

UNCLASSIFIED



AD NUMBER

AD-381 439

CLASSIFICATION CHANGES

TO UNCLASSIFIED

FROM CONFIDENTIAL

AUTHORITY

OCA; Apr 1979

19990317091

THIS PAGE IS UNCLASSIFIED

UNCLASSIFIED



AD NUMBER

AD-381 439

NEW LIMITATION CHANGE

TO

DISTRIBUTION STATEMENT: A

Approved for public release; Distribution is unlimited.

LIMITATION CODE: 1

FROM

No Prior DoD Distr Scty Cntrl St'mt Assgn'd

AUTHORITY

AFRPL ltr; Feb 5, 1986

THIS PAGE IS UNCLASSIFIED

AFRPL-TR-67-109

~~CONFIDENTIAL~~

NO. C80-7C4 -1. 481
DOCUMENT CONTROL

(TITLE UNCLASSIFIED)
FOAM CERAMIC
NOZZLE INSERTS

T. A. Greening
United Technology Center

TECHNICAL REPORT AFRPL-TR-67-109
APRIL 1967

Group 4
DOWNGRADED AT 3 YEAR INTERVALS
DECLASSIFIED AFTER 12 YEARS
DOD DIR. 5200.10

DDO
JUN 2 1967
A

In addition to security requirements which must be met, this document is subject to special export controls and each transmittal to foreign governments or foreign nationals may be made only with prior approval of AFRPL (RPPR/STINFO). Edwards, California 93523.

This document contains information affecting the national defense of the United States within the meaning of the Espionage Laws, Title 18, U.S.C., Section 793 and 794, the transmission or revelation of which in any manner to an unauthorized person is prohibited by law.

AIR FORCE ROCKET PROPULSION LABORATORY
RESEARCH AND TECHNOLOGY DIVISION
AIR FORCE SYSTEMS COMMAND, UNITED STATES AIR FORCE
EDWARDS, CALIFORNIA

UTC 2180-FR

~~CONFIDENTIAL~~

Reproduced From
Best Available Copy

19990317091

AD381439

SECURITY

MARKING

The classified or limited status of this report applies to each page, unless otherwise marked.

Separate page printouts MUST be marked accordingly.

THIS DOCUMENT CONTAINS INFORMATION AFFECTING THE NATIONAL DEFENSE OF THE UNITED STATES WITHIN THE MEANING OF THE ESPIONAGE LAWS, TITLE 18, U.S.C., SECTIONS 793 AND 794. THE TRANSMISSION OR THE REVELATION OF ITS CONTENTS IN ANY MANNER TO AN UNAUTHORIZED PERSON IS PROHIBITED BY LAW.

NOTICE: When government or other drawings, specifications or other data are used for any purpose other than in connection with a definitely related government procurement operation, the U. S. Government thereby incurs no responsibility, nor any obligation whatsoever; and the fact that the Government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation, or conveying any rights or permission to manufacture, use or sell any patented invention that may in any way be related thereto.

~~CONFIDENTIAL~~

NO. C80-7C4-1.48A
DOCUMENT CONTROL

(Title Unclassified)
FOAM CERAMIC NOZZLE INSERTS

T. A. Greening

Group 4
DOWNGRADED AT 3 YEAR INTERVALS
DECLASSIFIED AFTER 12 YEARS

In addition to security requirements which must be met, this document is subject to special export controls and each transmittal to foreign governments or foreign nationals may be made only with prior approval of AFRPL (RPPR/STINFO), Edwards, California 93523.

This document contains information affecting the national defense of the United States within the meaning of the Espionage Laws, Title 18, U. S. C., Section 793 and 794, the transmission or revelation of which in any manner to an unauthorized person is prohibited by law.

UTC 2180-FR

~~CONFIDENTIAL~~

When U. S. Government drawings, specifications, or other data are used for any purpose other than a definitely related Government procurement operation, the Government thereby incurs no responsibility nor any obligation whatsoever, and the fact that the Government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data, is not to be regarded by implication or otherwise, or in any manner licensing the holder or any other person or corporation, or conveying any rights or permission to manufacture, use, or sell any patented invention that may in any way be related thereto.

~~CONFIDENTIAL~~

FOREWORD

(U) This Final Report describes work performed under Contract AF 04(611)-11218 from 1 December 1965 to 13 February 1967 by United Technology Center (UTC) Sunnyvale, California, for the Air Force Rocket Propulsion Laboratory, Research and Technology Division (AFRPL, RTD), Edwards, California.

(U) This contract resulted from UTC Proposal No. 65-108, "Demonstration of Ability of Foam Ceramic Composites to Withstand Advanced Rocket Engine Propellant Environments," issued in response to the Air Force Request for Proposal (RFP), Purchase Request No. 3058636. It was accomplished under the technical direction of Capt. Richard W. Ogershok, AFRPL, RTD. The work reported in this document was conducted in the Engineering Materials and Process Department of UTC.

(U) This report contains no classified information extracted from other classified documents.

(U) This technical report has been reviewed and is approved.

Richard W. Ogershok, Capt., USAF
Project Engineer

~~CONFIDENTIAL~~

(This page is Unclassified)

UNCLASSIFIED

UTC 2180-FR

UNCLASSIFIED ABSTRACT

(U) Eight nozzle inserts were fabricated from United Technology Center's foam ceramic composites to define the operational limitations of these materials when used in liquid rocket engines employing either N_2O_4/N_2H_4 -UDMH or LF_2/N_2H_4 Blend propellants. The nozzle inserts consisted of the following:

A. Condition 1

N_2O_4/N_2H_4 -UDMH
 $Al_2O_3 + SiO_2$ fibers (two nozzles)
 $ZrO_2 + SiO_2$ fibers (two nozzles)

B. Condition 2

LF_2/N_2H_4 Blend
 $ZrO_2 + C$ fibers (two nozzles)
 $ZrC + C$ fibers (two nozzles).

(U) Thermal and mechanical property tests were conducted on the ceramic foam composites. The objective was to supply data for an Air Force computer program to predict materials performance in rocket engines, and to afford a basis for evaluating nozzle erosion from the firings in this program.

(U) The test firings were conducted by the Air Force Rocket Propulsion Laboratory, Research and Technology Division, Edwards, California. The classified results and posttest analyses are included in this report.

(U) In general, the erosion rates were high under the test conditions used in this program. However, the erosion was uniform causing little, if any, deviation from throat circularity. It was also established that the foam ceramic composite materials were insensitive to thermal shock. The ZrC composition has shown promise as a potential candidate for components in high thermal environments.

UNCLASSIFIED

UNCLASSIFIED

UTC 2180-FR

CONTENTS

<u>Section</u>		<u>Page</u>
I	INTRODUCTION	1
II	BACKGROUND	3
III	MATERIALS SELECTION	5
IV	DESIGN AND FABRICATION OF NOZZLE INSERTS	7
	1. Design	7
	2. Fabrication	7
	a. Process	7
	b. Nozzle Inserts	12
	c. Color Coding of Nozzles	12
	d. Thermocouples	15
V	PROPERTIES MEASUREMENTS	21
VI	TEST FIRING OF THE NOZZLE INSERTS	33
VII	POSTTEST ANALYSIS	39
	1. Performance of Nozzles	39
	a. Erosion	39
	b. Temperature Data	56
	c. Char Thickness	59
	d. Effect of Properties on Performance	60
	e. Ballistic Performance	62
VIII	CONCLUSIONS	63
IX	POTENTIAL AREAS OF APPLICABILITY	65
	APPENDIX: Materials R&D Report	67

UNCLASSIFIED

UNCLASSIFIED

ILLUSTRATIONS

<u>Figure</u>		<u>Page</u>
1	(U) Design of Nozzle Insert for Test Condition 1	8
2	(U) Design of Nozzle Insert for Test Condition 2	9
3	(U) Alumina Foam and Alumina-Silica Fiber Foam	11
4	(U) Nozzle Inserts for Test Condition 2	13
5	(U) The Eight Foam Ceramic Nozzles Fabricated	14
6	(U) Thermocouple Positions for Condition 1 Inserts	16
7	(U) Thermocouple Positions for Condition 2 Inserts	17
8	(U) Incorporation of Thermocouples	18
9	(U) Foam Ceramic Thermal Conductivity and Thermal Expansion Specimens	23
10	(U) Foam Ceramic Specific Heat Specimens	24
11	(U) Foam Ceramic Density Specimens	25
12	(U) Foam Ceramic Compressive Strength Specimens	26
13	(U) Foam Ceramic Tensile Strength Specimens	27
14	(U) Thermal Expansion Coefficient of Cured Impregnated Foam Ceramic Composites	28
15	(U) Specific Heat of Foam Ceramic Materials	29
16	(U) Thermal Conductivity of Foam Ceramic Materials	30
17	(U) Mechanical Properties of Foam Ceramic Composites	31
18	(U) Density of Foam Ceramic Materials	32
19	(U) AFRPL Flintstone Facility	34
20	(U) Test-Fired Ceramic Foam Nozzle Inserts	38
21	(U) Cross Section of Insert No. 1 - Al_2O_3 + SiO_2 Fibers	40
22	(U) Cross Section of Insert No. 2 - Al_2O_3 + SiO_2 Fibers	41
23	(U) Cross Section of Insert No. 3 - ZrO_2 + SiO_2 Fibers	42
24	(U) Cross Section of Insert No. 4 - ZrO_2 + SiO_2 Fibers	43

UNCLASSIFIED

UNCLASSIFIED

UTC 2180-FR

ILLUSTRATIONS (Continued)

<u>Figure</u>		<u>Page</u>
25	(U) Cross Section of Insert No. 5 - ZrO_2 + C Fibers	44
26	(U) Cross Section of Insert No. 7 - ZrC + C Fibers	45
27	(U) Cross Section of Insert No. 8 - ZrC + C Fibers	46
28	(U) Throat Contour Orientation Insert No. 1	47
29	(U) Throat Contour Orientation Insert No. 2	48
30	(U) Throat Contour Orientation Insert No. 3	49
31	(U) Throat Contour Orientation Insert No. 4	50
32	(U) Throat Contour Orientation Insert No. 5	51
33	(U) Throat Contour Orientation Insert No. 7	52
34	(U) Throat Contour Orientation Insert No. 8	53
35	(U) Char Layer, Condition 1 Inserts	54
36	(U) Char Layer, Condition 2 ZrC + C Fiber Inserts	55
37	(U) Typical Thrust and Chamber Pressure Curves for Foam Ceramic Insert Firings	57

UNCLASSIFIED

UNCLASSIFIED

TABLES

<u>Table</u>		<u>Page</u>
I	(U) Materials Selection	5
II	(U) Color Code for Nozzles	15
III	(U) Thermocouple Locations and Lead-Length Coding	19
IV	(U) Laboratory Tests Conducted	22
V	(U) Test Conditions	33
VI	(U) Data from Ceramic Nozzle Insert Test Firings	36
VII	(U) Performance Properties for Foam Ceramic Insert Firings	37
VIII	(U) Properties - Order of Merit	61

UNCLASSIFIED

UNCLASSIFIED

UTC 2180-FR

SECTION I

INTRODUCTION

(U) In response to Air Force Request for Proposal 3058636, United Technology Center (UTC) proposed to demonstrate the ability of its phenolic resin impregnated, foam-ceramic composites to withstand the environments of advanced rocket engines employing high-energy propellants. The program was to define the operational limitations of these materials when used as nozzle inserts in liquid rocket engine tests employing either N_2O_4/N_2H_4 -UDMH(50-50) and LF_2/N_2H_4 Blend. * Tests were to be conducted at AFRPL, Edwards Air Force Base, California.

(U) The proposal was accepted, and a contract (AF 04(611)-11218), "Foam Ceramic Nozzle Inserts," was issued.

(U) In accordance with the contract, four test materials were selected by mutual agreement with the Air Force, two for each of the two stated propellant systems. Physical, mechanical, and thermal property measurements were made on these materials to supply data for an Air Force computer program to predict material performance in rocket engines.

(U) Two nozzle inserts of each test material, a total of eight, were fabricated to Air Force-supplied designs. These inserts were delivered to the Air Force for test firing, after which they were returned to UTC for post-test analysis.

(U) Erosion and char measurements have been correlated with Air Force-supplied performance and thermocouple data. Conclusions have been made regarding the applicability of these materials for thrust chambers of advanced liquid propellant rocket engines.

* N_2H_4 Blend: wt-% - 66.7 N_2H_4 , 24.0 MMH, 9.3 H_2O

UNCLASSIFIED

UNCLASSIFIED

UTC 2180-FR

REVIEW FOR THE BUREAU, INFORMATION NOT FILLED.

SECTION II

BACKGROUND

(U) In the past it was generally realized that the use of refractory ceramics was being thwarted by lack of thermal shock resistance. Foam ceramic structures were developed at UTC in an attempt to overcome this problem. Early in this work techniques were developed for foaming a large variety of ceramic powders and for sintering these foamed materials into dense-walled structures. Sintering proved to be a difficult process with which to achieve reproducibility and uniform density or for maintaining a homogeneous structure. The resultant high degree of crystalline bonding also retained considerable thermal shock sensitivity in the structure. As a result, a means of chemical bonding rather than sintering was investigated. The material derived from chemical bonding would, of course, be a composite material rather than a porous form of a crystalline material. In general, composite types have less susceptibility to thermal shock failure than crystalline or diffusion bonded materials. Grain boundaries in a composite material tend to terminate thermal cracking as compared to propagation of such cracks through sintered, diffusion-bonded materials.

(U) Techniques were developed combining several well-known technologies of chemical bonding with UTC's foaming process. Through the use of proper combinations of binders, foaming agents, wetting agents, and catalysts the process currently employed to produce ceramic composites evolved. These materials were considered suitable for backup insulation of thrust chamber and nozzle sections where low weight, low conductivity, and ease of fabrication are important.

(U) For thermal barrier applications, the open pores of the foam structure did not restrict hot-gas flow, thus impregnation of the foamed structure with vaporizable and char-forming coolant-type materials (e.g., phenolic resin) became a next logical extension of this study.

(U) Thus, a new class of composite insulating materials was successfully developed and demonstrated at UTC. A large number of combinations of foam skeleton and impregnants were evaluated in oxy-acetylene and plasma torch tests. The principal demonstration of these new materials had been as insulation for high-energy hybrid mixer baffles. Then, in response to an Air Force Request for Proposal, UTC proposed to demonstrate performance of its phenolic resin-impregnated foam ceramic composites in liquid rocket environments employing both N_2O_4/N_2H_4 -UDMH(50-50) and LF_2/N_2H_4 Blend. Upon acceptance of the proposal a program was agreed upon, and work began on selection of materials, design, and fabrication of foam ceramic nozzle inerts for this stringent application.

UNCLASSIFIED

UNCLASSIFIED

UTC 2180-FR

THIS DOCUMENT IS UNCLASSIFIED EXCEPT WHERE SHOWN OTHERWISE

SECTION III
MATERIALS SELECTION

(U) Four test materials were selected by mutual agreement with the Air Force, two for each of the two propellant systems to be employed.

(U) Two alternate material selections were offered, one for each of the two propellant systems. Material selection was as shown in table I.

TABLE I
(U) MATERIALS SELECTED

<u>Condition 1</u>	<u>Condition 2</u>
$N_2O_4/.5N_2H_4 - .5UDMH$	LF_2/N_2H_4 Blend
Nozzle No. 1 $Al_2O_3 + SiO_2$ fibers	Nozzle No. 5 $ZrO_2 + C$ fibers
2 Same	6 Same
Alternate ZrO_2	Alternate $TiC + C$ fibers
Nozzle No. 3 $ZrO_2 + SiO_2$ fibers	Nozzle No. 7 $ZrC + C$ fibers
4 Same	8 Same

(U) The suggested choices were accepted by the Air Force, and the alternatives were not used.

(U) Reasons governing selection of the above materials included:

- UTC's extensive experience with Al_2O_3 foam and the high degree of success with such foams in mixers for hybrid motors
- Favorable test results in flame and plasma tests employing ZrO_2 foams both at UTC and in outside test agency facilities
- ZrC foam performance in fluorine atmospheres
- Indication of improved erosion resistance of foams with incorporation of appropriate fiber reinforcement additives.

UNCLASSIFIED

UNCLASSIFIED

UTC 2180-FR

EXCLUDED FROM THE GPO. DECLASSIFIED BY 1000.

SECTION IV

DESIGN AND FABRICATION OF NOZZLE INSERTS

1. DESIGN

(U) The nozzle inserts of each test material, a total of eight, were fabricated to Air Force-supplied design prints, No. X65C7745 and No. 65C7746; the former applying to Condition 1 inserts, and the latter to the Condition 2 inserts of table I. The two designs are shown in the order listed, in figures 1 and 2.

2. FABRICATION

a. Process

(U) Fabrication of the foam ceramic material is a two-fold process: producing the ceramic skeleton and impregnating the skeleton with a coolant material. Basic ingredients for fabrication of the chemically bonded foam structures are as follows:

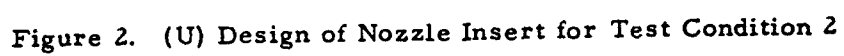
- A. Refractory powder or fiber (less than 325-mesh size)
- B. Liquid binder (e. g., sodium silicate, phosphoric acid, etc.)
- C. Wetting agent (to reduce surface energy of the refractory powder and inhibit coalescence of bubbles)
- D. Foaming agent (e. g., sodium peroxide which decomposes and produces a gas)
- E. Catalyst (to control and accelerate decomposition of the foaming agent)
- F. Green binder (to stabilize the foam structure during drying)
- G. Deflocculant (to maintain suspension of the refractory powder during casting).

UNCLASSIFIED



Figure 1. (U) Design of Nozzle Insert for Test Condition 1

UTC 2180-FR



UNCLASSIFIED⁹

UNCLASSIFIED

(U) The above materials are thoroughly mixed to form a slip, or suspension, of refractory powders in a liquid. The catalyst is the last ingredient incorporated so that premature foaming does not occur. The slip is cast into a mold made of an inert material such as plastic or metal sheet stock. The foaming action commences instantly and continues for up to 3 min at a diminishing rate. Cast foams are dried at a slow rate at room temperature. Acceleration of this process produces nonuniform structures. Mishandling of the foams during this drying period, while their strength is relatively low, would cause collapsing of the pores and slumping or cracking of the part. After complete drying, the foamed structure can be easily cut, machined, or sanded into appropriate modular parts. The dry, foam ceramic skeleton with compressive strengths of 300 to 400 psi, can be readily handled and assembled into an unlimited variety of geometric combinations.

(U) The height to which these foams can be produced is generally limited to 2 in., and there is no apparent limit to the diameter. This height limitation is caused by the low strength of the wet foam directly after casting. If excess material is foamed, the weight of the upper part causes collapsing of the lower pore structure. However, this condition imposes no problem from a practical viewpoint because the foam rings or plates are easily assembled into an unlimited variety of shapes and sizes by being bonded or mechanically held together. This modular method was employed to build units of sufficient size to permit machining to the design configurations required. Shear-resistant strength of the foams is improved by incorporating fibrous materials into the foamed skeleton. An alumina foam containing silica fibers is shown in figure 3, together with one containing no fibers.

(U) Control of the porosity and pore size of the foamed materials is maintained chemically. By the proper balance of foaming agent (hydrogen peroxide) and wetting agents, porosities ranging from 20% to 90% can be achieved. Pore sizes are dependent on the rate of decomposition of the foaming agent which is controlled by the amount of catalyst, type of wetting agents, temperature of reaction, and concentration of foaming agent. Pore sizes, e. g., of alumina, can be controlled in the range of 0.001- to 0.100-in. diameters. An average pore size diameter of 0.075 in. is generally used for foams to be impregnated and represents the target pore size for fabrication of the nozzle inserts for this program.

