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A STUDY FOR DEVELOPMENT OF AEROTHERMODYNAMIC TEST MODEL MATERIALS AND FABRICATION TECHNIQUE

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Prepared by
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16. Abstract

The work documented encompasses a literature survey, materials reformulation and tailoring, fabrication problems, and materials selection and evaluation for fabricating models to be used with the phase-change technique for obtaining quantitative aerodynamic heat transfer data. The study resulted in the selection of two best materials, Stycast 2762 FT, and an alumina ceramic. Characteristics of these materials and detailed fabrication methods are presented.

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FOREWORD

Results are presented of work performed by Lockheed's Huntsville Research & Engineering Center for NASA's Langley Research Center. This constitutes the Final Report for Contract NAS1-9983, a study for "Development of Aerothermodynamic Test Model Materials and Fabrication Technique."

Wind tunnel tests were performed in the Mach 8 Variable Density Facility at the Langley Research Center, Hampton, Virginia. Thermal properties measurements were subcontracted to Dynatech Corp., of Cambridge, Mass. Thermal conductivity measurements were performed by Materials Consultants, Inc., of Denver, Colorado. All other work reported here was performed at the Lockheed-Huntsville facility.

Acknowledgement is given to Messrs. R.A. Wyman and H.W. Childers for their contributions to the work.

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NOMENCLATURE

Symbols

$A\ell_2O_3$	alumina
C_aO	calcia
C_p	specific heat
h	heat transfer coefficient
K	thermal conductivity
ℓ	depth
P_o	total pressure
S_iO_2	silica
T	temperature
T_o	total temperature
t	time
ρ	density

Greek

α	thermal diffusivity, $K/\rho C$

Subscripts

AW	adabatic wall conditions
i	initial
pc	melting
o	total conditions
s	stagnation point
w	wall conditions
∞	free stream

A STUDY FOR DEVELOPMENT OF AEROTHERMODYNAMIC TEST MODEL MATERIALS AND FABRICATION TECHNIQUE

FINAL REPORT

By W.G. Dean and L.E. Connor
Lockheed Missiles & Space Company, Inc.

Section 1

INTRODUCTION AND SUMMARY

This report summarizes work performed and results obtained under Contract NAS1-9983, "A Study for Development of Aerothermodynamic Test Model Materials and Fabrication Technique." The work documented encompasses a literature survey, materials reformulation and tailoring, fabrication problems, and materials selection and evaluation.

The study resulted in the selection of two best materials for model fabrication. These materials were Stycast 2762FT, and an alumina ceramic. Appendix A presents a comparison of the characteristics of these materials as well as fabrication procedures.

The contract objective was to locate a range of commercially available materials that could be used without modification, or that could be modified for use in fabricating aerothermodynamic test models. Rules established for selecting the materials were based on the use of the materials for heat transfer testing by the phase-change data acquisition technique. Physical and thermal properties of Stycast 2762 were used as standards for comparison.

During the course of this contract, numerous materials were investigated. From these, four materials were determined to be acceptable within certain limitations and are recommended for use with the phase-change data acquisition technique. These materials are RTV-60 silicone rubber, Novimide 700, Stycast 2762FT, and alumina ceramic. Each material offers its own unique advantages as compared to each of the others. Since the RTV-60 rubber has been and is currently being used by various companies for fabricating test models, Lockheed's investigations were centered around the other three materials. The alumina ceramic is the best of the three, meeting or exceeding all contract requirements.

Section 2
TECHNICAL DISCUSSION

2.1 BACKGROUND AND OBJECTIVES

Since the advent of NASA space shuttle activities thermodynamicists have necessarily relied more and more on experimental testing techniques to provide quantitative and qualitative heat transfer data. The engineers are finding that state-of-the-art analytical techniques developed for simple shapes cannot be directly applied to complex space shuttle air frame designs. Data for a rapid gross evaluation of the space shuttle concepts are now being provided by experimental testing.

Detail design studies of thermal protection systems are now dictating future requirements for more qualitative experimental heat transfer data, particularly in vehicle regions of very high heating rates. These requirements are placing high demands on techniques for acquiring experimental heat transfer data. Therefore, efforts are continually being made to expand and improve such techniques.

The classical aerothermodynamic testing technique for obtaining heat transfer data for reentry vehicles is to run wind tunnel tests of thin-skin stainless steel models having thermocouples placed on the model in the regions of interest. Data are recorded electronically and reduced to temperatures and heat transfer coefficients. Personnel at Langley Research Center, however, have developed a relatively simple and very useful technique for obtaining experimental aerodynamic heating data. This technique, as explained in Ref. 1, consists of using a model made from a low-conductivity plastic, covered with a phase-change coating or paint. This coating is a surface temperature indicator which is known commercially as Tempilaq. It consists of wax crystals with a calibrated melting point suspended in an inert, volatile, nonflammable fluid.

When the model is aerodynamically heated in a wind tunnel, the phase-change coating melts when the model surface reaches the melt temperature. Knowing this melt temperature, the model material properties and time required to melt, the heat transfer coefficient can be determined for all body locations at which melting occurs by using a transient one-dimensional heat conduction equation.

$$\frac{\partial T}{\partial t} = \frac{K}{\rho C} \frac{\partial^2 T}{\partial \ell^2} \tag{1}$$

To solve this equation a set of boundary conditions which most nearly describe the actual tunnel test were used. In this case the boundary conditions are:

$$\begin{aligned} T(\ell, 0) &= T_i \\ T(\infty, t) &= T_i \\ \frac{\partial T(0,t)}{\partial \ell} &= \frac{h}{K}\left[T_{AW} - T(0,t)\right]. \end{aligned} \tag{2}$$

It was assumed that the coating temperature and model surface temperature were initially identical. The solution of the above equation yields the test time required for the surface temperature to reach T_{pc}, the coating melt temperature. Other assumptions are:

1. Depth of heat penetration into the model is small compared with the model thickness so that the model acts like a semi-infinite slab.
2. The model is initially isothermal.
3. The surface is subjected to an instantaneous step in aerodynamic heating at time zero and the heat transfer coefficient does not vary with time.
4. The product of model thermal conductivity, density, and specific heat does not vary with temperature.

The solution of the transient one-dimensional heat conduction equation with stated boundary conditions is:

$$T = \frac{T_{pc} - T_i}{T_{AW} - T_i} = 1 - e^{\beta^2} \operatorname{erfc}\beta \tag{3}$$

where

$$\beta = \frac{h\sqrt{t}}{\sqrt{\rho CK}} \quad . \tag{4}$$

It can be seen from Eqs. (3) and (4) that for a given set of tunnel conditions, the time for melting to occur is coupled with the materials' ρCK product. With a high ρCK product, it will take longer times to reach melt temperature and vice versa. Therefore, the magnitude of heat transfer coefficients desired to measure within allowable test times must be established so that the type of material to be used for model fabrication can be determined.

Data obtained with the phase-change technique are recorded on movie film. Data are reduced by hand tracing the melt contours over the body from the projected movie film.

The phase-change coating technique is a simple and accurate method for obtaining heat transfer data. Also, it offers several advantages over the standard thermocouple technique. Larger quantities of data can be obtained, and data can be recorded in model regions of interest where the thermocouples cannot be located. With respect to test time and support equipment required, the phase-change coating technique offers a more economical means of obtaining quantitative heat transfer data. Of course, this technique has limitations which are mainly centered around the thermal and physical properties of the material used for model fabrication. The low conductivity plastic normally used to cast these heat transfer test models is known commerically as Stycast 2762, manufactured and distributed by Emerson & Cuming, Inc., of Canton, Massachusetts. According to Ref. 2, Stycast 2762 is an expoxide casting resin with a usable temperature range of -90°F to $+500^{\circ}$F. For short periods it is usable at 600°F. Continued and prolonged exposure at temperatures $\overset{=}{>} 600^{\circ}$F will result in material charring and degradation. Most hypersonic wind tunnels, which test with high pressure flow, have a total temperature on the order of 900°F to 1400°F. Model shapes tested in hypersonic tunnels at these conditions experience very high localized heating rates, resulting in local skin temperatures approaching the flow total temperature. Therefore, the skin temperatures repeatedly become higher than the material's maximum service temperature, resulting in material breakdown. Charring and degradation of a test model, particularly in stagnation regions (nose tip, wind leading edges), tend to trip the local flow and produce local flow fields that are not representative of the true condition. Therefore, to increase the versatility of the phase change technique, effort was initiated with the specific objective of finding a high temperature material with 1000°F capability. The contract objectives are briefly explained below.

Efforts were begun with a literature survey to locate and identify commercially available nonmetallic materials that have the desired thermal and mechanical properties necessary for manufacturing aerothermodynamic wind tunnel test models. Materials which were to be considered are those which:

1. Are castable and/or machinable into complex shapes,
2. Are nonmetallic and have a low thermal conductivity,
3. Have high temperature resistance on the order of 600°F to 1000°F,
4. Have a ρCK product different from Stycast by no less than, nor greater than, a factor of two,
5. Are homogeneous and can be made with a uniformity in thermal properties,
6. Have smooth nonporous surface finishes,
7. Have structural capabilities at least as good as Stycast, and
8. Are economically feasible to fabricate.

Materials selected were to be based on these guidelines, and tradeoff effects among them were to be considered. Materials selected with the potential of thermal properties tailoring were then to be analyzed, reformulated, and tested. Resulting products were then to be used for fabricating models to be tested in the Langley Research Center Mach 8 Variable Density Facility. A materials' performance evaluation was to be undertaken and all efforts documented.

The following sections discuss all efforts that were performed to meet contract objectives, including conclusions and recommendations.

2.2 PHASE I: MATERIALS INVESTIGATION AND SELECTION OF CANDIDATES

A comprehensive literature survey was performed to locate available materials that can withstand high temperatures (up to 1000°F) and could be cast into complex shapes. Guidelines discussed in Section 2.1 were used to select candidate materials.

Lockheed-Huntsville personnel have direct access to the Redstone Scientific Information Center (RSIC) located on Redstone Arsenal. The RSIC maintains a library of up-to-date technical information through acquisition of Government and contractor reports, books, technical publications and reviews, national and international scientific conferences, etc. In addition, Lockheed-Huntsville has its own well equipped technical library as well as access to Lockheed-Sunnyvale's technical library. All provided useful information during the literature survey. The more productive efforts resulted through personal contact with commerical materials vendors. Lockheed-Huntsville personnel have compiled a wealth of information in the form of sales brochures and technical bulletins distributed by vendors over the past decade. Lockheed-Huntsville is on their current mailing lists for bulletins, etc., as new products, materials, and laboratory apparatus become available. During this survey, approximately 50 vendors were contacted and our materials requirements made known. In almost all cases, the vendors willingly discussed our wants in detail and are continually recontacting and informing us of new or promising materials. As a result of these efforts, over eighty different materials were researched for our particular application.

As the survey continued, it become apparent that the materials could be divided into two basic categories having unique characteristics. These materials were divided into (1) plastics, and (2) ceramics, and are discussed separately.

Results of the literature survey are summarized in Table 1. Data that were obtained on each material are listed as shown. The properties and fabrication procedures for each material were studied so that candidate materials could be selected that could possibly fill the requirements outlined in Section 2.1. Many materials were rejected on temperature capability alone, others because of expensive fabrication procedures. After all due considerations, the following materials were selected for further analysis.

<u>Plastics</u>

1. Stycast 2762 FT
2. Phenolic 1-33-1
3. Amide Polyimide AI-1137
4. Novimide 700

<u>Ceramics</u>

5. P1W Slurry
6. Grade A Lava
7. Porcelains
8. Silfrax-Fused Silica.

These materials were selected because they offered the best possible temperature capabilities, as well as desirable thermal and physical properties. They all could be cast or molded except for items 2, 3, and 6. The Phenolic and Amide Polyimide are laminating resins used with fiber glass layups. If these resins could be cured in a solid mass instead of in extremely thin layers, they could possibly be used. Grade A Lava can only be machined.

At the end of Phase I, a trip was made to Langley Research Center. Results and recommendations were discussed with the Contracting Officer's Representative (COR) and a decision was reached to study further the eight materials selected in Phase II. Additional materials were also to be considered if their properties were promising. A discussion of the work is presented in Section 2.3.

Table 1

LITERATURE SURVEY-HIGH TEMP AERO-THERMODYNAMIC MATERIALS

Material Designation	Cost ($/lb)	Manufacturer	Type Material	Maximum Service Temp. °F	Coeff. of Thermal Expansion (in/in/°F)	Tempilaq Absorption	Thermal Properties $\rho\ \frac{lb}{ft^3}$	 $C_p\ \frac{Btu}{lb\text{-}^{\circ}F}$	 $K\ \frac{Btu}{ft\text{-}hr\text{-}^{\circ}R}$	 $\rho C_p K\ \frac{Btu^2}{hr\text{-}ft^4\text{-}^{\circ}R^2}$	Structural Properties Tensile Strength (psi)	 Flexural Strength (psi)	 Compressive Strength (psi)
Stycast 2762 FT	–	Emerson & Cumings Co.	Epoxy	≈600	21×10^{-6}	No	130	~.20	.775	20.15	–	18,500	–
Supermica 620	–	Mycalex Corp.	Mica bonded with in-organic crystal-line	1200	–	–	235	–	.25	–	–	12,000	–
Silfrax	4.00	Carborundum Co.	Fused silica	3000	$.3 \times 10^{-6}$	Variable	115	–	–	–	–	1,800	6000
Glasrock S-820	.50	Glasrock Corp.	Fused silica	3000	$.45 \times 10^{-6}$	Variable	115	–	–	–	–	1,200	4-6000
P1W Slurry	–	Nalco Corp.	Fused silica	3000	$.4 \times 10^{-6}$	Some	–	–	–	–	–		
QF-180 Cement	–	Carborundum Co.	Cera-mic	–	–	–	–	–	–	–	–	–	–
QF-100 Cement	–	Carborundum Co.	Cera-mic	–	–	–	–	–	–	–	–	–	–
40-C Lavasil	–	Saxonburg Ceramics	Fused silica	3000	–	–	115	–	.5	–	–	700	–
Zircocast 60-D	–	Zirconia Corp. of America	Zirco-nia	4000	–	–	270	–	.5	–	–	1,500	–
Boron Nitride	–	–	–	–	–	–	–	–	16.	–	–	–	–
Silican Carbide	–	–	–	–	–	–	–	–	10-20	–	–	–	–
Silican Nitride	–	Remington Arms	–	–	–	–		–	.90	–	–	–	–
Silica Glasses (Quartz, Vitreous Vycor)	–	–	–	–	–	–	–	–	–	–	–	–	–
Borosilicate Glass	–	Pyrex Corp.	Pyrex	–	–	–	140	.30	.95	39.9	–	–	–
Lead Silicate Glass	–	–	–	–	–	–	190	.15	8.0	228.0	–	–	–
Grade A Lava	–	American Lava	Alum. Silicate	2000	–	No	142	–	.75	–	–	9,000	–
Sauereisen	–	Sauereisen Cements Co.		–	–	–	–	–	–	–	–	–	–
No. 33	–	Sauereisen Cements Co.	Silica Base	2000	–	–	165	–	2.5-3.0	–	–	–	–

Table 1 – (Continued)

Material Designation	Cost ($/lb)	Manufacturer	Type Material	Maximum Service Temp. °F	Coeff. of Thermal Expansion (in/in/°F)	Tempilaq Absorption	Thermal Properties				Structural Properties		
							$\rho \frac{lb}{ft^3}$	$C_p \frac{Btu}{lb\text{-}^\circ F}$	$K \frac{Btu}{ft\text{-}hr\text{-}^\circ R}$	$\rho C_p K \frac{Btu^2}{hr\text{-}ft^4\text{-}^\circ R^2}$	Tensile Strength (psi)	Flexural Strength (psi)	Compressive Strength (psi)
No. 29	–	Sauereisen Cements Co.	Zirconia Base	2000	–	...	230	–	3.6	–	–	–	–
No. DW-30	–	Sauereisen Cements Co.	Magnesite base	1000	–	–	200	–	2.0-3.0	–	–	–	–
No. 54	–	Sauereisen Cements Co.	–	2000	7×10^{-6}	–	145	–	–	–	–	–	–
Slip Case Porcelain	1.00	Bell Ceramics	Ceramic	2200	–	No	–	–	0.5-2.0	–	–	–	–
Dental Porcelain	100.0	Myerson Tooth Corp.	Ceramic	2000	–	No	–	–	0.5-2.0	–	–	Very Str.	–
Porox Porcelain	–	Ferro Corp.	Ceramic	1500	–	No	150	.18	0.75	20.25	4700	–	100,000
Arlcite Porcelain	–	Ferro Corp.	Ceramic	2400	–	No	206	.19	1.00	39.15	18,000	–	200,000
Std. Elec. Porcelain	–	Stupakoff Co.	Ceramic	2460	2.7×10^{-6}	No	147	–	.90	–	6,800	–	53,000
Commer. White Porcelain	–	Star Porcelain Co.	Ceramic	2900	3.2×10^{-6}	No	157	–	1.07	–	2,440	5,360	49,100
Nu-Blac Porcelain	–	Star Porcelain Co.	Ceramic	2880	2.9×10^{-6}	No	159	–	1.08	–	2,630	5,730	45,600
921 Insul. Porcelain	–	Star Porcelain Co.	Ceramic	2900	3.9×10^{-6}	No	179	–	1.29	–	2,910	6,310	56,600
Thermolain Porcelain	–	Star Porcelain Co.	Ceramic	2570	1.6×10^{-6}	Some	155	–	1.10	–	1,190	3,390	17,100
Vitrolain Porcelain	–	Star Porcelain Co.	Ceramic	2800	3.2×10^{-6}	No	151	–	1.04	–	3,330	6,760	67,300
Lavolain Porcelain	–	Star Porcelain Co.	Ceramic	2450	4.4×10^{-6}	No	173	–	1.14	–	3,670	7,370	54,100
Injecta Stone	1.00	Prevest Dental Matl.	Ceramic	>1000	–	Yes	–	–	–	–	–	–	3,000
P. V. Stone	1.00	Prevest Dental Matl.	Ceramic	>1000	–	Yes	–	–	–	–	–	–	3,800
Pyroceram	–	Dow Corning	Corning Ware	–	–	No	–	–	1.0	–	–	–	–
Mullite	–	Coors Porcelain Co.	$3Al_2O_3$ 25; O_2	3200	Very Small	No	186	–	1.4	–	18,000	500	–
Steatite	–	Superior Steatite Co. DU CO Ceramics Co.	$MgSiO_2$	1800	–	Slightly	161	–	1.4-1.9	–	–	11 - 20,000	–
Forsterite	–	American Lava	$2MgSiO_2$	1800	–	–	183	–	1.9-2.4	–	–	10,000	–

Table 1 – (Continued)

Material Designation	Cost ($/lb)	Manufacturer	Type Material	Maximum Service Temp. °F	Coeff. of Thermal Expansion (in/in/°F)	Tempilaq Absorption	Thermal Properties				Structural Properties		
							$\rho \frac{lb}{ft^3}$	$C_p \frac{Btu}{lb-^oF}$	$K \frac{Btu}{ft-hr-^oR}$	$\rho C_p K \frac{Btu^2}{hr-ft^4-^oR^2}$	Tensile Strength (psi)	Flexural Strength (psi)	Compressive Strength (psi)
Mycalex 2803	–	General Electric	Glass fused Mica	≈700	–	–	212	.13	0.3	8.26	–	14,000	–
Mycalex 2803	–	General Electric	Glass fused Mica	≈700	–	–	161	.24	0.21	8.12	–	14,000	–
Supermica 560	–	Mycalex Corp.	Mica bonded with inorganic crystalline	930	–	–	167	–	0.25	–	–	12,000	–
White Mica Brick	–	–	–	–	–	–	43	–	0.1	–	–	–	–
Synthetic Mica	–	–	–	–	–	–	–	–	1.4	–	–	–	–
Fiberfrax	–	Carborundum Co.	–	–	–	–	–	–	–	–	–	–	–
High Temperature Bonding Motar	–	Barcock & Wilcox Co.	–	–	–	–	–	–	–	–	–	–	–
1-33-1	4.00	American Reinforced Plastics Co.	Modified Phenolyic	700	–	No	120	≈.23	≈.13	3.59	–	12-14,000	–
Skybond 709	4.20	MonSanto Chem. Co.	Polyimide	700	–	No	70	–	.17-.23	–	–	–	–
AI-1137	–	Amoco Chem. Co.	Amide Polyimide	600-700	–	No	70	–	–	–	–	4,000	–
Proseal 700	4.50	Teledyne Coast Proseal	Synthetic Asbestos Rubber	500	–	No	70	–	–	–	–	–	–
Novimide 700	3.15	Isochem Resins Co.	Imide Novolac	700	3.5×10^{-5}	No	94	–	0.125	–	10,300	–	18,000
Novimide Catylst	4.90	Isochem Resins Co.	Imide Novolac	–	–	–	–	–	–	–	–	–	–

Table I – (Concluded)

Material Designation	Cost ($/lb)	Manufacturer	Type Material	Maximum Service Temp. ^{o}F	Coeff. of Thermal Expansion (in/in/^{o}F)	Tempilaq Absorption	Thermal Properties				Structural Properties		
							$\rho \frac{lb}{ft^3}$	$C_p \frac{Btu}{lb\text{-}^oF}$	$K \frac{Btu}{ft\text{-}hr\text{-}^oR}$	$\rho C_p K \frac{Btu^2}{hr\text{-}ft^4\text{-}^oR^2}$	Tensile Strength (psi)	Flexural Strength (psi)	Compressive Strength (psi)
PH 990 (Filler)	40.00	El Monte Chemical Co.	Phosphoiteilic Chloride	750	–	–	–	–	–	–	–	–	–
HAVEG 41F Cement	–	HAVEG Corp.	Digested Asbestos Phenol-phenolic	–	–	–	–	–	–	–	–	–	–
7501	14.00	Dow Corning Co.	2 part system	600	–	–	70	–	–	–	–	–	–
7522	14.00	Dow Corning Co.	Resins	600	–	–	70	–	–	–	–	–	–
RTV-60	–	General Electric	Silicone Elastomer	600	11.4×10^{-5}	No	92	–	0.18	–	–	800	–
HX901 Foam	–	Hexal Corp.	Polyimide Foam	700	–	–	–	–	–	–	–	–	–
Ceramacast 510	–	Aremco Products Inc.	Alumina Oxide Ceramic	3200	–	Yes	310	–	2.08	–	–	1,500	–
Lithafrax 2121	–	Carborundum Co.	Lithium Alum.	2300	2×10^{-6}	–	50	–	–	–	–	–	–
Lithafrax 2122	–	Carborundum Co.	Silicates	2300	2×10^{-6}	–	50	–	–	–	–	–	–
Alsimag 202	–	American Lava	Corderite 2MgO $2Al_2O_3$ $5SiO_2$	–	–	–	–	–	–	–	–	–	–
Teflon	–	DuPont	Thermo Plastic	500-600	5.5×10^{-5}	No	137	0.25	0.14	4.79	2000	–	600

2.3 PHASE II: MATERIALS MODIFICATION, FORMULATION, AND DEVELOPMENT, AND MODEL FABRICATION

The materials selected from Phase I efforts offered either the temperature capability or the possibility of thermal property tailoring so as to achieve the desired material. The materials were divided into: (1) plastics and (2) ceramics with each group offering its own unique qualities and fabrication procedures.

