$ 0.8609 PG/Carbitex/ Carbitex SL-3 28, 29, 30 (2.1867)(2.3012)(0.000368)RPG Altitude

SUMMARY OF AVERAGE THRUST CHAMBER THROAT EROSION DATA, TEST PERIOD II -- DECEMBER 1968 TO FEBRUARY 1969

NOTES:

(1) Run 10 (30 sec), Run 11 (60 sec), Run 18 (90 sec)

(2) Run 26 (47.5 sec), Run 27 (59.5 sec)

(3) Run 28 (71.3 sec), Run 29 (68.2 sec), Run 30 (15.7 sec)

(4) Hole burned in vicinity of throat

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## TABLE XV

EXTENDED RANGE FILM TEMPERATURE DATA, TEST PERIOD II TESTS

Run	Max.	Temp.	Run	Chember	Remarks
No.	°F	(°K)	sec	Cliamber	
11	2760	(1789)	65	POCO No. 2	Measurement taken at internal surface of exit
13	2730	(1772)	22.9	PG SL-11	External surface at throat
14	2880	(1855)	20.9	PG SL-1	External surface at throat
17	3150	(2005)	60	Pyrocarbide Multilaminar MRS-58	External surface at throat (See Note 1)
18	2700	(1755)	81.1	POCO No. 2	Same as Run 11

NOTES :

- 1. Temperature measurement questionable due to fogging of camera window.
- 2. Tests of the 100-pound FLOX/Methane engine.

ΧVΙ	
TABLE	

# TEST PERIOD III - 100 LB THRUST FLOX/METHANE ENGINE TEST DATA

SYAAMAAR		Performance and facility check. Throat carbon deposits.	Repeat Run 1 Oxidizer flow decay	$P_{\rm C}$ increase to 164.4 psia from nominal. Heavy throat deposits.	Oxidizer leak at injector plate. Data invalid. Throat deposits.	0/F Survey	P <sub>c</sub> monitor inoperative	High O/F due to fuel freezing. Transient data	Duration run. P <sub>c</sub> steady	Duration run. P <sub>c</sub> increased to 126.4 psia from nominal.	Oxidizer leak at injector plate. Run terminated. Slight throat deposit.	O/F variation due to oxidizer two phasing.	Duration run. P <sub>c</sub> increased to 131 psis then dropped back to nominal.	0/F - Deposition survey	
ц н	LBS	64.0	63.0	63.0	1	43.0	1	30.5	52.0	52.0	1 1	50.0	50.5	50.0	
 *: ::	<del>8</del> 2	96.7	96.6	95.9	1	4.46	· 1	73.0	7.66	98.2	l	104.0	96.0	83.1	
С* БЧТ/	SEC	6651	6631	6593	1	6219	1	4520	6804	67.65	I I	06TL	668 <b>1.</b>	5739	
P <sub>C</sub> (NOM)	PSIA	4.0LL	107.4	106.4	1	±1.°17	1	52 <b>.</b> 4	82.4	96.4	I	100 <b>.</b> 4	85.4	83 <b>.</b> 4	
0/F	(MON)	01.4	4.30	4.39	1	6.72	1	8.66	4.20	4.58	l E	6.00	5.20	5.62	
THROAT EROSION	RATE MIL/SEC	N "A "*	0.15	N.A.	N "A "	0°067				41.0		0	0.15	0.28	
RUN	SEC	20°0	23.0	26.1	10,0	20°0	10°0	20.0	60,1	82.0	5.0	40°0	105.0	30°0	
ATTON	INJECTOR	too n/s x244401	=	=	x24401 s/n ooz	100 N/S Tothtor	=	=		x244401 s/n ool	x24401 s/n 002	too n/s tottot	F	100 N/S 104442X	
CONFIGUI	CHAMBER	Poco No. 1 L $* = 14$ in.	E	Ŧ	Poco No. 2 L* = 10 in.	FG/TH/resin No. 1	F	=	Ħ	FG/TH/resin No. 2	Poco No. 2 L* = 10 in.	PB/Carbitex SL-1	=	Poco No. 2 L* = 10 in.	lable
ЪАТТЕ		69/6/1	69/6/L	69/6/L	7/10/69	69/TT/L	69/11/2	69/11/2	69/11/2	69/TT/2	7/15/69	7/16/69	7/16/69	7/16/69	Not Avail
RUN	°	н	N	m	t=	ŝ	9	4	ω	6	10	1	12	13	*