10
UNCLASSIFIED

UNCLASSIFIED

UTC 2180-FR

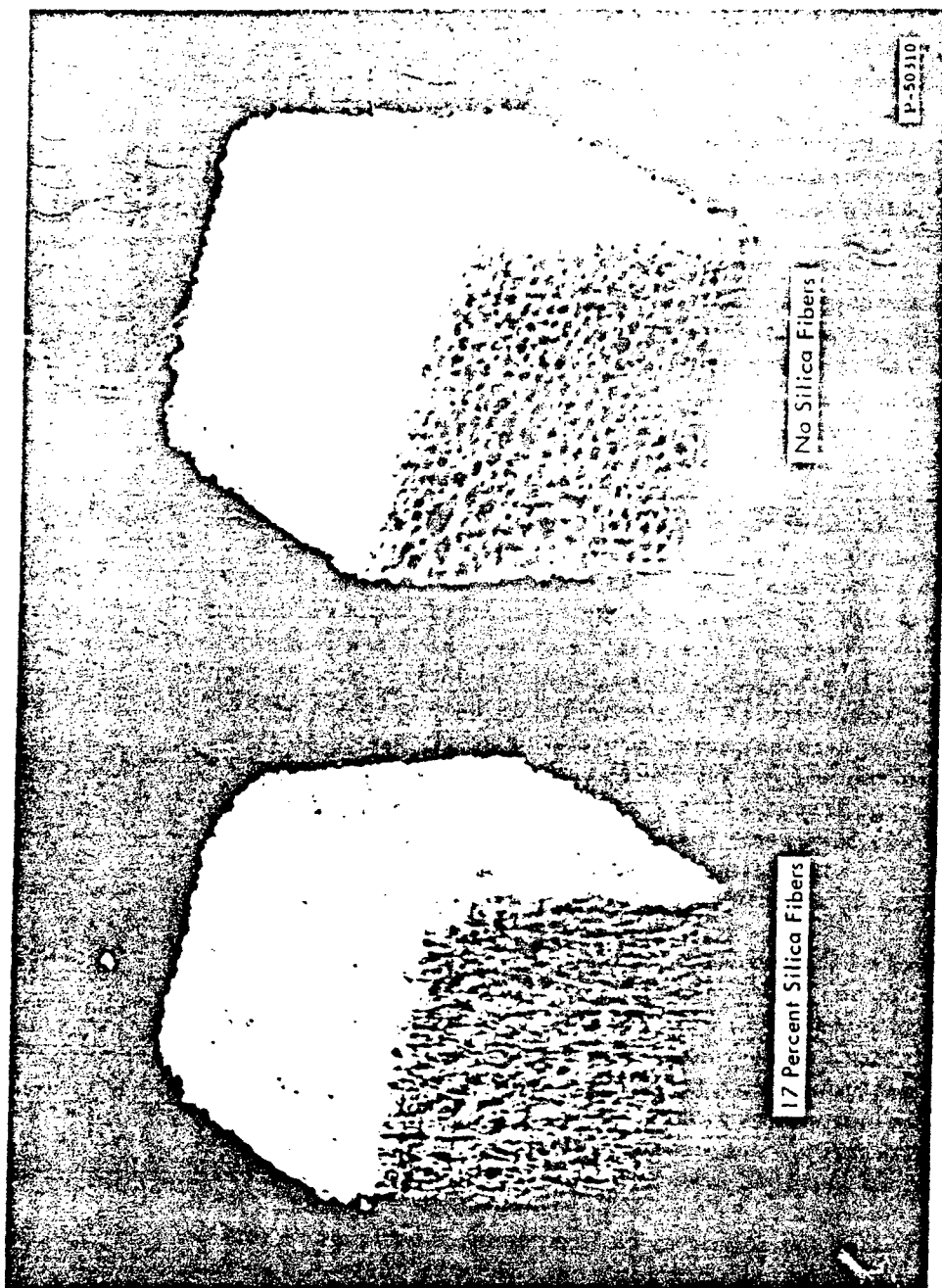


Figure 3. (U) Alumina Foam and Alumina-Silica Fiber Foam

11
UNCLASSIFIED

UNCLASSIFIED

(U) A vacuum is used to evacuate air from the pores of the skeleton, permitting entrance of impregnant. The impregnant is then cured or dried in place. Organic carriers or solvents are used in all instances with sodium silicate bonded foams.

(U) The impregnating has been with Monsanto SC-1008 phenolic resin. The process is carried out in containers that fit closely around the foam assembly and act as a resin bath. Evacuation and impregnation is conducted in a vacuum chamber. The resin is first devolatilized before curing.

(U) Curing of the phenolic resin is conducted in a recirculating-air oven at temperatures over 150°F for approximately 24 hours. This curing is followed by a postcuring treatment at 250°F for 12 hours, after which components are cooled. If curing is initiated before the devolatilization is sufficiently complete, exothermic reactions occur causing a blowing out of the resin. For an impregnated foam structure, this condition results in an excessive amount of unfilled pores, and this condition must be guarded against.

b. Nozzle Inserts

(U) The eight nozzle inserts required for this program were fabricated by the above method. Their structures were modular in nature. The inserts were delivered to the Air Force at Edwards Air Force Base, Rocket Propulsion Laboratory on 15 June 1966 for test firing as described in section VI. Figures 4 and 5 are prefire photographs of the completed nozzles.

(U) Figure 4 shows ZrC + C fiber and ZrO₂ + C fiber foam ceramic nozzle inserts from left to right.

(U) Figure 5 shows nozzle inserts fabricated from phenolic impregnated foams of: (left to right) Al₂O₃ + SiO₂ fiber, ZrO₂ + SiO₂ fibers, ZrC + C fibers, and ZrO₂ + C fibers.

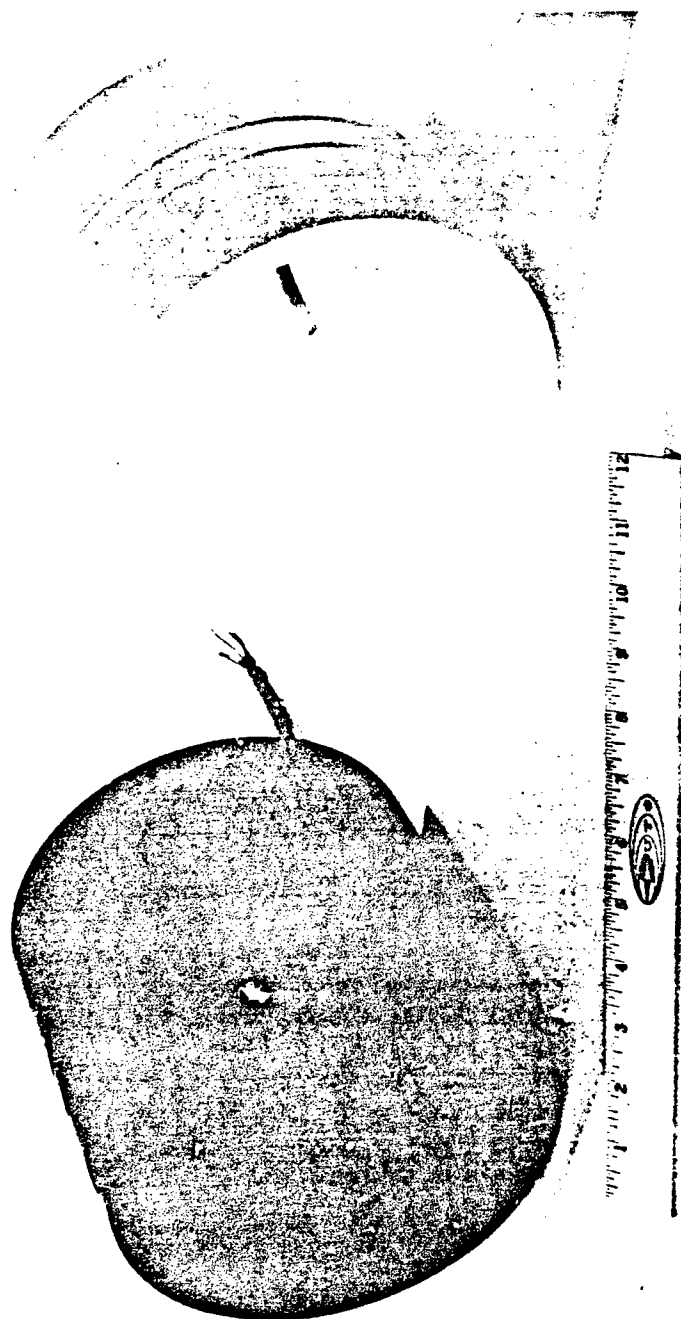
c. Color Coding of Nozzles

(U) Inserts were color coded as shown in table II.

12
UNCLASSIFIED

UNCLASSIFIED

UTC 2180-FR



R-70204

9765-3

Figure 4. (U) Nozzle Inserts for Test Condition 2

13
UNCLASSIFIED

UNCLASSIFIED

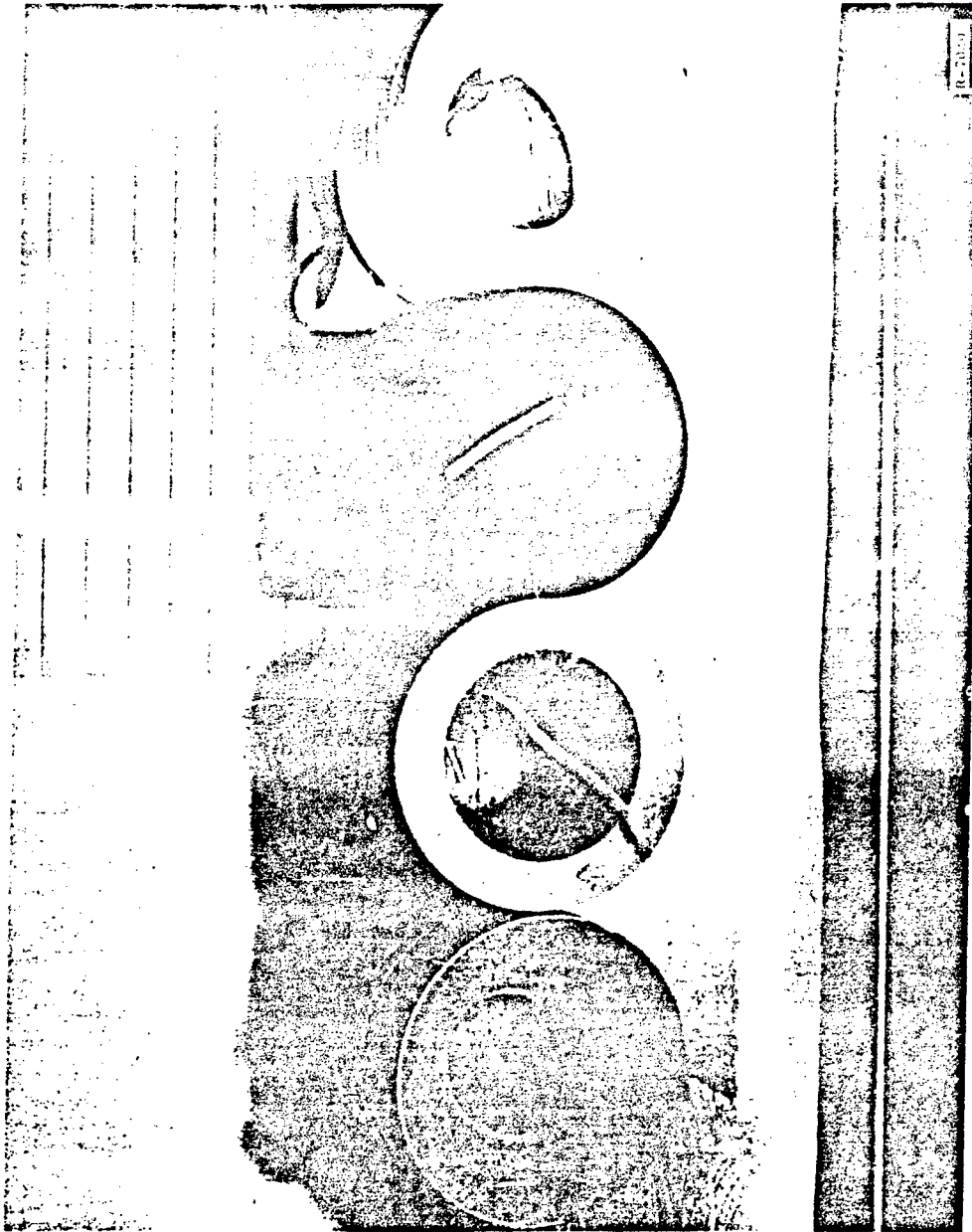


Figure 5. (U) The Eight Foam Ceramic Nozzles Fabricated

UNCLASSIFIED

UNCLASSIFIED

UTC 2180-FR

TABLE II
(U) COLOR CODE FOR NOZZLES

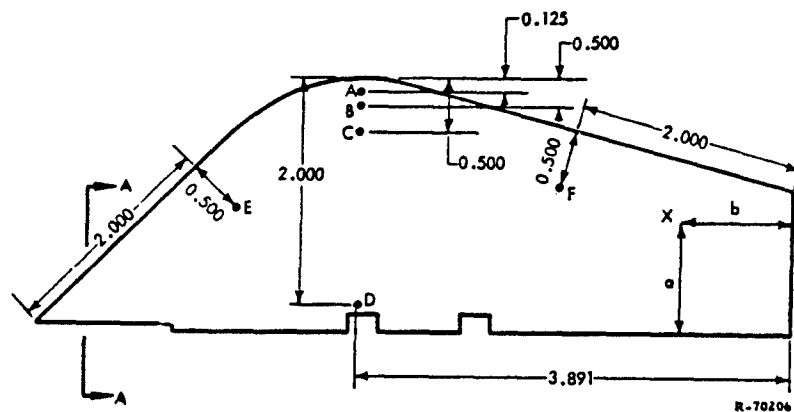
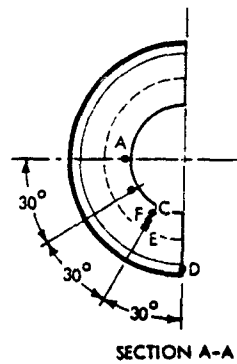
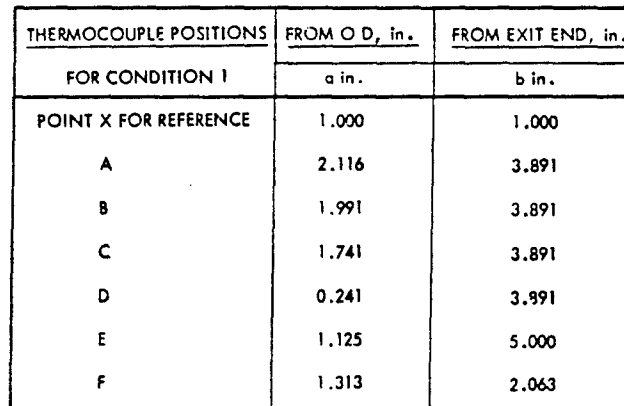
<u>Color Code</u>	<u>Nozzle No.</u>	<u>Composition</u>	<u>Print No.</u>
Red	1	$\text{Al}_2\text{O}_3 + \text{SiO}_2$ fibers	X65C7745
Red	2	$\text{Al}_2\text{O}_3 + \text{SiO}_2$ fibers	X65C7745
Grey	3	$\text{ZrO}_2 + \text{SiO}_2$ fibers	X65C7745
Grey	4	$\text{ZrO}_2 + \text{SiO}_2$ fibers	X65C7745
White	5	$\text{ZrO}_2 + \text{C}$ fibers	X65C7746
White	6	$\text{ZrO}_2 + \text{C}$ fibers	X65C7746
Black	7	$\text{ZrC} + \text{C}$ fibers	X65C7746
Black	8	$\text{ZrC} + \text{C}$ fibers	X65C7746

d. Thermocouples

(U) Six type K Chromel-Alumel thermocouples were potted in each nozzle at the locations indicated in figures 6 and 7 for Conditions 1 and 2, respectively, of table I. Figure 8 illustrates the incorporation of the thermocouples. Thermocouple leads were embedded in the inserts and permitted to protrude as a set from the annular outlet face of the nozzle in each instance. Leads were length coded from A (shortest) to F (longest). Table III lists thermocouple hole locations and approximate protruding lead length for reference.

UNCLASSIFIED

UNCLASSIFIED



UNCLASSIFIED

UTC 2180-FR

THERMOCOUPLE POSITIONS	FROM O D, in.	FROM EXIT END, in.
FOR CONDITION 2	a in.	b in.
POINT X FOR REFERENCE	0.500	0.500
A	1.611	4.214
B	1.486	4.214
C	1.236	4.214
D	0.236	4.214
E	0.781	5.406
F	0.766	2.094

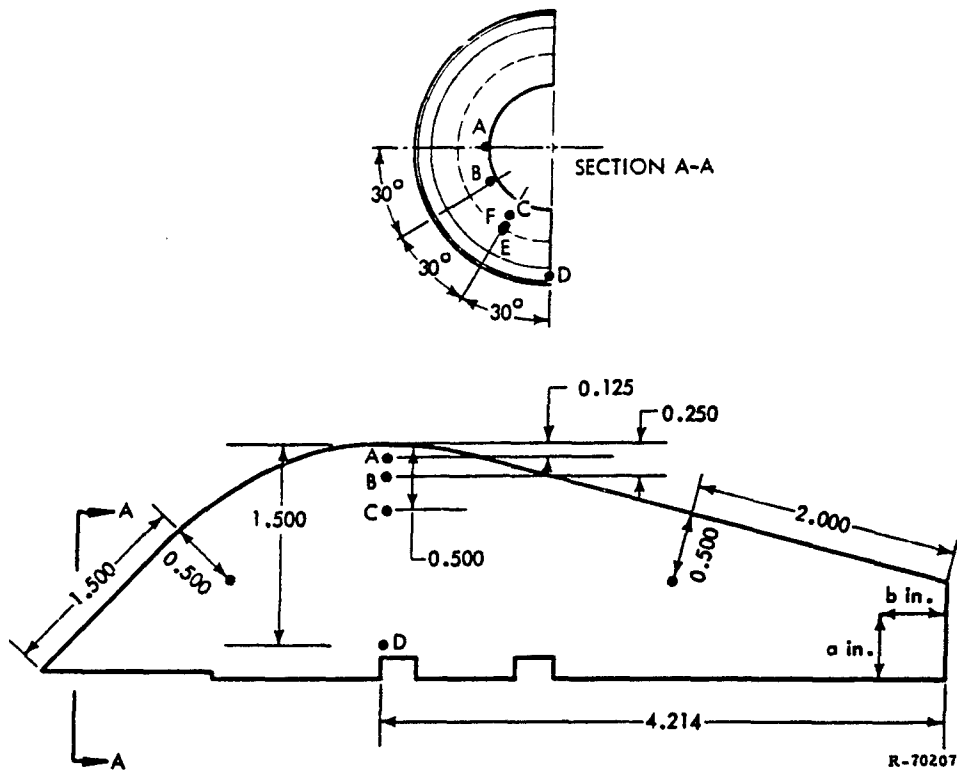
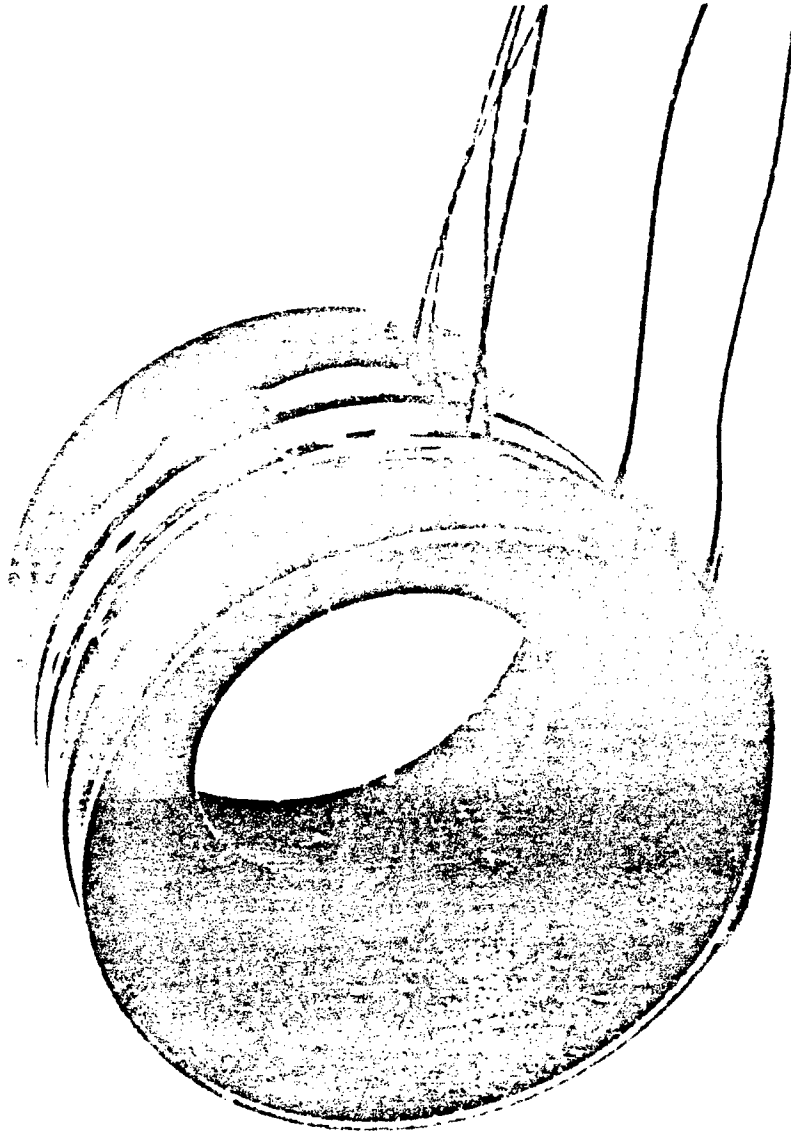


Figure 7. (U) Thermocouple Positions for Condition 2 Inserts

17
UNCLASSIFIED

UNCLASSIFIED



R-70208

Figure 8. (U) Incorporation of Thermocouples

5765-6

UNCLASSIFIED

UNCLASSIFIED

UTC 2180-FR

TABLE III
(U) THERMOCOUPLE LOCATIONS AND LEAD-LENGTH CODING

Thermocouple Location	For Print X65C7745		For Print X65C7746		Lead† Protruding
	a*	b†	a	b	
A	2.116	x 3.891	1.611	x 4.214	Shortest
B	1.991	x 3.891	1.486	x 4.214	Shortest + 1 in.
C	1.741	x 3.891	1.236	x 4.214	Shortest + 2 in.
D	0.241	x 3.891	0.236	x 4.214	Shortest + 3 in.
E	1.125	x 5.000	0.781	x 5.406	Shortest + 4 in.
F	1.313	x 2.063	0.766	x 2.094	Shortest + 5 in.

* Depth from OD, in.

† Location from outlet end, in.

‡ Protruding lead lengths, approximate in.

19
UNCLASSIFIED

UNCLASSIFIED

UTC 2180-FR

REMOVED FROM THE FILE. PROPERTY NOT TESTED.

SECTION V

PROPERTIES MEASUREMENTS

(U) Laboratory tests were performed on the materials employed for the selected nozzle insert compositions. Physical, mechanical, and thermal property measurements were made on these materials to supply data for an Air Force computer program formulated to predict material performance in rocket engines.

(U) Table IV lists the tests, the number of test specimens, and the conductor.

