DEFENSE DOCUMENTATION CENTER

FOR

SCIENTIFIC AND TECHNICAL INFORMATION

CAMERON STATION, ALEXANDRIA, VIRGINIA

6335

405 711

405711

405 711

BERYLLIUM OXIDE RADOME DEVELOPMENT
Final Engineering Report
Contract NOw-62-0592-c
Bureau of Naval Weapons
Period 15 March to 31 December 1962
National Beryllia Corp.
Haskell, New Jersey

BERYLLIUM OXIDE RADOME
DEVELOPMENT

Contract NOw-62-0592-c
Bureau of Naval Weapons
Washington 25, D. C.

Final Engineering Report
For the Period 15 March to 31 December 1962

National Beryllia Corp.
Haskell, New Jersey

Contract NOw 62 0592-c

Personnel

PROJECT MANAGER   - P. S. Hessinger

PROJECT ENGINEER   - K. H. Styhr

RESEARCH DIRECTOR - Dr. E. Ryshkewitch

TABLE OF CONTENTS

LIST OF ILLUSTRATIONS

LIST OF TABLES

(1.0) INTRODUCTION

1.1 Purpose of the Program

The utilization of ceramic radomes in high-speed missiles and air-craft has offered a means of overcoming a wide range of thermal and mechanical problems encountered by reinforced plastic structures. Among these problems are:

(a) High-temperature resistance;

(b) Rain-erosion resistance; and

(c) Dielectric homogeniety.

Ceramic radomes, on the other hand, have disadvantages such as higher weight, low impact resistance and high cost of fabrication. The latter factor is one of the most critical and is usually the result of the need for excessive grinding to reach close dimensional tolerances and the relatively low yield of acceptable pieces under production conditions.

Radomes have been fabricated from a number of ceramic materials including aluminum oxide, devitrified glass, and fused silica with relatively good success. Aluminum oxide radomes have been formed by slip-casting, flame-spraying and, most commonly, by isostatic pressing. Devitrified glass radomes are formed by relatively conventional glass casting techniques while fused-silica radomes are generally slip-cast to the required shape.

Each of these methods results in a preformed shape which must subsequently be heat-treated to mature the body, and then must be ground to reach the desired dimensional tolerances. Grinding costs can, in most cases, be reduced by machining the un-fired or pre-fired blank to tolerances approaching the final dimensions, with final grinding being done after high-firing.

Beryllium oxide holds strong potential as a radome material because of its unique combination of physical properties. In spite of the fact that it is more expensive than most ceramic radome materials and has handling limitations due to the toxicity of its powder form, it has characteristics which may offer the solution to several present and future radome problems.

Beryllia may be compared, in general, with other types of radome materials by examining the data in Table I. From the properties it is quite obvious that BeO offers the advantage of low density, high strength, and heat shock resistance in one material. As with the other ceramics, however, it must be fabricated at high-temperatures and diamond-ground to final dimensions. The objectives of this program are therefore:

  (A) To investigate and evaluate the various grades of BeO available for radome fabrication.

  (B) To determine the optimum compositions and material preparation methods for forming.

TABLE I
RADOME MATERIALS

Physical Property		Reinforced Plastic	Devitrified Glass	Fused Silica	Aluminum Oxide	Beryllium Oxide
1. Composition		Organic Resin and Glass Fibers	Glass and Crystalline Phase	SiO_2	Al_2O_3 (97%)	BeO (99.5%)
2. Safe Temperature Limit °F.		250° – 500°		1800°	2800°	4000°
3. Density g/cc		1.60 – 1.90	2.60	2.2	3.70 – 3.90	2.85 – 2.90
4. Modulus of Rupture, psi		30,000 – 50,000	20,000	15,000	30,000–60,000	20,000–40,000
5. Thermal Expansion in/in, °F	100°F	5×10^{-6}	4×10^{-6}	0.3×10^{-6}	3.5×10^{-6}	3.2×10^{-6}
	1000°F	---	2.7×10^{-6}	0.3×10^{-6}	4.3×10^{-6}	6.5×10^{-6}
6. Thermal Conductivity $BTU/FT^2 hr°F/in.$	100°F	2.9	14.5	10	230	1700
	1000°F	---	---	15	80	900
7. Thermal Shock Resistance		---	Good	Excellent	Fair	Excellent
8. Dielectric Constant 10^{10} cps	100°F	3.6	5.5	3.8	9.0	6.5
	1000°F	---	5.8	3.8	9.4	6.9
9. Loss Tangent 10^{10} cps	100°F	.001	.0003	.0001	.0005	.0004
	1000°F	---	.003	.0005	.002	.0005

(C) To utilize pressure casting as a means of forming
     BeO and determine the material and process parameters
     which control the properties of the finished pieces.

(D) To fabricate prototype radomes using the materials
     and process information established, so as to reduce
     final grinding to a minimum.

1.2 Phases of the Program

The goals of this program were achieved through the pursuit

of four distinct phases. A brief description of the work involved

in each of the phases is given in the following paragraphs. The

phases are discussed in detail in Section 2.

Phase I - Raw Materials

A number of types or grades of beryllia raw material, some

domestic and some imported, of varying chemical purity, particle

size and shape, cost, thermal history, and method of preparation

are available at this time.

The first phase of the program is an evaluation of beryllia

raw materials culminating in the selection of one to be used for

experimental radome fabrication. Studies are included of the

treatment of the as-received raw material selected in efforts to

improve its particle size distribution, sinterability, and other

parameters in order to optimize the final characteristics of the

completed radome.

Phase II - Slip and Body Preparation

The second phase of the program is directed towards the

preparation of a slurry or ceramic slip of beryllium oxide in a

suitable suspending medium which may then be formed into the desired radome shape. Variables such as composition, solids content, viscosity, hydrogen ion concentration (pH), and defloculating agents are evaluated and parameters selected for the preparation of a suitable material.

Phase III - Radome Molding Experiments

This phase of the program involves the evaluation of previous pressure casting experiments and the development of an experimental pressure casting facility for use with the potentially hazardous beryllium oxide materials.

This phase includes the design and construction of a pressure slip casting facility for the fabrication of prototype beryllium oxide radomes. A number of radomes were cast and fired with the aim of approaching final dimensions with a minimum of grinding.

(2.0) TECHNICAL DISCUSSION

2.1 Phase I - Beryllia Raw Materials

There are a number of types of beryllia available of varying grades of chemical purity, particle size, shape, and cost. Some of these are considered "sinterable" and some "unsinterable". However, this factor is, in actuality, an expression of the behavior of the powder under certain conditions of temperature and furnace atmosphere, and even the most "unsinterable" BeO powders can, under proper conditions, be fired into dense ceramic bodies. In general, the cost of BeO is directly related to chemical purity and sinterability. Present prices range from approximately $12.00 per pound as high as $25.00 per pound depending upon the user's requirements.

Table II lists several of the commercially available BeO powder types. In addition, there are several imported grades becoming available from French and Japanese sources which indicate good purity and reasonable fabricability. Table III shows the typical chemical analyses of these ceramic beryllia powders, and Table IV shows bulk density values obtained on powder samples.