2.3.1 Plastics

Stycast 2762 FT is essentially the same as Stycast 2762 but it is a more highly filled casting compound which features high strength and slightly higher thermal conductivity. Their typical properties as quoted from Refs. 2 and 3 are presented for comparison:

Stycast 2762

Specific gravity	2.1
Flexural Strength, psi at 70°F	10,800
Flexural Modulus, psi at 70°F	1.2×10^6
Coefficient of Thermal Expansion per °C	1.5×10^{-6}
Machinability - must be ground	
Thermal Conductivity Btu/ft^2/°F/hr/in	4.0

Stycast 2762 FT

Specific gravity	2.1
Flexural strength, psi at 70°F	18,500
Flexural Modulus, psi at 70°F	1.2×10^6
Coefficient of thermal expansion per °F	21×10^{-6}
Machinability - must be ground	
Thermal conductivity, Btu/ft^2/°F/hr/in	9.3

The filler in both Stycasts are the same, but 2762 FT filler is a much more finely ground and concentrated filler. This offers several advantages that are somewhat apparent from the preceding data. The coefficient of thermal expansion is reduced, thereby rendering a reduction in thermal cycling stresses. Also the volume cure shrinkage of a epoxy resin system is minimized by adding as much filler as possible. The highly concentrated filler also helps eliminate filler settling problems associated with long cure times. Stycast 2762 FT can be mixed and cured out as a more homogeneous material resulting in more constant materials properties throughout the casting. A detailed explanation of these effects is discussed in Ref. 4.

Lockheed-Huntsville has been successfully casting Stycast 2762 FT into complex test model shapes for the past two years. The models have been tested with the phase change data acquisition technique. Data acquired with these sets of models were analyzed and shown to give good repeatable results. The models have repeatedly withstood high temperature environments without degrading seriously. Based on comments from experienced test engineers and our own experiences, Stycast 2762 FT was found to be very good material, better than those previously used. However, it was desired to obtain other materials that offered a range of thermal properties and temperature capabilities superseding Stycast 2762 FT.

Phase I resulted in four plastics being considered for Phase II efforts. Stycast 2762 FT has its optimum properties, therefore it was decided that no reformulations or adding of fillers would be attempted. The Phenolic 1-33-1, Amide Polyimide AI-1137, and the Novimide 700 comprised the center of efforts for plastics in Phase II.

Phenolic 1-33-1, manufactured by American Reinforced Plastics Company of Los Angeles, California, and Amide Polyimide AI-1137, manufactured by Amoco Chemicals Corporation of Chicago, Illinois, are both high temperature resin systems. They are laminating varnishes that are used as "prepregs" that can be tailored to vacuum-bag, tension-wrap and press-mold.

Both systems contain 15 to 20% by weight of volatiles that outgas during cure cycles. These resin systems are cured in extremely thin layers to allow for the outgassing in order to achieve a solid homogeneous material.

To assess the feasibility of casting these resins into thick sections, attempts were made to find a proper cure cycle that could possible achieve the desired results. Phenolic I-33-1 was poured into cylinderical dishes and cured over the cycle as shown:

Temperature (°F)	Time (hr)
150	24
200	24
225	26
250	40
300	24
350	4
400	3
500	15

Amide Polyimide 1137 was also cured but at the following cycle:

Temperature (°F)	Time (hr)
150	17
180	26
200	38
220	24
240	24
250	24

These cycles were increased with smaller step sizes, hopefully to improve those recommended by the materials' manufacturers. The results were disappointing, as shown in Figs. 1 and 2. The toxic outgassing of the resin volatiles created large cavities or bubbles which rendered the material unusable for purposes of this study. A later attempt was made, using the AI-1137, to cast a model shape by curing single layer buildups over long

Fig. 1 — Phenolyic 1-33-1 (Post Cured)

Fig. 2 — Amide Polyimide 1137 (Post Cured)

periods of time. Again this met with failure, as shown in Fig. 3. Figure 4 also shows the same results when the material was cured in mass over a period of three weeks at 150°F.

Other work was directed toward locating and obtaining various fillers that could be added to the resin systems as well as to the Novimide 700. These fillers, hopefully, would not only affect the materials thermal properties in a desirable manner, but also increase their quoted maximum temperature capabilities. Those selected were:

Wollastonite
Micronized Silica
Lithafrax 2121
Lithafrax 2122
Alsimag 202
Antimony Oxide
Powdered Alumina
Mica
Zirconium Oxide.

These materials were selected because of their temperature capabilities, conductivities, and their compatibility for use as fillers with phenolics, silicone, polyesters, and epoxies.

Attempts were made to load the Amide Polyimide AI-1137 with Lithafrax 2122 and the Micronized Silica in order, hopefully, to reduce the bubbling action during the cure cycle. Unfortunately, the fillers had no effect, but a foamy mixture was left, which had little possibilities.

Efforts were then concentrated on the resin system called Novimide 700 which is manufactured by Isochem Resins Co. Novimide 700 is a two-part thermo-setting plastic consisting of an imide novolac filled with an oxide/trioxide of antimony. This plastic has most of the desirable characteristics outlined in Phase I and is easily castable. The only limitation is its maximum temperature capability. To extend the upper temperature range of Novimide 700, all the previously mentioned fillers were added. Cure cycles suggested by Isochem Company were used to load and cure the end products.

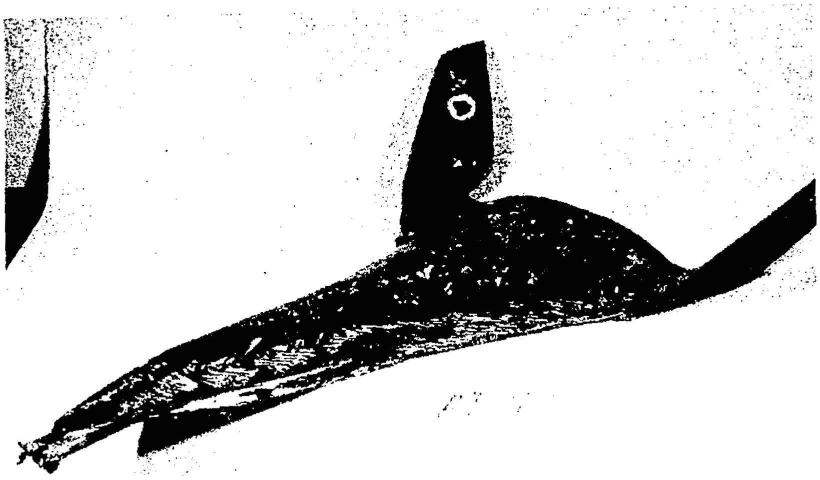


Fig. 3 — Amide Polyimide Post Cured in Thin Layers

Fig. 4 — Amide Polyimide Post Cured in Mass

Several tests were devised and run to compare the temperature capabilities of the various reformulated Novimide 700 mixtures. Lockheed-Huntsville has a Thermolyne Type 1500 Furnace with a 2200°F temperature capability for performing these tests. A DT 306 Integrating Digital Multimeter and a thermocouple junction box are used to monitor temperature response of slab thermocouples. Test samples consisted of cylindrical disks and rectangular slabs. The disks were subjected to a furnace temperature of 1000°F for one-minute; two-minute and three-minute durations and were observed for physical changes. A standard sample of Stycast 2762 FT was used for overall comparisons. Pictures were made of all samples before and after the tests. The rectangular slabs ($2\frac{1}{2} \times 2\frac{1}{2} \times \frac{1}{2}$ in.) had chromel-alumel thermocouples cast within their centers for monitoring internal temperature changes. A brief description of material formulations and tests that were performed are given below.

- Novimide 700 (without fillers) was cast into a wind tunnel model and several cylindrical disks. The models were cured at 300°F for seven hours and turned from their normal light gray to a dark tan color. This color change can possibly be attributed to RTV molds, or by contaminants in the oven. Several disks were subjected to 1030°F for one minute. There were signs of heat deterioration at the edges noted by a browning or slight charring.

- MP51 Micronized Silica was mixed with Novimide 700 to a ratio of 20% by weight. The mixture was then cast into several disks and a thermocouple slab. The disks were subjected to 1000°F with no visible signs of burning or surface change. The thermocouple slab was injected into the furnace at 750°F with the internal temperature monitored. At the end of 15 minutes, the internal temperature was 750°F but the surface was charred, and smoked considerably.

- Alsimag 202 (Corderite, 2 MgO 2 $A\ell_2O_3$) was mixed with Novimide 700 to a ratio of 40% by weight with disks and a thermocouple slab being cast. The disks were subjected to 1000°F for one minute and showed no signs of burning or surface discoloration. The thermocouple slab was placed in the furnace at 1000°F and timed. After three minutes the part ignited with the center thermocouple reading 500°F.

- Alsimag 202 was again added to Novimide 700 but by a 46% weight ratio and cast into disks. After curing, the disks were inserted into a 720°F furnace for 30 minutes. The test parts were still very hard but showed signs of swelling and was smoking slightly when removed. The surface changed from smooth to slightly "orange peel" and was charcoal black.

- Lithafrax 2121 was added to Novimide 700 by a 38% weight ratio and cast into cylindrical disks. After curing, the disks were subjected to 1000°F with no signs of heat deterioration. Other samples were placed in a 720°F furnace for 30 minutes and removed. The part was still very hard but smoked slightly with some swelling. The "orange peel" surface was present with a charcoal black discoloration.

- Lithafrax 2122 was added to Nominide 700 by a 43% weight ratio. Disks were cast and tested with the same results as Lithafrax 2121.

- Wollastonite was mixed with Novimide 700 to a ratio of 30% by weight. Wollastonite is a calcium metasilicate ($CaSiO_3$) mineral with a high melting point of 2800°F. Samples were cast, cured, and tested at one-, two- and three-minute intervals. The samples withstood heating for approximately 2.5 minutes and then ignited.

- Antimony Oxide was mixed with Novimide 700 to a ratio of 45% by weight. Samples were cast, cured and tested at the standard time intervals. A slight browning was noticed after two minutes with charring at the edges after three minutes.

- Mica Powder was mixed with Novimide 700 to a ratio of 11% by weight. Samples were cast, cured and tested at the standard time intervals. A very slight browning at the edges was noted after two minutes but the sample ignited after 2.75 minutes.

- Powdered Alumina was mixed with Novimide 700 to a ratio of 61% by weight. Samples were cast, cured and tested. Again browning at the edges was noted after two minutes with charring after three minutes.

- Zirconium Oxide was mixed with Novimide 700 to a ratio of 66% by weight. Samples were cast, cured and tested. Only a very slight browning was noticed after two minutes with a slight charring at the edges after three minutes.

- Stycast 2762 FT was cast, cured and tested to provide a sample for comparison. Slight browning was noticed after two minutes and charring at the edges after three minutes.

Typical results of the inhouse tests are shown in Fig. 5. The material shown is the Novimide 700 filled with Wallostonite. It was concluded that a more meaningful evaluation could be made by fabricating wind tunnel test models and conducting actual wind tunnel tests on them. This would eliminate the necessity of performing physical and thermal properties tests on all reformulated materials.

Shrinkage samples were made of all Novimide 700 formulations and cured at the same cycles. Measurements were made with results presented in Table 2.

2.3.2 Ceramics

- General

Early in Phase II it was decided to give strong consideration to ceramic materials for this application because of the following advantages:

- The raw materials and tooling are quite inexpensive. A typical porcelain slip sells for \$4 to \$5 per gallon. Molds are made of inexpensive gypsum-type plaster. Simple models could be poured by hand and fired in a simple \$200 kiln up to about 2300°F.
- With this simple tooling, quick turnaround or delivery time is obtainable.
- Some ceramics have relatively high strength as compared to Stycast. Its strength properties can be tailored by the firing process, e.g., higher strength can be obtained by firing to higher temperatures.
- These materials have the required thermal property values. These values are also variable by choice of firing temperature and raw material constituent ratios.
- These materials can be made impervious, i.e., they will not absorb Tempilaq or dye.
- These materials have temperature capabilities in excess of the required value of 1000°F.

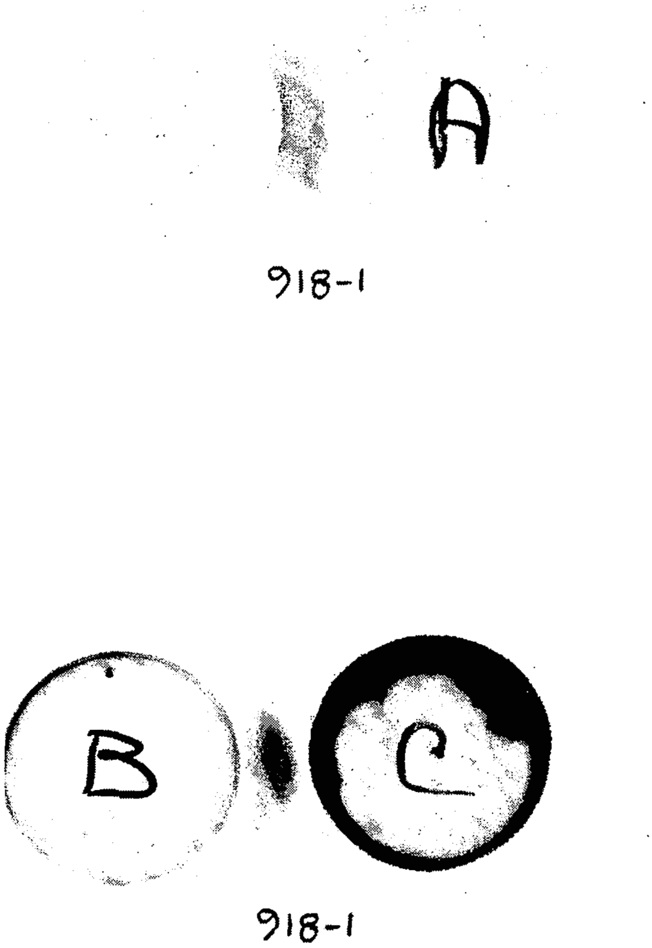


Fig. 5 - Novimide 700 Filled with Wallostonite Subjected to: A (1000°F 1 minute), B (1000°F 2 minutes), C (1000°F 3 minutes). Compared to Unfired Sample.

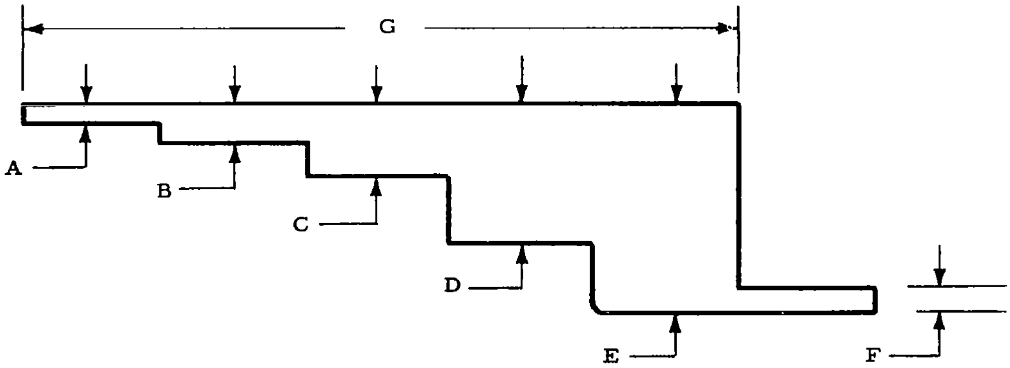


Table 2

SHRINKAGE SAMPLE COMPARISON

Specimen	Mold Mtl	A	B	C	D	E	F	G
Aluminum Master	585 RTV	.125	.275	.525	1.025	1.5235	.126	5.004
715 Mochem 700	RTV No. 2	.117	.257	.499	.960	1.495	.120	5.008
522 Stycast 2651 MM	RTV	.114	.255	.501	1.001	1.506	.114	5.023
320 Isochem 700 Lithafrax 2122 (30.6%)	RTV No. 2	.1205	.2675	.5145	1.0125	1.512	.125	4.975
Stycast 2762 ft 91-1	RTV No. 2	.1215	.2695	.517	1.0138	1.5125	.1252	4.995
Lithafrax 2121 915-2 Novimide 700	RTV	.1192	.2655	.512	1.009	1.515	.122	4.990
Alumina T61 916-2 Novimide 700	RTV	.1175	.2635	.507	1.001	1.504	.121	4.981
TB 916-1 Novimide 700	RTV	.117	.262	.507	1.005	1.505	.118	4.996
MP51 Silica 915-3 Novimide 700	RTV	.117	.263	.510	1.006	1.510	.120	5.000
Alsimag 202 915-1 Novimide 700	RTV	.117	.261	.505	1.0025	1.508	.120	5.002
Wollastonite 918-1 Novimide 700	RTV	.116	.261	.505	1.001	1.510	.119	4.997
Antimony 916-3 Novimide 700	RTV	.1133	.262	.502	.997	1.503	.117	4.997
MICA 917-1 Novimide 700	RTV	.120	.267	.5125	1.008	1.512	.1245	4.979
Stycast 2762 ft 921-1	RTV	.119	.261	.5025	.996	1.497	.1195	4.983

- The ceramic materials under consideration have a low coefficient of thermal expansion, hence a very good resistance to thermal shock. A sample of one porcelain material was heated to 1000°F, taken from the furnace, cooled in air briefly and immersed in cold running water without any failure.

In order to assist the reader who might not be familiar with ceramic materials and their processing, a brief, general discussion is deemed worthwhile at this point. This should help in the understanding of the following discussion of the development of ceramic models.

Table 3 gives a good overview of the breakdown of the many types of ceramic materials or bodies. This figure is from Ref. 5 which is recommended as a good source of general information. Also Ref. 6 is quite up to date and useful. As seen from the table there are many categories of ceramics, each having many subdivisions of composition percentages. The two types of porcelain which were subsequently pursued under this contract are Wollastonite (28) and high alumina (20).

In the manufacture of a finished ceramic material or product three steps are generally involved, which are:

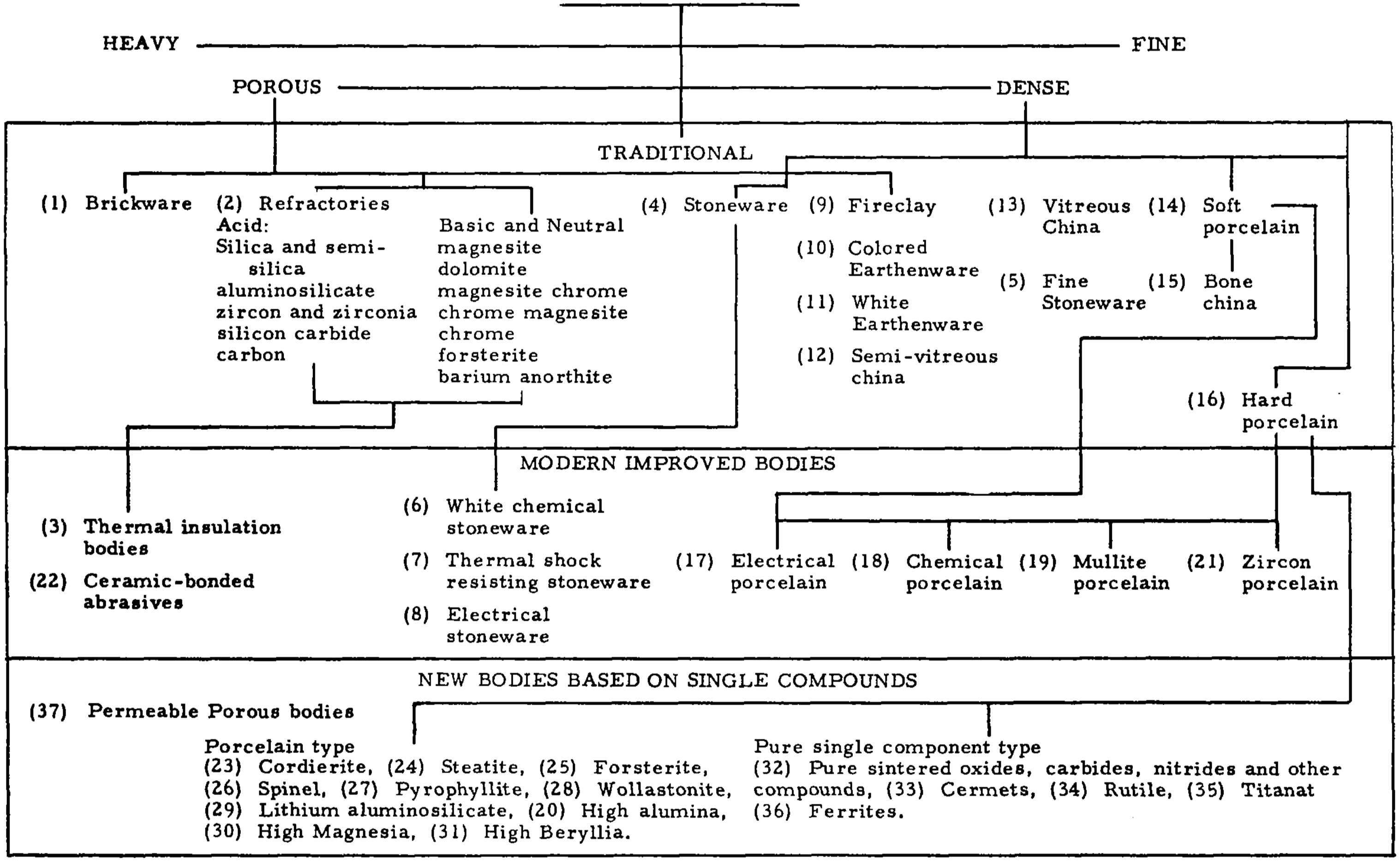
- Preparing and processing raw materials
- Forming raw materials into the desired shape
- Firing.