SST DATA	REMARKS		Repeat Run 13	$0/F$ , $F_n$ survey	Oxidizer leak at injector plate. Run terminated	Injector fuel/oxidizer momentum angle change performance check	Run aborted due to photographic difficulty	Minor throat deposits.	Injector performance check. Deep erosion streak in throat.	0/F deposition	Propellant flows not stabilized. Two phasing.	Same	Materials test	Injector fuel/oxidizer momentum angle change performance check	Same	Coating removed at throat but intact in chamber.
INE TH	ц н	Sa	51.5	43.0	1	32.5	1	37 • 5	40°0	56.0	26.0	38.0	144°5	49.0	52.0	29.5
EDNEE	*	<u>8</u>	81.7	86.1	I T	67.6	т 1	73.0	65.4	100.0	80.0	87.0	96.6	87.6	88.9	78.0
ETHANI	С* FrT/	別 の	5627	5949	1	1631	1	5006	58 <del>44</del>	6873	14757	5668	6584	6004	1019	5019
FLOX/M	P <sub>c</sub> (NOM)	PSIA	80.4	fe.4	I I	52.4	R I	52°14	72.4	2,101	67 °4	81.4	93.4	87.4	4°68	63.4
THRUST	_0/F	(MOM)	69°4	4.98	1	5.67	1	5.62	₩2°₩	6.2	10°15	7.57	6.22	6.0	5.97	67.7
100 LB	THROAT EROSION	RATE MIL/SEC	3 <b>1</b> °0	0.32	1.14	0°0†	0	14°0	0	0.22	0.19	0.26	0 <b>.</b> 14	0*06	0.18	5.0
- III Q	RUN DIRATION	SEC	30°0	60.0	7.0	20°0	15.0	61.0	20°0	60,0	10.0	15°0	35.0	20°0	45°0	10,0
ST PERIO	ATION	INJECTOR	TOO N/S		x244401 s/n 002	F	2	\$	200 N/S TO41401	100 N/S TO41401	x24401 s/n ool	=	=	s/N 002	=	TOttiZX
11E	CONF 1GUF	CHAMBER	Poco No. 2 L* = 10 in.	=	Carbitex SL-2	E	*	5	Poco No. 3 L* = 14 in.	8	3-piece chamber	E	=	PG/TH/FG No. 1	5	Tantalum- Tungsten/ HfC coating
	ה∆ת <del>ו</del> ד	27 7 7 7 7	7/16/69	7/16/69	7/18/69	7/23/69	7/23/69	7/23/69	7/23/69	7/23/69	7/24/69	7/24/69	7/24/69	7/25/69	7/25/69	7/25/69
	RUN	°0N	1 <sup>,1</sup>	5	16	17	18	67	50	ដ	22	દર	54	ୟ	26	27

TABLE XVI (Continued)

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## TABLE XVII

## SUMMARY OF STRESSES IN ZrC/PG COMPOSITE THRUST CHAMBERS

Zr-C	Temperature	ID	t	Circumfere (p	ntial Stress si)
Distribution	Distribution	(in.)	(in.)	Inside	Outside
0% inside 30% outside linear	Transient at 300 seconds into run 5280°F - in 2350°F - out	1.4	0.24	-10,000	28,500
30% inside 0% outside linear	Transient at 300 seconds into run 5280°F - in 2350°F - out	1.4	0.24	-80,500	39,200
30% constant	Transient at 300 seconds into run 5280°F - in 2350°F - out	1.4	0.24	-71,200	59 <b>,</b> 300
0% constant (Pure PG)	Transient at 300 seconds into run 5280°F - in 2350°F - out	1.4	0.24	-27,300	22,200
0% inside 12% middle 0% outside	Transient at 300 seconds into run 5280°F - in 2350°F - out	1.4	0.24	-20,800	26,700
0% inside 30% outside linear	4500°F - in 2250°F - out linear	1.4	0.24	-15,800	22,900
0% inside 30% outside linear	4500°F - in 2800°F - out linear	l.4	0.12	- 2,700	8,300
0% inside 30% outside linear	4000°F - in 2500°F - out linear	2.8	0.24	- 3,500	8,400