In considering the use of these materials for radome fabrication, the following factors were considered:

      (a) Chemical purity

      (b) Cost and availability

      (c) Uniformity of density

TABLE II

BERYLLIA POWDER SOURCES

Powder Nomenclature	Vendor	Ore Source	Processing Operation
Grade #1	Beryllium Corp.	Imported	Domestic
GC	Brush Beryllium Corp.	Imported	Domestic
UOX	Brush Beryllium Corp.	Imported	Domestic
AA	Mineral Concentrates, Inc.	Domestic	Domestic
AAA	Mineral Concentrates, Inc.	Domestic	Domestic

TABLE III
BeO TYPICAL CHEMICAL ANALYSIS

(Impurities in ppm)

Constituent Metallic	Beryllium Corp. Grade #1	Brush GC	Brush UOX	Mincon AA	Mincon AAA
Al	20	25	40	150	10
B	3	1	1	--	3
Ca	50	30	30	50	5
Cd	.5	ND	ND	--	1
Co	1	1	1	5	1
Cr	30	4	10	5	1
Cu	30	2	2	--	1
Fe	30	40	30	20	25
Li	1	1	1	5	3
Mg	10	20	40	50	5
Mn	10	2	4	20	5
Na	95	15	20	50	50
Ni	25	5	3	5	5
Si	120	30	45	250	10
S	--	--	700	100	50

TABLE IV
BeO (As-Received) Powder
Bulk Densities

Powder	No. Samples	Bulk Density g/cc	
		Min.	Max.
Berylco #1	30	0.14	0.22
Brush GC	40	0.13	0.21
Brush UOX	40	0.17	0.22
Mincon AA	5	---	---
Mincon AAA	5	---	---

(d) Powder handling characteristics

(e) Sintering behavior

Examination of each type of material indicated that as far as chemical purity was concerned, they could all be considered relatively pure and suitable for producing a 99.5% pure body with the possible exception of Mincon AA which is somewhat high in silicon and aluminum. The cost of the Brush UOX and Mincon AAA material is approximately 50% higher than the others and all are available commercially.

For the purposes of this program, it was decided to evaluate three of the materials and select one of them on the basis of composition and sintering studies as a standard for the continuing radome development. Sintering tests were therefore conducted on the GC, UOX and AA grades. Of these, on the basis of firing results, cost and uniformity, GC was selected for the development work as described in the following section.

In high-purity oxide materials thermal treatment of a portion of the as-received powders and subsequent blending with un-fired powders can be utilized as a method for controlling the desired range of particle size distribution. The following graph (Figure 1) shows particle size distribution curves, by sedimentation, for fired and unfired GC material along with calculated curves for blends of the two powders from the same lot. However, fired density determinations as shown in Table VII on electric fired blend specimens did not show the characteristic maximum of density expected with a blend of coarse and fine

FIGURE I
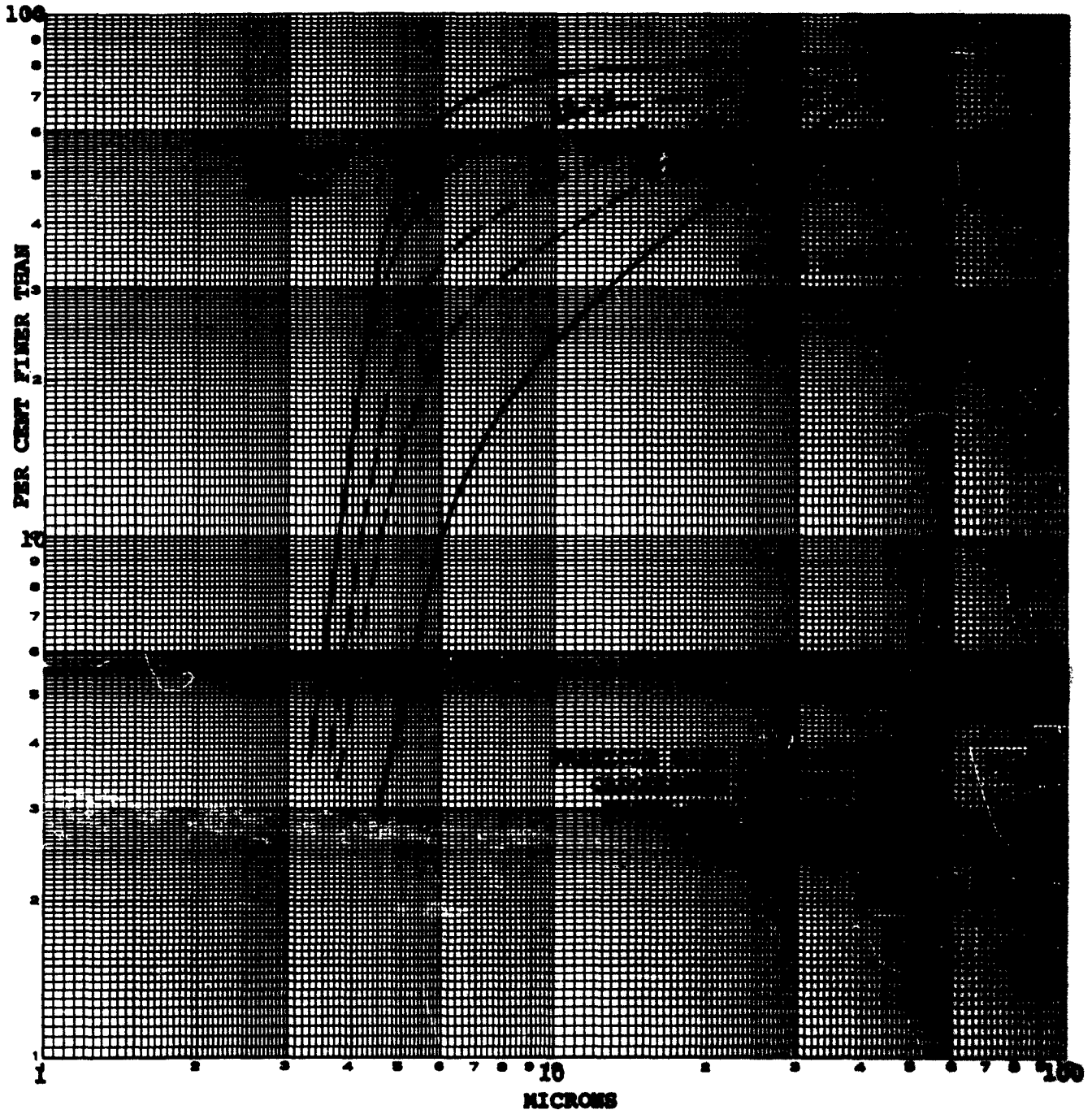
powders, raising a question as to the measurement technique.

A recent research report describing a comparative study of two grades of BeO shed some additional doubt on the particle size measurement technique. Reeve (10) estimated particle size by five different methods:

(1) An optical microscope

(2) X-ray line broadening techniques

(3) Surface area measurements by the BET method

(4) Electron microscopy

(5) Sieve analysis

His results indicated that "Particles" in the 1 to 20 micron range are really composed of sub-micron sized crystallites not resolvable using optical wave lengths and not readily deagglomerated under conditions of the hydrometer methods.

For these reasons a brief comparison of particle size determination method was initiated.

Figure 2 shows three curves for the same-as-received GC beryllium oxide material wherein particle size range is measured by three different methods

(1) Optical microscope

(2) The hydrometer method

(3) A commercial resistance change particle size determination method.*

It is apparent from the wide range of these particle size distribution curves that little correlation between these three