Each step is now discussed briefly. Reference 6 gives an excellent detailed treatment of each of these.

The preparation of raw materials involves mining from the earth, grinding, separating, screening, refining, processing, etc. This, of course, was not required in this development since any material in the quantities and grade we need can be purchased commercially. A typical example of the procedure would be fused silica. In processing, a high

Table 3*

CERAMIC BODIES



HEAVY — FINE

POROUS — DENSE

TRADITIONAL

(1) Brickware

(2) Refractories

Acid:
- Silica and semi-silica
- aluminosilicate
- zircon and zirconia
- silicon carbide
- carbon

Basic and Neutral
- magnesite
- dolomite
- magnesite chrome
- chrome magnesite
- chrome
- forsterite
- barium anorthite

(4) Stoneware

(9) Fireclay

(10) Colored Earthenware

(11) White Earthenware

(12) Semi-vitreous china

(13) Vitreous China

(5) Fine Stoneware

(14) Soft porcelain

(15) Bone china

(16) Hard porcelain

MODERN IMPROVED BODIES

(3) **Thermal insulation bodies**

(22) **Ceramic-bonded abrasives**

(6) White chemical stoneware

(7) Thermal shock resisting stoneware

(8) Electrical stoneware

(17) Electrical porcelain

(18) Chemical porcelain

(19) Mullite porcelain

(21) Zircon porcelain

NEW BODIES BASED ON SINGLE COMPOUNDS

(37) **Permeable Porous bodies**

Porcelain type
(23) Cordierite, (24) Steatite, (25) Forsterite,
(26) Spinel, (27) Pyrophyllite, (28) Wollastonite,
(29) Lithium aluminosilicate, (20) High alumina,
(30) High Magnesia, (31) High Beryllia.

Pure single component type
(32) Pure sintered oxides, carbides, nitrides and other compounds, (33) Cermets, (34) Rutile, (35) Titanat
(36) Ferrites.

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*From Ref. 5, Overview of Types of Ceramic Bodies silica beach sand is heated to its fusion temperature (about 3300°F) where it becomes a unified mass or "globe." This is cooled, broken, and ground (using a ball mill) to a fine white powder which resembles wheat flour. The fineness, purity, etc., to which the end product is taken depends upon the intended use.

The raw materials can be formed into desired shapes in various ways, such as:

1. Casting
   - a. Slip
   - b. Ram
   - c. Pressure
   - d. Centrifugal
2. Jiggering (like a potter's wheel)
3. Extrusion (limited to uniform cross-section parts)
4. Pressing
   - a. Dry or Semi-Dry — Using a die and 3,000 to 20,000 psi
   - b. Isostatic — (Using a rubber bag around the part, submerging it in a fluid and applied pressure)
5. Vibratory Compaction
6. Injection molding (transferring material from container to mold under high pressure)
7. Hot pressing (using heated molds or dies)
8. Other more exotic methods such as plasma spraying, explosive forming, etc.

Details on most of these are not given since the objective of this contract called for a simple, cheap method which could be transferred to other locations such as Langley. For this reason the slip casting method was chosen, which consists of making a plaster mold into which a slip or fluid-state material is poured. The slip is made by adding a liquid (such as water or alcohol) to the selected ceramic raw-material powder. This mixture is usually mixed overnight in a ball mill, then degassed and poured. The plaster mold absorbs the water from the slip, forming a moist crust on the inside walls of the mold. If a hollow model is needed the mold is emptied after a specified time, leaving a coating inside the mold. The longer the time before emptying, the thicker will be the walls. For a solid model like those made during this contract, the mold must be repeatedly refilled. After an initial drying period the plaster mold is removed from the green material, which is then air-dried for several days. The part can usually be worked, drilled, etc., during this green state after which it is to be fired. Although this process is straightforward in theory, there may be numerous practical problems. For example, skill and artistry are required to make the molds and parts. Since the molds are rigid, their "parting lines" must be accurately located. This can become a challenge on complicated shapes such as the space shuttle. For example, the part line ona fuselage mold must be exactly at the line of maximum diameter, otherwise the part will not slip out of the mold. (This problem is illustrated in making a spherical model. The part-line must be exactly at the equator, otherwise either one or the other of the halves will not be released.) This is quite critical for materials which have poor strength in the green state. The molds also must be quite smooth in order to allow for shrinkage during drying. If the part sticks, stresses and perhaps undetected fractures may occur in the green part. The fractures may or may not mend during firing, depending on a number of factors, including shape of the model, firing temperature, orientation of the fracture, and nature of the material.

After it is formed, the green part is ready to be fired. During the firing process a number of things happen. Initially any remaining water is driven off, then any organics are burned out. Also solid-phase reactions begin to take place. Phase changes may or may not occur, depending upon the materials and firing temperature. Generally speaking, some liquid phase of at least one of the constituents is needed to obtain good strength, and to obtain a vitreous body, a liquid phase is always required. Incidentally, the fact that the body may be impervious does not necessarily mean that it is non-porous. The body may have pores, which are closed and sealed off, preventing the absorption of moisture.

A classical porcelain body generally has three components: (1) a colloidal portion, (2) a flux, and (3) a refractory portion. The colloidal component serves as a binder to hold the part in shape after it has been formed. The amount of colloidal needed depends upon the forming method to be used and particle size distribution desired. A typical binder of this type is colloidal silica. Also, organic binders can be used such as polyvinyl alcohol, starch, wax, and phenolics. The flux constituent generally provides a glassy bond or liquid phase during the firing process which serves to bond the refractory portion particles together in a unified solid body. A typical flux is feldspar. The refractory portion provides the skeleton of the body, giving it strength and structural capability at higher temperatures. A classical refractory is finely ground quartz. The three basic constituents can be varied in percentages to give an almost infinite number of combinations. Selection of the components depends on a number of factors including availability, cost, forming method, properties desired, firing temperature and desired properties of the finished part.

Materials Considered

Initially a large number of ceramics were considered and their capabilities compared to the stated requirements for this application. After a preliminary screening, these were narrowed to a group of materials which can be assembled into the following four categories:

- Fused Silicas
- Modified Commericial Porcelains
- Aluminas
- Other Miscellaneous Materials.

Fused Silica: Fused silica materials have the advantage of being castable and economical. They also have the required thermal properties, excellent thermal shock resistance, and are homogeneous. Disadvantages are that their strength is lower than is desirable, and their surfaces are not impervious unless they are fired to a very high temperature. Because of their advantages, these materials were initially considered as strong candidates for this application. Materials were purchased and fabricated into samples and models. Typical models and specimens are shown in Figs. 6a, b, and c. Since this material is porous, attempts were made to find a glaze or coating to seal its surface. Numerous suppliers were contacted, but no commercially available coating was found. This material is difficult to glaze, because of its extremely low coefficient of thermal expansion.

When no available glaze could be located, the literature was searched and a method of glaze formulation and application was found. After several attempts, samples of fused silica were successfully glazed. Although fused silica meets many of the requirements when it is glazed, this material was later eliminated in favor of alumina materials (discussed later), because of the lower strength of fused silica and the fact that a glazed model violates the requirement for a homogeneous material. Fused silica can be fired to a temperature at which it becomes impervious. This high temperature firing was tried on samples, but proved to be undesirable, because of the large shrinkage during firing. Another possible solution to the surface porosity problem would be to clean the Tempilaq absorbed into the model surface during a test by heating the model in a furnace to say $2000^{\circ}F$ after each test. This course would mean that enough models must be provided for at least two days of testing; which is an undesirable requirement.

In summary, it was decided that the fused silica materials are only marginally acceptable.

Modified Commercial Porcelain Materials: Early in Phase II several commercially available porcelain slips were purchased and used to make

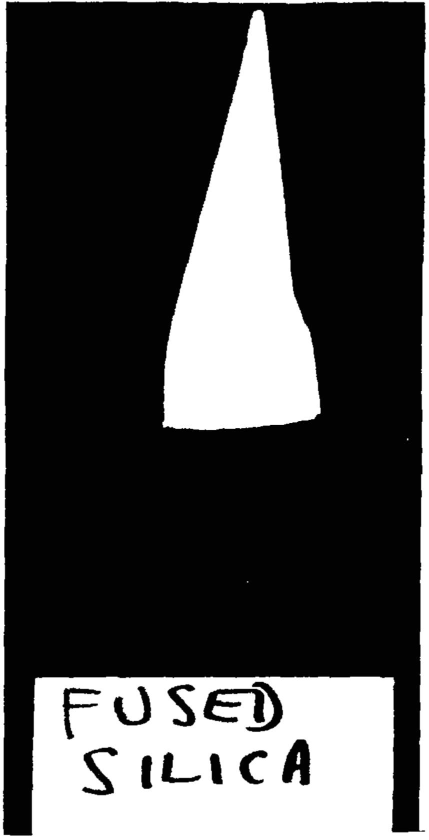

Fig. 6a – Typical Fused Silica Orbiter Model (Unfired)

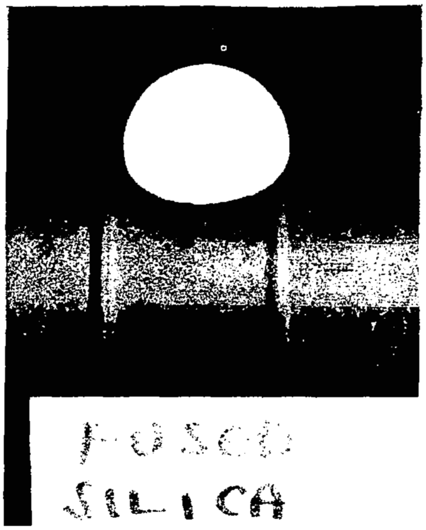

Fig. 6b – Typical Fused Silica Hemisphere Model (Unfired)

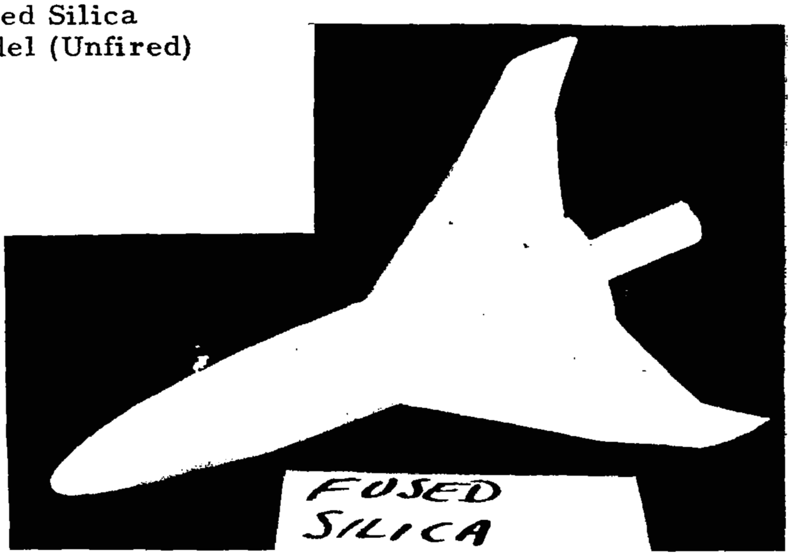

Fig. 6c – Typical Fused Silica Booster Model (After Firing) several samples and five-inch orbiter models. These included three white-type porcelains and one carbon-black type porcelain. Although specific thermal properties of commercial porcelain were not known at the time, these materials showed much potential early in Phase II. They have the advantages of being:

- Relatively straightforward to fabricate (requiring a plaster mold and a firing temperature of approximately 2300°F),
- Economical (costing about $4 to $5 per gallon for the slip),
- Temperature-resistant (much in excess of the required 1000°F),
- Impervious, (having relatively good surface finish).

A series of models of these materials was made and tested at Langley. Details of these tests are given under the Phase III discussion. During the tests, the material that performed best was the Carbon Black Porcelain; however, its strength properties were low. As a result, a task was undertaken to modify this material to improve its strength. A number of formulations were prepared in which this material was used as a base. Samples were poured and fired at various temperatures and flexural tests were performed. Results of the tests are given in Table 4. As a result of this work, formulation C-8 was chosen as a potential material for further use in making models. This selected formulation consists of 80% Carbon Black Porcelain slip and 20% Wollastonite.

Numerous models of various configurations were made. Typical models are shown on Figs. 7 through 10. Problems were encountered in firing the material when it was made into complex shapes. This is attributable to the very low strength of the material at the firing temperature required for verification. These problems are peculiar to each model shape and were worked out on a trial and error basis. (This material also shrinks about 15 to 20% upon firing.) A series of these models was successfully built and tested at Langley in March 1971 as discussed under the Phase III, Section 3.

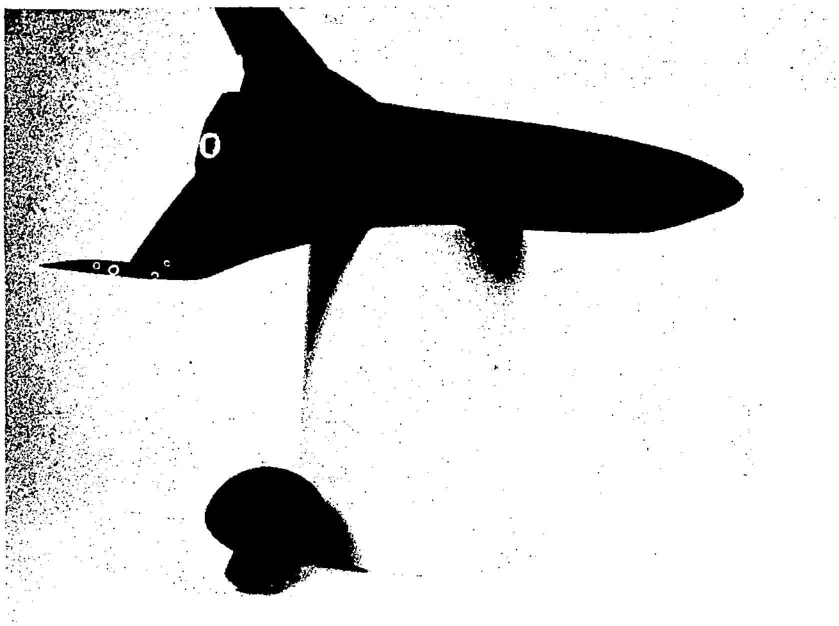

Fig. 7 — Typical C-8 Porcelain Booster and Hemisphere Model

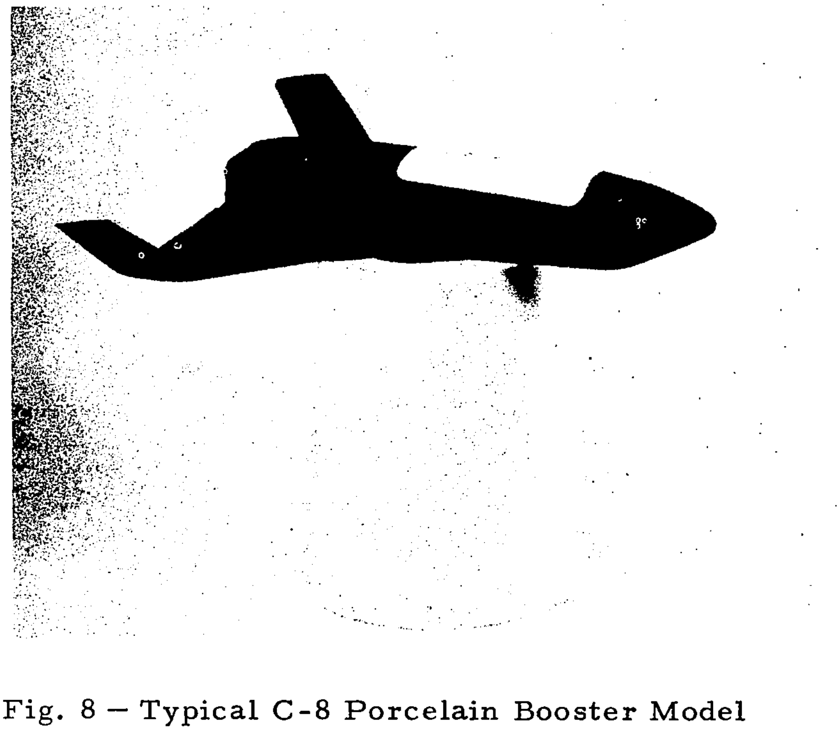

Fig. 8 — Typical C-8 Porcelain Booster Model

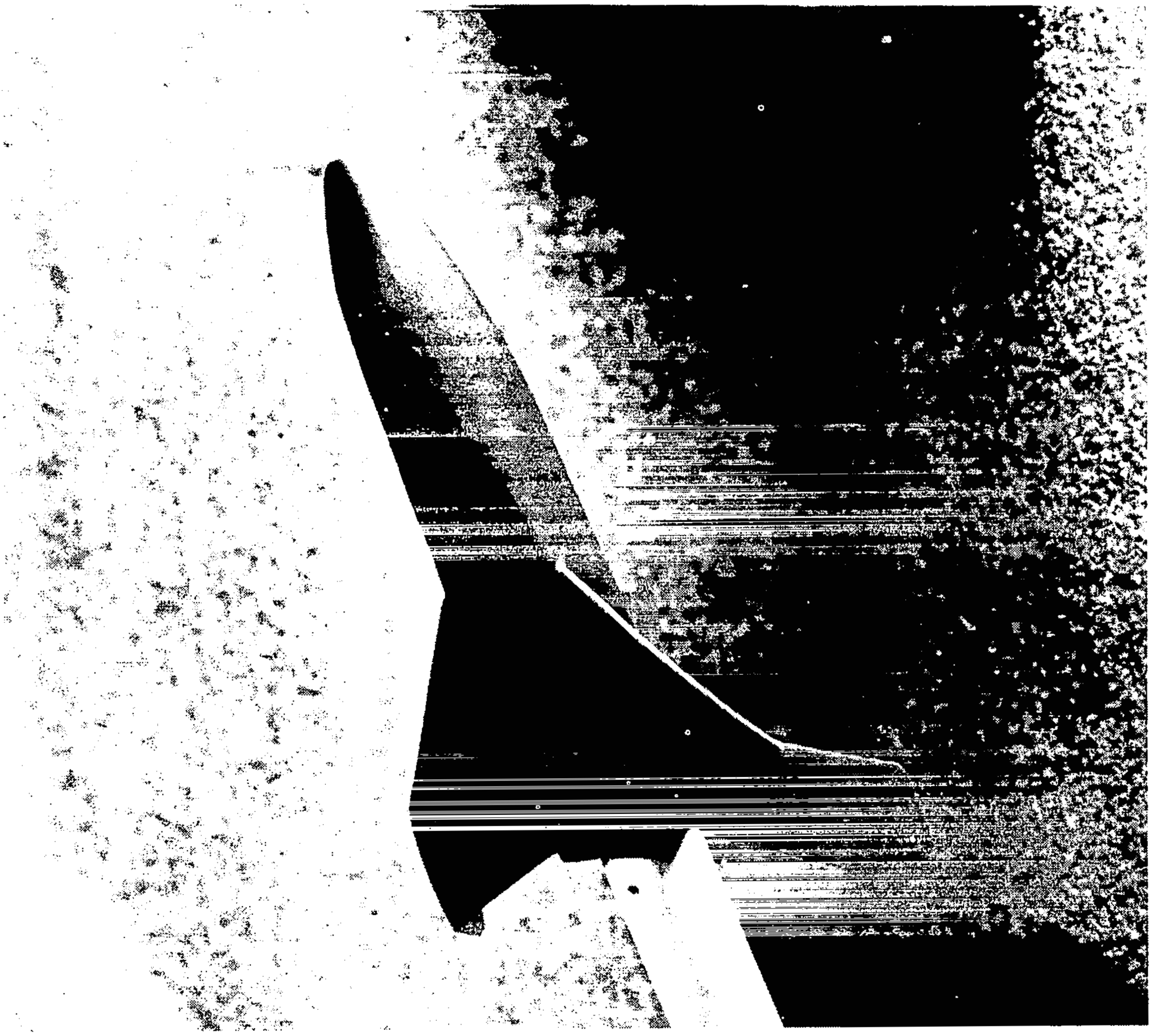

Fig. 9 — Typical C-8 Porcelain Model with Sting Installed Ready for Testing

Fig. 10 — Typical C-8 Porcelain Model with Sting Installed Ready for Testing

trial and error basis. (This material also shrinks about 15 to 20% upon firing). A series of these models was successfully built and tested at Langley in March 1971 as discussed under the Phase III, Section 3.

Alumina Material: Late in Phase II, it was decided that a material having higher strength and less firing shrinkage than C-8 would be desirable. A high-alumina material was thought to best meet these requirements. Since aluminas are available commercially only in the finished-part, fired state, the decision was reached that a formulation, tailored to requirements, using the basic raw materials, should be developed. A number of formulations were fabricated into samples and fired. Various binders were tried including the following:

- Emerson and Cumings Eccoseal W-19
- Union Carbide 2795 Epoxy
- Ethyl Silicate
- Sodium Silicate
- Potassium Silicate
- Colloidal Silica
- Polyvynl Alcohol
- Various Sauereisen Cements and Binders
- Georgia Kaolin
- Mogul Starch
- Marblette Corporation Maraset 655 Epoxy.