(U) The results of these tests are the subject of the R&D report required by line 7 of DD Form 1423 of the contract pertaining to this program. The R&D report is presented in appendix A of this report.

(U) The shape and size of the specimens for the various property measurement tests are illustrated in figures 9 through 13.

(U) A ready comparison of the properties reported in tables IX through XVI of appendix A can be found by reference to the set of bar graphs based on the data from these tables. The following bar graphs, figures 14 through 18, serve to elucidate the information given in the properties tables (IX through XVI, appendix A) as follows:

- A. Figure 14 illustrates relative thermal expansion coefficient data for the various foam ceramic compositions employed and is an analysis of table IX.
- B. Figure 15 combines the data of tables X and XI into a pictorial analysis of specific heat information.
- C. Figure 16 gives a comparison for thermal conductivity properties for the foam ceramic materials employed and reflects the data of tables XII and XIII.
- D. Figure 17 shows the mechanical properties comparison for the foam ceramic composites employed and as such reflects the data of tables XIV and XV.
- E. Figure 18 depicts densities of the pertinent ceramic foam composites with respect to virgin foam, impregnated and cured foam, and charred foams, as shown in table XVI.

UNCLASSIFIED

UNCLASSIFIED

TABLE IV
(U) LABORATORY TESTS CONDUCTED

Phase	Test	Materials x Orientation			Number of Specimens	Shape	Conductor
		x	Number	of			
Composite (impregnated fiber-foam)	Thermal Expansion	4 x 2 x 3	24	3/4-in. dia x 3-in.	24	3/4-in. dia x 3-in.	IITRI*
	Tensile Strength	4 x 2 x 3	24	4-in. x 1/2-in. x 1/2-in.	24	4-in. x 1/2-in. x 1/2-in.	UTC*
	Compressive Strength	4 x 2 x 3	24	1-in. cube	24	1-in. cube	UTC
	Density	4 x 3	12	1-in. cube	12	1-in. cube	UTC
	Thermal Conductivity	4 x 2	8	3/8-in. dia x 1/2-in.	8	3/8-in. dia x 1/2-in.	IITRI
Reinforcement (virgin foam)	Specific Heat	4 x 3	12	3/4-in. dia x 3-in.	12	3/4-in. dia x 3-in.	IITRI
	Density	4 x 3	12	1-in. cube	12	1-in. cube	UTC
	Thermal Conductivity	4 x 2	8	3/8-in. dia x 1/2-in.	8	3/8-in. dia x 1/2-in.	IITRI
Char (composite charred)	Specific Heat	4 x 3	12	3/4-in. dia x 3-in.	12	3/4-in. dia x 3-in.	IITRI
	Density	4 x 3	12	1-in. cube	12	1-in. cube	UTC

* The thermal part of the testing was subcontracted to Illinois Institute of Technology Research Institute, (IITRI) Chicago, Illinois, and the mechanical testing was conducted at UTC.

22
UNCLASSIFIED

UNCLASSIFIED

UTC 2180-FR

R-70209

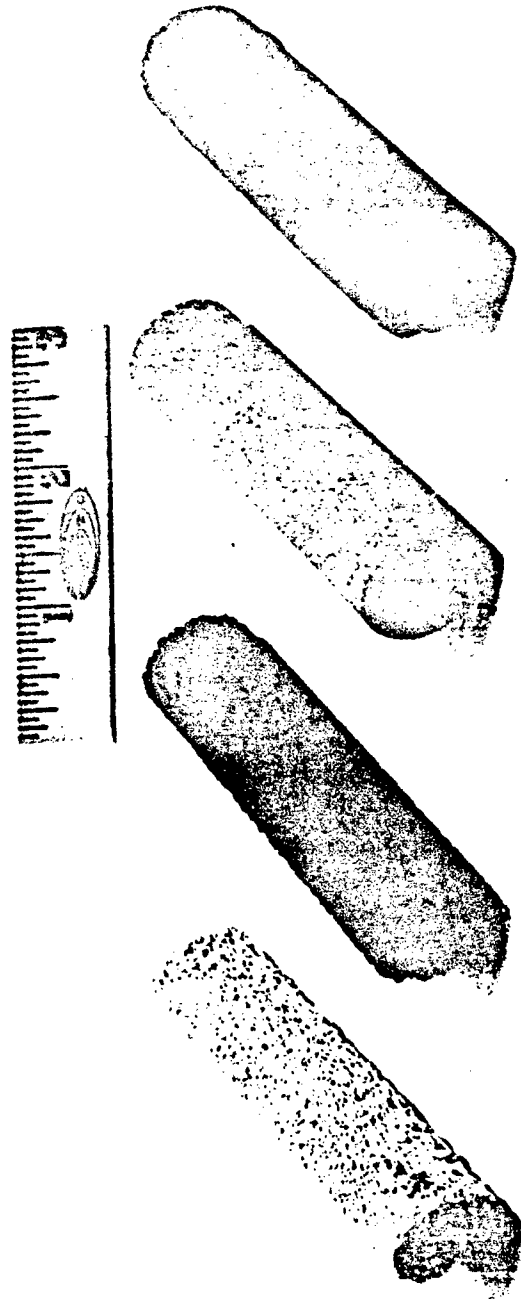


Figure 9. (U) Foam Ceramic Thermal Conductivity
and Thermal Expansion Specimens

5787-7

UNCLASSIFIED

UNCLASSIFIED

R-70210

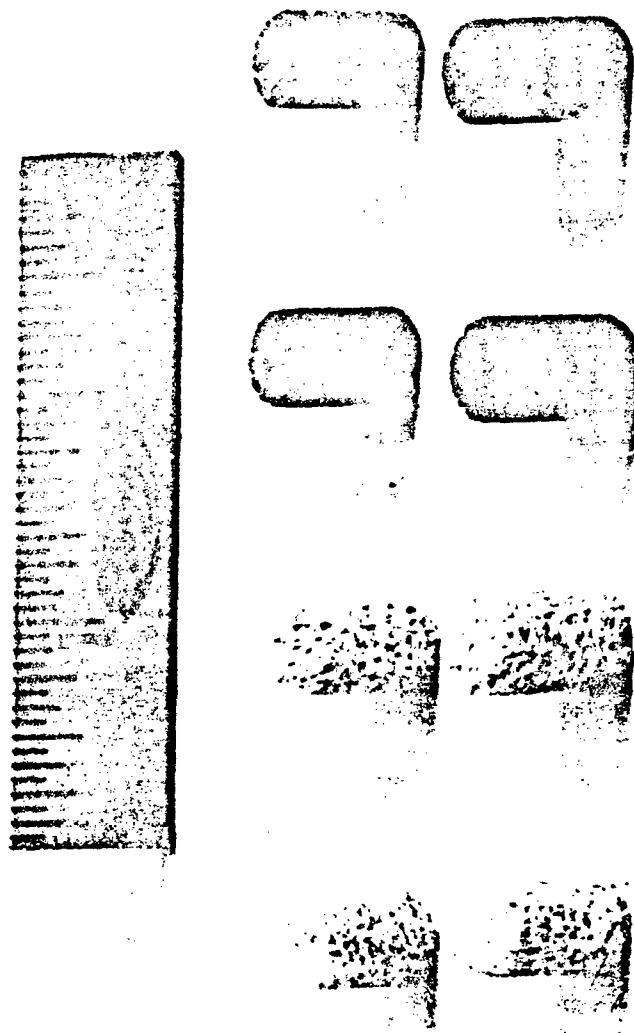


Figure 10. (U) Foam Ceramic Specific Heat Specimens

9787-8

UNCLASSIFIED

UTC 2186-FR

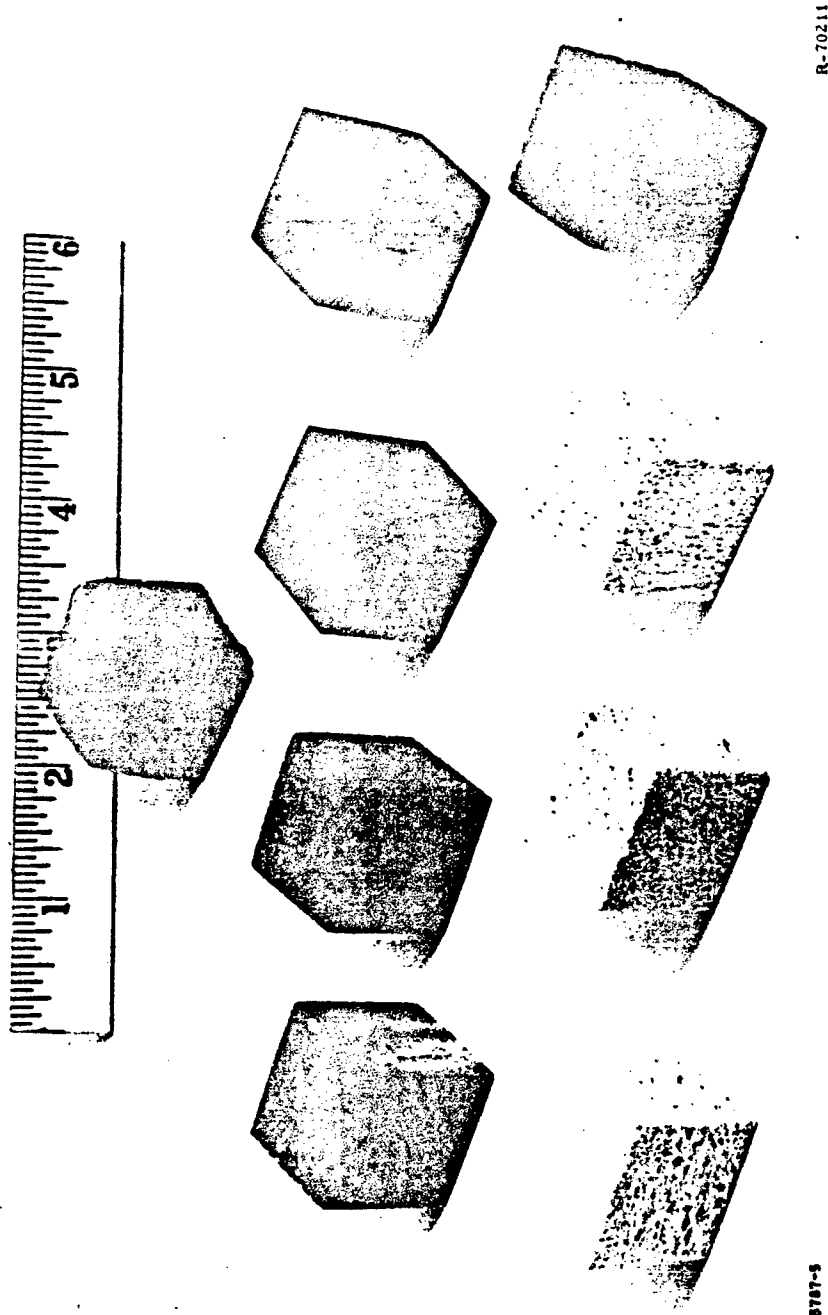


Figure 11. (U) Foam Ceramic Density Specimens

UNCLASSIFIED

UNCLASSIFIED



R-70212

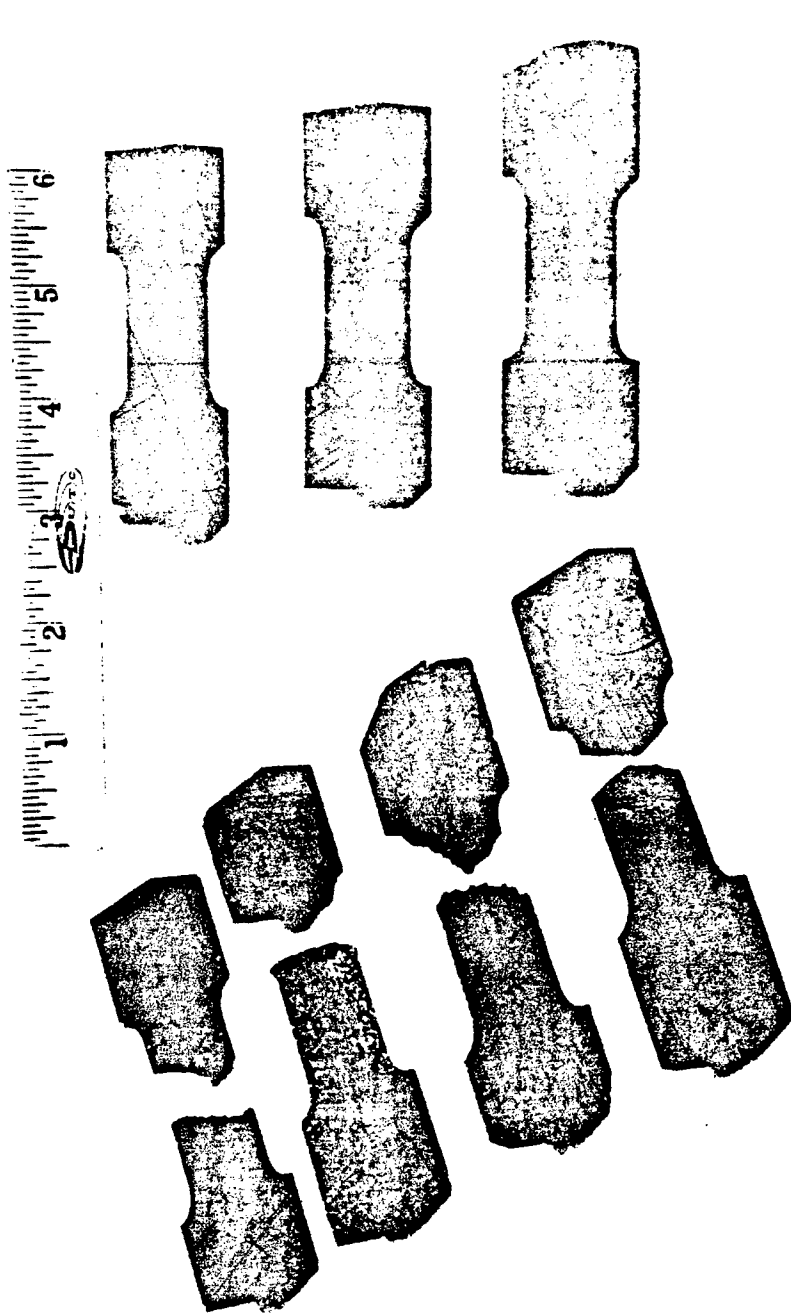
Figure 12. (U) Foam Ceramic Compressive Strength Specimens

3187-6

UNCLASSIFIED

UNCLASSIFIED

UTC 2180-FR



R-70213

Figure 13. (U) Foam Ceramic Tensile Strength Specimens

5787-2

UNCLASSIFIED

UNCLASSIFIED

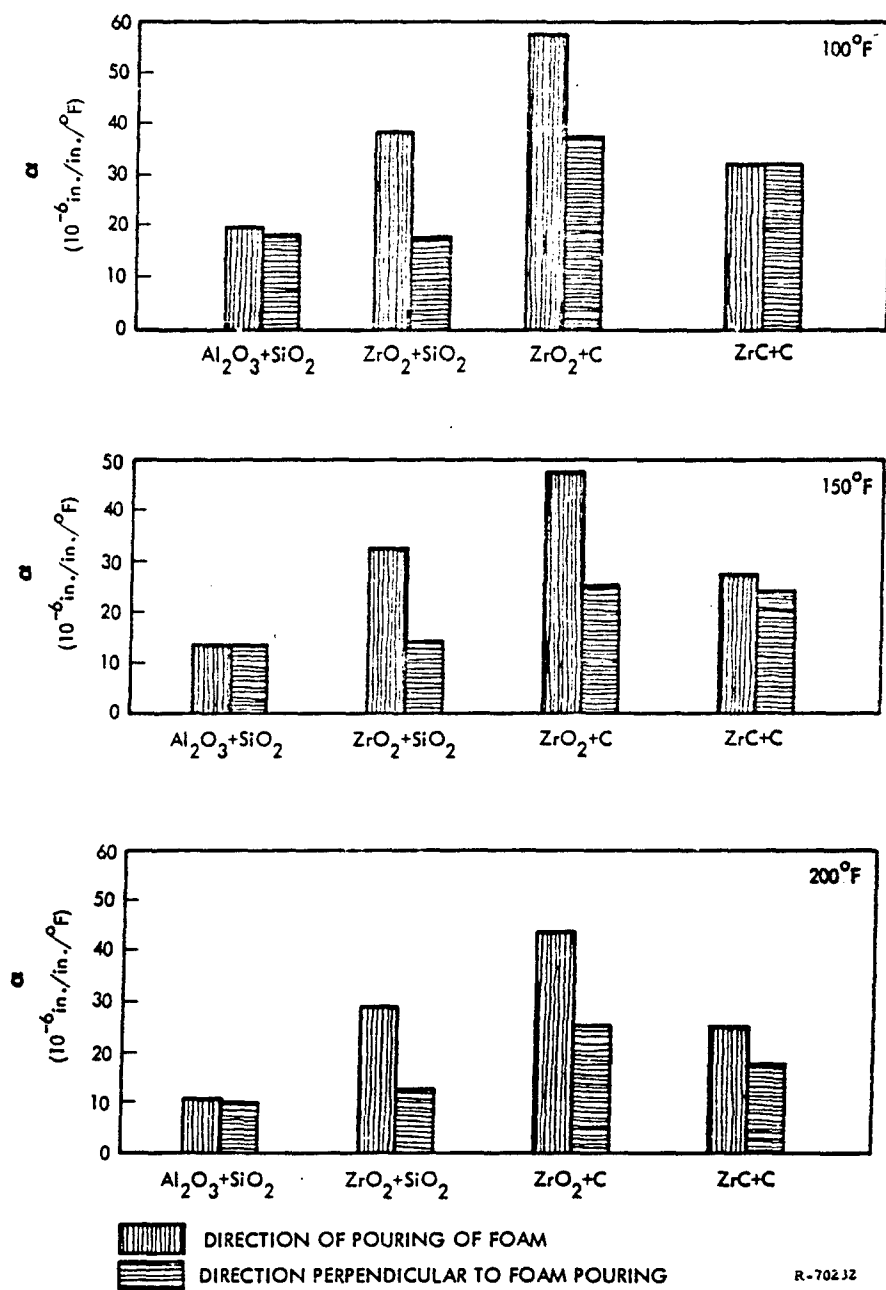
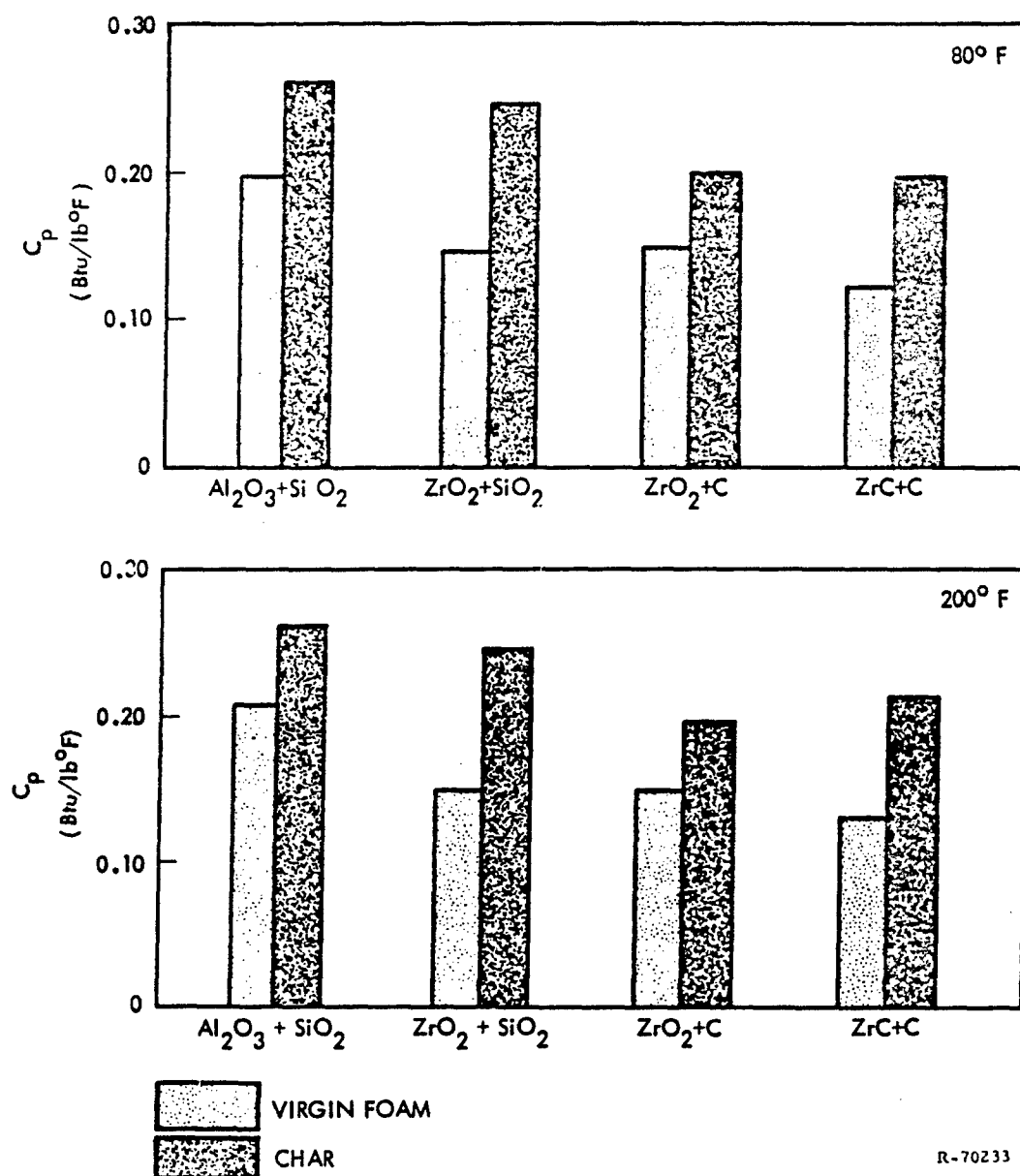