*The Coulter Counter, Coulter Electronic Sales Company

PARTICLE DIAMETER IN MICRONS
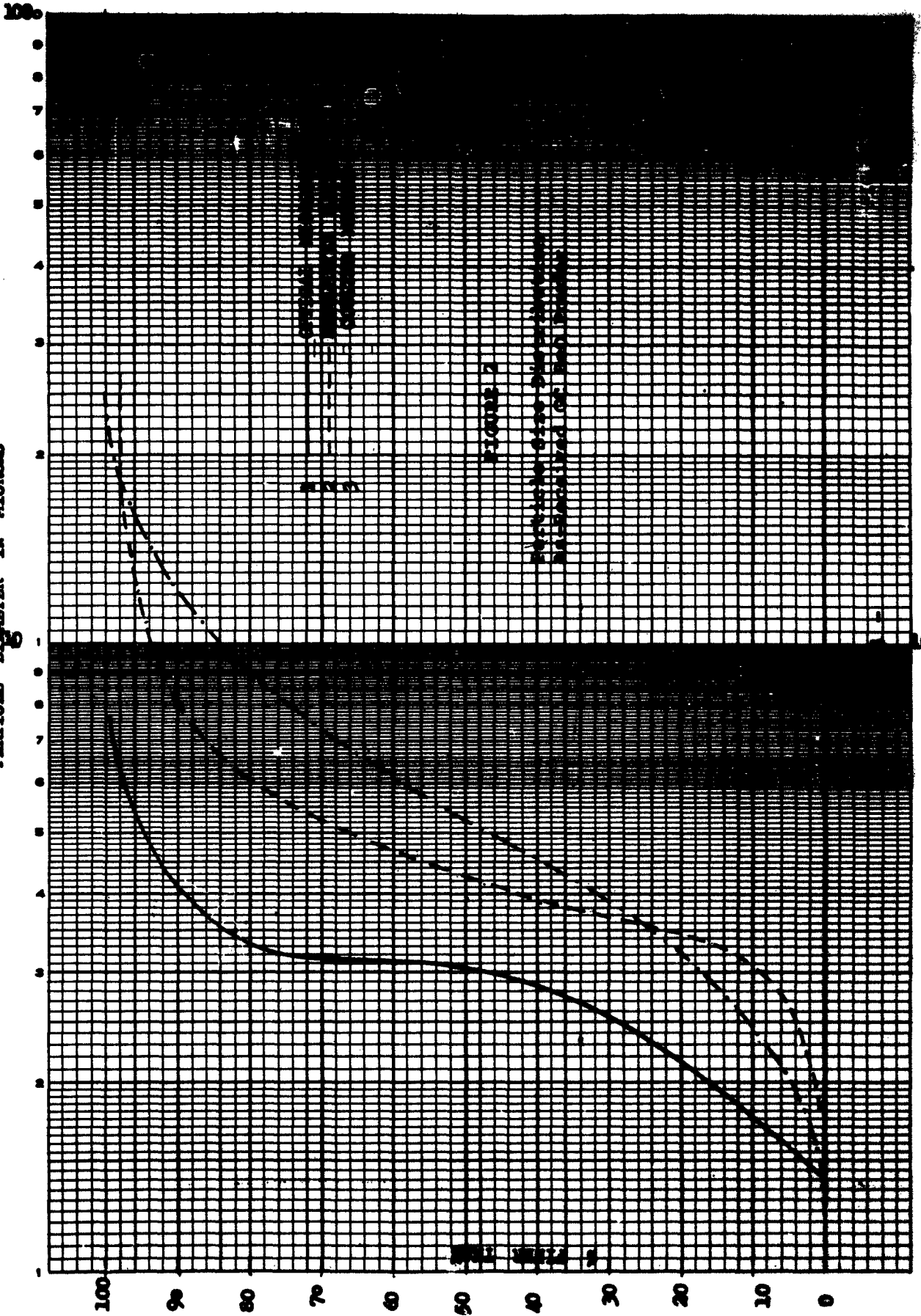
FIGURE 2

PARTICLE DIAMETER IN MICRONS

methods exists. Further, it seems apparent that all three of
these methods measure varying degrees of agglomeration, not
ultimate particle or crystallite size. Several additional types
of particle size measurement were given consideration in the
program. One is an air sedimentation method in which the particles
are first deagglomerated at supersonic velocities.** Another
is a measurement of surface area by the BET nitrogen absorption
method. A third is the Bahco Centrifical Micro Particle Size
Classifier. All of these measurements were hampered somewhat
by the precautions that must be taken when handling the beryllium
oxide powders, and some cases were abandoned due to toxicity
considerations.

A relatively simple comparative evaluation of the particle
size distribution of powders may be obtained using the bulk
density test. In this method, the powder is placed in a supported
glass funnel, aligned above a graduated cylinder of, for example,
50 cc and then by tapping or stirring is made to flow into the
cylinder until a measurable volume is reached. This amount
of powder is then weighed and bulk density determined by a simple
calculation. Chemically prepared un-calcined BeO powders usually
show values of 0.15 to 0.25 grams per cc, while calcined or fired
powders give higher readings due to their larger crystal size.
These usually range from 0.35 to 0.50 grams per cc. The calcining
of BeO powders prior to forming and firing is done to increase

**Micromerograph, Franklin Electronics Company

their crystal size from a range of 1-10 microns up to the 10-40 micron size. This calcining is usually done at temperatures of from 800°C to 1450°C for several hours and produces a powder of coarser grain and better handling characteristics.

In the control of particle size distribution, a wide range of distribution curves can be obtained by blending as-received and calcined powders. Particle size distribution can be quite accurately measured using a number of techniques. For the purposes of this program a sedimentation method was finally selected which is a modification of the ASTM test procedure D422-60T _Tentative Method for Grain-Size Analysis of Soils_. The procedure consists of dispersing 60 grams of BeO powder in 100 ml of water using about 1 per cent of a dispersant such as Darvan No. 7 Poly-electrolyte solution. The suspension is then agitated and the graduate placed in a constant temperature bath of 30°C. A hydrometer is placed immediately in the suspension and specific gravity readings taken at times of 1, 2, 5, 10 minutes etc., up to about 8 hours. From the dimensions of the hydrometer a depth L may be calculated for the particle size determined by the formula:

$$L = L_1 + 1/2 \ h - \frac{V_b}{A}$$

Where:

L_1 = distance from top of bulb to hydrometer reading, cm
L = effective depth, cm
h = overall length of hydrometer bulb, cm
V_b = volume of hydrometer bulb, cm^3
A = cross-section area of graduate, cm^2

A calibration curve is then prepared of L vs. specific gravity for a given hydrometer.

From the application of Stokes Law for sedimentation the average particle size at depth L can be caluculated using the formula:

$$d = \frac{18}{(D-1)g} \cdot \frac{L}{t}$$

Where:

d = average particle size, mm
D = specific gravity of particles
L = effective depth, cm
t = time, seconds
g = gravity constant, 980 cm/sec^2

From the actual specific gravity D of the particles, a curve can be plotted of specific gravity of the suspension vs. concentration of powder. This can be expressed as per cent settled or conversely, as per cent finer than a calculated particle diameter d. A typical reading is calculated as follows using as-received GC grade BeO powder.

Reading

Time (t) : 1800 seconds
Specific gravity of suspension: 1.0320
Hydrometer No. 133097

Calculations

(a) From graph: L = 15.4 cm at 1.0320 s.g.
    For BeO powder D = 3.008 g/cc
    therefore: d = 9.7 microns

(b) From graph at specific gravity 1.0320 the per cent settled is 21% or 79% of the particles are finer than 9.7 microns.

From this data curves can be plotted for BeO powders showing the entire range of sub sieve particle sizes.

While the sedimentation method was considered adequate for this exploratory work considerable additional effort would be required to develop a dependable and reproducible method for measurement and control of BeO particle size.

PHASE II

(2.2) COMPOSITION AND SLIP PREPARATION

Selection of the proper BeO body for pressure casting of radomes was based upon the requirements for maximum thermal conductivity, maximum mechanical strength, and minimum dielectric loss tangent. These requirements dictate the need for maximum purity in the body and careful control of grain size in both the starting material and the final fired body. The relationship between the particle size distribution of the unfired powder and that after firing is not a simple one. In general, however, it may be stated that the finer the grain size of the starting BeO powder, the more rapid the rate of grain growth at high temperatures. This may be somewhat obvious when one considers surface diffusion or evaporation-condensation as material transport mechanisms during sintering. Both of these processes are dependent on surface area which increased greatly with decreasing grain size. Other factors which influence the rate of grain growth are temperature, the presence of chemical impurities and water vapor, and oxidizing or reducing conditions in the furnace atmosphere. In BeO water vapor is a particularly critical factor, and for this reason, some data has been included comparing sintering in gas-fired and electric-fired furnaces where the differences in moisture content are sufficient to cause major variations in physical properties.