Table 4

RESULTS OF FLEXURAL STRENGTH TESTS ON MODIFIED COMMERCIAL PORCELAIN MATERIALS

Material Designation	Average Flexural Strength (psi)
C00	8636
C0	9081
C1	9388
C2	8217
C4	9183
C5	9027
C8	9876
C18	8203

After these binders were tried with various alumina and flux combinations, colloidal silica was selected as the binder best suited for this application. The final material selected consisted of the following approximate oxide percentages:

$A\ell_2O_3$	73%
SiO_2	21.2%
C_aO	5.3%

plus trace amounts of other oxides. This material proved to be very well suited for the present application. Numerous samples were fired and the optimum firing temperature found to be 2600°F. At this firing temperature a low shrinkage of approximately 2 to 3% is obtained. The material is essentially impervious, and has good strength. Models of alumina were more easily fired than the models of the C-8 porcelain material. Approximately sixty models of this material were poured using plaster molds. A number of these were fired and several were tested in the Langley Mach 8 Variable Density Tunnel (see Section 3, Phase III). Typical models of alumina are shown in Figs. 11 through 17.

In summary, alumina meets all requirements for this contract. Alumina is economical to fabricate, is strong, homogeneous, has acceptable thermal properties, can be cast, has a temperature capability well over the required 1000°F, and has a smooth nonporous surface finish.

Other Materials: Among many ceramic materials considered, one other is marginally acceptable. This material is called Grade A lava, which is an aluminum silicate and is mined from the ground in solid form. In the unfired state the material has a consistency similar to soapstone, and it is machinable. The fired material is nonporous. It also has little firing shrinkage. However, one problem with the material is that cracking occurs when it is fired in thick sections. Generally speaking this material is more adaptable to thin-section uses such as for circuit boards to electronics. To demonstrate the application of this material to this requirement, two representative models were made and fired. These are shown in Figs. 18 and 19. For application to model making

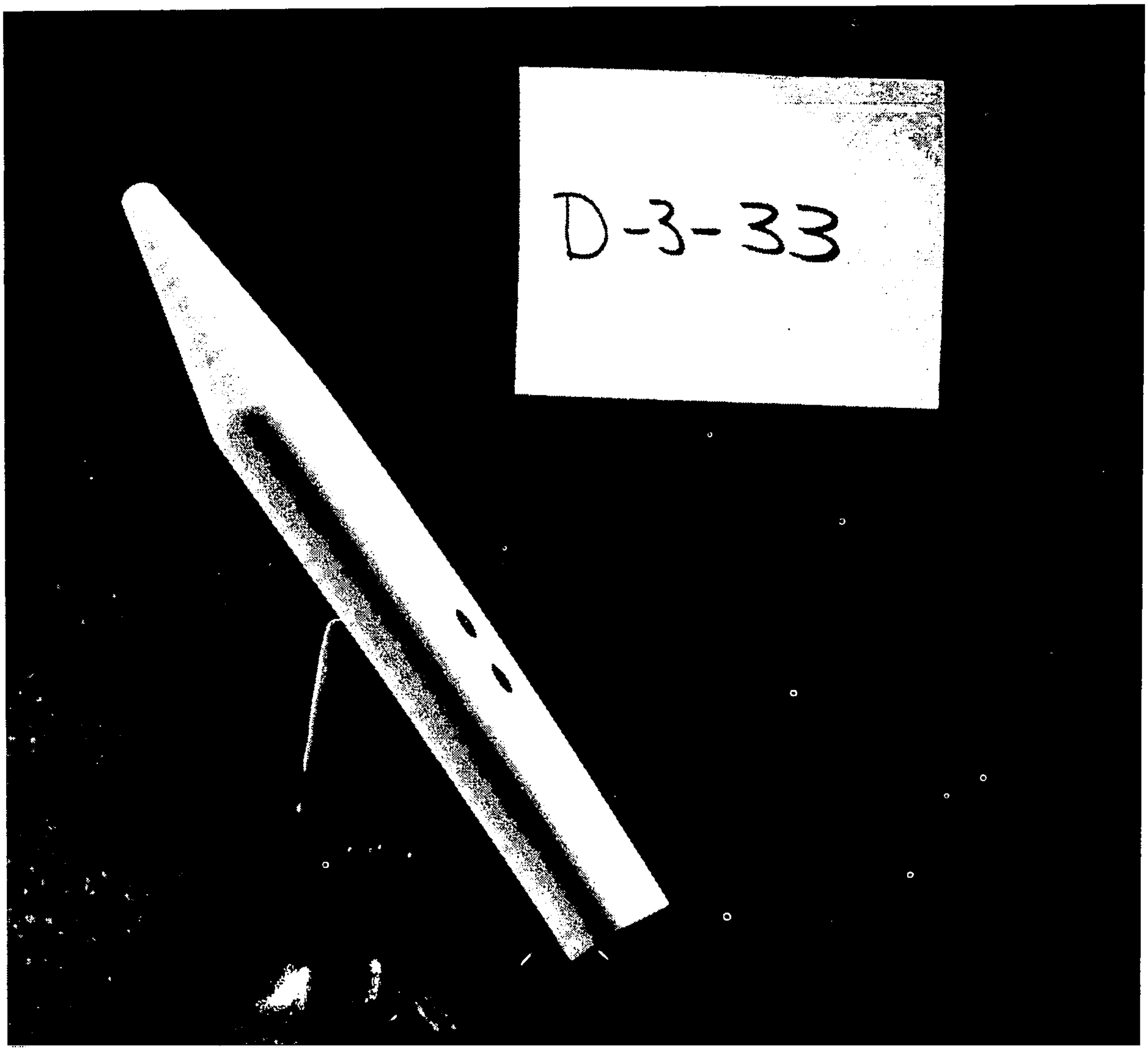

Fig. 11 — Alumina Model D-3-33 with Sting Mounted on Top Ready for Langley Test

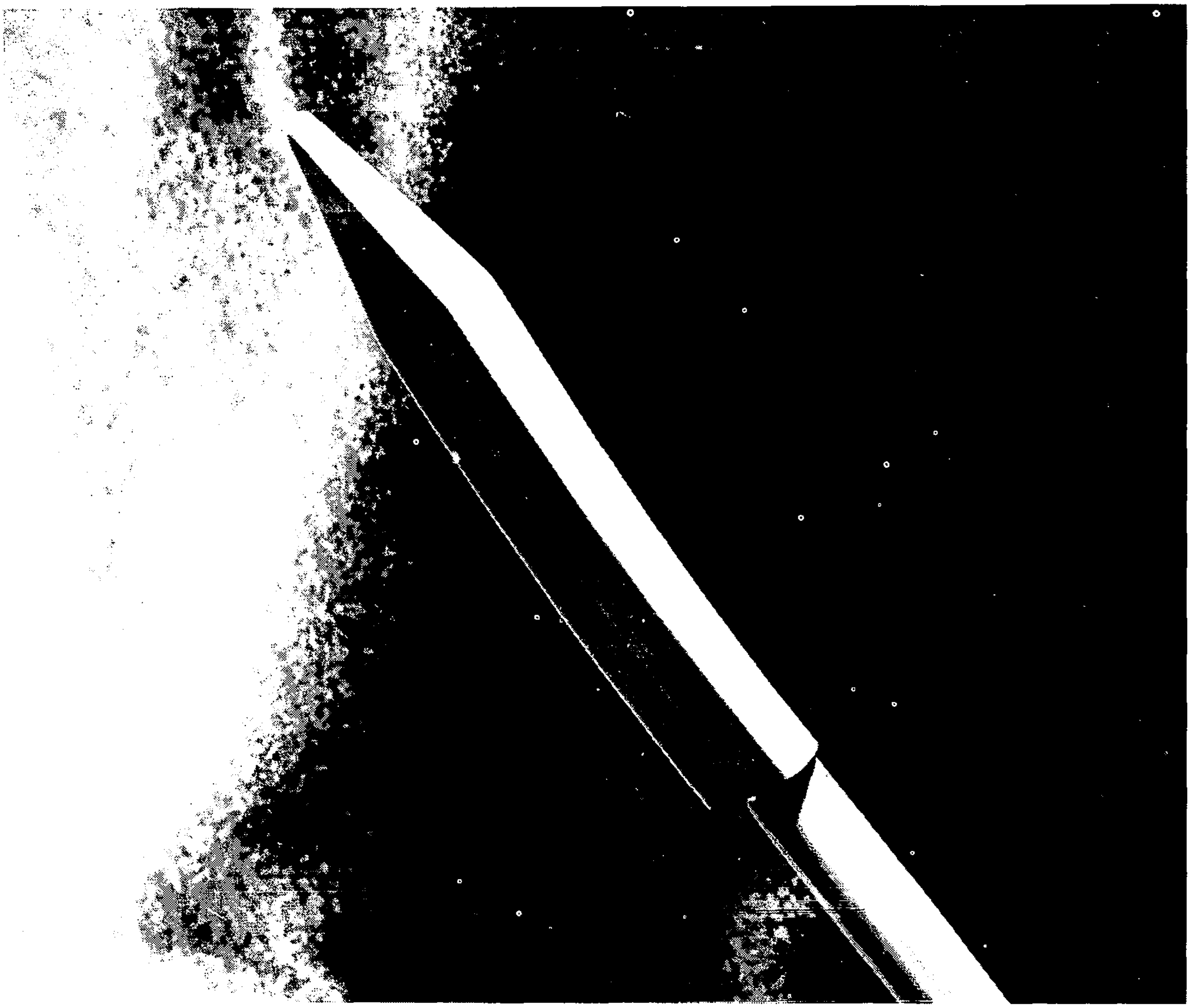

Fig. 12 — Typical Alumina Model with Sting Mounted at Rear

Fig. 13 – Typical Alumina Booster Model with Sting Mounted at Rear (Bottom View)

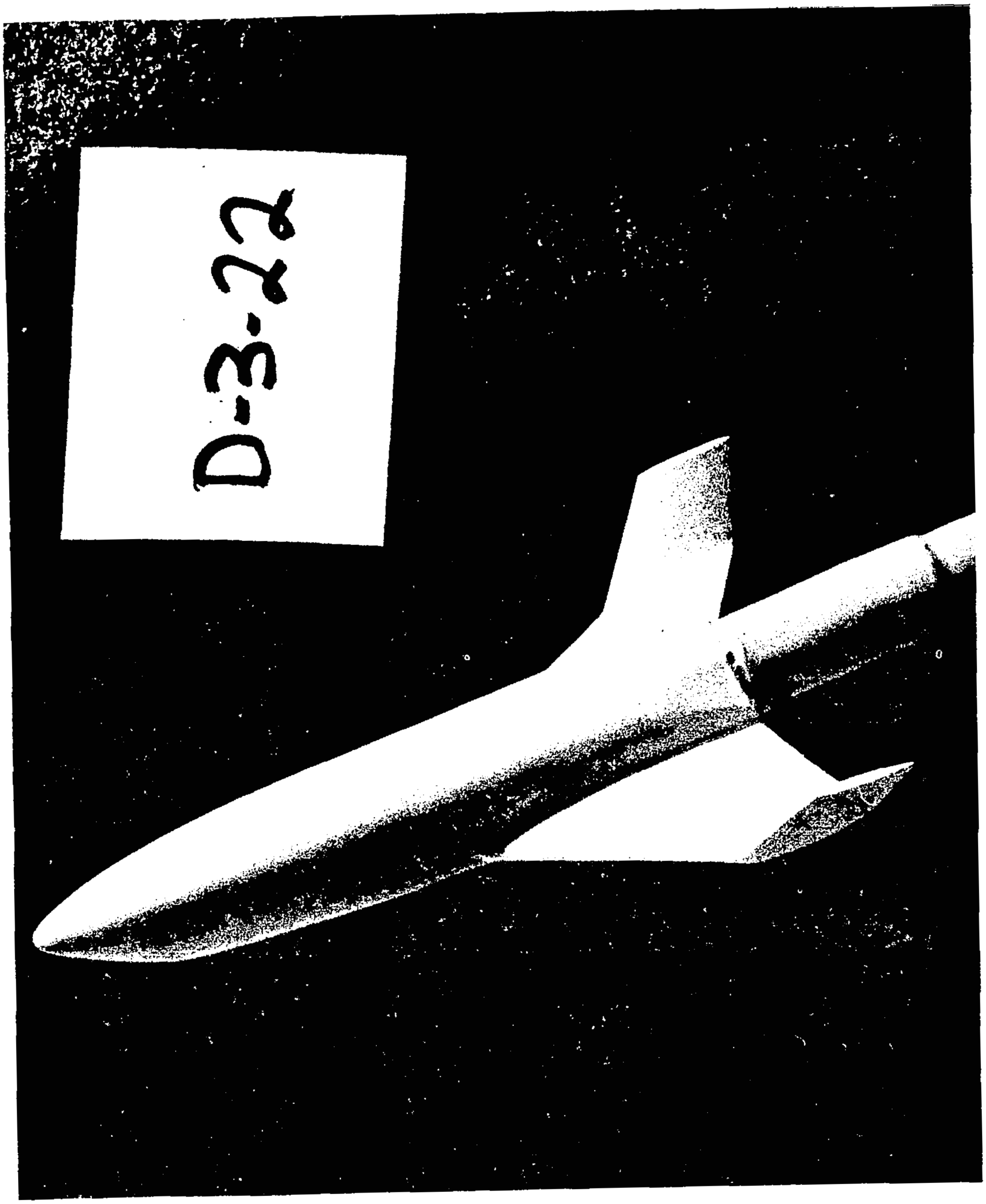

Fig. 14 -- Typical Alumina Booster Model with Sting Mounted at Rear (Top View)

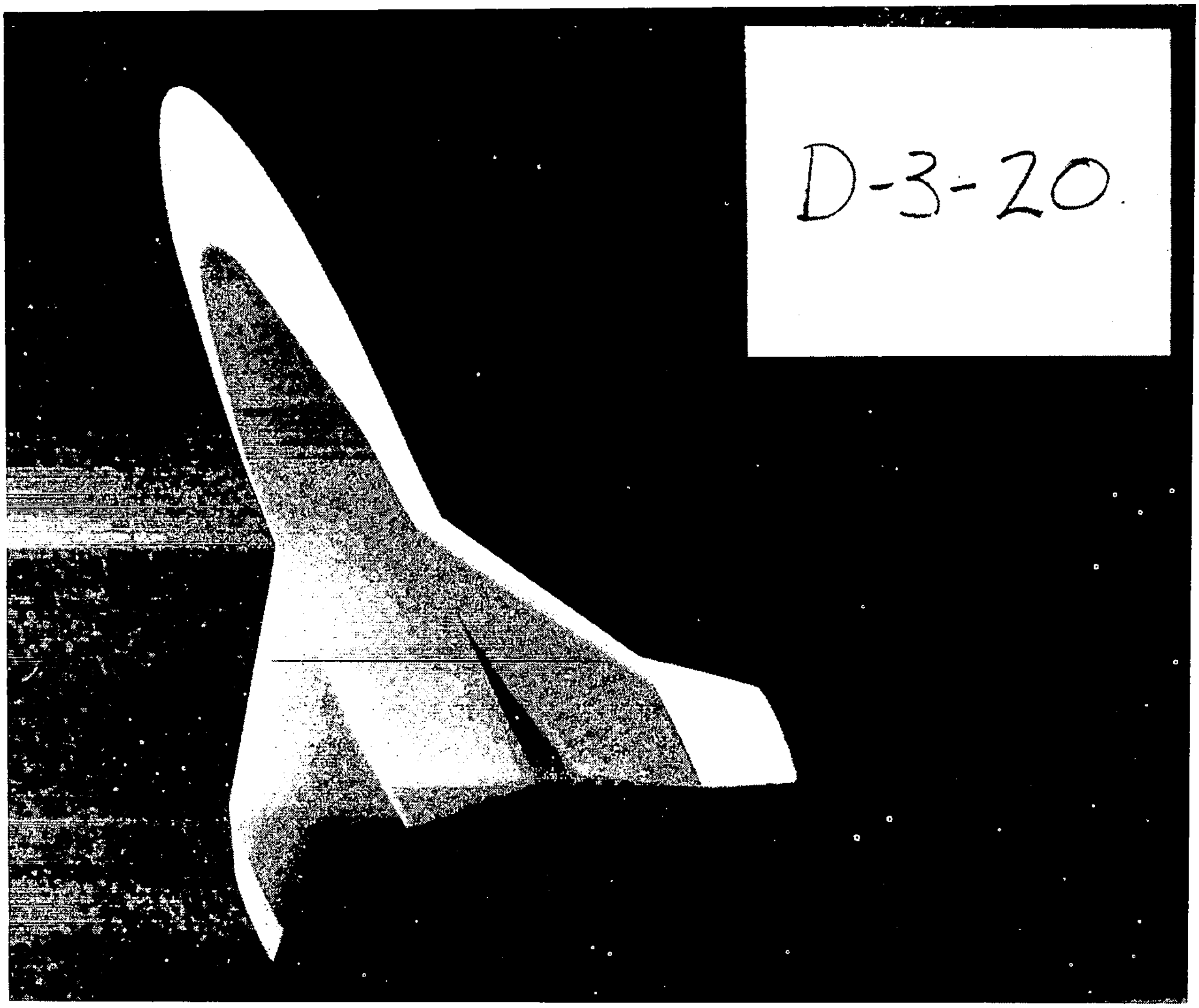

Fig. 15 – Typical Alumina Booster Model with Sting Mounted at Rear (Top View)

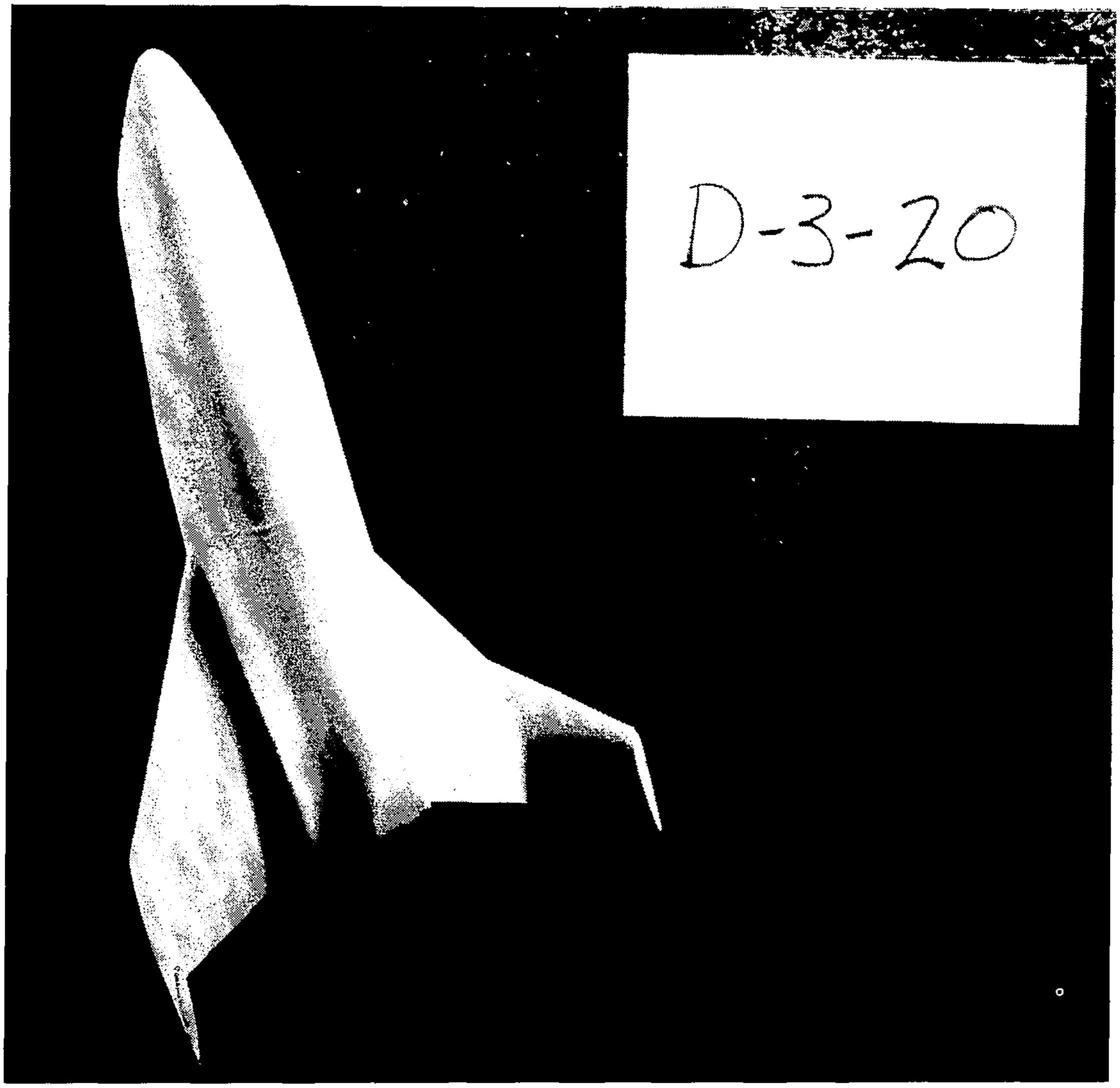

Fig. 16 – Typical Alumina Booster Model with Sting Mounted at Rear (Bottom View)