Figure 14. (U) Thermal Expansion Coefficient of Cured Impregnated Foam Ceramic Composites

UNCLASSIFIED

UNCLASSIFIED

UTC 2180-FR



R-70233

Figure 15. (U) Specific Heat of Foam Ceramic Materials

UNCLASSIFIED

UNCLASSIFIED

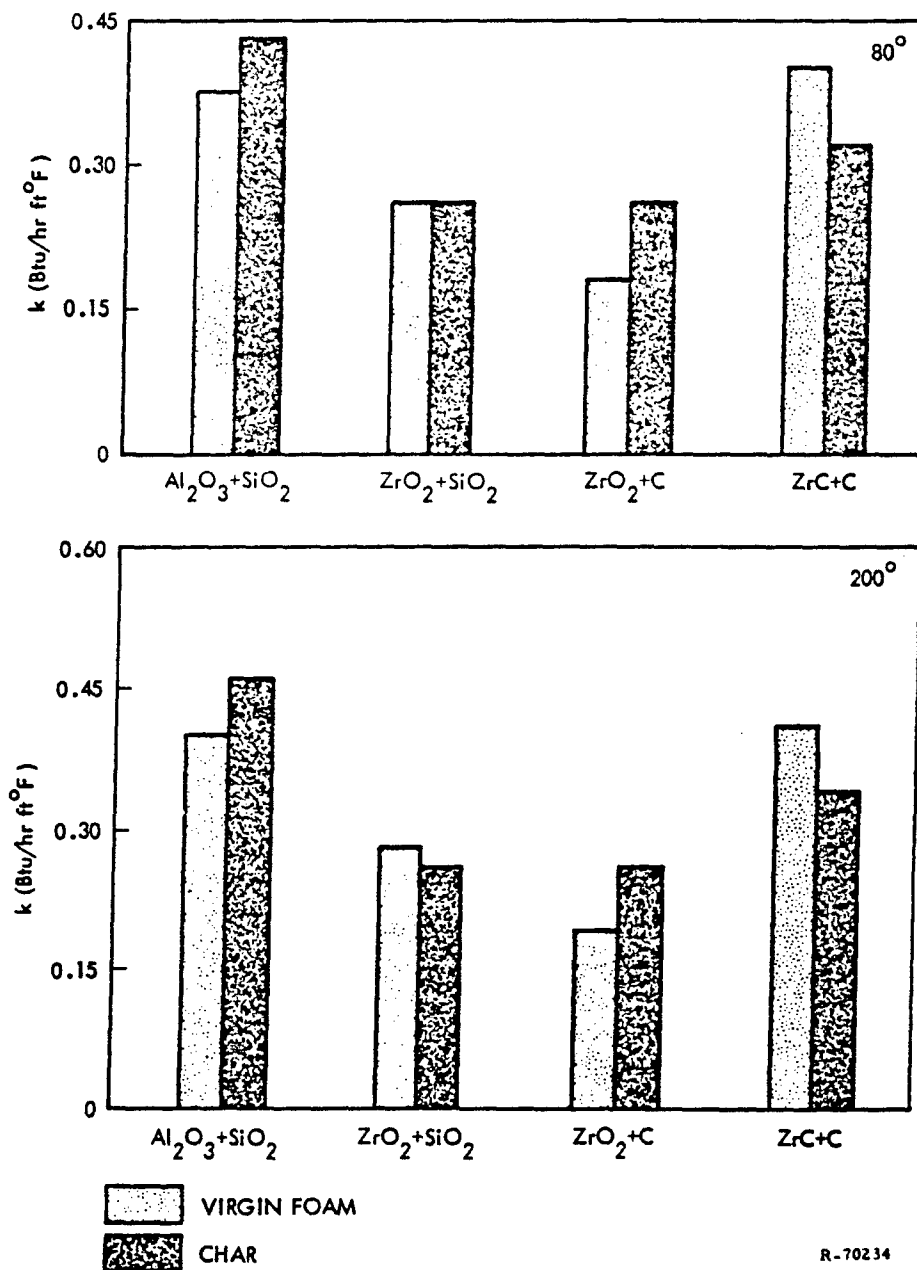


Figure 16. (U) Thermal Conductivity of Foam Ceramic Materials

30
UNCLASSIFIED

UNCLASSIFIED

UTC 2180-FR

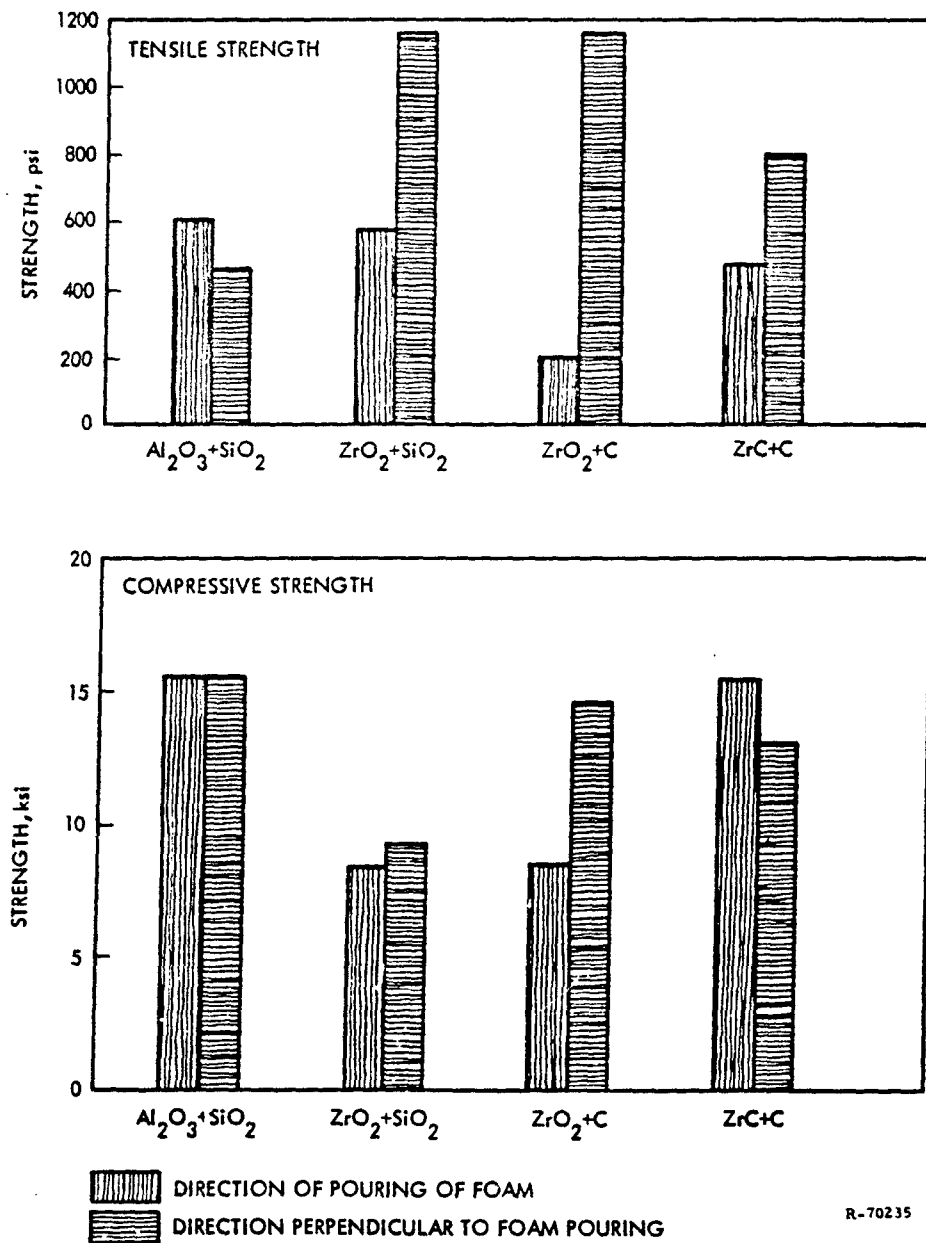
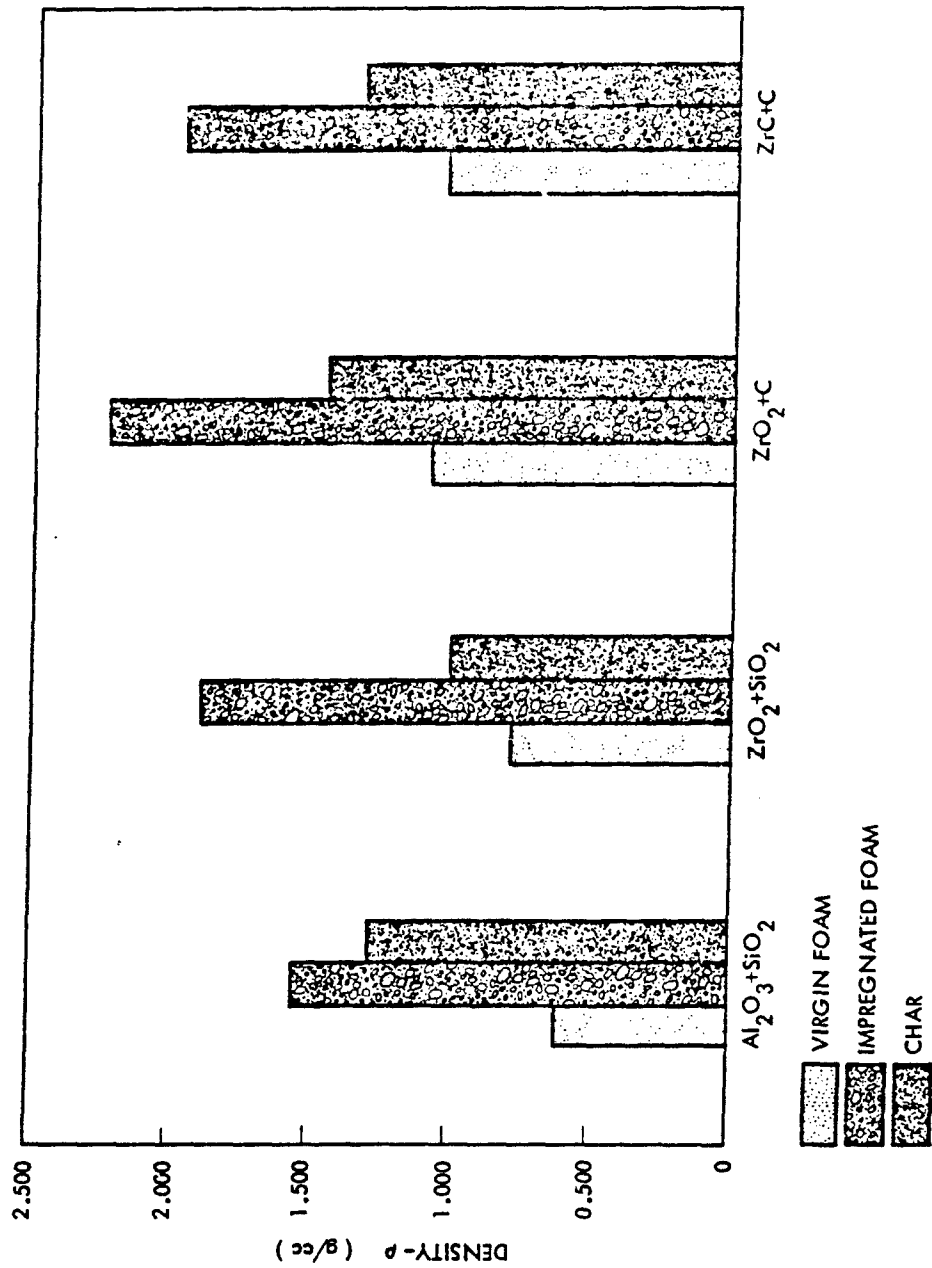


Figure 17. (U) Mechanical Properties of Foam Ceramic Composites

UNCLASSIFIED

UNCLASSIFIED



R-70236

Figure 18. (U) Density of Foam Ceramic Materials

UNCLASSIFIED

UNCLASSIFIED

UTC 2180-FR

SECTION VI

TEST FIRING OF THE NOZZLE INSERTS

(U) The nozzle inserts were designed and fabricated to fit the "Voramic" chamber barrels in accordance with AFRPL drawings X65C7745 and X65C7746. Representations of these drawings are shown in figures 1 and 2.

(U) The nozzle inserts were delivered to AFRPL and were test fired between August 1966 and December 1966, under the conditions outlined in table V, employing the compositions listed in table I. A photograph of AFRPL's Flintstone liquid motor facility where firings were conducted is shown in figure 19.

TABLE V

(U) TEST CONDITIONS

	<u>Condition 1</u>	<u>Condition 2</u>
Propellants	$N_2O_4/.5N_2H_4-.5UDMH$	LF_2/N_2H_4 Blend*
Chamber pressure, psia	500 (exhaust to 13.2)	200 (exhaust to 13.2)
Mixture ratio	2.0	2.0
Thrust, lb	5,000 (sea level)	3,750 (sea level)
Performance (theoretical), I_{sp}	96% (minimum)	96% (minimum)
Duty cycle/insert, sec	90 total (two restarts, cool to ambient)	90 total (two restarts, cool to ambient)
Chamber ID, in.	8	8

* N_2H_4 Blend: Wt-% - 66.7 N_2H_4 , 24.0 MMH, 9.3 H_2O .

33
UNCLASSIFIED

UNCLASSIFIED



Figure 19. (U) AFRPL Flintstone Facility

UNCLASSIFIED

CONFIDENTIAL

UTC 2180-FR

(U) Seven of the eight inserts were test fired. Data and results regarding firing times, chamber pressures thrust, throat diameters before and after firing, and temperature profiles are given in table VI. Pertinent performance data is extracted from table VI and compared with a calculated theoretical specific impulse, resulting in nozzle efficiency as presented in table VII. It should be realized that the theoretical value is based on throat areas enlarged by the observed erosion on a time-average basis.

(C) Nozzle insert No. 6, the second $ZrO_2 + C$ fiber insert, was not test fired because of the unusually high erosion rate found for the first $ZrO_2 + C$ fiber insert, unit No. 5.

(C) After the $Al_2O_3 + SiO_2$ fiber nozzle insert No. 1 initially was test-fired, the erosion rate was found excessively high at approximately 500 psi, and on a subsequent test the pressure was reduced to approximately 250 psi. The second $Al_2O_3 + SiO_2$ fiber nozzle insert was recontoured to a throat diameter of approximately 4.4 in. and chamber pressure was reduced to approximately 200 psi for test firing of that insert.

(U) The seven test-fired inserts are shown in figure 20.

CONFIDENTIAL

CONFIDENTIAL

TABLE VI
(U) DATA FROM FOAM CERAMIC NOZZLE INSERT TEST FIRINGS

COMPOSITE MATERIAL UTC NOZZLE NO. AIR FORCE CODE NO. RUN NO.	N ₂ O ₄ /N ₂ H ₄ - UDMH; Propellant				LF ₂ /N ₂ H ₄ Blend (5) Propellant			
	Al ₂ O ₃ + SiO ₂ Fibers		ZrO ₂ + SiO ₂ Fibers		ZrO ₂ + C Fibers		ZrC + C Fibers	
	1	2	3	4	5	6*	7	8
	310	311	312	313	314	315	316	317
19 Aug	22 Aug	19 Sep	12 Sep	12 Sep	31 Aug	3 Oct	3 Nov	21 Nov
1.75	10.78	9.82	4.80	4.82	9.70	9.91	7.95	9.26
Chamber Pressure, psi								
Maximum	492	280	299	252	290	158	195	209
Average	472	244	180	232	246	125	183	172
Thrust Developed, lbf								
Maximum	5,480	3,820	3,800	3,487	3,350	3,290	3,530	3,750
Average	5,420	3,700	3,680	3,420	3,260	3,000	3,480	3,380
Corrosivity(4)	0.40	0.55	0.49	0.49	0.63	0.00	0.00	---
Throat Diameter, in.								
Pretest	3.340(3)	3.550	4.377	3.340(3)	3.340(3)	4.350(3)	4.350(3)	4.350(3)
Posttest, Maximum	(1)	4.070	4.800	(1)	4.110	(1)	4.515	4.655
Posttest, Average	3.550	4.064	4.756	3.636	4.132	4.609	4.404	4.594
Erosion on Radius								
Total Amount, in.	0.105	0.257	0.190	0.148	0.166	0.532	0.027	0.122
Rate, in./sec	0.061	0.024	0.019	0.031	0.034	0.054	0.003	0.013
Maximum Temperature, °F								
Thermocouple A	(1)	950(2)	2,640(2)	410	1,950(2)	2,590(2)	1,720	1,560(2)
Thermocouple B	(1)	(1)	2,670(2)	88	150	2,500(2)	110	1,390
Thermocouple C	(1)	(1)	(1)	88	89	2,450(2)	80	125
Thermocouple D	(1)	(1)	(1)	---	---	---	73	(1)
Thermocouple E	(1)	(1)	(1)	91	89	(1)	76	(1)
Thermocouple F	(1)	(1)	(1)	87	88	605	76	135
Average Char Thickness, in.	(1)	0	0	0.020	(1)	0	0.150	0.100

* Not fired

NOTES:

- (1) Not obtained
- (2) Thermocouple was exposed by erosion of material causing destruction. Some traces show evidence of break and remake.
- (3) As fabricated dimension does not include sealing coat of lacquer.
- (4) Corrosivity as used at UTC is a measure of oxygen available in the chamber gases for the attack of carbon. It is calculated from theoretical composition.
- (5) Blend of N₂H₄ is by wt-%: N₂H₄ - 66.7 MMH - 24.0 H₂O - 9.3

CONFIDENTIAL

CONFIDENTIAL

UTC 2180-FR

TABLE VII
(U) PERFORMANCE PROPERTIES FOR FOAM CERAMIC INSERT FIRINGS

	Firing No.									
	<u>310*</u>	<u>311*</u>	<u>317*</u>	<u>314*</u>	<u>316-1*</u>	<u>316-2*</u>	<u>316-3*</u>	<u>320†</u>	<u>327*</u>	<u>332†</u>
Average chamber pressure	472	244	180	246	270	232	199	125	183	172
Average mixture ratio	1.342	2.146	1.843	2.497	1.809	1.808	1.809	1.464	1.678	†
Average expansion ratio	2.36	2.05	1.35	2.09	2.32	1.95	1.72	1.58	2.18	2.09
Average delivered specific impulse	233.0	219.2	211.4	221.0	230.0	225.1	219.8	253.3	271.2	270.4
Theoretical specific impulse	247.3	240.8	235.7	235.2	246.6	238.7	231.5	264.4	284.1	†
Efficiency	94.2	91.0	89.7	94.0	93.3	94.3	94.9	95.8	95.5	†

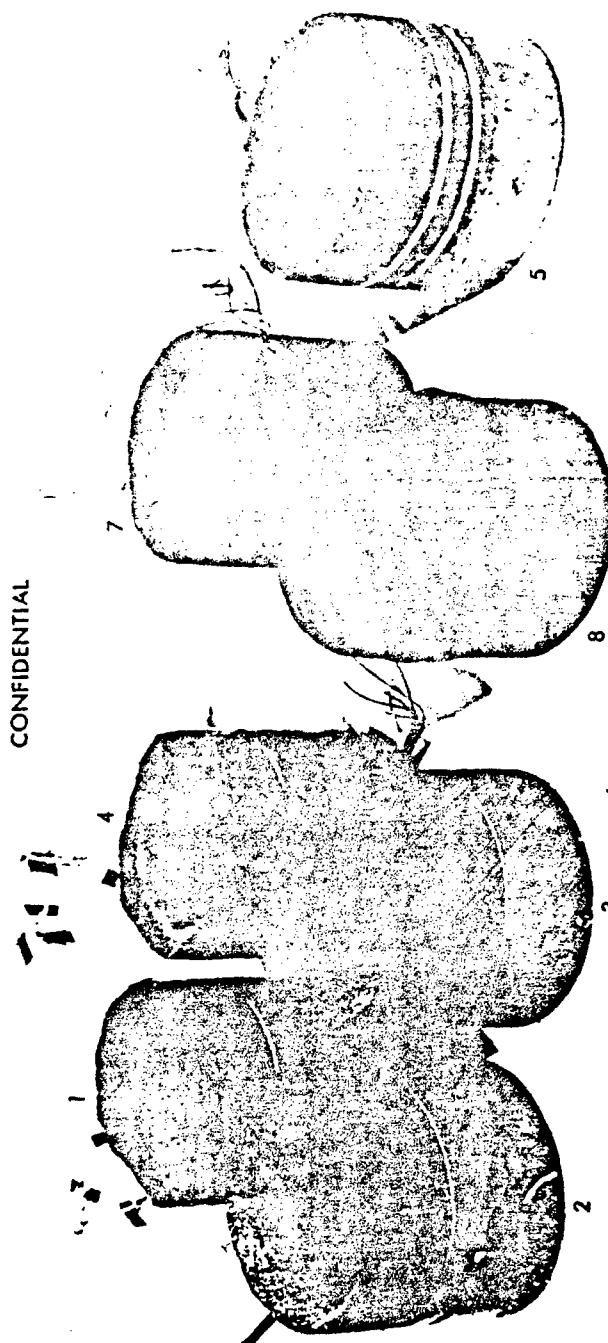
* Propellant is $N_2O_4/.5N_2H_4 - .5$ UDMH

† Propellant is $F_2/66.7 N_2H_4 - 24.0$ MMH - 9.3 H_2O

† Inconsistencies in propellant flows and O/F ratio precluded analysis of this data.