TABLE XVIII

HEAT TRANSFER AT THROAT FOR  $OF_2/B_2$  H<sub>6</sub>

 $P_c = 100 PSIA$   $D_t = 2.6 INCH$ 

	HEAT TRANSFER Coefficient BTU/IN <sup>2</sup> sec °F	HEAT FLUX To 1000°F Wall Btu/in <sup>2</sup> /sec	BOUNDARY Temperature °F
RMU REFORI 6039-F, 1966 Test data: Pc = 150 psia D2 = 2 22 incu	0.90 X 10 <sup>-3</sup>	NOT REPORTED	NOT REPORTED
UT = 3.32 INCH L* = 30 0/F = 2.6 TO 3.2 CR = 2.75 C*Eff = 89% TO 96% MARQUARDT ANALYSIS (BARTZ EQ.) Df = 2.6 INCH	1.0 X 10 <sup>-3</sup>	e.	0009
0/F = 3.0 Aerojet Report 659-F, 1969 Test data: P <sub>c</sub> = 110 PSIA			
UT = 2.67 TEST 007 - BARRIER COOLING - 1.5:1	1.6 X 10 <sup>-3</sup>	6 LO 9	5600-6400
0/F (BARKIER) = 1.4 T <sub>6</sub> (Barrier) = 4600°F (Theor.) 0/r (fucing) = 4500°F (Theor.)	2.2 X 10 <sup>-3</sup>	5 5 7	3400
U/F (ENWINE) = 4.13  Sp = 87% Tect die 5 dabbieb fooi ing - 1 6.1	2.48 X 10 <sup>-3</sup>	6.2	3400-3600
0/F (BARRIER) = 0.68 T <sub>6</sub> (BARRIER) = 3300°F (THEOR.)	4.7 X 10 <sup>-3</sup>	 D	UNSATISFACTORY (1766)
TEST 006 - FILM COOLING - 8:1 Rocketdyne - Aiaa Paper 69-505 Bocket v Cooling	0.7 TO 1.4 X 10 <sup>-3</sup>	5.4	6200
TEST DATA: $P_c = 100 PSIA$ TEST DATA: $P_c = 100 PSIA$ $D_t = 4.2 INCH$ O/F = 3.5 $I_{Sp} = 93\%$	NOT REPORTED	4 9	NOT REPORTED ( $\approx 6000$ )

Report S-989

V7826-3A

XIX	
TABLE	

0/F = 3.0P. = 100 PSIA

COMBUSTION PRODUCTS OF OF2/B2 H6

ບ				Ň	0		
			WO	LE FRACTION	٩S		
	7000°R	6000°R	500°R	4000°R	3000°R	2000°R	1000°R
60	0.00058	0.00010	0.00001	0°0000	0*0	0.0	0*0
8 Т	0.06708	0.06171	0.03243	0.00301	0.00000	0.0	0.0
8F2	0.00764	0.01023	0.00873	0.00161	0.00002	0*0	0°0
B F 3	0.00216	0°00918	0.03873	0.07778	0.16267	0.19666	0.22549
HØ	0.00004	0.00002	0.00000	0.0000	ũ°ũ	0°0	9°0
BHF2	0.00061	0.00267	0.00983	0°01464	0°0624	0.00015	0.0
N H G	0.00008	0.00013	0.00012	0.00002	0.00000	0.0	0.0
BH303	0.00000	0.00000	0.00000	0.00000	0.00001	0.00005	0.00010
06	0*06440	0.02757	0.00626	0.00055	000000*0	0°0	0.0
80 10 10	0.16129	0.23008	0.27560	0.28941	0.05696	0.00008	0.0
80F2	0.00004	0.00003	0.00001	0.00000	0.00000	0°0	0.0
802	0.00069	0.00016	20000°C	0°0000	0°0	0*0	0*0
820	0.00003	0.00002	0.00001	0*00000	0.0	0.0	0.0
8202	0.00047	0.00076	0.00387	0.00074	0.00000	0.0	0.0
8203(L)	0.0	0.0	0.0	0°0	0*05941	0°08328	0°0
B203(S)	0.0	0.0	0.0	0.0	0.0	0.0	0.10845
8203	0.00016	0.00024	0.00030	0.00096	0.00007	0.0	0°0
8303F3	0*0	00000 *0	000000 * 0	0.00025	0*03290	0.03015	0.00007
L	0.00506	0.00052	0.00002	0°0000	0°0	0.0	0.0
3	0.23706	0.09199	0.01995	0.00179	0.00003	0°00000	0.0
HBO	0.000048	0.00038	0.00017	0.00004	0°0000°0	0°0	0°0
H802	0.00098	0.00126	0.00133	0.00338	0.00240	0.00001	0.0
u I	0.21602	0.18553	0.11690	0.04761	0.02939	0.732	9.00395
H2	0.22985	0.37496	0.48831	0.55675	0.64603	0.66407	0.64756
HZO	0.00273	0.00220	0.00130	0.00144	0.00386	0*00820	0.01438
H3B306	0.0	0.0	0.0	0.0	0.00000	0.00005	0.00001
0	<b>0</b> 000055	0*0003	00000 * 0	0.0	0*0	0.0	0°0
I	0.00161	0.00023	0.00001	0.00000	0.00000	0°0	0.0
ADDITIONAL	PPODICTS	WHICH BY		ati on Yau	82	ÊN2	HU 2
WERE CONSID	DERED BUT V	WHOSE D2	• . • .	л г. У П У		П Эсг	2
MOLE FRACT	TONS WERE	LESS			0	J	
THAN 0.0000	05:						