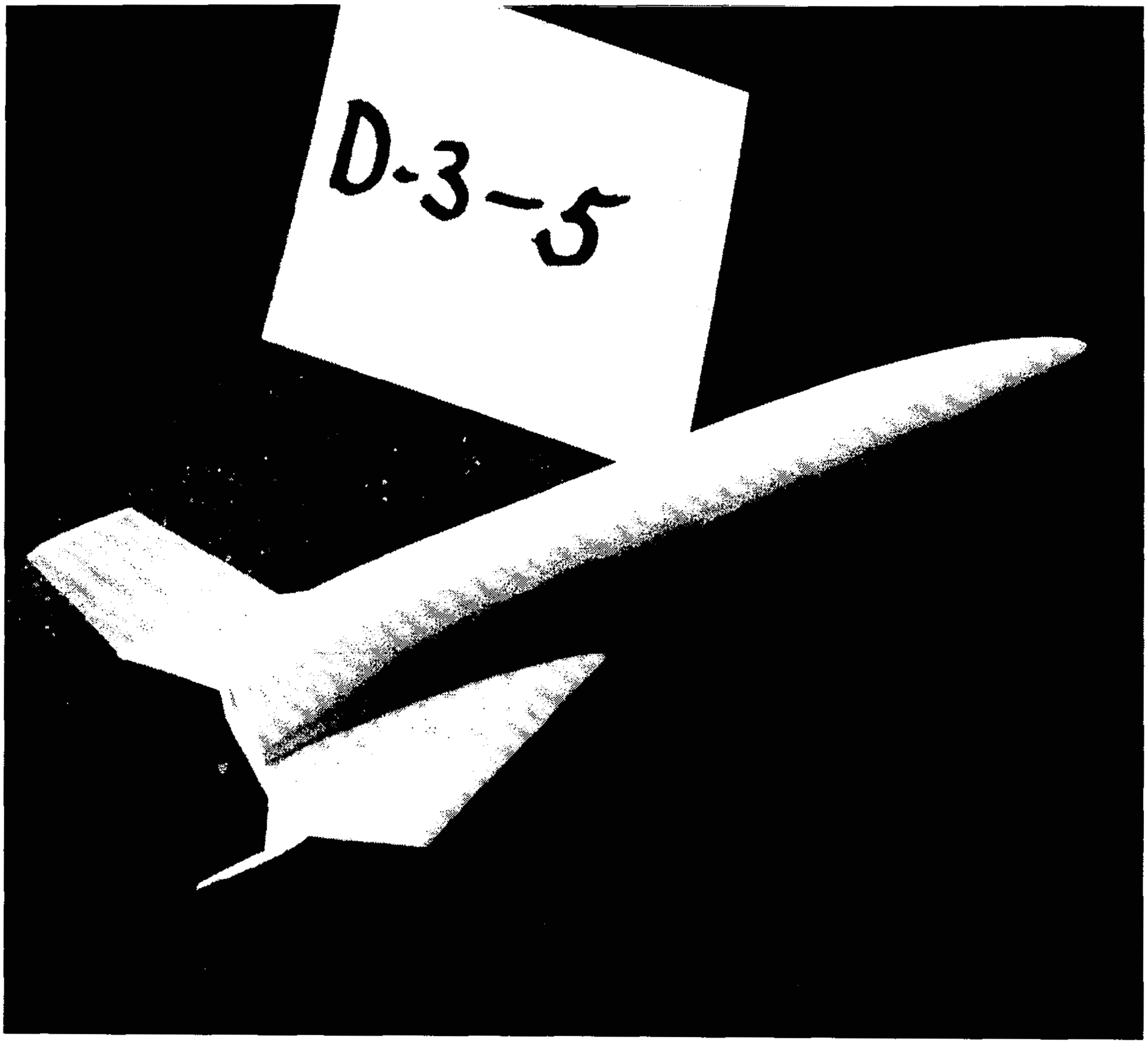

Fig. 17 – Typical Alumina Booster Model Before Sting was Mounted

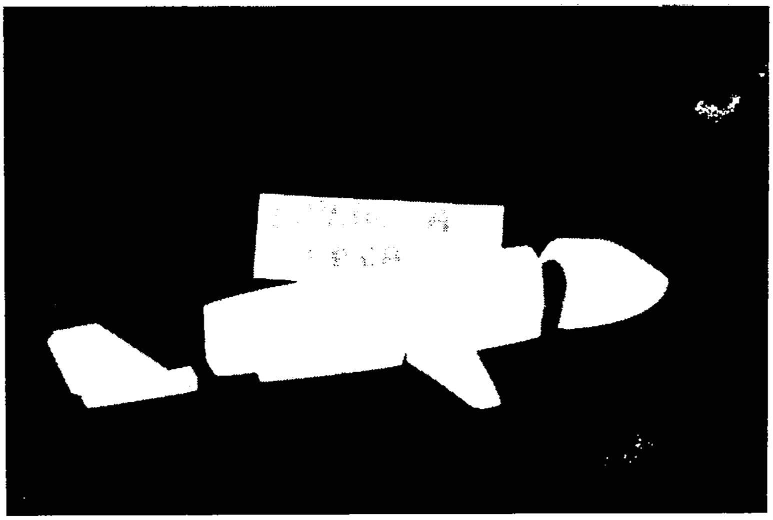

Fig. 18 — Grade A Lava Model After Machining and Firing, Ready to be Cemented Together

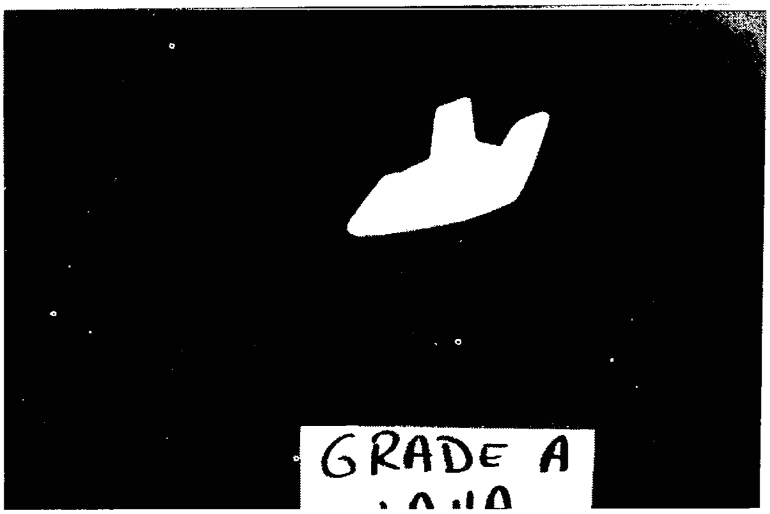

Fig. 19 — Small One-Piece Grade A Lava Model After Machining and Firing

each part, such as a wing or fuselage, must be made and fired separately and cemented or mechanically attached to the other parts as shown in Fig. 18. The approximate properties of this material are as follows:

$K = 0.9$ Btu/ft-HR-$^{\circ}$R
$C \cong 0.25$ Btu/lb-$^{\circ}$R
$\rho = 145$ lb/ft^3
Flexural Strength = 9000 psi
Maximum use temperature = $\sim 2000^{\circ}$F.

As seen, the thermal properties are very similar to Stycast but the strength is low. In summary this material is only marginally acceptable.

2.3.3 Thermal Shock Tests of Ceramic and Plastic Models

In order to make a qualitative comparison between the thermal shock resistance of Stycast, alumina, and the C-8 materials, the following tests were performed.

The three models shown in Fig. 20 were used in the tests, which were performed by heating the models in a furnace to a constant temperature and holding at that temperature for approximately one hour. Each model was then removed from the oven and submerged in water at room temperature. The first temperature used was 300°F. Subsequent tests were performed at 100°F increments up to 800°F. Results were as follows: After the 300°F and 400°F tests were performed, none of the models showed cracks; however, after the 500°F test, the Stycast cracked as shown in Fig. 20b. After the 600°F test there were more cracks in the Stycast model and the cracks were more pronounced (see Fig. 20c). The C-8 and alumina models did not change. After the 700°F tests, the alumina model showed significant cracking and it broke when it was handled. The C-8 showed no cracks. After the 800°F tests, the C-8 model had cracks but did not break (Fig. 20d). The Stycast was not tested at 700°F or 800°F.

In summary, the relative resistance of these three materials to thermal shock in order of best-to-worst is: (1) C-8; (2) alumina; and (3) Stycast.

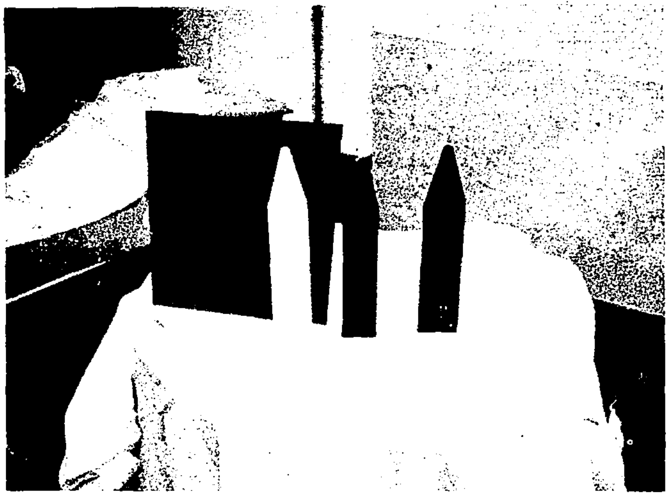

Fig. 20a - Thermal Shock Resistance Models Before Tests

Fig. 20b - Thermal Shock Resistance Test Results (After 500°F Test)
(Left to Right: Alumina Ceramic, C-8 Modified Porcelain, Stycast 2762FT)

Fig. 20c — Thermal Shock Resistance Test Results (After 600°F Test)

Fig. 20d — Thermal Shock Resistance Test Results (After 800°F Test)

2.4 PHASE III: MATERIALS PERFORMANCE AND EVALUATION TESTS

2.4.1 Wind Tunnel Tests at Langley Research Center (Plastics and Ceramics)

A materials screening test program was conducted from 26 to 28 October 1970 at the Variable Density Tunnel, Langley Research Center. Primary objectives of the tests were to determine the relative performances of the various materials cast in a representative model shape, with the materials under actual test conditions. The overall purpose of Contract NAS1-9983 was to find a castable material that will withstand 1000°F high-pressure flow without surface degradation or material failure. Novimide 700 (an imide novalac) was the highest temperature castable plastic that was found (other than Stycast 2762 FT) that came close to filling requirements.

In an attempt to improve Novimide's thermal properties and give it additional strength, various fillers were added to Novimide and cast into model shapes. Two Stycast 2762 FT models were also cast to provide standards for comparisons. Two model shapes were cast: 5-inch orbiter and an 8-inch booster. Examples are shown in Figs. 21 and 22. The orbiter had a much smaller nose radius and presented a more severe test for the materials. The following plastic formulations were cast:

8-Inch Booster

Batch Number	Mixture
10-15	Stycast 2762 FT
828	Novimide + Lithafrax 2121
826	Novimide
99-1	Novimide + Alumina
98-1	Novimide + TB (Antimony Trioxide)
910-1	Novimide + Lithafrax 2121 (different %)

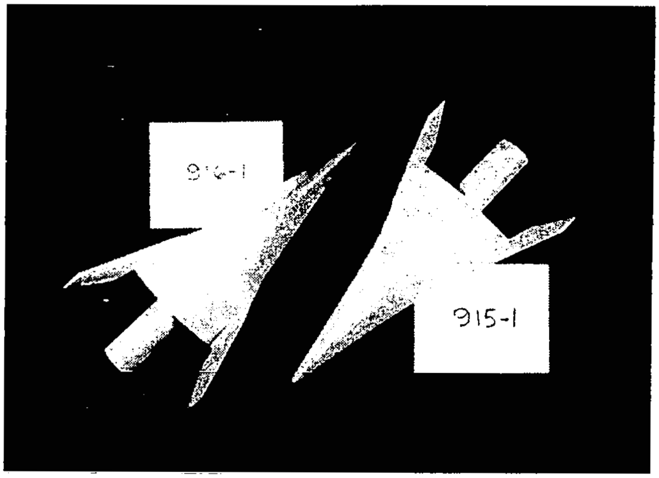

Fig. 21 — Test Models; 5-Inch Orbiters

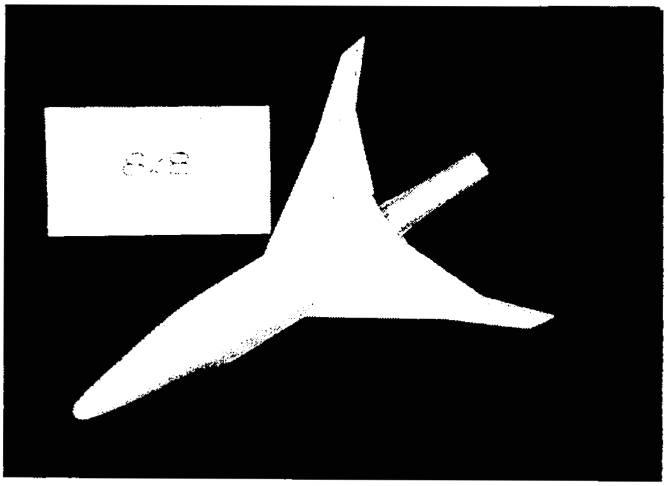

Fig. 22 — Test Model; 8-Inch Booster

5-Inch Orbiter

Batch Number	Mixture
921-1	Stycast 2762 FT
912-1	Novimide
916-1	Novimide + TB (Antimony Trioxide)
915-1	Novimide + Alsimag
918-1	Novimide + Wollastonite
916-3	Novimide + Antimony
923-1	Novimide + TAM (Zirconium Oxide)
922-1	Novimide + Mica

Also models of the 5-inch orbiter were cast from several commercial porcelain slips and fired at 2300°F. These models were also provided and tested as candidate materials. The following models were made.

Number	Description
1	White Orchid Porcelain
2	55-S Porcelain
4	White Orchid Porcelain
5	Carbon Black Porcelain
6	White Orchid Porcelain

It was decided to run all models at the same test conditions and then compare surface finishes. The initial test conditions were:

P_o = 500 psig

T_o = 1000°F

T_{pc} = 500°F

time = 15 sec

α = 0 deg

The six 8-inch boosters were tested first with Stycast performing the best. It had very little evidence of any surface change while the other five models had nosetip and wing/fin leading edge scorching with slight charring. These initial results were somewhat dissapointing, therefore it was decided to test the porcelain models next.

The porcelains performed extremely well under initial test conditions with the exception of Porcelain No. 1 breaking in half after twelve (12) seconds of testing. The porcelain slip used for Model No. 1 is a very brittle material with poor shock impact resistance. Also an air pocket in the material caused a weak point in its construction. All porcelain models were completed within a two-week period, and a great deal of effort was not put into their fabrication. Results of these tests demonstrated that porcelain

- Is non-porous
- Is castable
- Has an acceptable ρCk product, and
- Has 1000°F capability.

The five (5) inch plastic orbiters were then tested at the same conditions with all models except Stycast having some scorching and charring of the nosetip and fin leading edges. The models were ranked according to their performance and are:

<u>Best to Worse</u>

1. Stycast
2. 922-1
3. 918-1
4. 915-1
5. 916-1
6. 916-3
7. 923-1
8. 912-1

It was concluded after all models had been tested, that porcelain offered the best material for high temperature capability. To determine their performance under more severe conditions, the pressure was increased to $P_o = 1000$ psig. The porcelain models were then tested with the following results:

Porcelain No. 6 - wing broke under loading after 12 seconds.

Porcelain Nos. 4, 5, 2 - performed extremely well.

The wing on model No. 2 was broken while being prepared for its test. On all porcelain models, there were no signs of charring or surface degradation. The melt contours obtained indicated that the porcelains had ρCK products well within acceptable limits. The only apparent problem was in handling due to the brittleness of the material.

The 5-inch orbiter of Stycast and model No. 922-1 was tested at P_o = 1000 psig with Stycast performing the better of the two. The test pressure was then increased to P_o = 1500 psig for maximum severity. The Stycast orbiter was tested at these conditions with evidence of charring of the nosetip and leading fin edge.

Porcelain model No. 6 was then tested at 1500 psig and performed extremely well with no charring or scorching. Porcelain model No. 4 was broken while being prepared for testing and model No. 5 broke in half after testing for 9 seconds. The porcelain's brittleness has been recognized as a pending problem and efforts had been initiated to solve this problem. Wollastonite, previously mentioned, had been added to Newell No. 55 porcelain slip and a nosetip model cast and fired. This model was mounted in a sting and tested at P_o = 1000 psig with extremely good results. There was no evidence of charring or scorching and the model did not break.

It was concluded that overall test results indicated the porcelain was the best material if the brittleness problem could be solved. Also the Stycast 2762 FT models, according to Mr. J. L. Hunt of Langley, were of superior quality as compared to other Stycast models tested in the Mach 8 Variable Density Tunnel. To further evaluate Lockheed-Huntsville Stycast models, two severe runs were made on the 8-inch booster at a P_o = 1500 psig. It performed extremely well but the nosetip began to char with the fin leading edges charring back approximately 1/8 inch. Two other runs were then made to obtain relative values of ρCK between Stycast and booster No. 99-1. According to the melt contour progressions, they were approximately the same value. Evidence of this is shown in Figs. 23 and 24. Black and white polaroid photographs were made after each test documenting the materials' performances.

Following the completion of the first test program, it was decided that efforts should increase toward solving fabrication problems of porcelain, with a minor effort continuing in plastics. Subsequently, a series of alumina ceramic, C-8 porcelain and Stycast 2762FT models was fabricated and delivered to Langley Research Center during March 1971. Several models of a delta wing shuttle booster configuration were fabricated of the ceramic and porcelain, while Stycast was used to fabricate two models as shown in Figs. 25 and 26. These models were later tested at the most severe conditions available in the Mach 8 Variable Density Tunnel. The results are discussed later.

The delta wing models fabricated from alumina ceramic and C-8 porcelain presented many fabrication problems that should be noted. A discussion of them here should help to enlighten future model fabricators concerning problems encountered and eventual solutions. The solutions were reached through applied engineering judgements and trial and error procedures. Lockheed makes no pretentions that they are the ultimate in optimum solutions for fabrication, but they did work. By referring to Figs. 13 through 17, the reader can ascertain the complexity of the shape, and envision some of the fabrication problems, particularly if he is familiar with porcelain and ceramics.

The problems in fabrication of C-8 porcelain and alumina ceramic models can be summarized as follows: correct mold fabrication, model pour rate, model removal, model drying rate, and model firing sequence.

Fig. 23 — Model 99-1 (Novimide and Alumina) Tested at P_o = 1500 psig, T_o = 1000°F After 10 Seconds

Fig. 24 – Model 10-15 (Stycast 2762 FT) Tested at P_o = 1500 psig, T_o = 1000°F After 10 Seconds