CONFIDENTIAL

CONFIDENTIAL



R-70215

6033-22

Figure 20. (U) Test-Fired Ceramic Foam Nozzle Inserts

CONFIDENTIAL

CONFIDENTIAL

UTC 2180-FR

SECTION VII

POSTTEST ANALYSIS

(U) A posttest analysis was conducted by UTC to evaluate nozzle performance under the two conditions previously described. Erosion and char depth profiles are shown in the photographs of the nozzle inserts in axial cross section (figures 21 through 27) and throat contours (figures 28 through 34). Numerical values for erosion and char depth are given in table VI as are values for erosion rates. Condition and depth of the char are shown in figure 35 which is typical of the materials fired under condition 1, and figure 36 which is typical of the result of $\text{ZrC}_2 + \text{C}$ fiber nozzles subjected to condition 2. In both photographs the char zone and eroded surface appear below the scale.

(U) Figure 37 presents a replot of pressure and thrust data from firing No. 311, which employs the $\text{N}_2\text{O}_4/\text{N}_2\text{H}_4$ - UDMH propellant with the $\text{Al}_2\text{O}_3 + \text{SiO}_2$ fiber nozzle insert No. 1, run No. 2. This replot is representative of firings in this program. Because cavitating venturis were used to control propellant flows, the pressure loss reflects the opening of the throat as a result of erosion.

1. PERFORMANCE OF NOZZLES

a. Erosion

(C) In general, it can be seen that inserts fabricated from $\text{Al}_2\text{O}_3 + \text{SiO}_2$ fibers eroded less than $\text{ZrO}_2 + \text{SiO}_2$ fiber inserts with $\text{N}_2\text{O}_4/\text{N}_2\text{H}_4$ - UDMH propellant. The erosion rates for the former varied from approximately 20 to 25 mils/sec on the diameter at chamber pressures from 200 to 250 psi; and at 60 mils/sec at 475 psi while the erosion rates for the latter at 250 psi were in the order of 34 mils/sec on the diameter.

(C) Under condition 2, the tests employing liquid fluorine and hydrazine blend ($\text{LF}_2/\text{N}_2\text{H}_4$ Blend), one of the $\text{ZrO}_2 + \text{C}$ fiber inserts eroded at approximately 55 mils/sec, with chamber pressures of 125 psi. This rate of erosion was considered so abnormally high that the second insert of this composition was not fired. The high observed erosion rates were caused by nonretention of char which will be discussed below under c., Char Thickness.

CONFIDENTIAL

CONFIDENTIAL

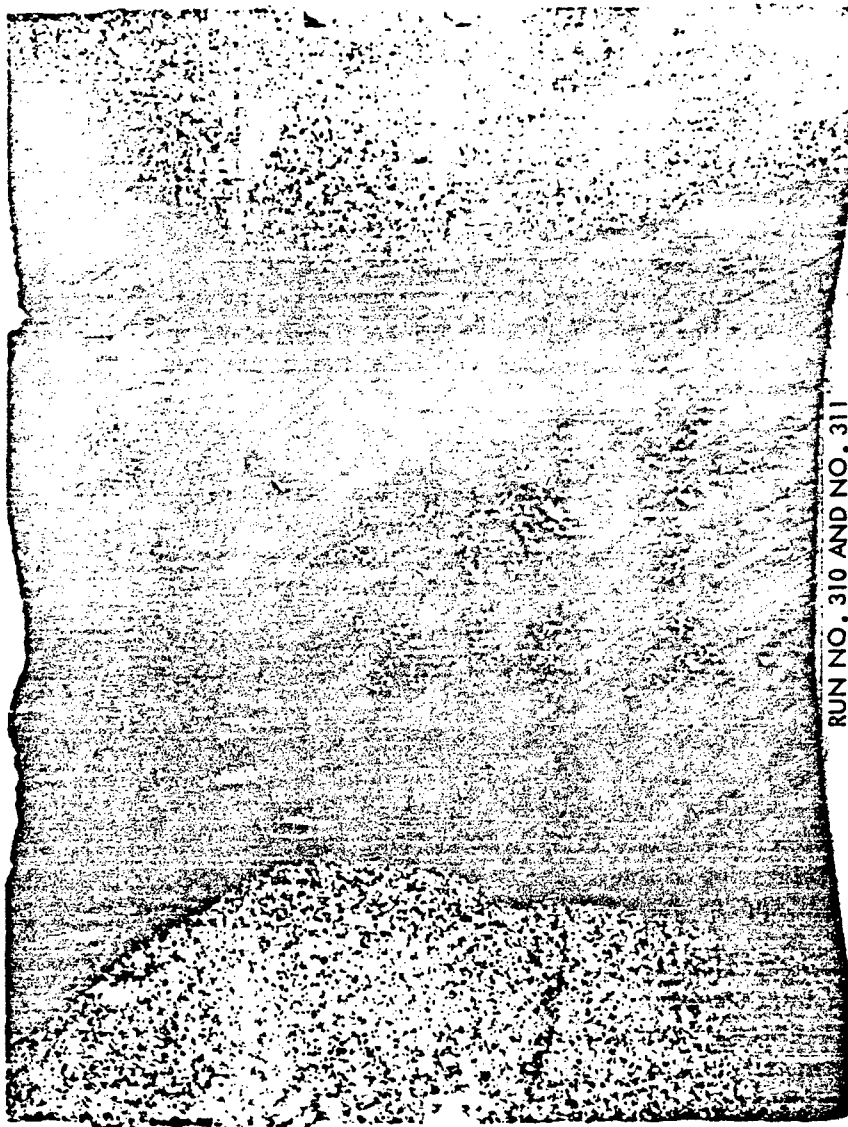


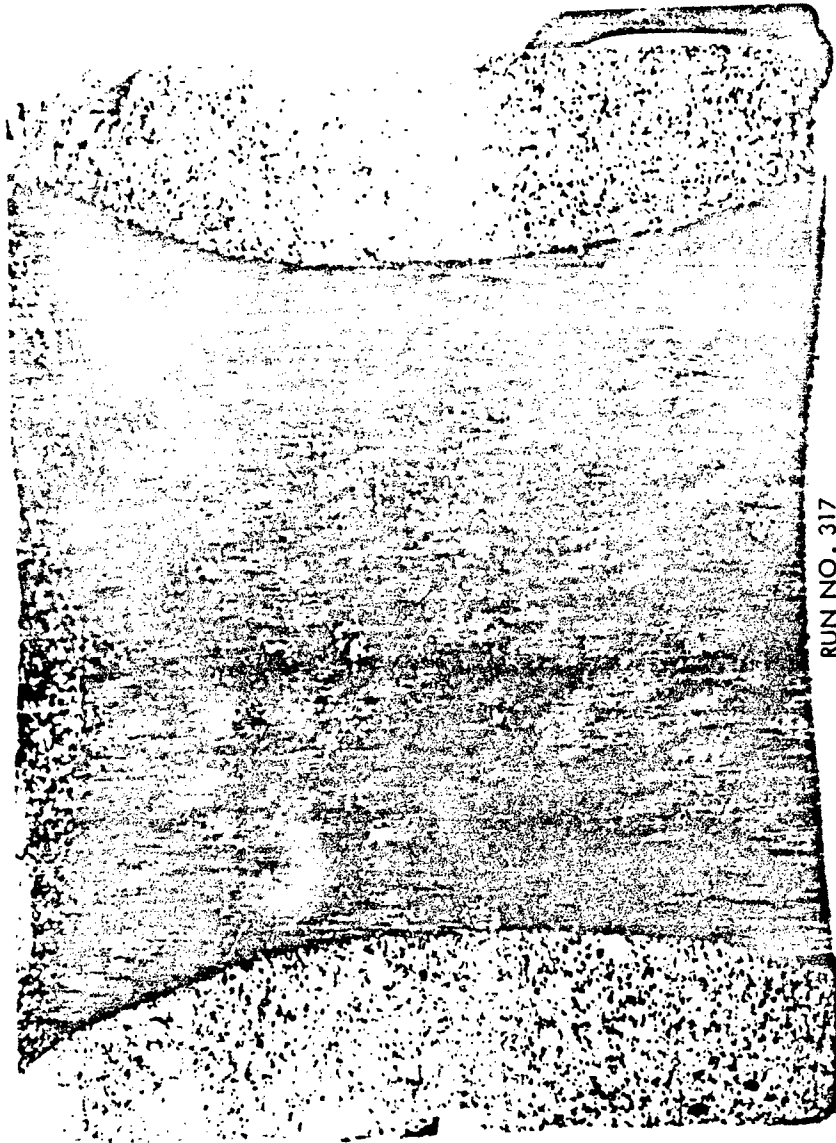
Figure 21. (U) Cross Section of Insert No. 1 — Al_2O_3 + SiO_2 Fibers

CONFIDENTIAL

(This page is Unclassified)

UNCLASSIFIED

UTC 2180-FR



R-70217

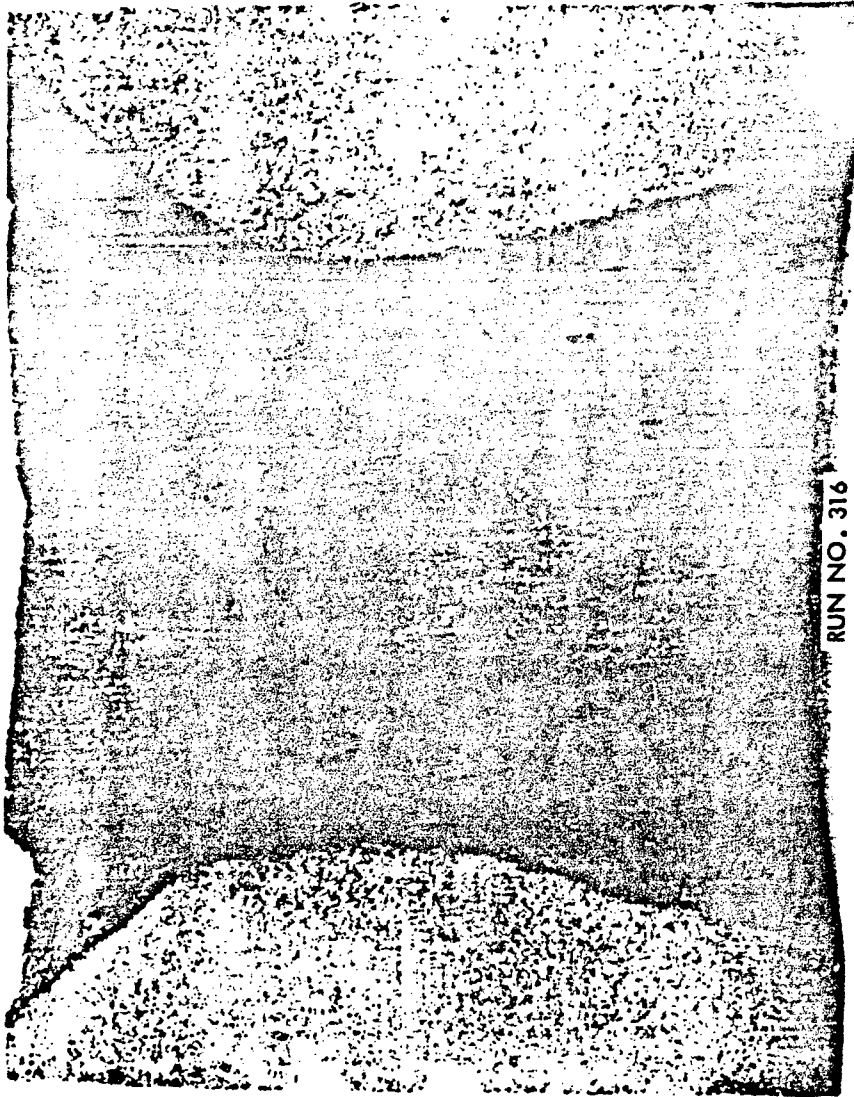
RUN NO. 317

6034-4

Figure 22. (U) Cross Section of Insert No. 2 - Al_2O_3 + SiO_2 Fibers

UNCLASSIFIED

UNCLASSIFIED



6014-3

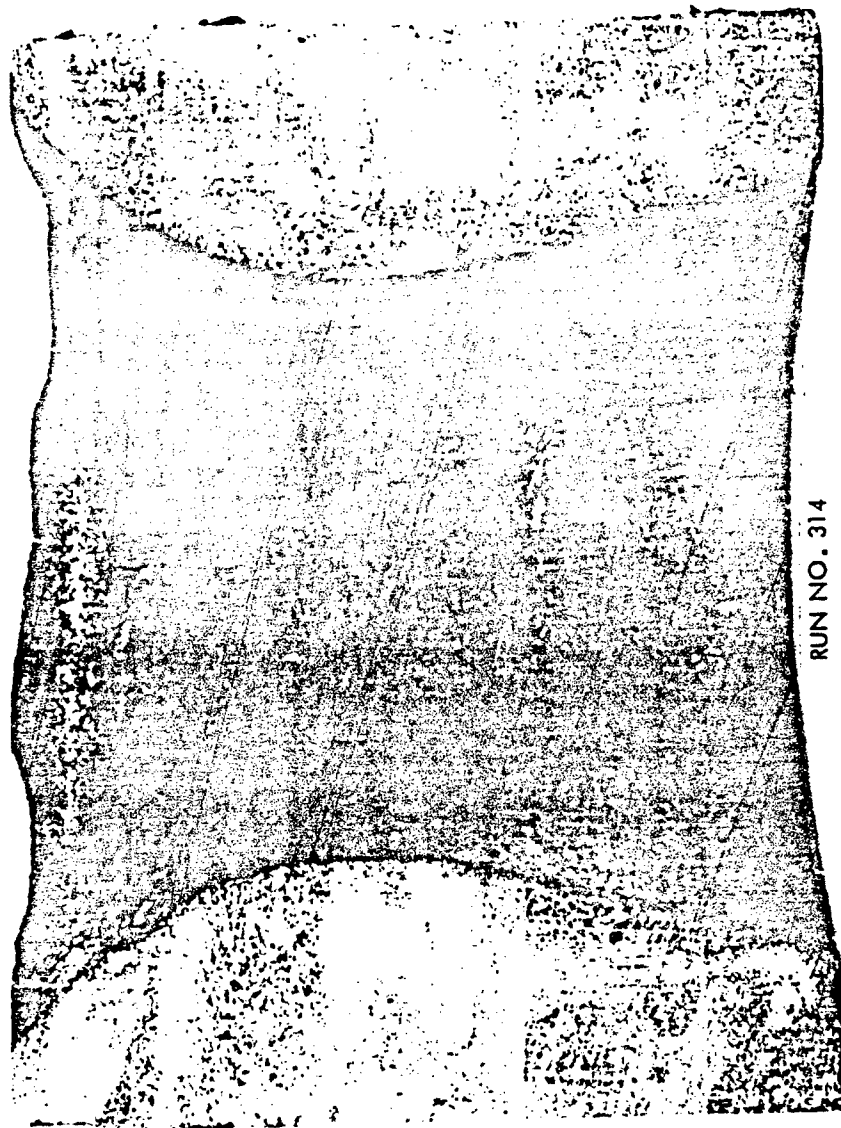
K-70218

Figure 23. (U) Cross Section of Insert No. 3 - ZrO_2 + SiO_2 Fibers

UNCLASSIFIED

UNCLASSIFIED

UTC 2180-FR



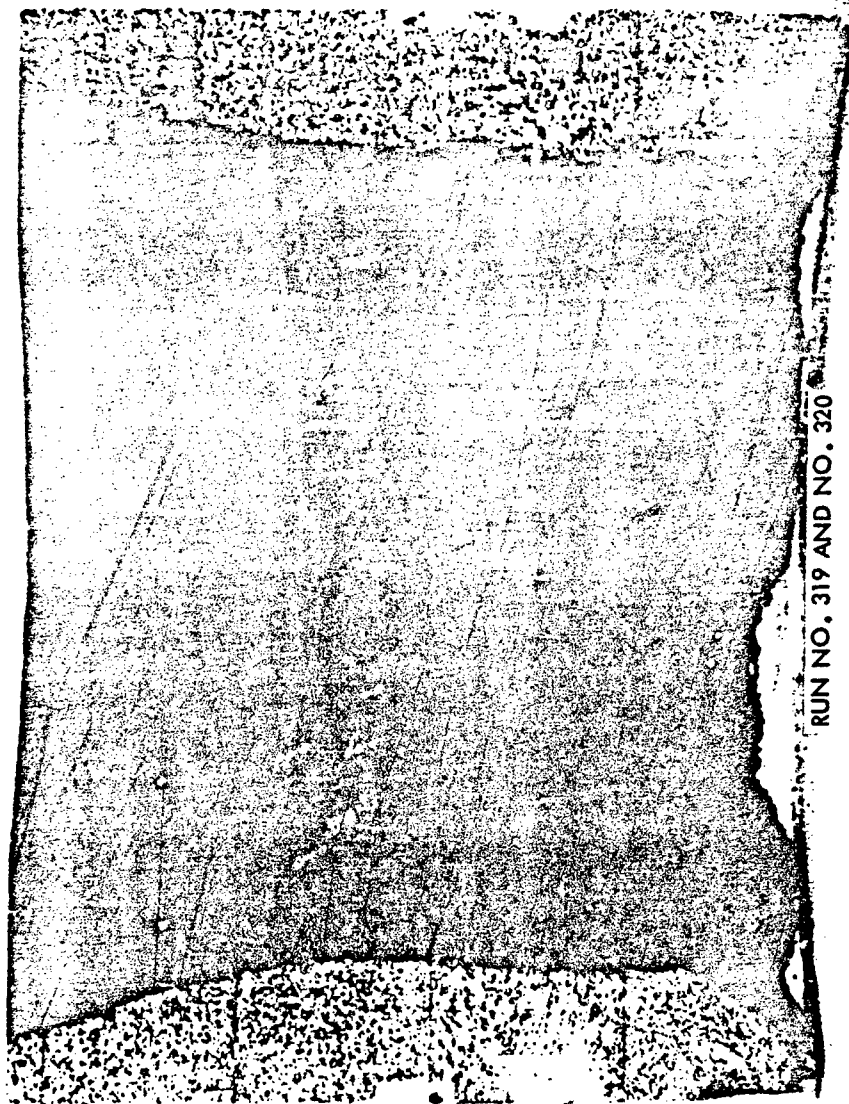
R-70219

Figure 24. (U) Cross Section of Insert No. 4 - ZrO_2 + SiO_2 Fibers

6034-7

UNCLASSIFIED

CONFIDENTIAL



R-70220

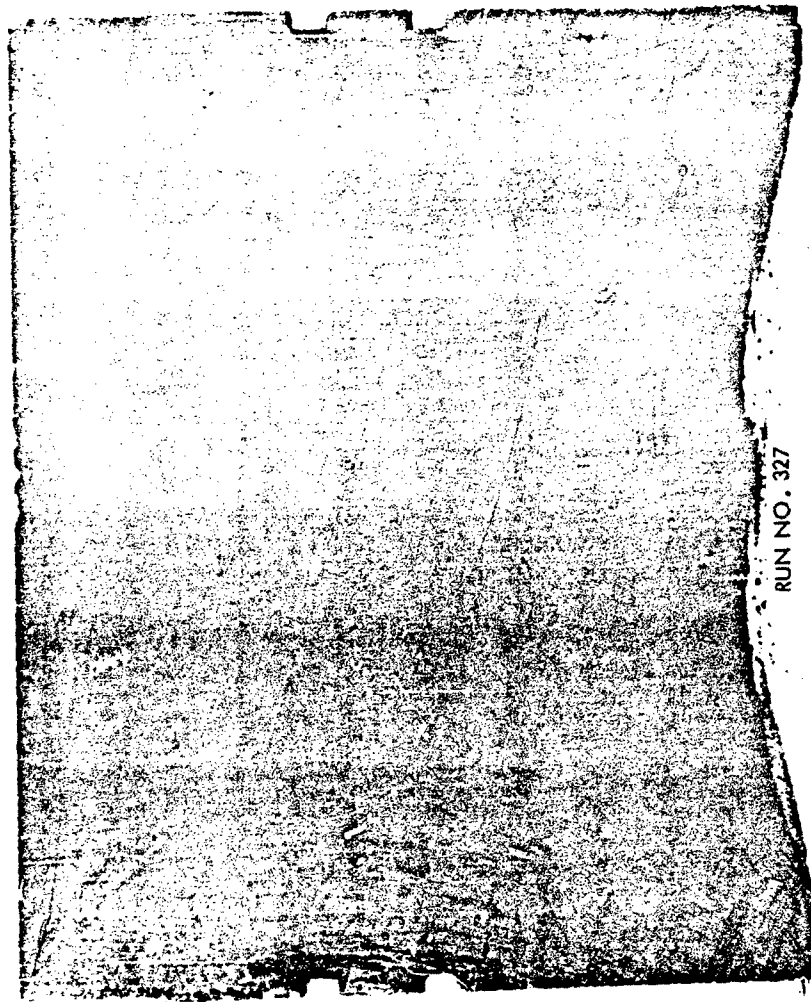
6034-5

Figure 25. (U) Cross Section of Insert No. 5 - ZrO_2 + C Fibers

CONFIDENTIAL
(This page is Unclassified)