TABLE XX

P <sub>c</sub> = 100 1 Weight Rati	SIA = 20.1	GRAPHIT	T EQUILIBRI	IN WITTH OF	0/B2H6		
			IOW	LE FRACTION	S	ne manager an	
хнству ПСПУ	7000°R	6000°R	5000°R	4000°R	3000°R	2000°R	1000°R
ø	0.00012	0.00002	0° 000 00	0.00000	0.0	0.0	0.0
L.	0.00959	06800*0	0.00315	0.00087	0000000	0°0	0.0
₹¥ ₩ @	0.00077	0.00090	0.00069	0.00017	0.00000	0.0	0.0
8F3	0.00015	0.00049	0.00245	0.00308	0.00361	0.00465	0.00548
I	0.00001	0° 00000	0°0000	0.00000	0.0	0.0	0
8 H B H B H B H B H B H B H B H B H B H	0°0004	0.00020	0.00071	0.00150	0.00092	0.00000	0,0
8H2	0.00001	0.00002	0.00001	0.00001	0.00000	0.0	0.0
Ca	00000 0	0.00001	0.00001	0.00001	0.00000	0.0	0.0
80	0.00001	0.00004	0.00022	0.00233	0.00134	0.00000	0,0
8202	0.0	0.00000	0.00000	0.00001	0.00000	0.0	0.0
8203(L)	0.0	0.0	0°0	0.0	0.00126	0.00192	0°0
8203(S)	0.0	0.0	0.0	0.0	0.0	0.0	0.00264
8303F3	0.0	0.0	0.0	0.00000	0.00073	0.00071	0.00000
C(S)	0.95405	0.96179	0.96678	0.97019	0.97592	0.97815	0.98347
U	0.00024	0.00001	0.00000	0.0	0.0	0.0	0.0
C84(L)	0.0	0.0	0.00084	0.0	0.0	0.0	0.0
CR4(S)	0.0	0.0	0.0	0.00065	0.0	0°0	0.0
С Ц	0.00010	0.0000	0°0000	0.0	0.0	0.0	0.0
L L	0.00003	0.00000	0° 00000	0.0	0.0	0.0	0.0
CH3	0000000	0°00001	000000 * 0	0000000	00000 * 0	0000000	0.0
CH &	0.00000	0.0000	0°0000	0.00001	0.00008	0.00172	0 00777
00	0.00820	0.00808	0.00792	0.00577	0.00079	0.00020	000000°0
C2	0.00034	0° 00000	00000000	0*0	0.0	0.0	6.0
Can	0.00682	C. 00089	0.00003	0.0000	0.0	0.0	0,0
C2HF	0.00001	00000 *0	0.00000	0.0	0.0	0.0	0.0
C 2H2	0*00240	0°00179	0.00048	0.00005	000000	0.00000	0
C3	0.00101	0.00002	0.00000	0.0	0.0	0.0	0,0
u	0.00016	0.00001	0.00000	0°0000	0.0	0.0	0.0
-	0.00704	0.00297	0.00059	0.00005	0.00000	0000000	0.0
HB02	0.0	0.00000	0* 00000	0.00000	0.00001	000000.0	0,0
上.	0.00449	0.00364	0.00278	0.00049	0.00010	0.00024	600000°6
H2	0.00441	0.01020	0.01334	0.01479	0.01522	0.01233	0.00020
H20	0.00000	0.00000	0*00000	0°00000	0.00000	0.00007	0.00034
ADDITIONAL	PRODUCTS W	HICH B(S)	B(L)	BH3 BH30	13 BOF2	802 B2	R 20
WERE CONSIL	ERED BUT WI	HOSE CF2	CF3	CF4 CH2	COF	COF2 CO2	C2F2
MOLE FRACT	IONS WERE LI	ESS C20	C302	F0 F02	F2	1B0 HC0	H0 2
THAN 0.00000	<b>)</b> 5:	H383	0 90	0H 02			