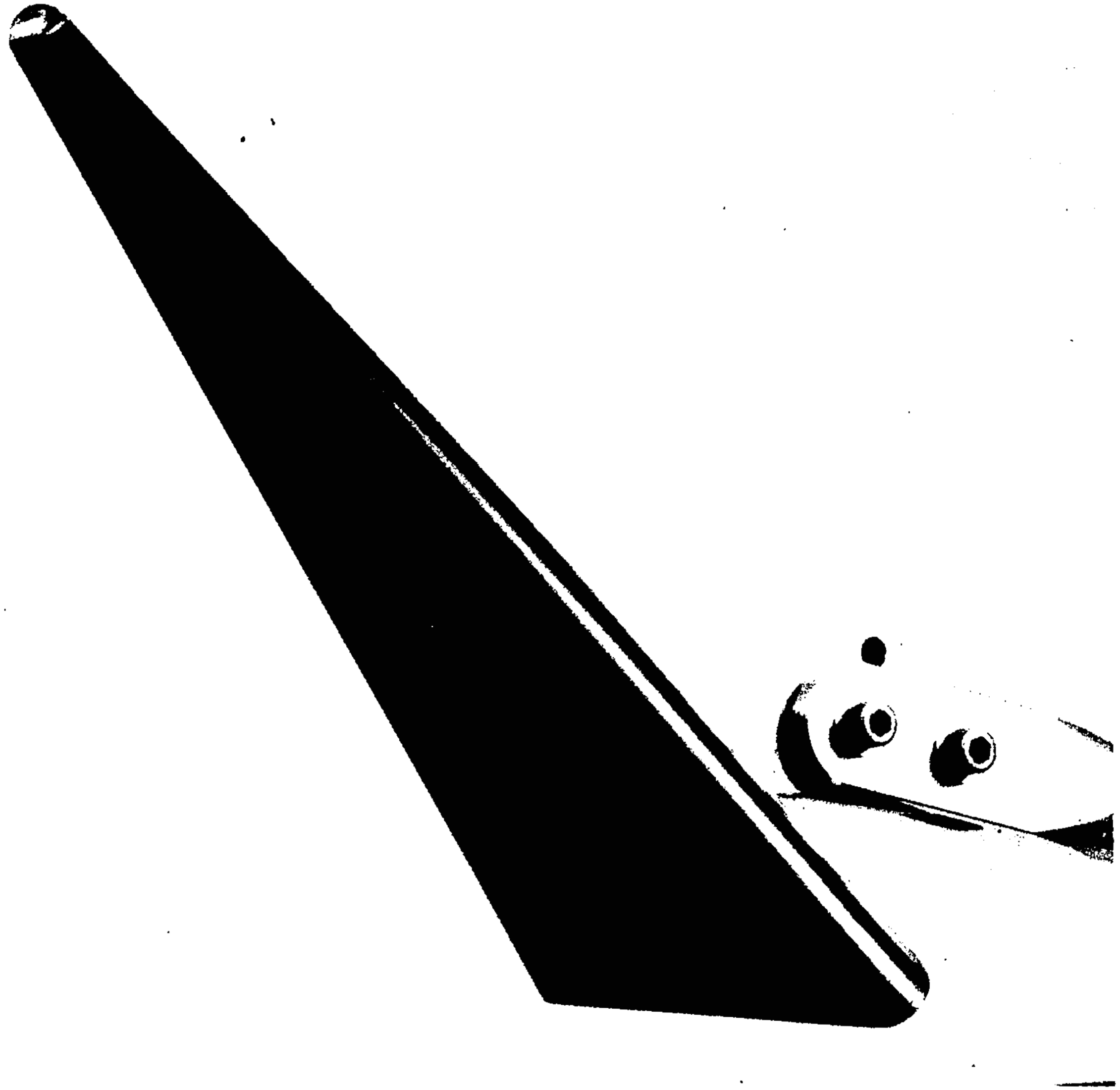

Fig. 25 - Langley Delta Body, Stycast 2762FT

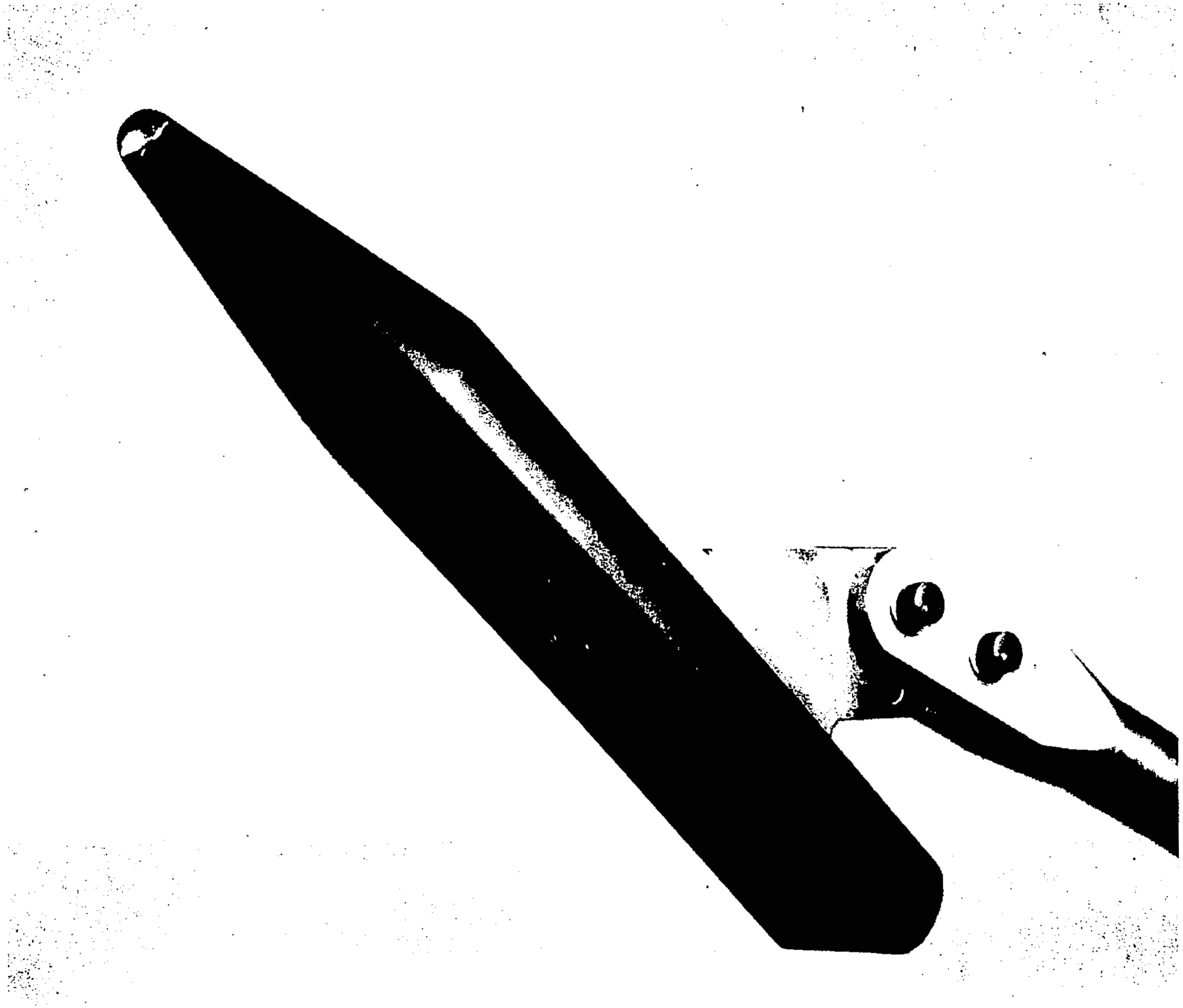

Fig. 26 - Langley Transition Model, Stycast 2762FT

Molds for fabricating either C-8 porcelain or alumina ceramic models are made the same way. A pattern model of the desired shape must be provided, with Gypsum mold plaster used to fabricate the molds. This material is available at any commercial or wholesale ceramic outlet store. The plaster is sifted thoroughly to remove all lumps, then mixed with water to make a pourable fluid. To provide a smooth mold surface, the mix is degassed in a vacuum chamber for approximately 5 to 7 minutes before it is poured.

The plaster sets up or hardens within 20 minutes after it is mixed, therefore, everything (pattern, mold box) must be ready for use before mixing is begun. The hardening takes place during an exotherm reaction and it can be noted by an abundance of heat being discharged.

Multiple-part molds must be prepared because of the rigidity of the plaster, and they must be fashioned in a way the molds will not hang or catch on any part of the models when they are removed from the molds. This is basically a trial-and-error procedure which can be perfected only through experience.

Attempts were made to fabricate two-, three-, and four-piece molds for the shuttle booster shape under discussion. Again, the trial and error technique was used to find the right mold design. The three- and four-piece molds that were developed provided the best results. A typical three-piece mold is shown in Fig. 27. Tincture of green soap was used as a mold parting agent and it is highly recommended for this use.

Next, the model is poured. First, the mold is assembled and held together with large rubber bands. The slip is then mixed and poured into the mold until a solid model is formed.

Many problems were encountered in obtaining a perfectly shaped model without sags, cracks, or surface blemishes. By trial and error,

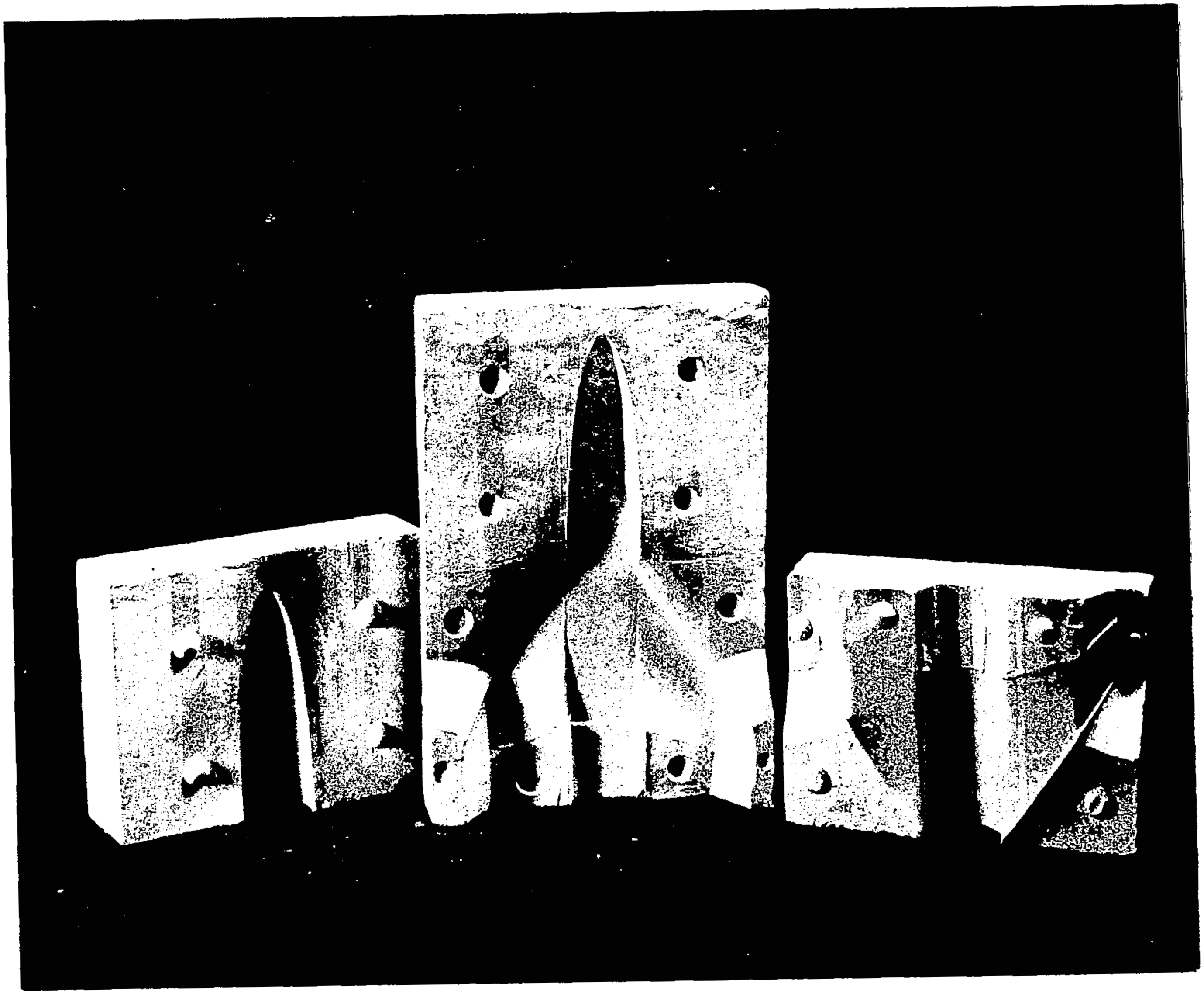

Fig. 27 - Three Piece Plaster Mold for Space Shuttle Model

model slip pouring rate was found to be important and also that the mold must be very dry before pouring is begun. The mold/model hole should be quickly filled with water and then emptied before the slip is poured. The model material drying rate is initially slowed down enough so that an entire model can be poured before fast drying sets up. When the model pouring is begun, care must be taken to have plenty of slip available so that there is no break in the pouring. Otherwise, a surface blemish will result. The mold is continuously filled until the whole model is solid. The model is then left in the mold for 15 to 45 minutes or until the model is dry enough to be removed from the mold without sagging or breaking. The mold is then gently removed, piece by piece, until the model is clear of all portrusions, etc.

The next major problem encountered was model breaking or cracking during the drying cycle. The model must be completely dry before it is fired, otherwise, the model will rupture, when heat is applied to it. Initially, the delta wing booster models were air dried at room temperature (70°F and 40% relative humidity). Many of the models cracked, especially at wing/body junction. Some of the cracks were very minute and were not noticeable until the models were fired. Differences in the thicknesses of the various model parts were observed to produce various drying rates which caused cracks to develop. The drying rate of the models was controlled by sealing the models in airtight plastic containers for six to nine days until most of the moisture contained in the thick sections had slowly reduced to such a level that the models would dry in the air without cracking. Of course, there were still "fatalities," but there were not as many. A humidity-controlled drying chamber was therefore decided to be a necessity for fabricating ceramic models which have very thin and thick sections.

A humidity-controlled chamber was therefore designed and assembled during the last stages of the contract performance; however, because of tight schedules and approaching contract completion, the chamber was not used. The chamber will be available for future use on Langley models.

Models of simple shapes (like the Viking Lander) were fabricated and air-dried. The fatality rate for this shape was much less than for the first models cast. This model is discussed in Section 2.5.

After the fabrication procedures had been developed to provide a clean ceramic or porcelain model, the last task was to fire the models at their vitrification temperature. A simple Paragon floor kiln was used to fire the C-8 porcelain models, but the alumina ceramic required a much higher temperature than the Paragon kiln was rated to accommodate. The Paragon kiln proved to be unreliable in producing repeatable firing temperatures. Some models would become too hot and melt, and some would not vitrify completely. Lockheed purchased a $2900^{\circ}F$ furnace which has temperature controls that can be programmed for various temperature-time cycles. This furnace has proven to be helpful and it gives very repeatable results. Discussions with various ceramic companies were valuable in providing insight for determining temperature time rates and soak times for alumina ceramics. After various trial and error attempts, a firing cycle was determined. The cycle encompassed a Δ temperature increase at $50^{\circ}F$ per hour up to $2550^{\circ}F$. A soak time of three hours at $2550^{\circ}F$ is then held to allow all parts, regardless of thickness, to vitrify completely and to reach the desired state.

Alumina ceramic models were successfully fired in this furnace which provided the bulk of the test models for testing in the Mach 8 Variable Density Tunnel at Langley Research Center. Several attempts were made to use this furnace for firing C-8 models, but results were disastrous. The vitrification temperatures somewhat established in the Paragon kiln, proved to be too high when they were obtained in the $2900^{\circ}F$ furnace. Models would melt and ruin the support structures in the furnace. When very promising results became a reality with the alumina ceramics, all efforts with the C-8 porcelain were discontinued, even though several C-8 models had been successfully fired. These models were presented to Langley for testing.

The test series on the C-8 porcelain and alumina ceramic models were performed over a two week period during the last of March 1971. The models included:

- Two alumina ceramic boosters
- Two C-8 porcelain boosters
- Two alumina ceramic Langley configurations.

The two alumina Langley configurations were the same as those shown in Fig. 26. A typical mold and a model of this configuration are shown in Fig. 28.

All models were tested at various tunnel conditions with the maximum at:

$$P_o = 1500 \text{ psia}$$

$$T_o = 1000^\circ F$$

$$T_{pc} = 400^\circ F$$

$$\text{Time} = 35 \text{ seconds.}$$

All models performed quite well. There was no charring or degradation. The alumina ceramics models were excellent, indicating that the material was acceptable and promising. Typical results from the tests are shown in Figs. 29 and 30. This is an alumina ceramic delta wing booster with 400°F paint that had been tested at the stated test conditions.

The two Stycast models presented in Figs. 25 and 26 were fabricated from Stycast 2762FT and they had stainless steel nosetips. These models have been tested by Mr. Charles Johnson at Langley Research Center in the Mach 8 Variable Density Tunnel. The test conditions were:

$$M_\infty = 8.0$$

$$P_o = 2800 \text{ psig}$$

$$T_o = 900^\circ F$$

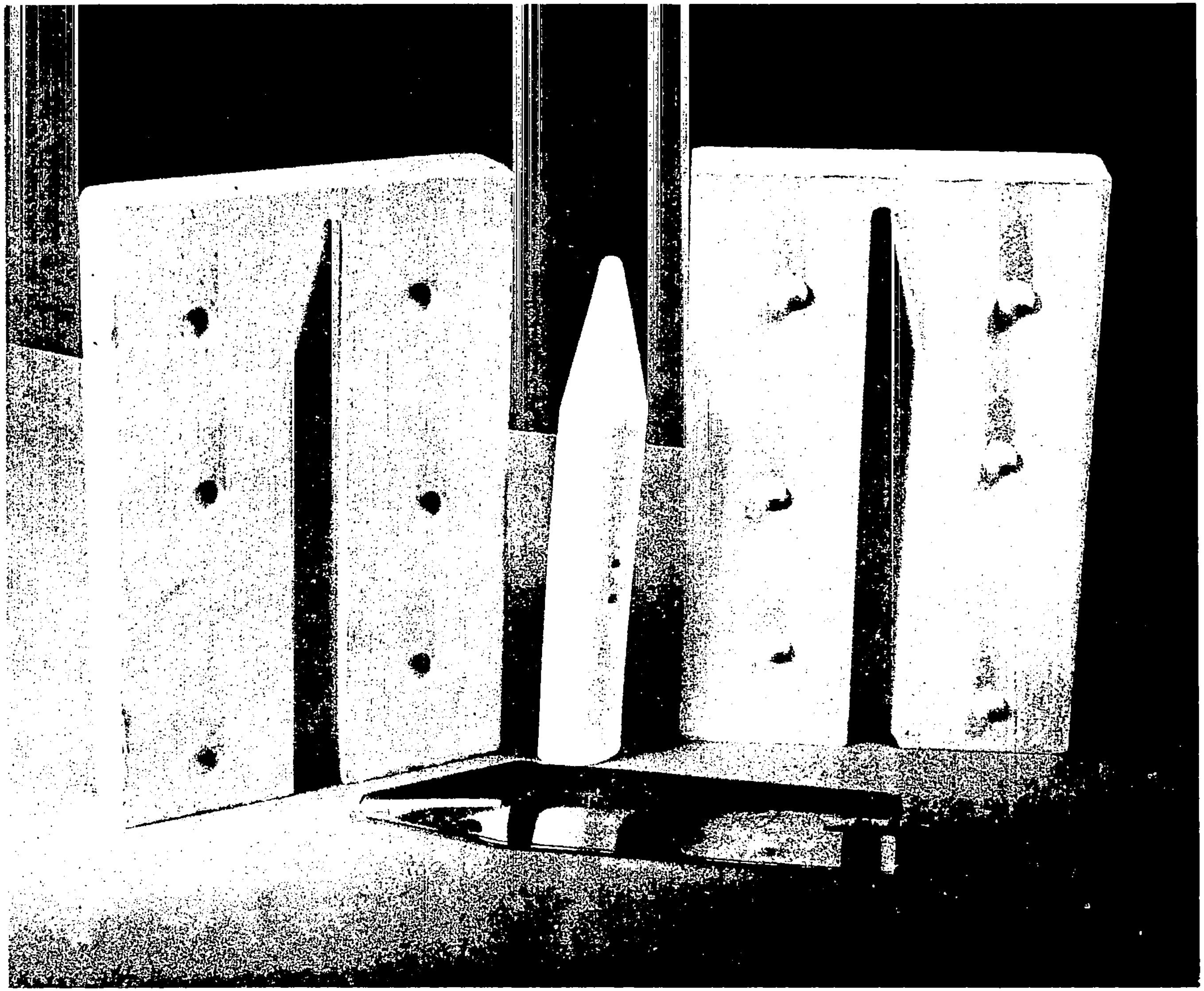

Fig. 28 - Langley Research Center Alumina Ceramic Model

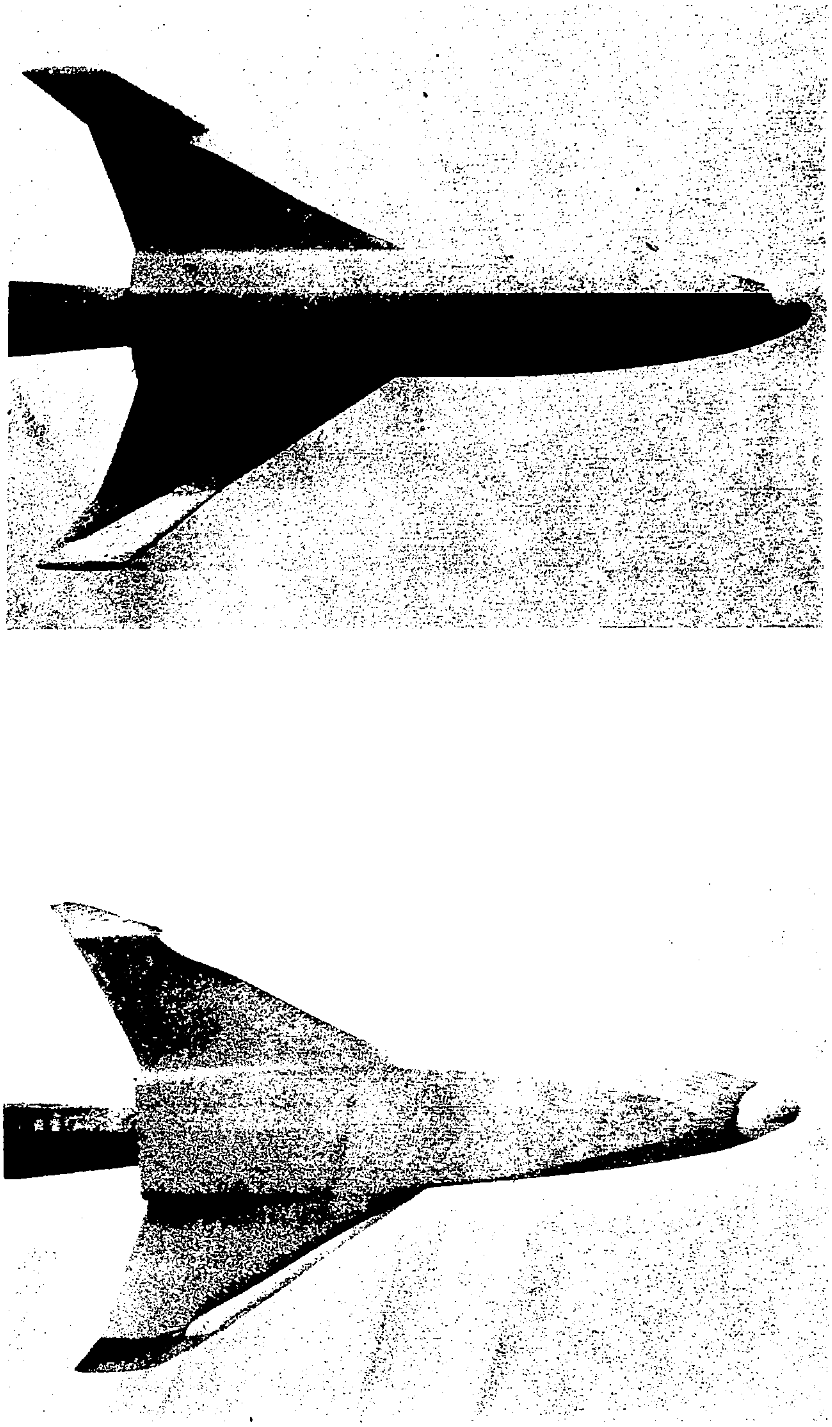

Fig. 29 - Alumina Ceramic Model, Top and Bottom View After Testing

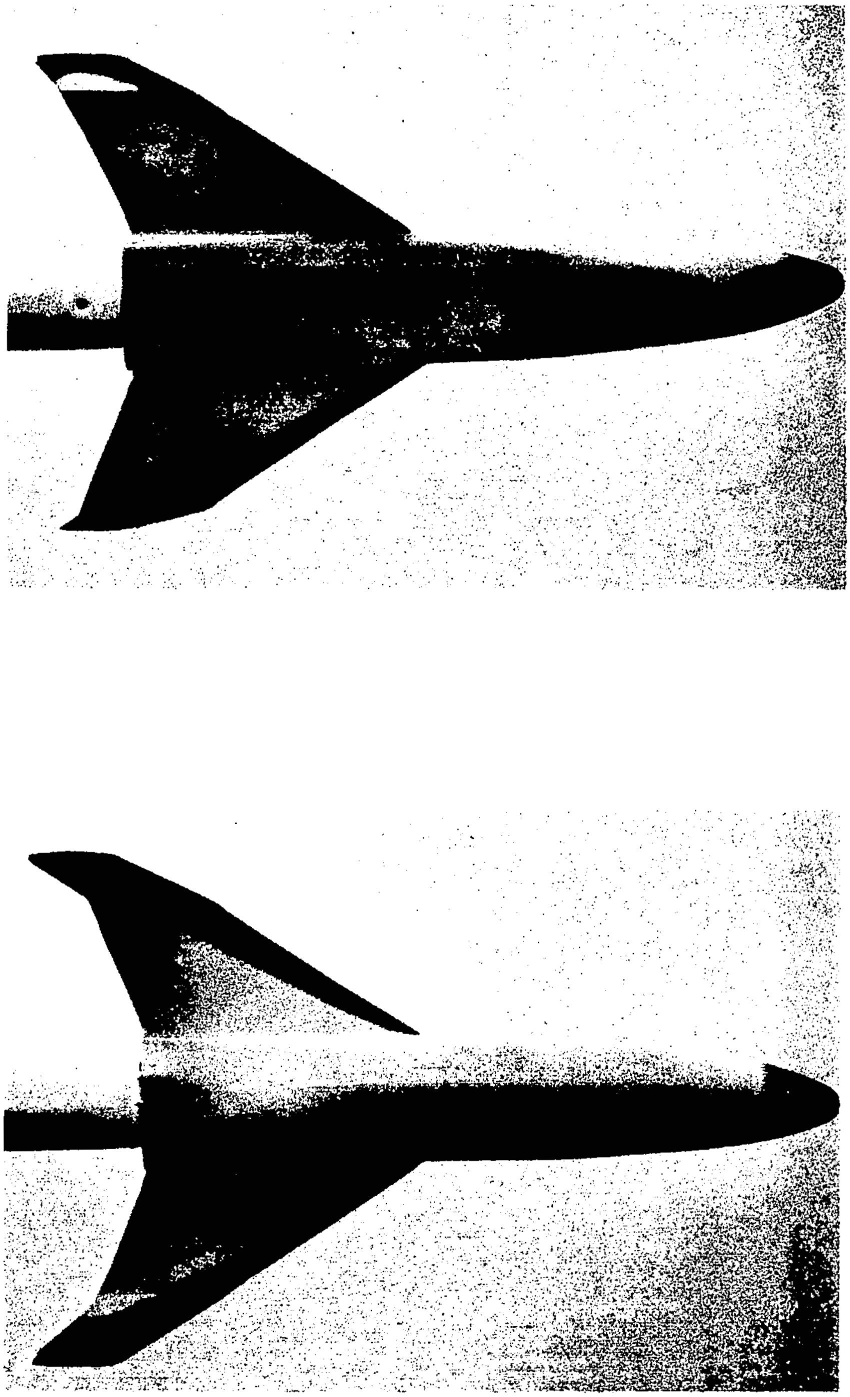

Fig. 30 - C-8 Porcelain Model, Top and Bottom View After Testing

$$T_{pc} = 450^{o}F$$

$$\text{Time} = 8 \text{ seconds.}$$

They represent the maximum conditions available in this test facility. According to Mr. Johnson, the Stycast 2762FT performed extremely well and a wealth of information was obtained. These models were tested a number of times and they still remain in excellent condition. Overall results from tests performed with Stycast 2762FT models for Langley Research Center, Marshall Space Flight Center, and Manned Spacecraft Center, have established a high level of confidence in this material for use with the phase-change technique.

Since the Tempilaq paints used in paint testing is usually white, the white alumina ceramics were modified slightly by adding cobalt oxide to give the material a dark blue color. This is discussed further in Section 2.5.1.

2.4.2 Thermal Properties Tests

Cured samples of Stycast 2762FT, C-8 modified porcelain, white alumina ceramic, and cobalt oxide/alumina ceramic were fabricated to measure their respective thermal properties of density, specific heat, and thermal conductivity.

Lockheed performed the measurements for density and specific heat, while Dynatech, Inc., of Cambridge, Mass. was subcontracted to measure thermal conductivity. Conductivity measurements were also performed by Materials Consultants, Inc. of Denver, Colorado. Lockheed-Huntsville is now buying a Tharmal Conductivity Comparative Apparatus from Materials Consultants, Inc., and they performed the measurements reported here as a courtesy for allowing them a 30-day extension on their delivery date.

Densities were measured by using weights and volumes for the various samples. Specific heats were measured using a calibrated steady-state calorimeter. (See Appendix B.) The following values were obtained:

C-8 Modified Porcelain

$$\rho = 150 \text{ lb/ft}^3$$

$$C_p = 0.201 \text{ Btu/lb}^\circ\text{F at } T = 125^\circ\text{F}$$

Stycast 2762FT

$$\rho = 131 \text{ lb/ft}^3$$

$$C_p = 0.16 \text{ Btu/lb}^\circ\text{F at } T = 80^\circ\text{F}$$

White Alumina Ceramic

$$\rho = 182.8 \text{ lb/ft}^3$$

$$C_p = 0.1968 \text{ Btu/lb}^\circ\text{F at } T = 128^\circ\text{F}$$

Cobalt Oxide-Alumina Ceramic

$$\rho = 157.6 \text{ lb/ft}^3$$

$$C_p = 0.12 \text{ Btu/lb}^\circ\text{F at } T = 80^\circ\text{F (See Appendix A.)}$$

Thermal conductivity measurements for the white alumina ceramic that were performed by Dynatech, Inc., provided the following values:

Temperature ($^\circ$F)	Thermal Conductivity (Btu/ft hr-$^\circ$F)
104	2.0
212	1.9
392	1.76
572	1.62
752	1.53
1004	1.45
392 (cooling)	1.79

(See Appendix C)

These measurements were performed with Comparative Apparatus that has a quoted accuracy of ±5%.

Thermal conductivity measurements performed by Material Consultants, Inc., are presented as follows:

Stycast 2762FT

Temperature (°F)	Thermal Conductivity (Btu/hr-ft°F)
150	0.967
250	0.930
375	0.969

Five samples were measured with less than one percent difference between any two samples. (See Appendix C.)

2.4.3 Flexural Strength Tests

ASTM procedures were used for these tests and produced the following results:

White Alumina Ceramic

Sample Number	Force at Time of Fracture (lb)	Flexural Strength (psi)
29	4,080	22,500
47	4,800	26,800
49	4,700	26,200

Cobalt Oxide/Alumina Ceramic

Sample	Forces at Time of Fracture (lb)	Flexural Strength (psi)
1	139	9550
2	136	9690

These values are lower than expected in that a value at least as great as Stycast was sought, which is approximately 18,000 psi. This lower value possibly resulted from the lack of complete vitrification which was observed after the models were tested. This can be easily increased by holding at the maximum temperature for a longer time.

Section 3
RESULTS AND RECOMMENDATIONS

Efforts expended under Contract NAS1-9983 have resulted in four materials recommended for use with the phase change testing technique. Studies have indicated that RTV-60 Silicone Rubber, Novimide 700, Stycast 2762FT, and alumina ceramic all have desirable properties for fabricating heat transfer test models, but they must be used within their respective limitations.

3.1 RTV-60 SILICONE RUBBER

General Dynamics Convair have been successfully fabricating RTV-60 Silicone Rubber models for the last several years. Many heat transfer tests have been conducted with this type model and they have yielded good results. RTV-60 has a very low diffusivity, making it an excellent material for measuring very low heating rates. On the other hand, this low diffusity makes it a poor material for measuring high heating rates, in that melt times are extremely short, which induces large errors in data reduction.

RTV-60 has a 700 to 800^{o}F capability and tends to soften as the maximum service temperature is approached. The material is weak structurally and must be bonded to steel inserts. Models require steel leading edges and nose tips. One familiar with model fabrication will recognize the increasing costs associated with increasing model complexity, particularly when metal work is concerned.

Lockheed spent very little time on this material since the limitations and applications are fairly well known. No data have been found on the material's thermal properties variation with temperature so comments on this characteristic would be unsubstantiated.

3.2 NOVIMIDE 700 (Ref. 7)

Novimide 700 was tested as potential model material with somewhat promising results, except for its inability to take temperature over 600°F. In its original state, Novimide 700 has a very low diffusivity on the order of RTV-60 Silicone Rubber. Many fillers were added in an attempt to increase its maximum service temperature, but all efforts resulted in poor results. The materials' $\rho C_p K$ product was the only property that was really changed. This was good, but it is felt that Novimide 700 in the original state offers a casting compound for fabricating test models that could be used for measuring low heating rates within allowable test times. In other words, Novimide 700 is a substitute material for RTV-60. It offers several advantages over RTV-60 that should be noted. First, it is castable, and can be machined, drilled, finished, and polished without special equipment. It has the strength necessary for model wings, fins, protrusions, etc., so that model failure due to loading would not be a problem if it is used within certain practical limitations. Steel inserts and supporting structures are not needed, although steel nose tips could be added if severe testing conditions are to be experienced. Models fabricated from Novimide would be relatively cheap for the reasons listed, and they could be cast with little effort.

The material's homogeneity was studied in detail. Samples which were cut and inspected showed no filler settling problem. This can be attributed to the very fine filler used by Isochem Resins Co. Settling problems have been noted with other materials. A brief discussion is presented later.

3.3 STYCAST 2762FT (Ref. 3)

Stycast 2762FT has proven to be the best commercial casting compound for fabricating heat transfer test models. This epoxy material meets all the contract objectives except that for temperatures. It has a +600°F capability that has been proven many times.

Stycast 2762FT is an epoxy resin loaded with a very fine alumina filler. It is a two part system having very desirable thermal and physical properties. The material can be cast in a closed mold and cured at elevated temperatures. Its thermal and physical properties can be fairly well duplicated. Polished Stycast 2762FT has the surface finish and homogeneity required, but it does require special tools for working. The cost of complex model shapes is largely influenced by the amount of hand-working required to produce a good model surface finish. Also, the types and location of sting supports are influencial on costs.

Stycast 2762FT models for various shuttle configurations have averaged from \$800 to \$2000 each with the price varying according to complexity. Also, increasing the number of models per configuration, brings the average cost down. Several models can be cast from the same mold without losing any tolerances.

3.4 ALUMINA CERAMIC/COBALT OXIDE ADDITIVE

Lockheed personnel have developed an alumina ceramic material, that when fired to the appropriate temperature, is a very strong, high-temperature material. This is highly suitable for casting wind tunnel models for testing with the phase-change technique. It meets all contract objectives in that it:

- Has a high temperature capability greater than 1000°F
- Is castable into complex shapes
- Is nonmetallic and has a fairly low thermal conductivity
- Has a $\rho C_p K$ product different than Stycast but no greater than a factor of two
- Has smooth non-porous surface finishes
- Has structural capabilities greater than Stycast
- Is economically feasible to fabricate.

A short discussion as to the mixture and as reiteration of its fabrication process is presented later. Alumina ceramic test models can be made a dark blue by adding cobalt oxide to the mixture. This was requested by the Langley Contracting Officer's Representative so melt contour resolution could be improved when it is used with white Tempilaq. Alumina ceramic models have been fabricated and are being tested at Langley Research Center by Mr. Charles Johnson.

3.5 FABRICATION PROCEDURES

A short description of the fabrication procedures for Novimide 700, Stycast 2762FT, and alumina ceramic models are offered so as to inform interested personnel as to time and effort required to fabricate a heat transfer test model.

3.5.1 Novimide 700 and Stycast 2762FT

Since Novimide 700 and Stycast 2762FT test models are fabricated with the same procedures, these steps are discussed only once.

As previously discussed, these materials are casting compounds that can be heated, mixed, and cast in a closed mold. The initial step in fabricating such models is to have a pattern model for mold preparation. This pattern can be made from wood, plastic, aluminum, or other material. Preferably from a cast-per-unit basis and scheduling this pattern should be provided by the contracting personnel. (Note that all part lines, scratches, or discontinuities on pattern models will show up on the cast product.) Lockheed has full capabilities in facilities and personnel for fabricating most model shapes.

Sting locations are determined for the proposed test models before mold preparation is begun. A rigid mold box is prepared from steel plating and the pattern model hung or suspended from the end, bottom or side, however the mold is to be prepared. Usually two or three piece molds will be prepared, but this is strictly determined through model shape and experience.

Silicone Rubber RTV 585, the material used for the mold, is a two part system. It is mixed according to its instructions, degassed, and then poured into the box. A 72-hour period is allowed for room cure. Liquid green soap used for a mold parting-agent, works extremely well. Additional mold parts are made as needed until the complete mold is prepared. Alignment pins, usually common stock material, are used to maintain all tolerances and to give a clean line with minimum flash. Usually a mold can be used to cast two to four models without any deterioration. The required parts for a complete fabrication of Stycast 2762FT models are shown in Fig. 31. The same applies for Novimide 700.

To clean flash lines, protrusions, or drill holes in Stycast 2762FT, grinding stones, wet and dry sandpaper, and carbon tools are needed. Only through use of Carborundum stones combined with wet and dry sandpaper, can Stycast 2762FT be finished and polished for testing. The required handwork is a major contributing factor to model cost, therefore emphasis must be placed on preparing molds that will require little hand finishing. Novimide 700 is also an abrasive material, but it can be worked by more conventional techniques, thereby reducing model costs.

3.5.2 Alumina Ceramic

For alumina ceramic models, as for other types, pattern models must be provided or fabricated for mold preparation. Alumina ceramic shrinks approximately 4%, therefore if scaled sizes must be adhered to, the pattern model must be fabricated oversize.

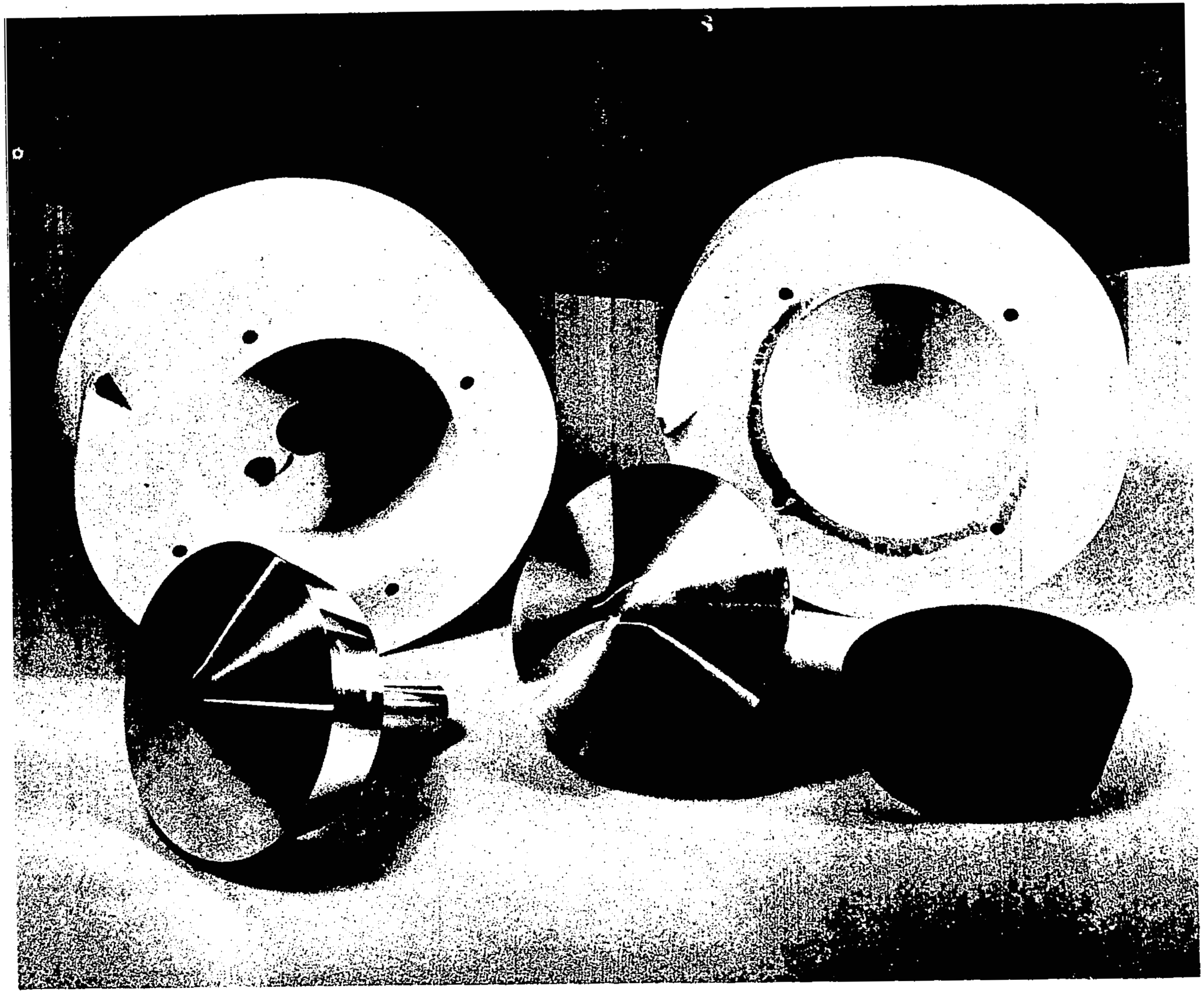

Fig. 31 - Fabrication of Stycast 2762FT Models

The alumina ceramic material is prepared according to the following composition:

	1752 gms	$A\ell_2O_3$
	264 gms	Wollastonite
	264 gms	Electro Chem
	50 gms	Talc (96-38)
	2330 gms	Total Powder
+	5%	Cobalt Oxide
	290 gms	Nalco 1030
	4 drops	Ultramet/2.0
	32 drops	Dow Corning Antifoam B Emulsion
	400 gms	Distilled Water

The mixture is placed in a standard ceramic mill jug and turned on a ball mill for approximately 18 hours. This turning mixes the ceramic to a homogeneous state with all particles completely wet. This gives a very nice pourable ceramic slip ready for casting.

Gypsum mold plaster is used to prepare the model mold as described in Section 2.4. The molds are dried for seven or eight days before casting, then assembled. After the inside of the mold cavity is wetted quickly, the slip is poured into the mold and is added continuously until a solid model is formed. The model is then removed and dryed, prefired for working, and then fired up to 2550°F.

Stings are attached only after the model is completely fired and finished. In case the surface finish is not as smooth as desired, it can be finished with carborundum stones and wet and dry sandpaper. This process is the same as that required by Stycast 2762FT models.

Stings are attached by potting with RTV585 Silicone Rubber. Other rubbers are available for this, but only RTV585 has been used. This is important in that the rubber can relieve an expansion by metal inserts and will help reduce model breakage from impacts. Several ceramic models are currently being fabricated under Contract NAS1-10937 whereby this sting attachment method will be used.

Figure 32 shows the various parts involved in the fabrication of alumina ceramic models.

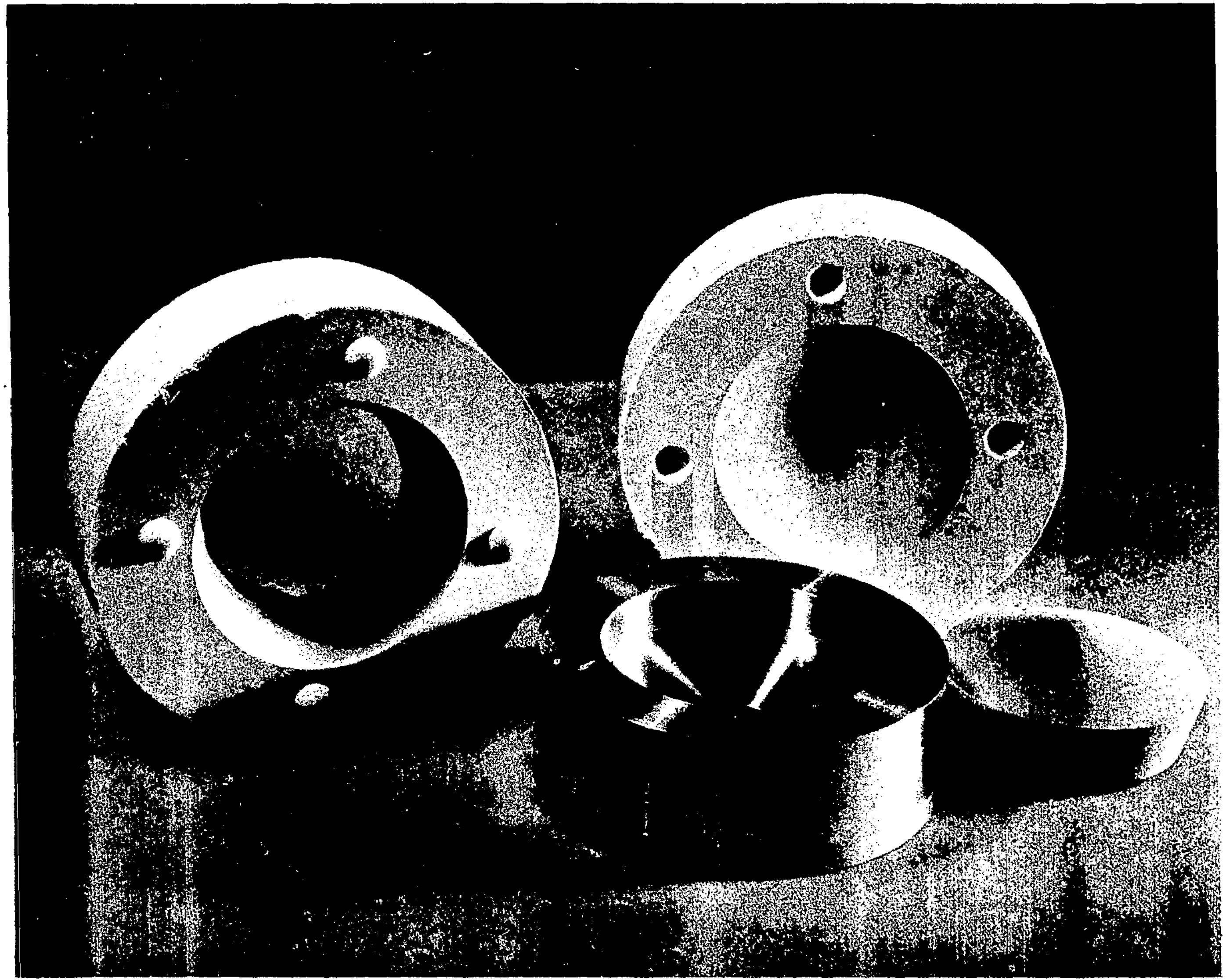

Fig. 32 - Fabrication of Alumina Ceramic Models

Appendix A

STYCAST 2762FT AND ALUMINA CERAMIC COMPARISONS AND FABRICATION TECHNIQUES

A.1 INTRODUCTION

As a result of this study it has been determined that two materials are best suited for aerothermodynamic model fabrication. These are Stycast 2762 FT and the alumina ceramic formulated during the study. This section presents a summary of comparisons of the characteristics of these materials as well as the fabrication techniques for both materials.