(A) Composition

In order to obtain maximum thermal conductivity and re-
fractoriness in the body, it was decided to try to maintain at
least 99.5% purity in the finished ware. In producing such a
high-purity material, the problem of reaching high density, that
is - freedom from porosity, becomes increasingly difficult. The
general approach is to utilize small percentages of inorganic
chemical additives which aid sintering and control grain growth
at elevated temperatures. Such additives must be very carefully
selected on the basis of sintering tests and determination of
their effect on mechanical and dielectric properties.

For the purpose of this study, several additives were selected
on the basis of their refractoriness and low dielectric loss
characteristics. These were $Al2O_3$ aluminum oxide, MgO magnesium
oxide, $MgAl_2O_4$ spinel, and ZrO_2 zirconia. No alkalis or siliceous
additives were used because of their known deleterious effect
on loss tangent at elevated temperatures. The additives were
introduced in amounts .125 and .250 mol per cent to a standard
BeO GC grade calcined powder. The powder used had a bulk density
of 0.32 - 0.34 g/cc. A mill batch of 100 grams was used and the
additive introduced as the powdered oxide. A quantity of 200 ml
of water was added and the batch milled for two hours. The batch
was then removed from the mill, vacuum filtered and then dried at

100°F for 48 hours. The dried cakes were then crushed and a small quantity of gum solution added as a temporary binder. The powder was then put through a 20 mesh screen and compacted in a steel die at 12,000 psi. The samples were then ready for firing tests. The following Table (V) lists the experimental radome composition batches tested.

(B) **Sintering Tests**

Densification of the ceramic body is dependent not only on particle size distribution and chemical purity, but also on the chemistry of the furnace atmosphere. For this reason sintering tests were conducted in three furnace conditions:

(1) Gas-fired; water-vapor present and oxidizing

(2) Electric-fired; essentially dry and oxidizing

(3) Hydrogen; dry and reducing

The samples were fired to maximum density and then evaluated for shrinkage, density, grain size and modulus of rupture. The results of the tests are shown on Table VI.

Examination of the sintering test data indicates that good densities can be obtained in gas-firing with most compositions. Fair results were obtained in hydrogen firing, and poor results in electric oxidizing firing. The latter, however, is the most desireable from the point of radome firing because of the ease of temperature control absence of water vapor, and freedom from

TABLE V

EXPERIMENTAL RADOME BATCHES
BeO-Grade GC (Calcined 1450°C)

Batch	Additive	Mol %	Wt. %
RX1	None	--	--
RX2	Al_2O_3	.125	0.5
RX2A	Al_2O_3	.250	1.0
RX3	MgO	.125	0.2
RX3A	MgO	.250	0.4
RX4	$MgAl_2O_4$	.125	0.53
RX4A	$MgAl_2O_4$	.250	1.07
RX5	ZrO_2	.125	0.62
RX5A	ZrO_2	.250	1.23

TABLE VI

RESULTS OF BeO RADOME COMPOSITION
SINTERING TESTS

Composition No.	Green Density g/cc	Hydrogen - 3000°F.		Electric-Fired Air - 3000°F.		Gas-Fired Air - 3400°F.	
		Shrinkage %	Density g/cc	Shrinkage %	Density g/cc	Shrinkage %	Density g/cc
RX 1	1.84	8.6	2.78	2.5	2.00	12.7	2.83
RX 2	1.87	8.4	2.66	2.4	1.98	11.6	2.81
RX 2A	1.88	8.8	2.82	2.4	2.01	11.9	2.83
RX 3	1.90	10.5	2.73	9.5	1.90	12.0	2.85
RX 3A	2.10	11.3	2.82	7.5	1.89	12.4	2.87
RX 4	1.73	--	--	14.2	2.68	15.8	2.83
RX 4A	1.71	--	--	16.1	2.87	16.4	
RX 5	1.88	9.2	2.71	1.89	2.27	11.3	2.80
RX 5A	1.89	9.9	2.70	1.88	2.29	11.7	2.83

distortion. For this reason, additional effort was placed on developing a composition suitable for electric firing using GC grade material. This was achieved by preparing blends of as-received and calcined powder so as to achieve a controlled particle size distribution. While the $MgAl_2O_4$ RX4 body showed good results, it utilized over 1.0 per cent additive which was undesirable. Therefore, the RX3A body was selected for additional radome work. A second series of experimental RX3A bodies was then made up comparing various BeO blends using GC and UOX grade materials as follows:

Batch No.	Blend
RX3B	75% Calcined GC/25% of as-received GC
RX3C	50% Calcined GC/50% as-received GC
RX3D	25% Calcined GC/75% as-received GC
RX3E	100% as-received GC
RX3BU-1	100% UOX Calcined (1250°C)
RX3BU	75% Calcined UOX/25% as-received UOX
RX3CU	50% Calcined UOX/50% as-received UOX
RX3DU	25% Calcined UOX/75% as-received UOX
RX3EU	100% as-received UOX

The firing tests on these bodies is shown in Table VII. It is interesting to note that the utilization of controlled particle size blends allowed densification of GC material at 3000°F in electric firing. The UOX material, on the other hand, while densifying at 3000°F, when fired to 3400°F blistered badly and was quite non-uniform. Attempts were also made to pre-fire similar pieces to 2200°F; however, this was ineffective. It is

TABLE VII

RESULTS OF BeO RADOME CONTROLLED
PARTICLE SIZE SINTERING TESTS

Batch No.	Gas Firing 3400° F. Oxidizing		Electric Firing 3000°F. Oxidizing	
	Shrinkage %	Density g/cc	Shrinkage %	Density g/cc
GC Series				
RX3	12.4	2.87	9.5	2.50
RX3B	13.2	2.86	11.7	2.69
RX3C	14.7	2.89	13.9	2.76
RX3D	16.0	2.89	15.7	2.82
RX3E	16.4	2.81	17.3	2.87
UOX Series				
RX3BU-1	19.7	2.59*	18.4	2.87
RX3BU	20.1	2.85	17.9	2.89
RX3CU	19.7	2.56	18.6	2.86
RX3DU	19.8	2.59	18.3	2.85
RX3EU	19.9	2.72	17.8	2.86

*Samples bloated badly internally in this series.

suspected that this phenomena may be due to residual sulfur which is indicated in the raw material analysis. In general, the GC material appeared the most stable and uniform and was used for subsequent experimental work.

Phase II - <u>Slip Preparation</u>

The basic body RX3E selected from Phase I data was prepared in ceramic slip form under a variety of conditions. To determine quantitatively the effect of pH variation on the viscosity of the beryllia slip at various solids content levels, the following procedure was followed.

A Brookfield Model RVT 7 viscosimeter was used to determine slip viscosity while pH readings were obtained using the Beckman Model G pH meter. The slip samples used in each case had a minimum volume of 600 milliliters so as to obtain a maximum accuracy of viscosity readings. Varying amounts of concentrated ammonia and hydrochloric acid were added to the slip and stirred using mechanical agitation with glass agitators and containers. Measurements of pH and viscosity were taken only after the system had come to equilibrium, usually 20 to 30 minutes after each addition. A curve was determined from a pH of 1 to a pH of 11 for three different solids contents; 33% solids, 42% solids, and 50% solids.