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Report S-989

	HLTW
	EQUILIBRIUM
	NTTRIDE
	BORON
	20:1
<b>FN LA</b>	10 10

0/F = 3.0	A T D		TABLE	IIXX a			
rc = 100 r Weight Rati	0 = 20:1	BORON NTI	RIDE EQUILI	CBRIUM WITH	OF <sub>2</sub> /B <sub>2</sub> H6		
			V	OLE FRACTI	ONS		
	7000°R	6000°R	5000°R	4000°R	3000°R	2000°R	1000°R
BíL)	0.55277	0.61682	0.00085	0.0	0.0	0.0	0.0
8	0.05478	0.03336	1000000	0.00000	0.0	0.0	0.0
T V C	0.02180	0.02099	16410*0				ູ
01 K	00000-0	00000	0.00045	0.00395	0.00783	0.00937	0.01093
	0.00098	0.0018	0.00001	0.0000	0.0	0.0	0.0
BHr 2	000000	0.0000	0.00067	0.00148	0-00048	0.00001.	0.0
8H2	0.00048	0.00047	0.00029	0.00001	0.0000.0	0.0	0.0
BH3	0 • 00000	0.00000	0.00001	0.00000	0.00000	0.0	с°0
BN(S)	0.0	0.0	0.92817	0.94556	0.95203	0.95275	0.95175
N N N	0.00897	0.00092	0.000.00	0.0000	0.0	0.0	c.o
80	0.00994	0.00913	0.00175	0.00006	0.00000	0.0	¢.0
80F	0.00009	0.00076	0.01227	0.01553	0.00273	0.00000	0.0
82	0.00039	0.00001	00000000	0.0	0*0	0.0	0.0
820	06000000	0.00066	0.00009	0.00000	0.0	0.0	0.0
B202	0.00002	0.00022	0.00095	0.00016	0.00000	0.0	0.0
B203(L)	0.0	0°0	0*0	0.0	06200.0	00400-0	0°0
8203(S)	0.0	0.0	0.0	0.0	0.0	0.0	0.00546
8203	0.00000	0.00000	0.00003	0.00005	0.0000	0.0	0.0
8303F3	0.0	0.0	00000000	0.00001	0.00158	0.00144	0.00000
<u>u</u> .	0.00001	0.00000	0.00000	0.0000	0.0	0.0	0.0
T	0.02728	0.01143	0.00131	0.00010	0.0000	0.00000	0.0
HBO	0.00002	0.0004	0.00004	0.00000	0.0000.0	c.0	0.0
M802	0.00000	0.00000	0.00003	0.00010	0.00007	0.0000	с, о
ΗF	0.00009	0.00023	0.00121	0.00130	0.00088	0.00041	0.00002
Ĩ	0.00680	0.01512	0.02972	0.03033	0.03125	0.03175	0.03135
HZO	0.00000	0.00000	0.00001	0.00002	0.00007	0.00010	0.00001
Z	0.00016	0.00001	00000.0	0.0	0.0	0.0	C•0
ž	0.00001	0.00000	0.00000	0.0	0.0	0.0	0.0
NH3	0.00000	0.0000	0.0000	0.00000	0.0000	0.00000	0.00045
N2	0.31450	) C.31950	0.00651	0.00056	0.00016	910000.0	0.00003
ADDITIONAL WERE CONSID	PRODUCTS ERED BUT	WHICH B(	SI BH3C	<b>33 BOFZ</b>	802	F0 F02	F2 H02
MOLE FRACT	IONS WERE	LESS H3	3305 NF	NF2	NF3	NH2 NO	NOF NO2
THAN 0.00000	5:	IZN		<b>D</b> .	H	٥Z	