A.2 COMPARISONS OF MATERIAL CHARACTERISTICS

Table A-1 contains a list of characteristics of these materials with pertinent comments. This table is generally based on the basic requirements for the materials to be selected as stated for this contract and listed on page 6 of this document.

A.3 CERAMIC MODEL FABRICATION PROCEDURE

A.3.1 Introduction

Fabrication of ceramic heat-transfer test models by the Lockheed-Huntsville shops consists of five basic parts as follows:

- Preparing slip
- Preparing molds
- Pouring models
- Drying models
- Firing models.

Each of these is discussed separately in the following subsections.

Table A-1
COMPARISON OF MODEL MATERIAL CHARACTERISTICS

Material Characteristic	Stycast 2762FT	Alumina Ceramic
Castability/Machinability	Castable	Castable. Machinable in the prefired state after firing to approximately 1900°F.
Non-metallic	Yes	Yes
Low conductivity	Yes	Yes
Temperature resistance of the order of 600 to 1000°F	600°F capability	Can be used up to approximately 2000°F
Have a ρCK product different from Stycast by no less than nor greater than a factor of two	ρCK = 14.6 to 29.0 Btu^2/ft^4-$^\circ F^2$-hr ρ = 130.0 to 145.0 lbm/ft^3 C = 0.16 to 0.22 Btu/lbm-°F K = 0.7 to 0.9 Btu/ft-hr-°F *	ρCK_{white} = 69.5 Btu^2/ft^4-$^\circ F^2$-hr ρCK_{blue} = 37.9 Btu^2/ft^4-$^\circ F^2$ ρ_{white} = 183 lbm/ft^3 ρ_{blue} = 158 lbm/ft^3 C_{white} = 0.19 Btu/lbm-°F C_{blue} = 0.12 Btu/lbm-°F K_{white} = 2.0 Btu/ft-hr-°F *
Homogeniety	Good	Good
Smooth non-porous surface	Yes	Yes
Structural strength	Flexural strength = ~ 18,500 psi	White material flexural strength = ~ 26,000 psi; Blue material flexural strength = ~ 9,600 psi (note: this can be increased with an improved firing cycle – see page 2-67).
Economical to fabricate	Yes	Yes – materials cost about same as Stycast. Fabrication somewhat more expensive.
Dark color (added at time of contract extension)	Yes, black	Yes, medium blue.

*Approximate values at room temperature

A.3.2 Preparing Slip

The dry constituents given in Table A-2 are weighed and mixed. (Table A-2 also lists the supplier of each of these constituents.)

Next, the following liquids are weighed and mixed:

1. 290 grams Nalco 1030 colloidal silica, (obtained from Nalco Chemical Company, Chicago, Ill.)
2. 400 grams distilled water.
3. 2 drops Ultrawet 60-L, (obtained from Nalco Chemical Company, Chicago, Ill.) Use small eyedropper.
4. 32 drops Antifoam B Emulsion. (obtain from Dow Corning, Midland, Mich.)

Next, these liquids are mixed with the dry constituents. They are mixed by hand using a spatula. A stone, or ceramic, container is recommended rather than plastic or paper. This mixture is then turned on a ball mill for 16 to 20 hours.

A.3.3 Preparation of Molds

Rigid molds are made using gypsum plaster. Approximately 70 to 80 pounds of water per 100 pounds of dry plaster are used to make a pourable mixture. These are mixed by hand, degassed, and poured. The plaster sets up in 10 to 20 minutes depending upon the mass involved. Larger quantities set up faster.

Molds for complex shapes must be made in parts to allow removal of the model. Tincture of green soap, obtainable at local drug stores, is used as a mold parting agent. No mold release is used on the master model while making the mold. Care should be taken to make the mold such that it is easily taken apart after the model is poured since these models are quite fragile when in the green state. A hole must be provided for pouring the slip into the mold.

Table A-2

CERAMIC MODEL SLIP CONSTITUENTS

Weight (grams)	Material Name/Designation	Supplier
1752	Alumina R-122-DCM	Reynolds Company, Atlanta, Georgia
264	Wollastonite P-1	Hardwick Standard Chemical Company Atlanta, Georgia
264	Ground fused silica Glassgrain flour GP-7, Grade 1, 325 mesh	Glassrock Company, Atlanta, Georgia
50	Talc, Cercron, CF 96-38	Williams Color & Pigment Company, Pfizer Minerals, Pigments & Metals Division, N. Y., N. Y.
116	Cobalt Oxide 70/71%	Ceramic Color & Chemical Mfg. Co., New Brighton, Pa.

Molds must be thoroughly dry before use in pouring models. This takes one to two weeks at room temperature and humidity, depending on the size and shape of the mold. Heat and air circulation aid in drying the mold faster. Do not let the mold get hotter than 150°F.

A.3.3 Pouring Models

Before pouring, the slip should be degassed. This can be done using an ordinary vacuum jar. The slip is then poured into the assembled mold. Care should be taken to let the slip pour directly onto the bottom of the mold and build toward the top to help prevent air bubbles being trapped. Vibration at a low level can be used while pouring. However, the mold should not be vibrated after the model starts to harden. After the initial filling of the mold, the liquid level will decrease and the mold has to be refilled several times.

The model is then left in the mold for approximately one to two hours before taking the mold apart. The exact time depends on the model and mold size and shape and is determined only by trial and error.

A.3.4 Drying Models

Next, the model is removed from the mold and allowed to dry. Drying should begin at a high humidity level and gradually reduced until the model is completely dry. This usually takes 6 to 10 days depending on the model size and shape. The model should not be touched or handled until completely dry. It should be adequately supported. Air should be allowed to all surfaces for uniform drying.

A.3.5 Firing Models

Next, the model is fired. If there is any machining or working of the model, it should first be prefired. This is accomplished using the Paragon Kiln, firing to approximately cone 019. After machining, the model is fired to 2600°F in the Blue-M furnace at 50°F per hour, holding four hours, and then cooling at 50°F per hour. Care should be taken to place the model such that when it softens at the high temperatures it does not sag. Care should also be exercised to minimize temperature gradients in the model during firing. This is done by enclosing it with a radiation shield such as a ceramic cylinder or by burying it in a high-temperature setting sand.

A.4 STYCAST MODEL FABRICATION PROCEDURE

A.4.1 Introduction

Fabrication of Stycast models by the Lockheed-Huntsville shops consists of four basic parts:

- Preparing molds
- Preparing Stycast Resin/Catalyst mixture
- Pouring models
- Curing models.

Each of these steps is now discussed separately.

A.4.2 Preparing Molds

Flexible room temperature vulcanizing (RTV) rubber molds are used for pouring these models. Dow Corning RTV 585 is used with the catalyst as furnished by the supplier. Resin/catalyst ratio used is 10 to 1. The mixture is degassed with a vacuum jar before pouring. Molds are allowed to fully cure for several days before using. The larger the mold, the longer the cure time must be. Liquid soap is used as a parting agent between mold parts. Usually, a two-piece or three-piece mold is used to allow removal of the finished model. A pour hole or injection hole is left for pouring the Stycast into the mold. Alignment pins are used to hold the parts together.

A.4.3 Stycast Resin-Catalyst Mixture

Stycast 2762 FT is used in an "as received" condition, i.e., no additional filler is added. The resin and catalyst are mixed in accordance with the manufacturer's instructions which come with the product. The resin and catalyst are usually warmed and thoroughly stirred before weighing and mixing. After mixing, the Stycast is degassed before pouring.

A.4.4 Pouring Models

Pouring can be done either through a pour hole or by injection, depending upon the configuration. The mold should be tilted to allow air to escape if necessary. Vibration can also be used to help eliminate any bubbles in the model. The Stycast should be poured slowly to give a uniform buildup in the mold.

A.4.5 Curing Models

Curing consists of two parts — precuring and post-curing. The precuring is done with the model in the mold for 16 hours at 160°F. Post-curing is done with the model out of the mold. The post-cure cycle consists of six hours at 200°F, six hours at 300°F, and 12 hours at 350°F. The model is then ready for possible machining, final finishing, and then testing.

Appendix B

THE DESIGN AND OPERATION OF A STEADY STATE CALORIMETER FOR MEASURING SPECIFIC HEAT AT CONSTANT PRESSURE

A specific heat calorimeter was developed during this study to determine accurately and inexpensively the specific heat at constant pressure (C_p) of solid model materials as a function of temperature. The principle of operation of the calorimeter is quite simple. The sample is placed inside an aluminum container of known mass and specific heat. The container is insulated with multilayer insulation (MLI) consisting of alternate layers of aluminum foil reflective shields and polyester paper spacer sheets. The container is then placed in a vacuum chamber which is evacuated to a pressure less than 1×10^{-5} mm Hg. The combined effect of the MLI and vacuum is to reduce the heat losses from the calorimeter to a minimum.

A known quantity of heat energy is added to the sample and container via a 50 ohm Minco foil resistance heater. A monitored Variac 0-120 volt alternating current source is used for power. After the sample and container have reached thermal equilibrium, the following energy balance is applicable:

$$Q_{HTR} = M_{Al}\,\Delta h_{Al} + M_{sample}\,\Delta h_{sample} + Q_{losses} .$$

Solving for Δh_{sample} yields:

$$\Delta h_{sample} = \frac{Q_{HTR} - Q_{losses} - M_{Al}\,\Delta h_{Al}}{M_{sample}}$$

The quantities on the right side of the equation are calculated using the following relations:

$$Q_{HTR} = \int_{t_i}^{t_f} IV\,dt$$

where t is time,

I is current, and

V is voltage.

$$Q_{losses} = \int_{t_i}^{t_f} \dot{Q}_{losses} \left(T(t)\right) dt$$

where Q_{losses} is a calibrated function of temperature, T, which is a known function of time, t, in a particular test.

M_{Al} is the mass of the aluminum container determined from mass balance measurements.

$$\Delta h_{Al} = \int_{T_i}^{T_f} C_{p_{Al}} \, dT$$

where $C_{p_{Al}}$ is the known specific heat of the aluminum container.

M_{sample} is the mass of the sample determined from mass balance measurements.

Several data points are obtained over the room temperature to 375°F temperature range yielding the sample specific enthalpy as a function of temperature, based on the datum that h = 0 at T = room temperature.

A plot of h_{sample} vs T is then constructed from the data (Fig. B-1).

Since:

$$C_p = \left(\frac{\partial h}{\partial T}\right)_p = \left.\frac{dh}{dT}\right|_{p\,=\,\text{const.}}$$

graphical differentiation yields $C_{p_{\text{sample}}}$ as a function of temperature (Fig. B-2).

In the test system, temperature measurements are made of the sample and of various points of the container using copper-constantan absolute thermocouples which are referenced to a Con-Ohmic 150°F thermocouple reference junction. Voltage readings are made with a Fluke differential microvolt null meter.

Regarding accuracy, a sample of aluminum alloy 6061-T6 was tested for specific heat at approximately 100°F and the measured value was within 2% of the reported values in the literature.

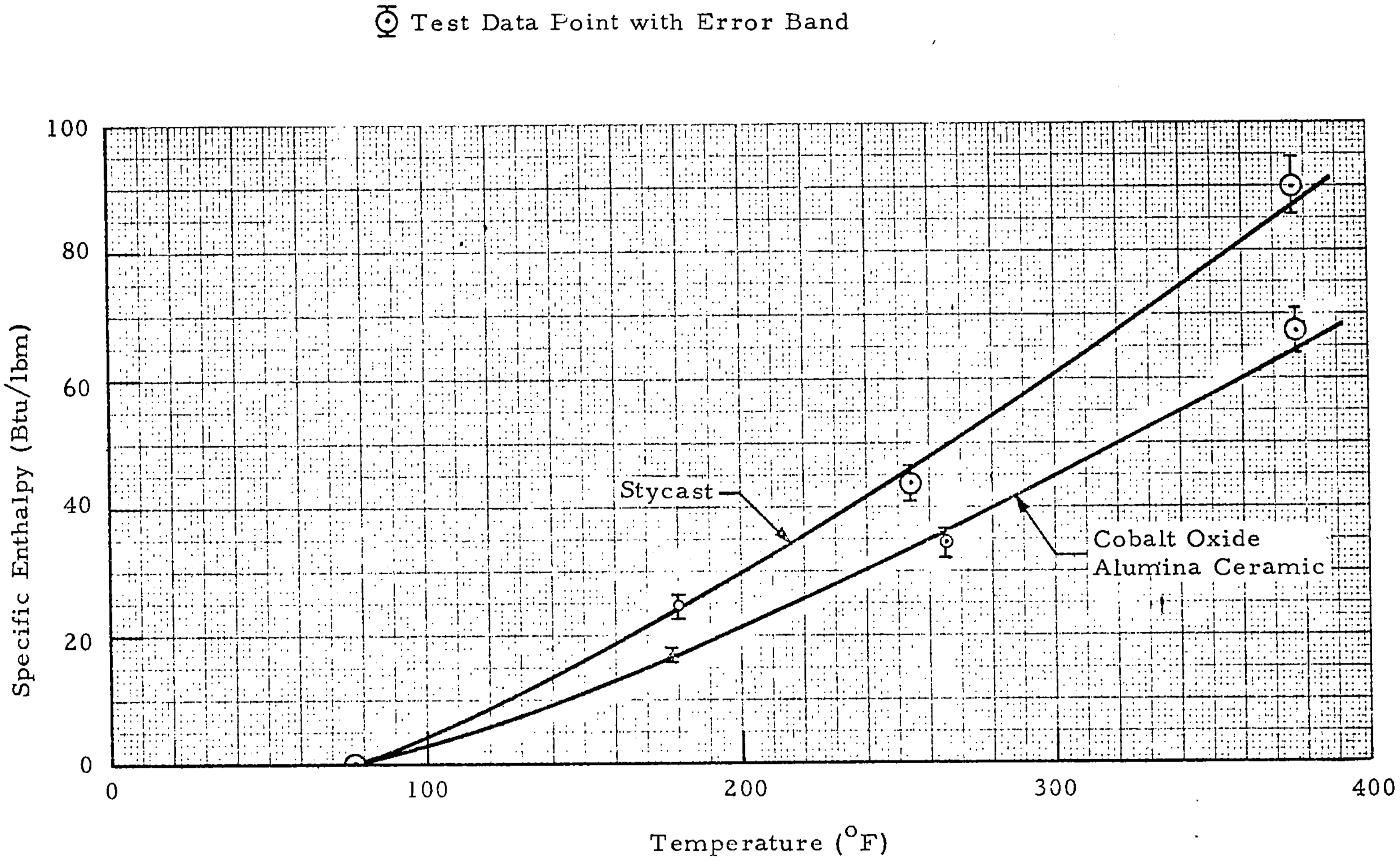

Fig. B-1 - Enthalpy vs Temperature for Two Model Materials

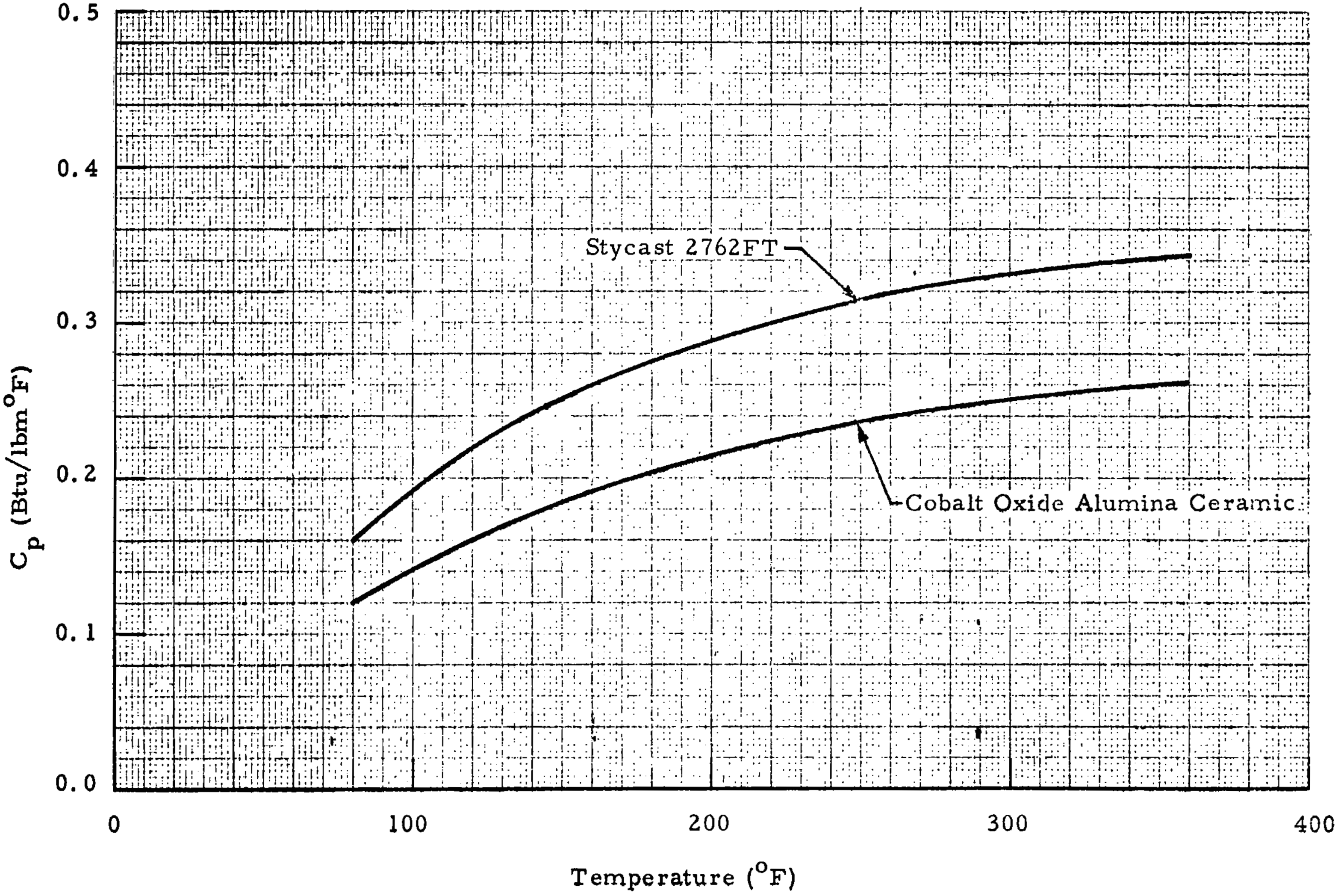

Fig. B-2 - Specific Heat at Constant Pressure (C_p) for Two Model Materials

Appendix C

THERMAL CONDUCTIVITY MEASUREMENTS

Report on

THE THERMAL CONDUCTIVITY OF A CERAMIC MATERIAL

The material supplied for evaluation over the approximate temperature range 40 to 540C was a high alumina ceramic. It was supplied in the form of a slab approximately 63 mm square and 44 mm thick with flat smooth faces. A groove 0.8 mm wide and deep was cut across the surface of each face. There was an indication of a crack in the sample along and in from one edge.

For the purpose of the test, fine gauge thermocouples were cemented into the grooves in the surface of the sample. The sample was placed between two similar samples of known thermal conductivity with the same type of thermocouple instrumentation. To minimize contact resistance each surface was coated with a thin layer of silicone grease. The composite specimen was placed between the plates of an upper heater and a lower heat sink and a reproducible load was applied to the top of the complete system. A guard tube which could be heated was placed around the system and the whole of the interspace and surrounds filled with a good heat insulating powder. The apparatus was evacuated and backfilled with dry nitrogen gas. By means of suitable adjustments to the power in the various heaters and of the cold sink temperature, a steady distribution was maintained in the system and undue radial heat loss prevented by keeping the guard tube at a temperature close to the average temperature of the sample. At equilibrium conditions the temperatures at various points in the system were evaluated from the thermocouple readings and the heat flow in the specimen derived in terms of that flowing through each standard material.

The thermal conductivity was calculated from a knowledge of the mean energy, the temperature difference across the sample and the known dimensions.

The results obtained for the particular sample tested are given in the following table.

Thermal Conductivity of a Ceramic Material (Test Density 2460 Kg m^{-3})

Mean Temperature, C	Thermal Conductivity	
	W m^{-1} $degK^{-1}$	Btu ft^{-1} hr^{-1} $degF^{-1}$
40	3.45	2.0
100	3.3	1.90
200	3.05	1.76
300	2.8	1.62
400	2.65	1.53
540	2.5	1.45
200 on cooling	3.1	1.79

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