It is apparent that various beryllia raw materials behave differently in slip form, that is, materials prepared by different

methods with different sintering additives will yield far different

viscosity curves at similar per cents solids content levels. As

an example, Figure 2 shows the pH vs. viscosity curve for three

slips of the RX3E composition. This may be compared with Figure 3,

which shows five pH vs. viscosity curves for Berylco raw material

which, in addition, had been calcined prior to milling with the

sintering additives [11]. It is apparent that the viscosity of

the 50 weight per cent solids content RX3E material is comparable

to a much higher weight per cent solids content calcined material.

On the basis of these data it is concluded that a slip of the

RX3E mix at about 40 to 45% solids content defloculated either

at a pH of 2 with hydrochloric acid or a pH of greater than 10.5

with ammonia, will be satisfactory for the pressure slip casting

experiments. In order to achieve a higher solid content, as

required for maximum green cast densities, RX3E material was

also prepared by pre-calcining and re-milling. Care must be

taken, however, to retain the good sintering characteristics so

as to allow electric firing at $3000^{\circ}F$ after pressure-casting.

The slip approach successfully employed on high alumina at

Rutgers [12,13] of fully maturing the raw material and then remilling

to a fine particle size slip in order to decrease drying shrinkage

was evaluated for high purity beryllium oxide casting slip.

The casting slip developed, Mix RX3E was extruded, fired to 96% of

theoretical density and subsequently remilled to an average particle

NATIONAL BERYLLIA CORP.
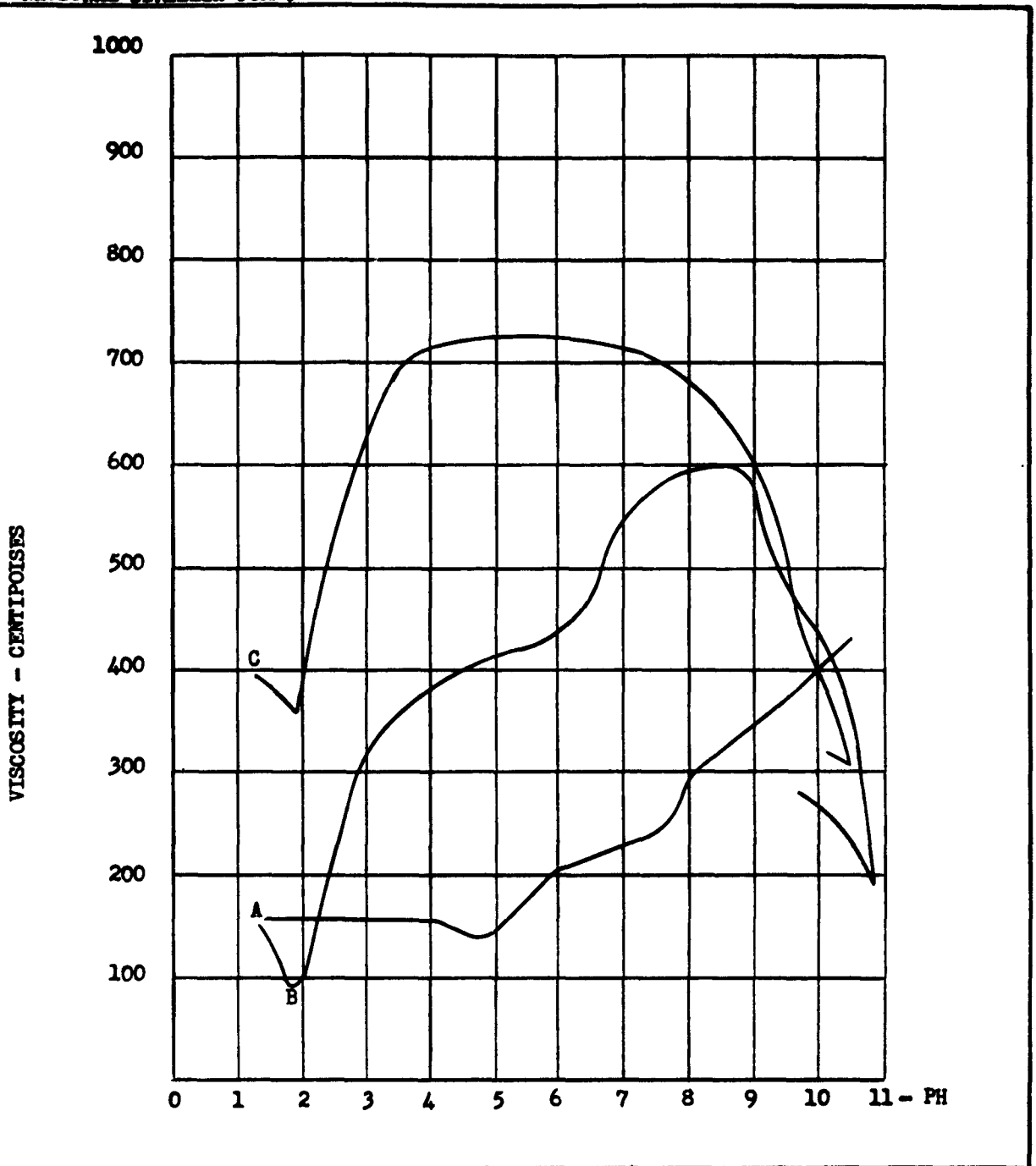
EFFECT OF PH ON VISCOSITY OF BERYLLIA SLIP UNCALCINED, AS-RECEIVED POWDER MILLED WITH SINTERING ADDITIVES

A - 33 w/o SOLIDS
B - 42 w/o SOLIDS
C - 50 w/o SOLIDS

FIG. 3

NATIONAL BERYLLIA CORP.

VISCOSITY - CENTIPOISES
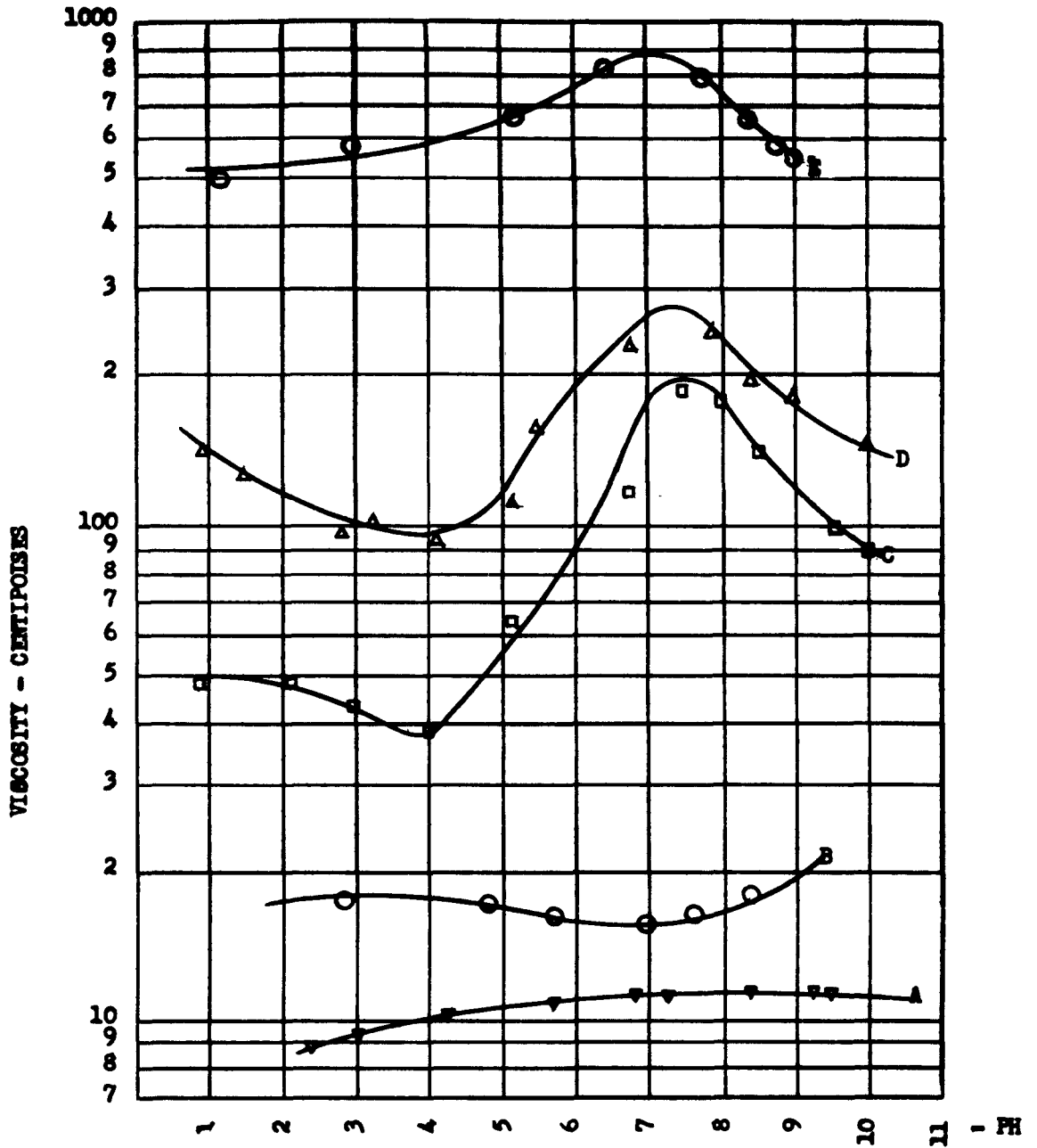
EFFECT OF PH ON VISCOSITY OF BERYLLIA SLIP CALCINED AND MILLED
WITH SINTERING ADDITIVES