## TABLE XXIII

TUNGSTEN EQUILIBRIUM WITH  $OF_2/B_2H_6$ 

O/F = 3.0 P<sub>c</sub> = 100 PSIA T = 6500°R Weight Ratio = 20:1

SPECIES	MOLE FRACTION
H2	0.303943
BLF101	0.196605
BlFl	0.672809E-01
FlHL	0.206151
W	0.493160E-05
H201.	0.261625E-02
BlF3	0.427957E-02
BlF2	0.903003E-02
BlHl	0.287915E-04
BIHIOI	0.456680E-03
B1H1.02	0.117679E-02
BLH2	0.108439E-03
B1H2O2	0.130790E-06
BLH3	0.719595E-06
BLH303	0.172300E-08
B1.02	0.349316E-03
B201	0.274693E-04
во	0.457590E-01
B2	0.884583E-08
венб	0.282430E-15
B202	0.635878E-03
B203	0.208589E-03
B3F303	0.180200E-08
B3H306	0.642487E-15
B5H9	0.242782E-32

SPECIES	MOLE FRACTION
BLOH14	0.0
F1H101	0.341074E-08
FlOl	0.825796E-09
F2	0.103262E-08
Fl	0.177728e-02
F201	0.420241E-14
ш	0.158610
H1.02	0.131397E-08
HIOL	0.700285E-03
H202	0.187245E-09
Ol	0.206367E-03
02	0.701233E-06
03	0.397906E-14
WF	0.907279E-05
WF6	0.818037F-15
WO	0.284071E-04
W02	0.453357E-05
W03	0.309831E-07
W206	0.884190E-12
· W308	0.602580E-17
W309	0.215580E-19
W4012	0.477467E-27
B203*	0.238419E-06
W*	0.491960
W02*	-0.108033E-06

\* Solid

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Report S-989



FIGURE 2. Layouts of Structural Thrust Chambers for Pyrobond Fabrication Studies



FIGURE 3. Raytheon Multilamina Chambers





AXIAL DIRECTION D



FIGURE 5. Schematic Diagram of Filament Orientation in a Carbitex Thrust Chamber

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FIGURE 6. Dimensions of Pyrolytic Graphite/Carbitex Composite Test Cones



FIGURE 7. Pyrolytic Graphite/Carbitex Test Cones



NEG. 9312-1





FIGURE 9. Variation of Circumferential Modulus of Elasticity of Carbitex with Wrap Angle





FIGURE 11. Variation of Axial Strength of Carbitex Cones with Wrap Angle



FIGURE 12. Dimensions of PG/Thornel Test Sample Spool





FIGURE 14. PG/Thornel NOL Ring Specimens for Tensile Tests

8-6816 'DEN



FIGURE 15. Results of Tensile Tests of PG/Thornel NOL Ring Specimens



A. Full Cross Section (10X)



B. Polarized Light (50X)

FIGURE 16. Microstructure of PG/Thornel Ring Specimen LC

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A. Full Cross Section (10X)



B. Polarized Light (50X)

FIGURE 17. Microstructure of PG/Thornel Ring Specimen 2C



A. Full Cross Section (10X)



B. Polarized Light (50X)

FIGURE 18. Microstructure of PG/Inornel Ring Specimen 3C





A. Full Cross Section (10X)



B. Polarized Light (50X)

FIGURE 19. Microstructure of PG/Thornel Ring Specimen 4C
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A. Spool No. 1, Polarized Light (750X)



B. Spool No. 2, Polarized Light (750X)

FIGURE 20. Photomicrographs of Representative Sections of PG Infiltrated Thornel, Spools Nos. 1 and 2

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B. Spool No. 4, Polarized Light (750X)

FIGURE 21. Photomicrographs of Representative Sections of PG Infiltrated Thornel, Spools Nos. 3 and 4





FIGURE 22. PG/Thornel/PG Chamber

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V7314-2



FIGURE 23. PG Infiltrated Thornel-50 Carbon Yarn

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Report S-989

104



1



01-9896 '93N



V8493-7

•

V8493-8





Copper Impregnated Graphite after Cyclic Heating

FIGURE 28.