UNCLASSIFIED

UTC 2180-FR



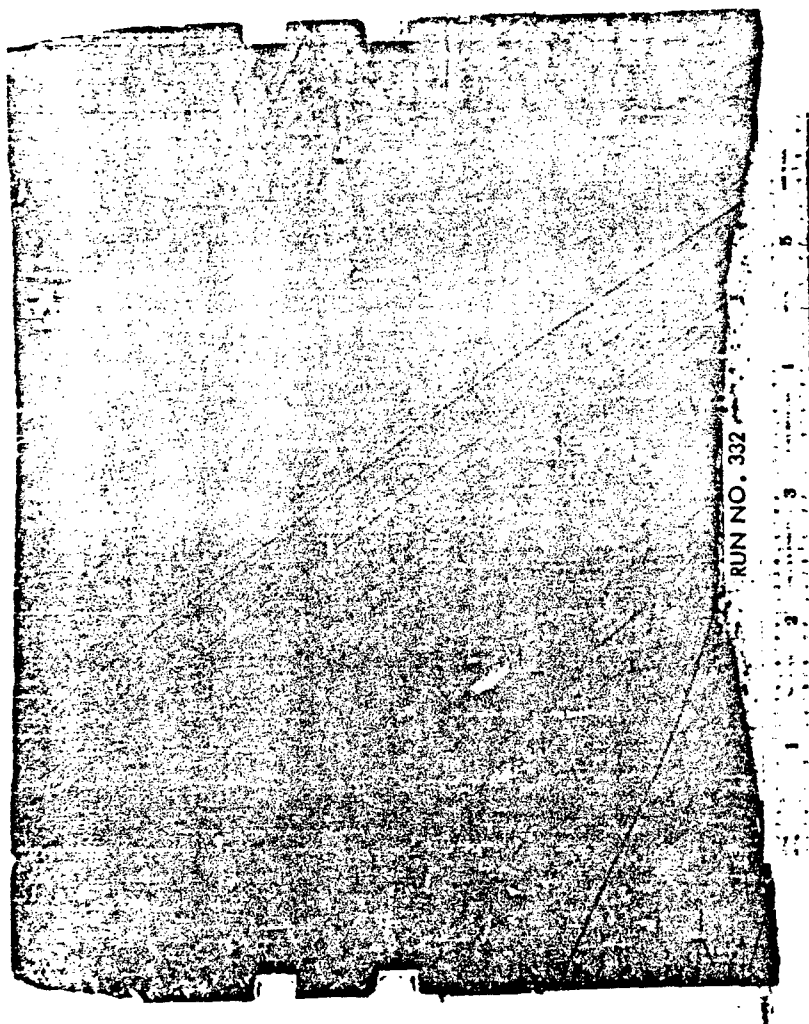
R-70221

Figure 26. (U) Cross Section of Insert No. 7 - ZrC + C Fibers

6034-1

UNCLASSIFIED

UNCLASSIFIED



R-70222

Figure 27. (U) Cross Section of Insert No. 8 - ZrC + C Fibers

UNCLASSIFIED

UNCLASSIFIED

UTC 2180-FR

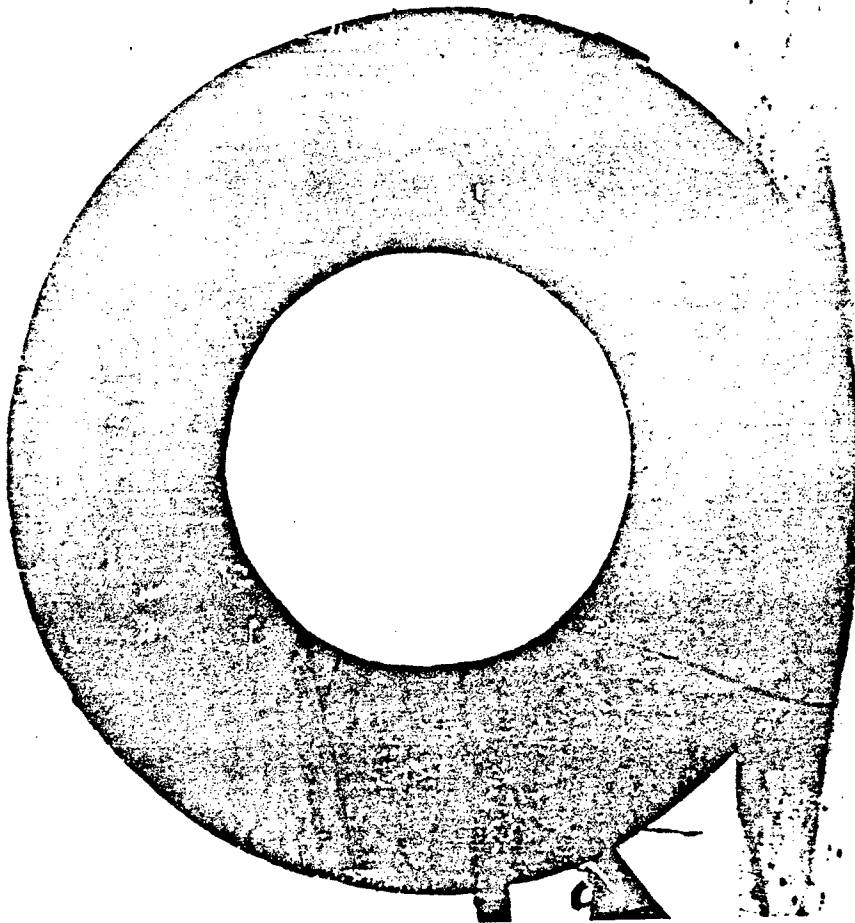
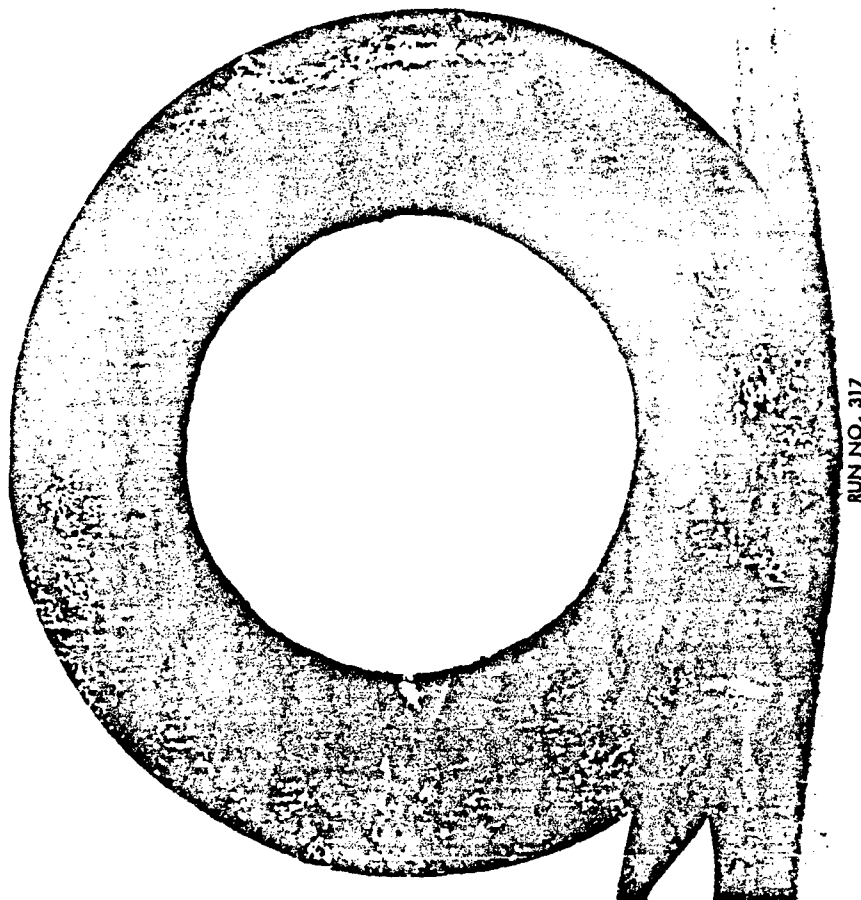


Figure 28. (U) Throat Contour Orientation Insert No. 1

UNCLASSIFIED

UNCLASSIFIED



R-70224

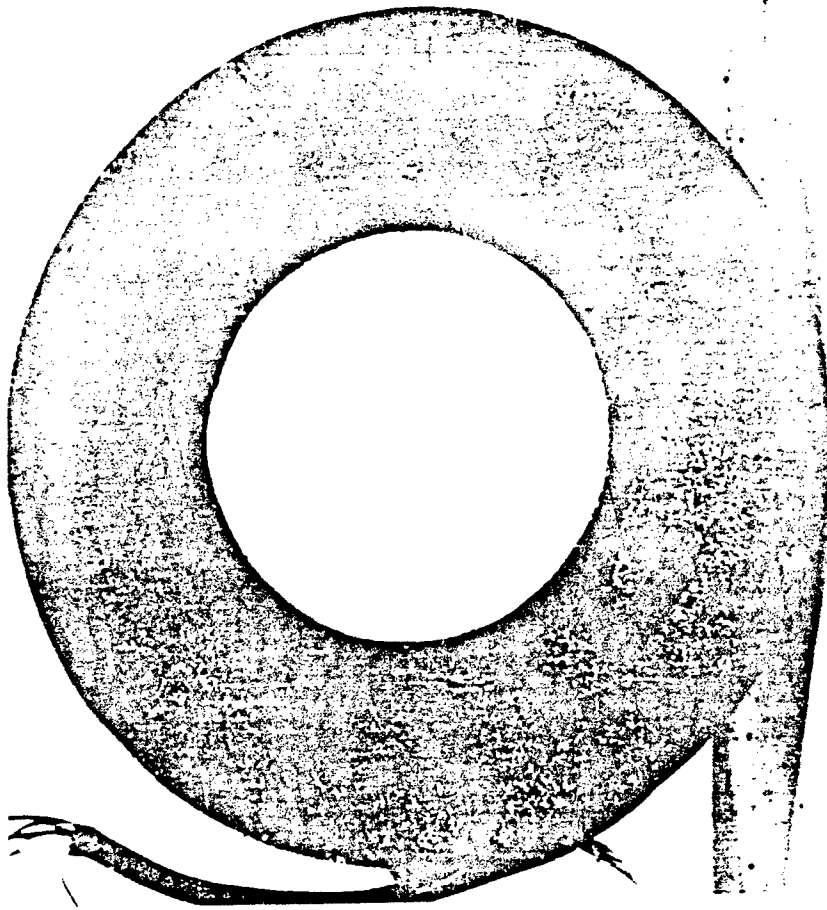
403-7

Figure 29. (U) Throat Contour Orientation Insert No. 2

UNCLASSIFIED

UNCLASSIFIED

UTC 2180-FR



RUN NO. 316

R-70225

6037-6

Figure 30. (U) Throat Contour Orientation Insert No. 3

UNCLASSIFIED

UNCLASSIFIED

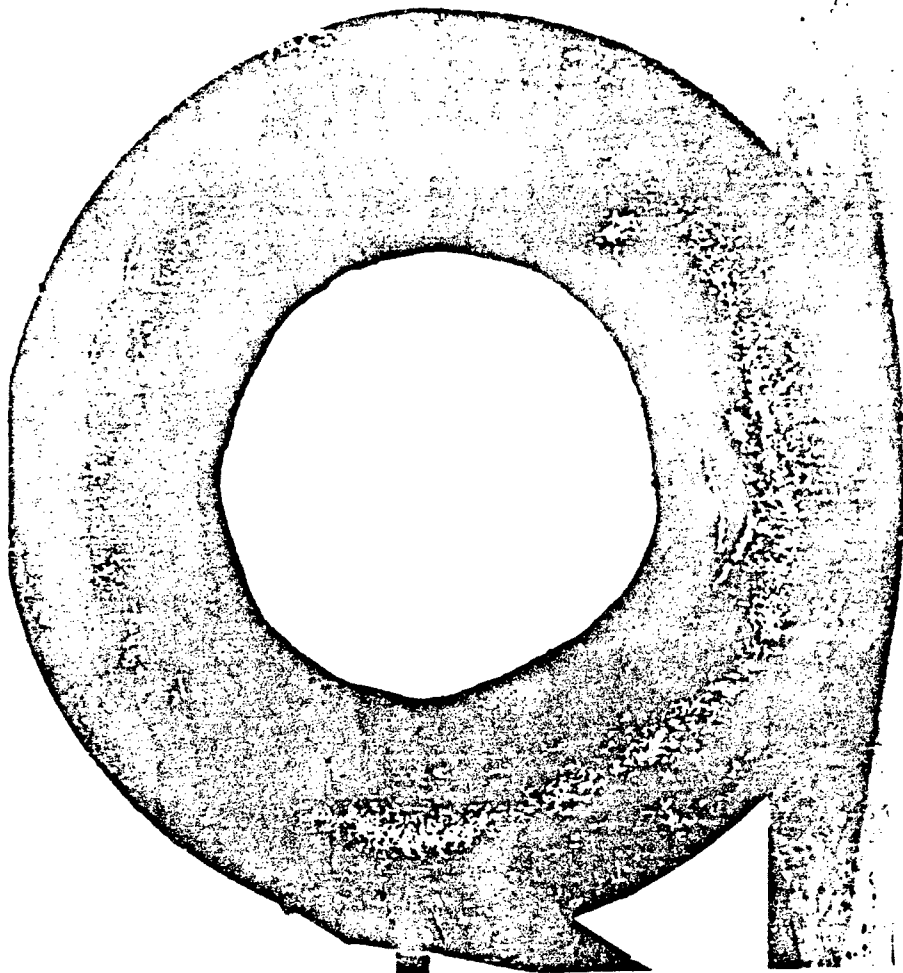
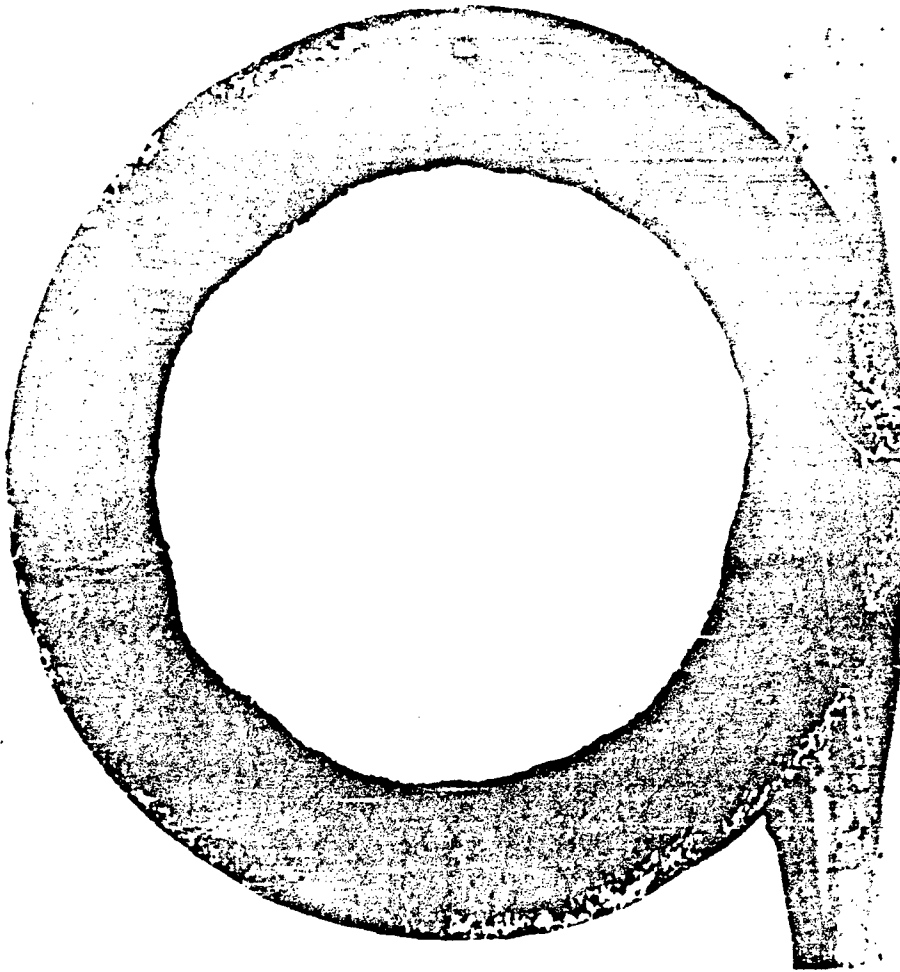