A — 10 w/o SOLIDS   D — 60 w/o SOLIDS
B — 25 w/o SOLIDS   E — 65 w/o SOLIDS
C — 50 w/o SOLIDS

FIG. 4

size between 3 and 5 microns with a sizable fraction under one micron. Fabrication experiments on this material yielded a shrinkage of 13% as compared with a shrinkage of 19 to 20% for the normal RX3E casting slip.

In order to maintain the purity of the BeO slip, alumina or porcelain ball mills may not be used. Beryllia mills and beryllia grinding media are feasible but expensive. Rubber lined mills with beryllia grinding media are more practical but relatively slow. Steel ball mills and steel balls are fast and the iron contamination easily and completely removed by magnetic separation followed by acid washing. This latter procedure was used in all "preprocessed" slip evaluation.

Essentially three kinds of high purity BeO slip were considered in this study.

    1. Raw casting slip

    2. Slip milled from calcined raw material

    3. Slip milled from "preprocessed" fired body.

An excellent casting slip was developed based on the RX3E composition.

    99.6 parts Brush GC BeO, 95% (1 to 10μ)
    0.4 parts reagent grade MgO

This material may be suspended in water using Darvan #7 electrolyte at a pH of 1.5 to 2.0 and a specific gravity of 1.30 to 1.43.

When the beryllia powders are calcined to 1100 - 1300°C prior to milling with the MgO sintering aid, the specific gravity may be increased to 1.43 - 1.58 while still maintaining reasonable viscosity and otherwise good rheological properties.

Preprocessing of the body, that is extrusion, firing, steel milling and purification of the RX3E ingredients allows a further increase in specific gravity to over 1.60. The efforts expended in obtaining this increase in solids content resulted in a disappointingly small decrease in green and total shrinkage, and no discernable increase in densification.

In spite of the relatively simple laboratory casting equipment utilized in the program, it is significant that the dimensional control as shown in Figure 13 was achieved. While the dimensions shown are only for one selected radome, it does indicate that by a casting method a beryllia radome blank can be made having a concentricity of .012" on a 3.7" base diameter and a maximum wall thickness variation of .012 around the periphery. In addition, the maximum wall thickness variation over the entire surface and 5.6" length of the radome was .030". Based on these measurements it is felt that future improvements could be made and that a substantial reduction of grinding costs could be achieved under production conditions.

PHASE III

(2.3) MOLDING EXPERIMENTS

A laboratory pressure casting experimental unit was first set up for the purpose of conducting preliminary screening tests on various beryllia compositions to obtain information applicable to design of radome casting equipment. A series of plaster molds were fabricated suitable for forming discs having a green diameter of 3 inches and a thickness of approximately 1/2 inch. Pieces formed from such molds are suitable for both dielectric testing and mechanical property determination. (Figure 5).

The basic mold cavity was formed in the plaster block having external dimensions of approximately 6 inches square and 3 inches thickness. The open face of the mold was fitted with a steel pressure plate containing a hole for introduction of the slip, and an air vent. A rubber gasket was provided to prevent leakage of slip between the steel plate and mold face. The external surfaces of the plaster block were then coated with an organic finish so as to allow buildup of pressure within the system.

The entire mold and plate assembly was then combined using steel clamps. A Nelson laboratory pump was used to provide up to 30 psi (2 atmospheres) pressure to the slip reservoir forcing the material into the mold cavity.

FIGURE 5

EXPERIMENTAL DISC MOLD

Initial experiments were conducted using 99.5% Al_2O_3 in the form of a slip containing 65 percent solids and having pH of 4.0. Starting material for the slip was -325 mesh Al_2O_3 which had been ball milled for two hours prior to casting with a HCl deflocculant. No difficulty was encountered in filling the mold cavity, and it was relatively easy to achieve densities in the fired pieces at least equivalent to a standard slip cast body.

Some variations were noted in mechanical strength. Normal production slip cast aluminum oxide (RC5) has a strength of approximately 35,000 psi, while the pressure cast material ranged from 24,000 to 28,000 psi. It was felt, however, that this difference was due to minor variations and the relatively crude setup used for these initial experiments. In making the test pieces with alumina, several pressure leaks were developed in the plaster block and around the rubber gaskets between the steel plate and mold base. This situation must not exist when beryllium oxide raw materials are being handled because of the potential health hazard. Any escape of beryllium oxide slurry would have to be immediately removed and scrubbed down because, on drying the slip would yield a fine powder and could therefore be inhaled.

Rather than invest further time and materials in the encapsulation of the original mold cavity to prevent such escape of the beryllium oxide slurry, it was decided to proceed with an experimental radome design configuration. This would have several advantages:

TABLE VIII

RESULTS OF INITIAL PRESSURE CASTING EXPERIMENTS USING
99.5% Al_2O_3 MIX (FIRED 3200°F.)

Test No.	Description	Per Cent Solids	Shrinkage %	Density g/cc	Density % Theor.	Absorption %	Modulus of Rupture psi
RC 1	No pressure	65	21.0	3.83	96.5	0.00	21,800 - 20,800
RC 2	15 psi; blended mix only	65	21.0	3.59	90.4	0.04	24,800 - 28,700
RC 3	15 psi; ball milled mix	65	18.7	3.89	93.0	0.03	14,100 - 19,000
RC 4	30 psi	65	17.8	3.88	97.7	0.15	23,400 - 28,200
RC 5	No pressure (prod)	65	ND	3.87	97.5	0.00	30,000 - 36,200
RC 6	30 psi; with vacuum	65	ND	3.87	97.5	0.00	24,400 - 28,300
RC 7	30 psi; with vacuum	65	19.1	3.85	97.0	0.00	22,200 - 23,000
RC 8	No pressure; Darvan Additive	77	16.9	3.84	96.7	0.00	24,500
RC 9	No pressure; Aerosol Additive	77	16.5	3.87	97.5	0.00	21,300 - 28,000

(a) Anticipated problems in the mold release of radome shapes would be encountered, could be analyzed and overcome before final design of the radome facility was considered

(b) Shrinkage data for specimens in the shape and approximate size of the radome would be available yielding better engineering data for the design of a useful radome mold

(c) The external shell, base plate, pressing mechanism and other portions of the equipment could be applied to the prototype radome configuration.