1-6716 .03N

108

Report S-989

V 84 93-10



FIGURE 29. Composite Chamber FLOX/LPG











112

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FIGURE 33. 100-pound Thrust Carbitex 713 Chambers





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FIGURE 35. X-Ray Photograph of PG/Carbitex Thrust Chamber with RPG Nozzle Extension 115 115



## FIGURE 36. 100-pound Thrust FLOX/LPG Engine Assembly

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PG Liners Used for PG/Thornel Composite Altitude Thrust Chambers FIGURE 38.

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FIGURE 41. Filament Winding Setup for PG1 Thornel Thrust Chambers







FIGURE 42. Electrodeposited Nickel on RPG/PG Cylinders before Machining

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A. Electrodeposited Nickel Layer on RPG after Machining (Full Size)



B. Interface between Nickel and RPG (50 X)

FIGURE 43. Electrodeposited Nickel on RPG/PG Cylinder after Machining



 $\begin{array}{l} \text{OD} = 3.15 \text{ in.} \\ \text{ID} = 2.15 \text{ to } 2.35 \text{ in.} \\ \text{L} = 0.94 \text{ in.} \\ \text{t}_{\text{PG}} = 0.04 \text{ in.} \\ \text{t}_{\text{RPG}} = 0.45 \text{ in.} \\ \text{t}_{\text{Ni}} = 0.04 \text{ to } 0.08 \text{ in.} \end{array}$ 

FIGURE 44. Test Specimen Used for Thermal Cycling Test of Bond of Electrodeposited Nickel on RPG

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A. Experimental Arrangement for Thermal Cycling Tests



B. Thermal Cycling Test in Progress

FIGURE 45. Setup for Thermal Cycling Test of Electrodeposited Nickel on RPG 125







Boxed Area (750 X)

FIGURE 47. Photomicrographs of Plasma Sprayed Nickel - RPG Interface

Final Configuration of the 100-pound Thrust FLOX Methane Injector, S/N002

FIGURE 48.



128



FIGURE 49. Final Configuration, S/N 002 Injector



1-2916 '93N



FIGURE 51. Erosion Pattern of PG Chamber S/N 006 after Test Run 45

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FIGURE 53. Water Flow Test of a Modified Injector Showing Film Spreading in a Lucite Chamber,  $\Delta P = 80$  psi

6



FIGURE 54. Spray Pattern from Cold Flow Tests of the 100-pound Thrust FLOX/Methane Injector with 18 Fuel Film Holes






PG/Carbitex Chamber with RPG Nozzle Extension after 322 Seconds Firing FIGURE 56.

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Report S-989



FIGURE 57. HFC Coated Tantalum Tungsten Chamber 137



FIGURE 58. Predicted Transient Temperature Distribution in a ZrC Composite Chamber Fired with FLOX/Methane



Inner Surface Stresses in a ZrC Composite Chamber Fired with FLOX/Methane

FIGURE 59.



FIGURE 60. Outer Surface Stresses in a ZrC Composite Chamber Fired With FLOX/Methane

V8493-11









SENSIBLE HEAT CAPACITY - Btu/Ib

V8493-14

144

V 84 93-1 5



V8493-9



FIGURE 66. Enthalpy of Gaseous Diborane

V 84 93-16







FIGURE 68. Combustion Products - OF  $OF_2/B_2^{H_6}$ 

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FIGURE 69. Heat Transfer Mechanisms in an Endothermically Cooled Pyrolytic Graphite Nozzle

Report S-989

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- MODERATE AMOUNT OF B2H6 FILM COOLING
- INERT CHAMBER BELOW 4000°F
- TUNGSTEN THROAT BELOW 5500°F
- COMPOSITE GRAPHITIC EXPANSION NOZZLE VERY LOW REACTION RATES IN LOW PRESSURE REGION

FIGURE 70. Composite Materials Chamber

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