Figure 31. (U) Throat Contour Orientation Insert No. 4

UNCLASSIFIED

UNCLASSIFIED

UTC 2180-FR



RUN NO. 319 AND NO. 320

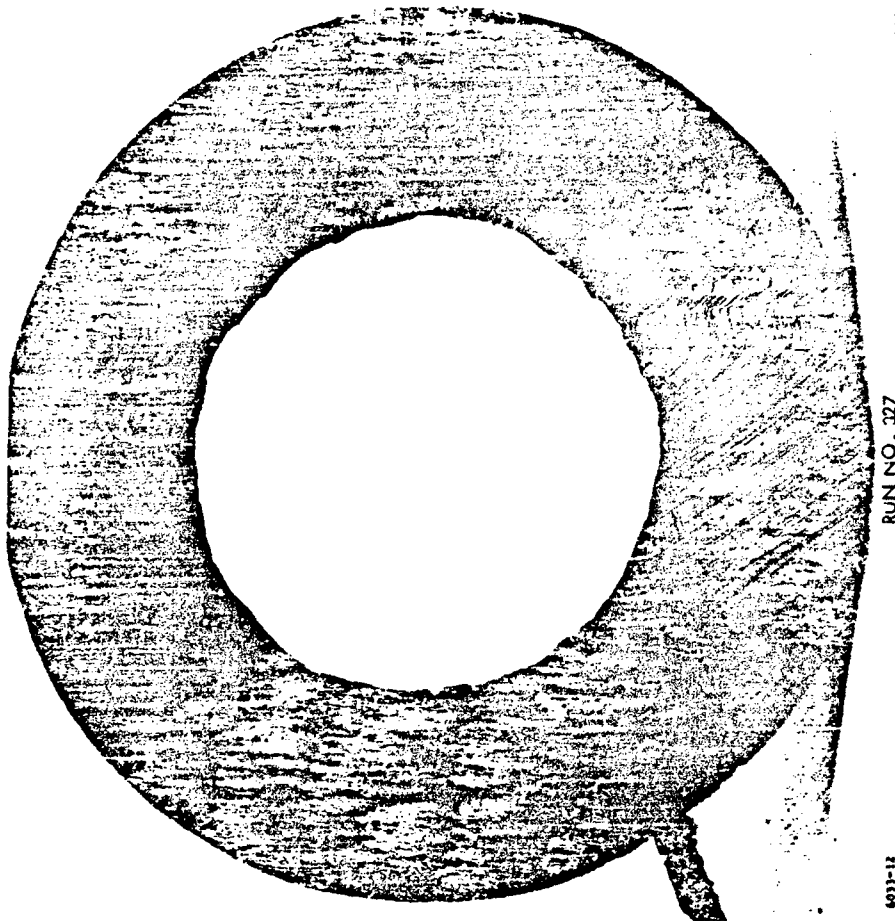
A-70227

6033-4

Figure 32. (U) Throat Contour Orientation Insert No. 5

UNCLASSIFIED

UNCLASSIFIED



R-70228

RUN NO. 327

6013-12

Figure 33. (U) Throat Contour Orientation Insert No. 7

UNCLASSIFIED

UNCLASSIFIED

UTC 2180-FR

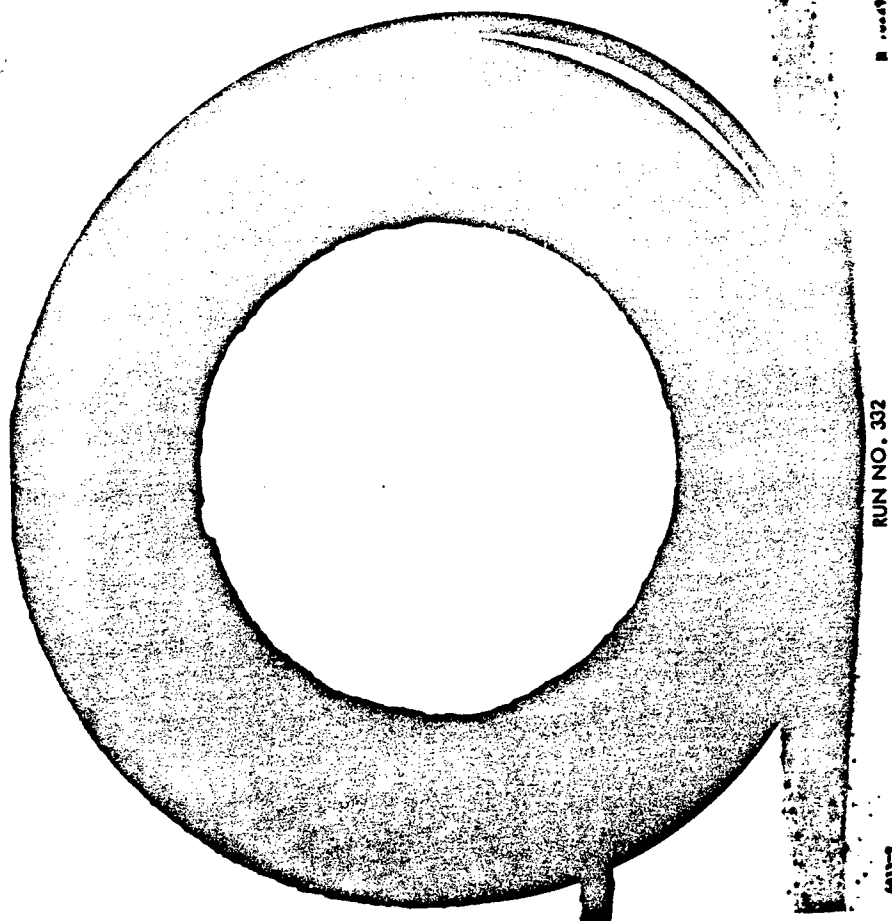


Figure 34. (U) Throat Contour Orientation Insert No. 8

UNCLASSIFIED

UNCLASSIFIED

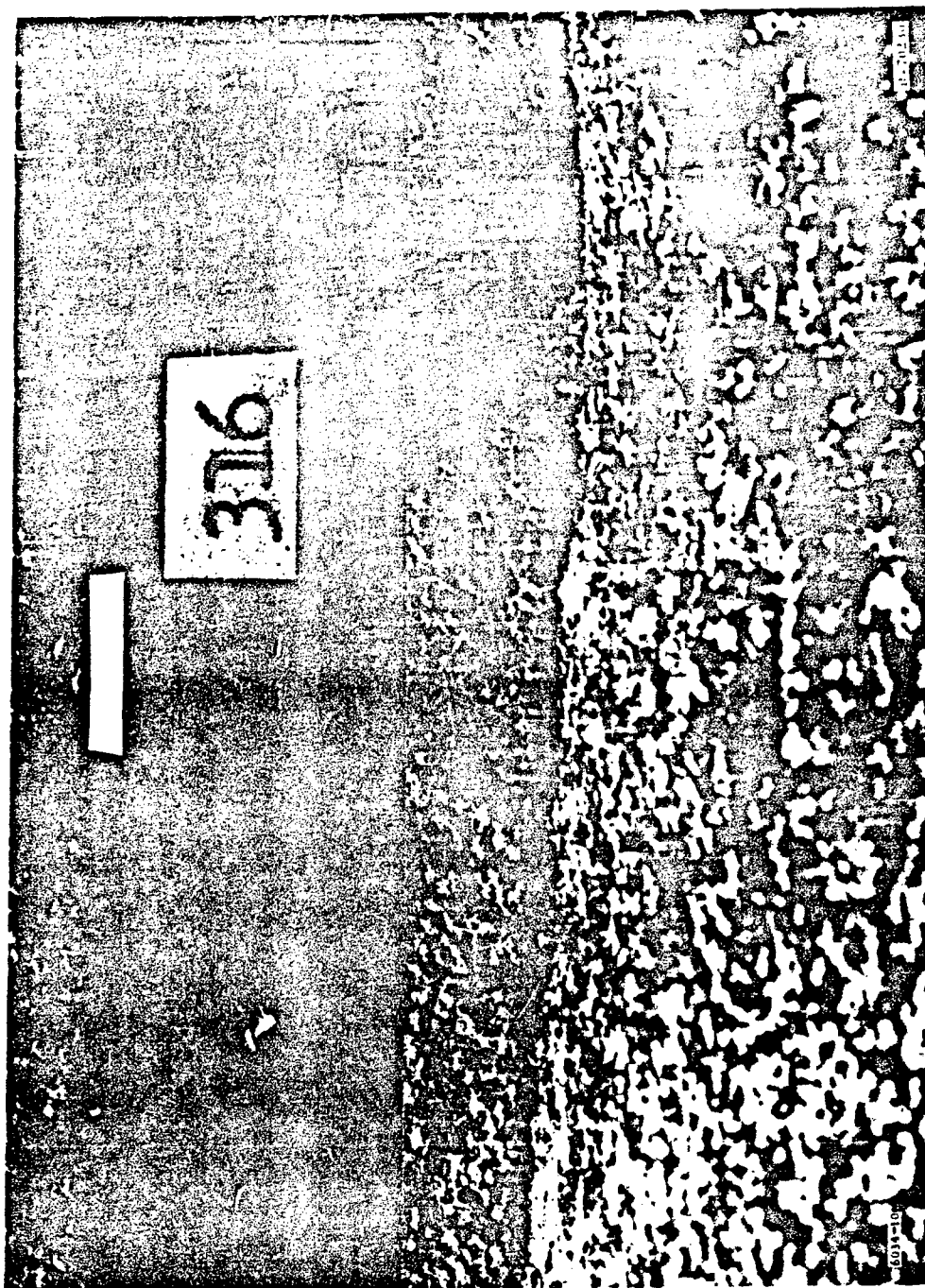


Figure 35. (U) Char Layer, Condition 1 Inserts

UNCLASSIFIED

CONFIDENTIAL

UTC 2180-FR

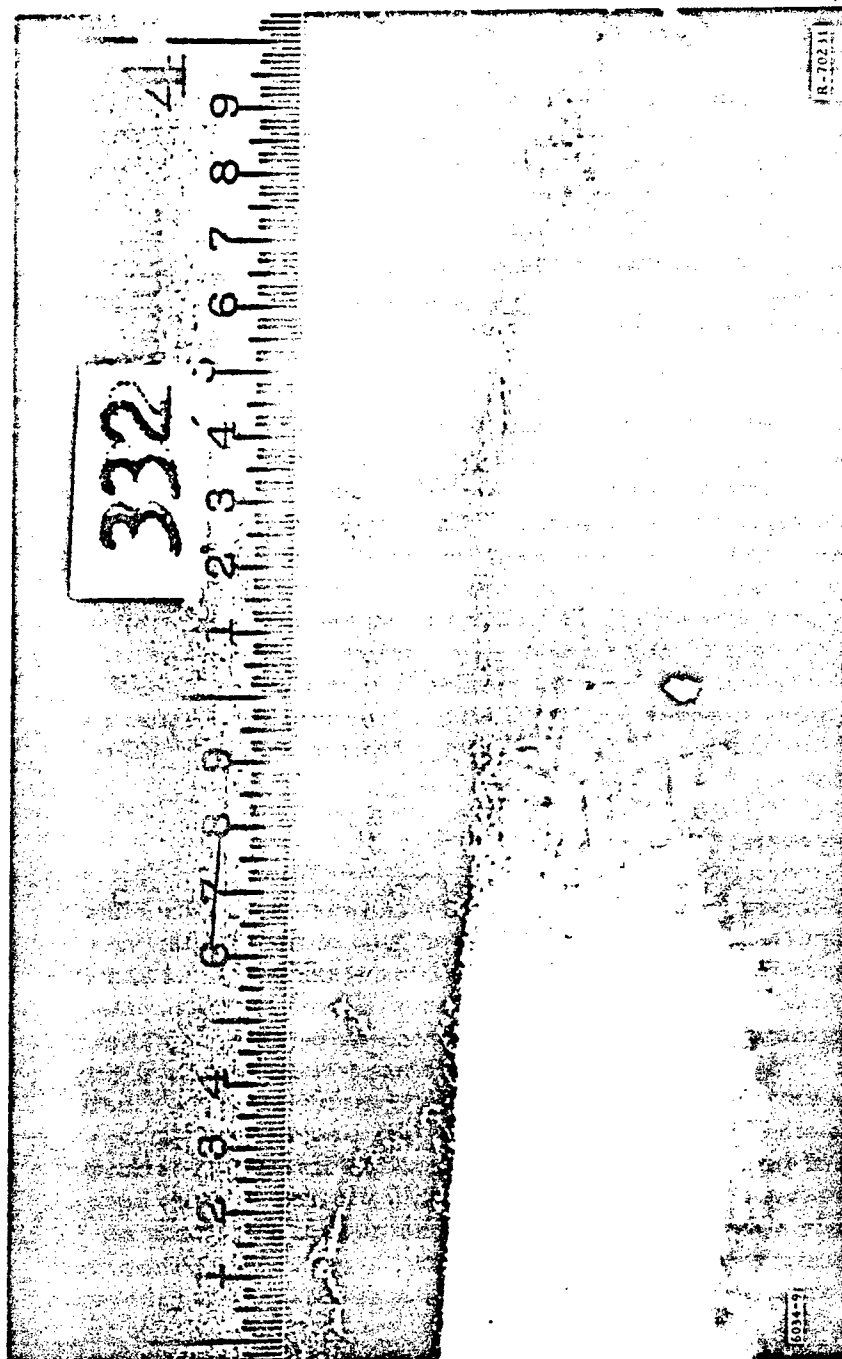


Figure 36. (U) Char Layer, Condition 2 ZrC + C Fiber Inserts

CONFIDENTIAL

(This page is Unclassified)

CONFIDENTIAL

(C) The ZrC + C fiber inserts tested under condition 2 appeared to perform the best of the inserts fired. Erosion rates were 3 to 13 mils/sec at pressures of approximately 175 psi.

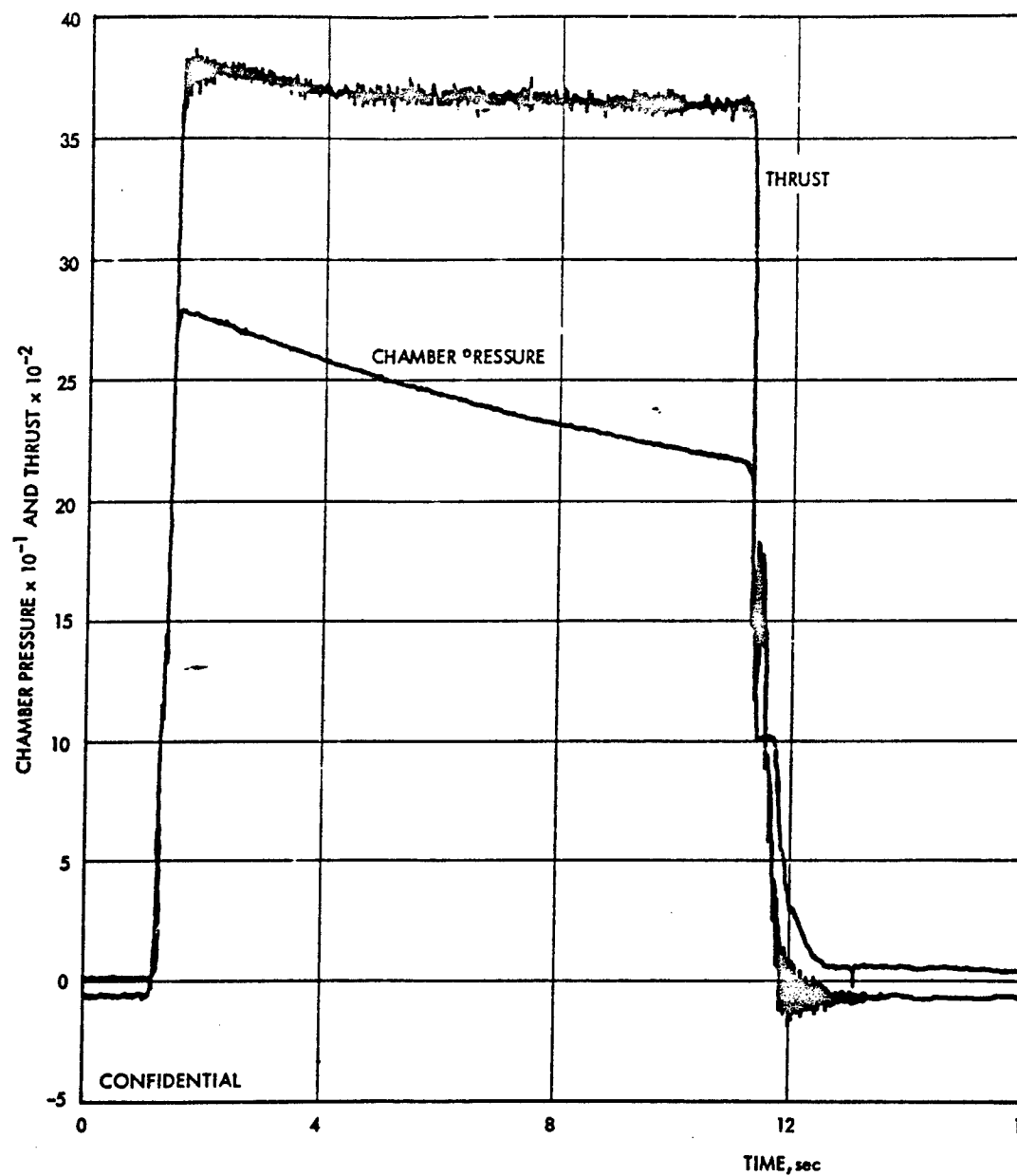
(U) The mechanism of char erosion is generally accepted to be the attack of carbon by corrosive gases in the flow, mainly CO_2 and H_2O , as controlled by the rate of the diffusion of these species through the boundary layer and by the rate of adsorption on the surface. Once the temperature of the char exceeds air induction temperature (about 3,600°F in this case), the corrosion reaction rates are very fast and the above-mentioned steps are controlling, taking but a few seconds in firings of the sort involved in this program. However, adsorption as a controlling step is bypassed by the porosity of the surface of any but the densest chars.

(U) The diffusion rate is a function mainly of mass velocity (a function of pressure) and corrosivity of the gas mixture. The calculated corrosivity of chamber gases based on the mole fraction relationship of the combined ($\text{CO}_2 + \text{H}_2\text{O} + 2\text{O}_2 + \text{O} + \text{NO} + \text{OH}$) molecules to the total molecules in the propellant gases is reported in table VI. The erosion rates reported in table VI can be compared with the average pressures and corrosivities and can be seen to correlate in a general way. However, this applies only when the char is retained, and in most of these firings there is evidence that the char was lost from failure of the reinforcement. This is discussed under section c. Char Thickness.

b. Temperature Data

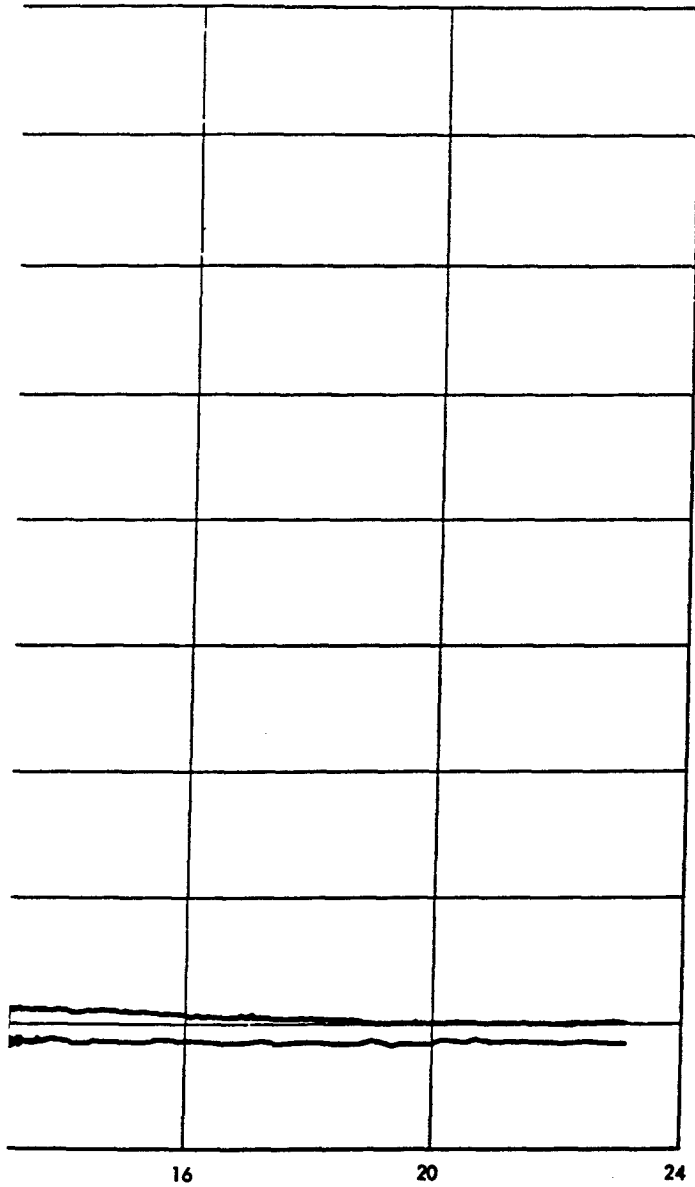
(C) A review of the thermocouple locations, as shown in figures 6 and 7, combined with an inspection of the erosion rate and temperature data, from table VI, reveals that low temperatures are maintained at the thermocouple-bead location almost until the moment of exposure by erosion or by charring when the char is not retained. This performance attests to the highly insulative properties of the foam ceramic materials. The erratic quality of the temperature data from this program is characteristic of that obtained with ablating materials as the technology of the use of thermocouples for this purpose has not been adequately developed. This is because of such problems as (1) maintaining contact with the material, (2) applying the proper contact pressure, and (3) carburization of the thermocouple weld.

CONFIDENTIAL



CONFIDENTIAL

UTC 2180-FR



R-70237

Figure 37. (U) Typical Thrust
and Chamber Pressure Curves
for Foam Ceramic
Insert Firings

CONFIDENTIAL

UTC 2180-FR

c. Char Thickness

(C) Only superficial charring occurred in the $\text{Al}_2\text{O}_3 + \text{SiO}_2$ fiber insert firings, and the $\text{ZrO}_2 + \text{SiO}_2$ fiber inserts charred to a depth of some 40 mils beyond the eroded surface contour after approximately 15 sec of firing.

(C) A 100- to 150-mil char layer was formed on the $\text{ZrC} + \text{C}$ fiber inserts in approximately 8 sec. These units displayed a completely different charring trend than the $\text{ZrO}_2 + \text{C}$ fiber inserts which charred only superficially. In the same length of time, of course, the ZrO_2 eroded rapidly and the ZrC eroded relatively slowly.

(C) Fiber incorporation did not seem to materially improve performance of the oxide foams. Apparently, an appreciable number of fibers tended to curl up into balls during the foam mixing operation. This reduced the total effectiveness of the fibers as reinforcement and negated their potential for shear and spalling resistance improvement in the char layer.

(C) Ablation resistance is fundamentally dependant upon the retention of char, without which erosion is rapid and irreproducible. The means of holding the char is through the effectiveness of the ceramic foam augmented by incorporation of fibers, which are hopefully oriented in a direction to act as ties across the pyrolysis zone. There are two limitations to fiber or foam reinforcement function (1) melting point, and (2) reactivity in the char. Alumina ($3,600^\circ\text{F}$), zirconia ($4,900^\circ\text{F}$), and silica ($3,000^\circ\text{F}$) all have melting points high enough to be useful in rocket nozzles, but apparently only silica is inert enough for this service as recently demonstrated in laboratory tests. This applies whether the fibers or foam are alumina (or zirconia) and they are attacked by the char, or the fibers are carbon attacked by the oxides.

(C) The observed retention of char by the zirconium carbide-carbon system is consistent with this viewpoint because there is no reaction between phases. It is apparent that silica melts before it reaches temperatures at which carburization reactions are important kinetically. Therefore, no evidence of reactivity would be anticipated.

CONFIDENTIAL

CONFIDENTIAL**d. Effect of Properties on Performance**

(U) Referring to the bar graphs of the property measurement data, figures 14 through 16, an order of merit can be assigned within each set of properties reflecting resistance to erosion if the role of that property in erosion of the foam structure is known. Table VIII is a compilation of a set of such orders of merit. It should be pointed out that the basis for evaluating erosion-proneness is conjectural. However, there appears to be a basis for the assumption that lower thermal expansion, and in some instances lower thermal conductivity, promote resistance to erosion. Similarly, higher specific heat and higher tensile and compressive strengths appear to contribute to erosion resistance. The role of density is difficult to define. If the volume of ceramic and fiber portions is constant from composition to composition, which is the goal during fabrication of the foams, then the density is directly a function of the density of the ceramic and fiber additions. On the other hand, the change of density between foams of various compositions is more often attributable to degree of resin penetration related to adsorptive, absorptive, or catalytic nature of the skeletal foam materials as well as to staging of the resin. The order of merit for density chosen for this program is given on the basis of a knowledge of the degree of impregnation of the foams concerned and is a value judgment.

(U) It can be seen that for each firing test condition the material displaying the better order of merit numbers eroded less than the material displaying the poorer order of merit numbers, i. e., Al_2O_3 inserts performed better than ZrO_2 inserts under condition 1 and ZrC inserts performed better than ZrO_2 inserts under condition 2.

(U) The influences permitting or inhibiting erosion are too numerous and/or too subtle to be defined alone by the properties measured in this program.

(U) If any factor were to be singled out as significant, it might be the compressive strength property which is high in the best performing materials for each condition.

(U) The roles of fiber orientation, fiber content, volatile constituents, and density are difficult to define, measure, or control.

CONFIDENTIAL

(This page is Unclassified)

CONFIDENTIAL

UTC 2180-FR

TABLE VIII

(U) PROPERTIES - ORDER OF MERIT

To be used for	Condition 1		Condition 2	
With propellant	N_2O_4/N_2H_4 -UDMH		Liquid F_2/N_2H_4 Blend*	
Foam compositions	Al_2O_3 + SiO_2 Fibers	ZrO_2 + SiO_2 Fibers	ZrO_2 + Carbon Fibers	ZrC + Carbon Fibers
Nozzle numbers	1 and 2	3 and 4	5 and 6	7 and 8

<u>Property</u>	<u>Order of Merit Relative to Erosion Resistance</u>			
Thermal expansion (see figure 14)	1	2	4	3
Specific heat (see figure 15)	1	2	4	3
Thermal conductivity (see figure 16)	4	2	1	3
Tensile strength (see figure 17)	3	1	4	2
Compressive strength (see figure 17)	1	4	3	2
Density (see figure 18)	1	2	4	3

* Blend of N_2H_4 : wt-% - 66.7 N_2H_4 , 24.0 MMH, 9.3 H_2O

61
CONFIDENTIAL
(This page is Unclassified)

CONFIDENTIAL**e. Ballistic Performance**

(C) The nozzle efficiency reported in table VII is another indicator of material performance. This is aside from the amount of erosion, because the theoretical specific impulse calculated for these efficiencies is based on a theoretical throat having the time-average area of the actual firing. Therefore, it reflects the uniformity of erosion and smoothness of the surface because lack of these qualities generate weak shocks that erode ballistic performance. It can be seen that the efficiencies do correlate generally with the uniformity that has been reported.