2.3.1 BeO Radome Molding

A schematic sketch of the experimental pressure casting radome mold is shown in Figure 6. It was built on an aluminum base plate containing holes for attachment of the radome mold, insulated feed-thoughs for an internal electric heater and a reservoir groove and intake for the BeO slip from a threaded hole on the side of the base plate. Two graphite cores were made, one with an outside diameter (O.D.) dimension identical to the O.D. dimension of the desired green radome. The other with the O.D. dimension identical with the internal diameter dimension of the desired green radome. The difference between the O.D.'s of the two graphite cores then represents the thickness of the green beryllia radome.

To fabricate the mold itself the larger graphite core is centered on the base plate by means of a central positioning screw. Porous tubing is then wound around this form supported on a suitable

PLASTER MOLD

POROUS
TUBES

SLIP OUT OVER
FLOW AND AIR BLEED

#600016
STEEL SLEEVE

RUBBER GASKET

#600014
MOLD FORM

SUCTION & BLOW OFF

STEEL TOP
PLATE #600017

#600015
Graphite Mold

SLIP RESERVOIR

600018
PLEXIGLASS

#600013
ALUMINUM BASE
PLATE

SLIP IN

POWER LEAD WIRE

INSULATORS
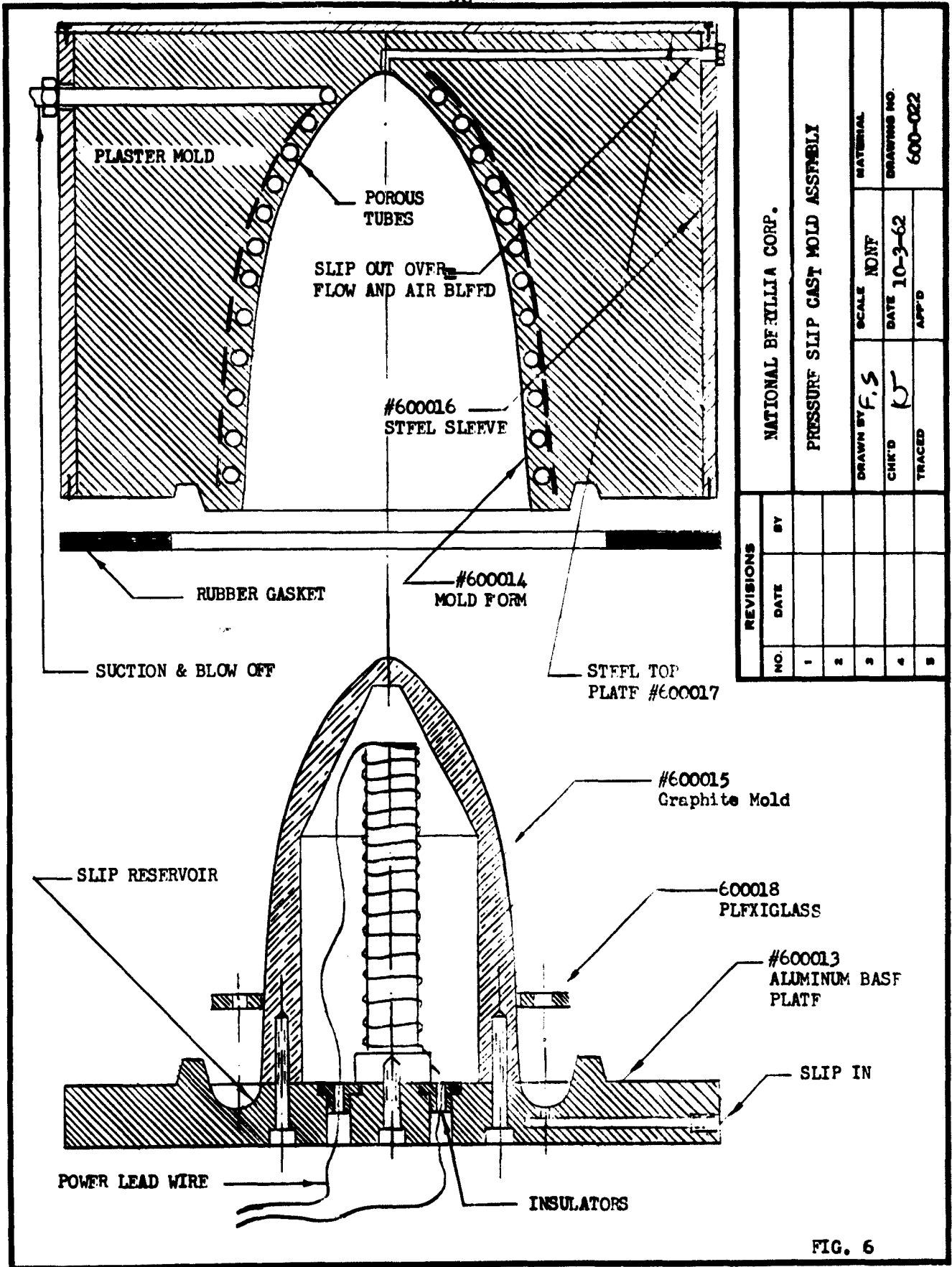
NATIONAL BERYLLIA CORP.

PRESSURE SLIP CAST MOLD ASSEMBLY

	SCALE NONE	MATERIAL
DRAWN BY F. S	DATE 10-3-62	DRAWING NO. 600-022
CHK'D	APP'D	
TRACED		

REVISIONS		
NO.	DATE	BY
1		
2		
3		
4		
5		

FIG. 6

wire screen and led to one side near the top. The steel outer
shell is then placed on the base plate. The porous tubing is
attached to one of the treated holes shown in the shell. A piece
of tubing is also led from the top of the graphite core to another
hole in the outside diameter of the steel shell. Plaster of Paris
(or hydrostone) is then poured into the shell and allowed to set.
The base plate and graphite core are removed as a unit and then
separated (Figure 7).

The smaller graphite core representing the I.D. of a resulting
green beryllium oxide radome is then attached to the base plate
by means of four set screws. Inside this smaller graphite shape
is placed a ceramic cylinder on which is wound a low temperature
heating element.

In operation, the top mold cavity is suspended in the top of
a suitable press. The base plate containing smaller graphite cores
is placed underneath the mold on a suitable movable press plate.

To operate, the plate is raised into the suspended cavity until
a tight seal is affected via a rubber gasket between the base plate
and the bottom of the suspended cavity.

The beryllia slip is then forced from a pressure tank by air
or tank nitrogen pressure into the side of the base plate, through the
reservoir, through the plexiglas distribution ring (shown in
Figure 6) and up an on over the smaller graphite core. Entrapped
air and finally beryllia slip are forced through the top central
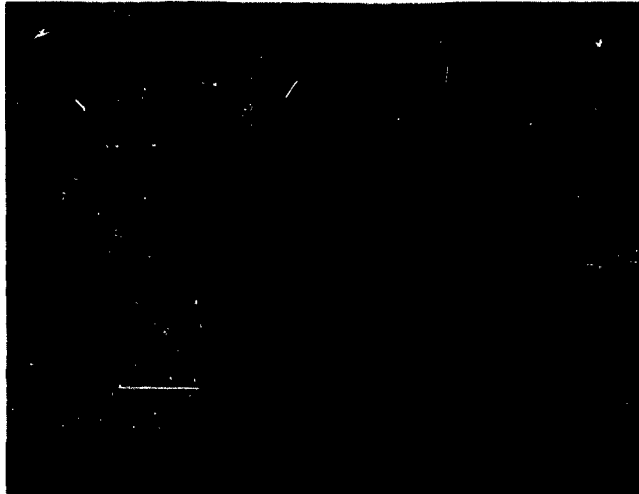
FIGURE 7

RADOME MOLD

FIGURE 8

PRESSURE SLIP CASTING APPARATUS

tube and out through the side of the shell into a suitable receptacle and then this channel closed. A slight vacuum applied through the porous tubing and the porous plaster remove the water and air from this slip. After release of the slip pressure, electrical energy supplied to the heating element encased in the graphite core heat the graphite and soften a thin wax coating placed previously on the graphite shape. The base plate may then be removed leaving the green radome shape held in the cavity by vacuum still applied through the porous tubing. A flat board or pallet may then be placed under the cavity, the vacuum removed and if necessary air pressure used to eject the green radome shape out of the cavity and on to the pallet where it may then be removed for drying and firing.