(C) However, it is not known why the efficiency of the three pulses appears to rise in firing No. 316 instead of falling as the throat erodes, except to say that the same phenomenon has been observed in multiple-pulse solid propellant motors.

CONFIDENTIAL

CONFIDENTIAL

UTC 2180-FR

SECTION VIII

CONCLUSIONS

(C) In general, erosion rates for the foam ceramic composites were high under the test conditions. It is apparent that these materials cannot be recommended for use as nozzle inserts for normal liquid rocket motor applications. A possible exception is the ZrC composition, which if upgraded from its present 3 to 13 mils/sec erosion rate could be potentially applicable.

(C) The erosion mode of the foam ceramic inserts makes them potentially interesting, particularly if their erosion resistance can be improved. This refers to their obvious ability to erode uniformly so as to provide little or no deviation from circularity in the throat of the insert as a result of firing. The significance of this phenomenon will be immediately recognized for its potential value in minimizing requirements for roll control and thrust vector control devices where no deviation from circularity is obtained in the nozzle throat.

(U) A comparison of the thermal and mechanical properties measurements with erosion data indicates that there may be a basis for predictability of performance for foam ceramic materials.

(U) Improvement in techniques for fiber inclusion into the foams, both with respect to the number of fibers and their proper disposition, is needed in order to upgrade properties of the foams. This could involve inclusion of patterns of fibers, weaves, or felts. In addition, metal and ceramic honeycomb reinforcements should be investigated as means for upgrading the foams.

(U) Specifically, foam ceramics show promise in spite of their generally high erosion rates in the tests performed under this contract. It should be kept in mind that these composites are prepared from inexpensive materials at ambient temperatures and pressures, are light in weight, easily fabricated, and may be readily produced in complicated shapes which can be tailored to requirement both with respect to shape and composition.

(U) The components so produced are insensitive to thermal shock, can be used in stop-start applications, behave well under heat soak conditions, retain a protective char layer in low shear stress environments, under static and low gas-flow conditions, and serve as an exemplary thermal insulator.

CONFIDENTIAL

CONFIDENTIAL

(U) In addition, components produced by foam ceramic methods can be readily repaired in the field at ambient temperatures and pressures and can be restored to service in a matter of minutes.

(U) When the effectiveness of the reinforcement of foam ceramic composites is sufficiently improved, these materials could be useful in high mass velocity environments such as are encountered in rocketry.

CONFIDENTIAL

(This page is Unclassified)

UNCLASSIFIED

UTC 2180-FR

SECTION IX

POTENTIAL AREAS OF APPLICABILITY

(U) Potential areas of applicability are as follows:

- A. Exit cone liners
- B. Thermal insulation, in both the unimpregnated and impregnated condition in static or relatively low gas-flow areas
- C. Nozzles for systems requiring known and controlled pressure drop where programming through nozzle erosion characteristics is more feasible than change of grain shape or burning rate
- D. Hybrid rocket motor mixers
- E. Where a charred reradiation surface can help in thermal control
- F. For safety systems sensitive to high pressure rise where low pressure, modest, gas- or liquid-flow is normal
- G. For systems where uniform erosion of nozzle throats is required and where deviation from circularity of the throat causes severe thrust vector or roll control problems
- H. Internal heat shields for solid and liquid motors
- I. Motor case liners
- J. Low-pressure thrust chambers
- K. As backing insulation in blast tubes and nozzles
- L. As base region insulation.

UNCLASSIFIED

UNCLASSIFIED

UTC 2180-FR

ALL INFORMATION CONTAINED HEREIN IS UNCLASSIFIED

APPENDIX

MATERIALS R&D REPORT

⁶⁷
UNCLASSIFIED

UNCLASSIFIED

UTC 2180-FR

APPENDIX

MATERIALS R&D REPORT

(U) This report is submitted to satisfy requirements of line item 7 of DD Form 1423 of the contract pertaining to the "Foam Ceramic Nozzle Inserts" Program.

1. PROPERTIES MEASUREMENTS

(U) Laboratory tests were performed on the materials employed for the selected nozzle insert compositions. Physical, mechanical, and thermal property measurements were made on these materials to supply data for an Air Force computer program formulated to predict material performance in rocket engines.

(U) Table IV of the report lists the tests, the number of test specimens, their shape and size, and the test-conducting agency.

(U) The shape and size of the test specimens is shown in figures 9 through 13, section V, of the report.

2. PROPERTIES DATA

(U) The following tables present the data for the properties measured in accordance with table IV of the report. Table IX lists the measured coefficients of thermal expansion for the impregnated and cured foam ceramic composites at 100°, 150°, and 200°F. Table X lists the specific heat characteristics for the foam ceramic compositions listed, measured prior to impregnation of the foam. Table XI lists the specific heat characteristics for foam ceramic composites after charring of the composite. Table XII lists the thermal conductivity for the foams prior to impregnation with phenolic resin. Table XIII lists the thermal conductivity for the foam ceramic components after charring of the composite. Table XIV lists values of tensile strength for impregnated and cured foam ceramic composites. Table XV lists values of compressive strength for impregnated and cured foam ceramic composites. Table XVI lists densities for virgin foam, impregnated and cured foam, and charred foam, respectively.

3. PROPERTIES COMPARISON

(U) A comparison of the properties reported in tables IX through XVI can be found by reference to the set of bar graphs based on the data from these tables (see section V).

UNCLASSIFIED

UNCLASSIFIED

TABLE X

(U) THERMAL EXPANSION OF FOAM CERAMIC MATERIALS
FOR CURED IMPREGNATED FOAM COMPOSITES

$$\alpha = \text{in./in./}^{\circ}\text{F}$$

Type	Sample No.	Direction*	α at 100°F	α at 150°F	α at 200°F
Al ₂ O ₃ + SiO ₂ fibers	1	A	16.2	11.3	8.2
	2	A	19.3	15.3	14.0
	3	A	<u>20.3</u>	<u>15.1</u>	<u>10.7</u>
		Avg	18.6	13.9	11.0
	4	B	17.6	14.4	10.5
	5	B	22.3	15.7	11.1
	6	B	<u>13.3</u>	<u>9.9</u>	<u>7.1</u>
		Avg	17.7	13.3	9.6
ZrO ₂ + SiO ₂ fibers	7	A	38.0	32.4	28.6
	8	A	50.3	40.9	39.6
	9	A	<u>27.3</u>	<u>22.6</u>	<u>17.8</u>
		Avg	38.5	32.0	28.7
	10	B	15.0	10.5	8.0
	11	B	19.2	16.9	12.0
	12	B	<u>18.2</u>	<u>14.2</u>	<u>17.4</u>
		Avg	17.5	13.9	12.5

* Direction A — in pouring direction
Direction B — 90° to pouring direction

UNCLASSIFIED

UNCLASSIFIED

UTC 2180-FR

TABLE IX

(U) THERMAL EXPANSION OF FOAM CERAMIC MATERIALS
FOR CURED IMPREGNATED FOAM COMPOSITES (Continued)

 $\alpha = \text{in./in./}^\circ\text{F}$

Type	Sample No.	Direction*	α at 100°F	α at 150°F	α at 200°F
ZrO ₂ + C fibers	13	A	49.3	39.3	38.5
	14	A	81.3	66.9	61.5
	15	A	<u>40.8</u>	<u>34.6</u>	<u>30.8</u>
		Avg	57.1	47.0	43.6
	16	B	34.6	29.1	13.9
	17	B	55.8	28.6	21.9
	18	B	<u>23.3</u>	<u>18.3</u>	<u>13.5</u>
		Avg	37.9	25.3	16.4
ZrC + C fibers	19	A	38.0	32.9	31.6
	21	A	<u>26.6</u>	<u>21.4</u>	<u>17.8</u>
		Avg	32.3	27.2	24.7
	22	B	29.2	25.6	18.2
	23	B	45.6	28.9	21.2
	24	B	<u>23.6</u>	<u>17.7</u>	<u>13.8</u>
		Avg	32.8	24.1	17.1

* Direction A — in pouring direction

Direction B — 90° to pouring direction

UNCLASSIFIED

UNCLASSIFIED

TABLE X
(U) SPECIFIC HEAT OF FOAM CERAMIC MATERIALS
FOR VIRGIN FOAMS

Type	Sample No.	Temperature	
		80°F	200°F
$\text{Al}_2\text{O}_3 + \text{SiO}_2$ fibers	33	0.197	0.211
	34	0.198	0.212
	35	<u>0.197</u>	<u>0.211</u>
	Avg	0.197	0.211
$\text{ZrO}_2 + \text{SiO}_2$ fibers	37	0.149	0.155
	38	<u>0.148</u>	<u>0.154</u>
	Avg	0.148	0.154
$\text{ZrO}_2 + \text{C}$ fibers	39	0.148	0.153
	40	0.147	0.152
	41	<u>0.148</u>	<u>0.154</u>
	Avg	0.148	0.153
$\text{ZrC} + \text{C}$ fibers	42	0.131	0.135
	43	0.121	0.124
	44	<u>0.122</u>	<u>0.125</u>
	Avg	0.125	0.128

UNCLASSIFIED

UNCLASSIFIED

UTC 2180-FR

TABLE XI
(U) SPECIFIC HEAT OF FOAM CERAMIC MATERIALS
FOR CHARRED COMPOSITES

<u>Type</u>	<u>Sample No.</u>	<u>Temperature</u>	
		<u>80°F</u>	<u>200°F</u>
$\text{Al}_2\text{O}_3 + \text{SiO}_2$ fibers	53	0.253	0.262
	54	<u>0.264</u>	<u>0.270</u>
	Avg	0.258	0.266
$\text{ZrO}_2 + \text{SiO}_2$ fibers	56	0.245	0.250
	57	0.247	0.254
	58	<u>0.236</u>	<u>0.240</u>
	Avg	0.243	0.248
$\text{ZrO}_2 + \text{C}$ fibers	59	0.194	0.200
	60	0.197	0.203
	61	<u>0.201</u>	<u>0.210</u>
	Avg	0.197	0.204
$\text{ZrC} + \text{C}$ fibers	62	0.209	0.218
	63	0.197	0.205
	64	<u>0.192</u>	<u>0.200</u>
	Avg	0.199	0.208

UNCLASSIFIED

UNCLASSIFIED

TABLE XII

(U) THERMAL CONDUCTIVITY OF FORM CERAMIC MATERIALS
FOR VIRGIN FOAMS

Btu/hr-ft-°F

<u>Type</u>	<u>Sample No.</u>	<u>Direction*</u>	<u>Temperature</u>	
			<u>80°F</u>	<u>200°F</u>
$\text{Al}_2\text{O}_3 + \text{SiO}_2$ fibers	25	A	0.34	0.35
	26	B	<u>0.43</u>	<u>0.45</u>
		Avg	0.38	0.40
$\text{ZrO}_2 + \text{SiO}_2$ fibers	27	A	0.26	0.27
	28	B	<u>0.27</u>	<u>0.29</u>
		Avg	0.26	0.28
$\text{ZrO}_2 + \text{C}$ fibers	29	A	0.17	0.18
	30	B	<u>0.19</u>	<u>0.20</u>
		Avg	0.18	0.19
$\text{ZrC} + \text{C}$ fibers	31	A	0.37	0.39
	32	B	<u>0.44</u>	<u>0.43</u>
		Avg	0.40	0.41

* Direction A - in pouring direction
 Direction B - 90° to pouring direction

UNCLASSIFIED

UNCLASSIFIED

UTC 2180-FR

TABLE XIII

(U) THERMAL CONDUCTIVITY OF FOAM CERAMIC MATERIALS
FOR CHARRED COMPOSITES

Btu/hr-ft-°F

<u>Type</u>	<u>Sample No.</u>	<u>Direction*</u>	<u>Temperature</u>	
			<u>80°F</u>	<u>200°F</u>
$\text{Al}_2\text{O}_3 + \text{SiO}_2$ fibers	45	A	0.41	0.43
	46	B	<u>0.45</u>	<u>0.48</u>
		Avg	0.43	0.46
$\text{ZrO}_2 + \text{SiO}_2$ fibers	47	A	0.26	0.25
	48	B	<u>0.26</u>	<u>0.27</u>
		Avg	0.26	0.26
$\text{ZrO}_2 + \text{C}$ fibers	49	A	0.22	0.23
	50	B	<u>0.29</u>	<u>0.28</u>
		Avg	0.26	0.26
$\text{ZrC} + \text{C}$ fibers	51	A	0.29	0.30
	52	B	<u>0.35</u>	<u>0.37</u>
		Avg	0.32	0.34

* Direction A — in pouring direction
Direction B — 90° to pouring direction

UNCLASSIFIED

UNCLASSIFIED

TABLE XIV

(U) TENSILE STRENGTH OF FOAM CERAMIC MATERIALS
FOR CURED IMPREGNATED FOAM COMPOSITES

"Dog Bone" - 1/2 in. x 1/2 in. x 4 in. Specimen

<u>Type</u>	<u>Sample No.</u>	<u>Direction*</u>	<u>Tensile Strength lb/in.²</u>
Al ₂ O ₃ + SiO ₂ fibers	65	A	96 [†]
	66	A	688
	67	A	512
			Avg 600
	68	B	440
	69	B	472
	70	B	443
			Avg 453
	71	A	712
	72	A	448
ZrO ₂ + SiO ₂ fibers	73	A	568
			Avg 576
	74	B	1,120
	75	B	1,224
	76	B	1,112
			Avg 1,152

* Direction A - in pouring direction
 Direction B - 90° to pouring direction

† Specimen alignment

UNCLASSIFIED

UNCLASSIFIED

UTC 2180-FR

TABLE XIV

(U) TENSILE STRENGTH OF FOAM CERAMIC MATERIALS
FOR CURED IMPREGNATED FOAM COMPOSITES (Continued)

"Dog Bone" - 1/2 in. x 1/2 in. x 4 in. Specimen

<u>Type</u>	<u>Sample No.</u>	<u>Direction*</u>	<u>Tensile Strength</u> <u>lb/in.²</u>
ZrO ₂ + C fibers	77	A	17 [†]
	78	A	208
			Avg 208
	80	B	880
	81	B	1,248
	82	B	1,350
			Avg 1,159
ZrC + C fibers	83	A	481 [‡]
	84	A	0
	85	A	444
			Avg 462
	86	B	500
	87	B	808
	88	B	967
			Avg 792

* Direction A - in pouring direction
Direction B - 90° to pouring direction

† Specimen cracked prior to testing

‡ Broke in grips (alignment questionable)

UNCLASSIFIED

UNCLASSIFIED

TABLE XV

(U) COMPRESSION STRENGTH OF FOAM CERAMIC MATERIALS
FOR CURED IMPREGNATED FOAM COMPOSITES

1 in. x 1 in. x 2 in. Specimen Size

<u>Type</u>	<u>Sample No.</u>	<u>Direction*</u>	<u>Compression Strength lb/in.²</u>
Al ₂ O ₃ + SiO ₂ fibers	90	A	16,500
	91	A	13,450
	92	A	16,500
			<u>Avg 15,483</u>
	93	B	15,625
	94	B	15,350
	95	B	2,600 [†]
			<u>Avg 15,487</u>
ZrO ₂ + SiO ₂ fibers	96	A	9,350
	97	A	8,050
	98	A	7,700
			<u>Avg 8,367</u>
	99	B	9,450
	100	B	10,500
	101	B	7,850
			<u>Avg 9,267</u>

* Direction A — in pouring direction
 Direction B — 90° to pouring direction

† Poor Specimen

UNCLASSIFIED

UNCLASSIFIED

UTC 2180-FR

TABLE XV

(U) COMPRESSION STRENGTH OF FOAM CERAMIC MATERIALS
FOR CURED IMPREGNATED FOAM COMPOSITES (Continued)

1 in. x 1 in. x 2 in. Specimen Size

Type	Sample No.	Direction*	Compression Strength lb/in. ²
ZrO ₂ + C fibers	102	A	12,400 [†]
	103	A	7,650 [†]
	104	A	5,500 [†]
			Avg 8,517
	105	B	14,850
	106	B	15,950
	107	B	13,000
			Avg 14,600
	108	A	15,000
	109	A	15,150
ZrC + C fibers	110	A	16,150
			Avg 15,433
	111	B	15,550
	112	B	11,800
	113	B	11,600
			Avg 12,983

* Direction A — in pouring direction
Direction B — 90° to pouring direction

† Scatter reflecting fiber alignment

79
UNCLASSIFIED

UNCLASSIFIED

TABLE XVI
(U) DENSITY OF FOAM CERAMIC MATERIALS

Material		Virgin Foam		Impregnated Foam		Char	
Foam + Base	Fiber Reinforcement	Sample No.	Density g/cc	Sample No.	Density g/cc	Sample No.	Density g/cc
Al ₂ O ₃	Silica	1	0.616	4	1.579	7	1.179
		2	0.619	5	1.493	8	1.259
		3	0.593	6	1.580	9	1.407
		Avg	0.609		Avg		Avg
ZrO ₂	Silica	10	0.770	13	1.901	16	1.018*
		11	0.778	14	1.907	17	0.985*
		12	0.795	15	1.857	18	0.994*
		Avg	0.781		Avg		Avg
ZrO ₂	Carbon	19	1.049	22	2.341	25	1.481
		20	1.099	23	2.317	26	1.453
		21	1.088	24	2.052	27	1.410
		Avg	1.079		Avg		Avg
ZrC	Carbon	28	1.003	31	2.041	34	1.396
		29	1.036	32	1.936	35	1.270
		30	1.056	33	1.936	36	1.304
		Avg	1.032		Avg		Avg

* Char appeared to bloat

UNCLASSIFIED⁸⁰

UNCLASSIFIED

Security Classification

DOCUMENT CONTROL DATA - R & D

(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)

1. ORIGINATING ACTIVITY (Corporate author) United Technology Center Division of United Aircraft Corporation P. O. Box 358 Sunnyvale, California		2a. REPORT SECURITY CLASSIFICATION CONFIDENTIAL
3. REPORT TITLE FOAM CERAMIC NOZZLE INSERTS		2b. GROUP 4
4. DESCRIPTIVE NOTES (Type of report and inclusive dates) Final Report (Preliminary), 1 December 1965 to 13 February 1967		
5. AUTHOR(S) (First name, middle initial, last name) Greening, Thomas A.		
6. REPORT DATE March 1967	7a. TOTAL NO. OF PAGES 91	7b. NO. OF REFS None
8a. CONTRACT OR GRANT NO. Contract AF 04(611)-11218 b. PROJECT NO. 3058 c. d.	9a. ORIGINATOR'S REPORT NUMBER(S) UTC 2180-FR 9b. OTHER REPORT NO(S) (Any other numbers that may be assigned this report) AFRPL-TR-67-109	
10. DISTRIBUTION STATEMENT In addition to security requirements which must be met, this document is subject to special export controls and each transmittal to foreign governments or foreign nationals may be made only with prior approval of AFRPL (RPPR/STINFO), Edwards, California 93523.		
11. SUPPLEMENTARY NOTES N/A	12. SPONSORING MILITARY ACTIVITY Air Force Rocket Propulsion Laboratory Research and Technology Division Air Force Systems Command, USAF Edwards, California	
13. ABSTRACT (UNCLASSIFIED) (U) Eight nozzle inserts were fabricated from United Technology Center's foam ceramic composites to define the operational limitations of these materials when used in liquid rocket engines employing either N_2O_4/N_2H_4 -UDMH or LF_2/N_2H_4 Blend propellants. The nozzle inserts consisted of the following: A. Condition 1 N_2O_4/N_2H_4 -UDMH $Al_2O_3 + SiO_2$ fibers (two nozzles) $ZrO_2 + SiO_2$ fibers (two nozzles) B. Condition 2 LF_2/N_2H_4 Blend $ZrO_2 + C$ fibers (two nozzles) $ZrC + C$ fibers (two nozzles). (U) Thermal and mechanical property tests were conducted on the ceramic foam composites. The objective was to supply data for an Air Force computer program to predict materials performance in rocket engines, and to afford a basis for evaluating nozzle erosion from the firings in this program. (U) The test firings were conducted by the Air Force Rocket Propulsion Laboratory, Research and Technology Division, Edwards, California. The classified results and posttest analyses are included in this report.		

DD FORM 1 NOV 66 1473

81

UNCLASSIFIED
Security Classification

UNCLASSIFIED

Security Classification

14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
FOAM						
CERAMIC						
NOZZLES						
ABLATIVE						

UNCLASSIFIED

Security Classification

13. Abstract (Continued)

(U) In general, the erosion rates were high under the test conditions used in this program. However, the erosion was uniform causing little, if any, deviation from throat circularity. It was also established that the foam ceramic composite materials were insensitive to thermal shock. The ZrC composition has shown promise as a potential candidate for components in high thermal environments.

END

DATE
FILMED

7 - 67

THIS REPORT HAS BEEN DELIMITED
AND CLEARED FOR PUBLIC RELEASE
UNDER DOD DIRECTIVE 5200.20 AND
NO RESTRICTIONS ARE IMPOSED UPON
ITS USE AND DISCLOSURE.

DISTRIBUTION STATEMENT A

APPROVED FOR PUBLIC RELEASE;
DISTRIBUTION UNLIMITED.