The assembly as shown in Figure 8 employed a three gallon, 60 psi pressure spray tank, polyethylene lined, and fitted with a rotational pressure seal for an externally powered stirring mechanism. Slip placed in this tank may be first de-aired by vacuum, then forced out by pressure applied by a nitrogen bottle and regulator. Allignment was achieved by constructing a six element Dexian frame between two 1/4" thick steel end plates with a total height just enabling it to fit into a high velocity stainless steel hood. The female mold was suspended from the top end plate with a cavity positioned downward. The male core and base plate were free to move in the vertical direction confined laterally by the

six element frame and propelled by a central screw thread rod

protruding through the bottom of the hood, threaded through the

bottom end plate and fastened by a double acting bearing to the

bottom of the aluminum base plate.

Several initial runs with this apparatus using alumina slip

showed that a bolted frame did not possess sufficient strength

to maintain positive allignment after some use. For this reason

a welded steel frame was constructed which satisfactorily solved

this problem.

A number of attempts were made to cast radomes with this

mold. In all cases, pieces were cast satisfactorily but problems

were encountered in removing the cast piece from the mold. The

anticipated problem of adherence of the cast piece to the male

graphite core was encountered. A thin wax layer applied to the

core prior to a run and softened prior to removal of the piece

by use of the heating element did not function satisfactorily. Stick-

ing of the specimen in the female plaster mold was also encountered

both when the plaster mold was wet and quite dry. A thin colloidal

graphite coating aided in release of the specimen initially, however

continued use of reverse gas pressure to eject the piece from the

female mold seriously erroded or pitted the plaster surface. Two

further problems were encountered:

(1) Excessive reverse gas pressure tended to drive water
from the mold into the piece thereby softening it
and allowing it to sag or slump, (a defect noted
previously with the Rutgers equipment).

(2) The external connection to the porous tubing coil was at the apex end rather than the base end of the cone.

Reverse pressure appeared to push in the top or apex of the specimen and bulge it outward at the base, thereby perhaps, preventing an easy release.

The experience with mold design Number 3 resulted in the design of mold Number 4 with the following changes:

(1) The porous tubing was encased in a thin layer of Hydrostone, a harder, more dense errosion resistant mold material, in turn surrounded by casting plaster.

(2) The connection to the porous tubing was led from the base rather than the apex end of the cone to the external fitting.

During the first run, however, a leak developed between the porous tubing connection and the base plate through a crack in the Hydrostone core rendering the porous tubing ineffective for reverse pressure or vacuum use.

Mold design Number 5 was similar to mold Number 4 in that a Hydrostone core was also used. Several partially successfull radomes were cast with this apparatus. Increased casting time was noted due probably to the increased density of the Hydrostone over normal casting plaster. An increase in the tendency to drive water from the mold back into the cast piece when reverse gas pressure was used was also noted. Errosion or pitting of the casting surface still occurred though to a lesser extent than with
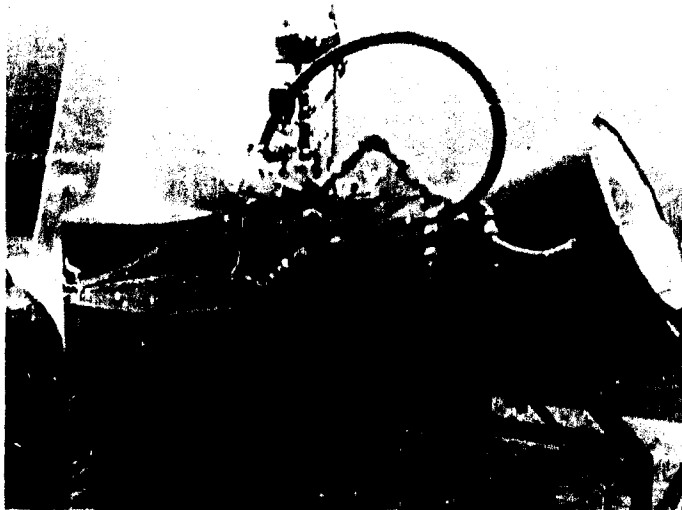
FIGURE 9

CORELESS PRESSURE CASTING APPARATUS

FIGURE 10

REVISED PRESSURE CASTING APPARATUS

normal plaster. An estimate of mold life would, therefore, not be high.

The major problem with this design was sticking of the piece to the male graphite core. Coating the core with Vaseline, grease, colloidal graphite or stearic acid did not satisfactorily solve the problem. Shrinkage of the case piece onto the core appeared to be the difficulty inherent in this design.

To determine if the pressure rather than the core was causing this difficulty the following experiment was run. A female mold of casting plaster was fabricated and fitted with a 2" wide rubber gasket and steel plate on its open end. Fittings for slip injection and air venting were incorporated into the steel plate. The plate and mold were pressed together to prevent slip leakage and slip injected under pressure into the entire cavity with no core within the chamber. After three minutes at pressure the apparatus was disassembled and the excess slip drained as in normal drain casting. The piece released nicely and produced a perfect radome with about .125 inch wall thickness. Several small holes were visible through the piece however, presumably due to the gas under pressure proceeding through the slip, through the cast piece, out through the porous plaster mold into the atmosphere. This mold was modified by coating the exterior surface with an impervious organic finish which in subsequent runs greatly decreased the number of these gas holes. This mold is shown in Figure 9. Several pieces were cast at

pressures between 15 and 50 pounds per square inch slip pressure, however, pressures in excess of 30 psi caused failure of the impervious coating.

The last mold to be constructed combined the best features of all previous devices and is shown in Figure 10. This mold is encased in a steel shell to prevent gas escape and has a Hydrostone core to decrease errosion tendencies. External fittings to the encased porous coils are provided at both the apex and base end of the cone. Castings may be made either with a core in place or without the core, as a pressure drain cast device. The shell was mounted on a tilting table to facilitate specimen removal and so that it could be used either with the cone apex facing upward or downward.

Several pieces were cast using mold Number 7 at pressures up to 50 pounds per square inch. Again whenever the graphite core was employed the problem of sticking of the specimen to the core was encountered. Increased casting time was again noted in comparing the Hydrostone core to normal plaster. Casting time was not significantly decreased by applying a vacuum to the porous tubing. The vacuum did have the apparent disadvantage of increasing slip penetration into the female mold thereby hampering ready release of the specimen from the mold.

A series of seven molds was fabricated during the course of this study. More than 50 attempts at casting were made at pressures

from 15 to 50 pounds per square inch resulting in a number of satisfactory radomes as shown in Figure 11. Three pieces were also normally drain cast at atmospheric pressure for comparison purposes using the same casting slip.

Three radomes pressure slip cast at 15, 20, and 30 pounds per square inch and one radome normally drain cast at atmospheric pressure were fired together in the same furnace so that all four received as nearly identical thermal treatment as possible. The results of this comparison firing to $3300^{\circ}F$ in a 10 3/4 hour cycle are listed in Table IX. It may be seen that there is no apparent increase in density or decrease in shrinkage as pressures are increased in the pressure slip casting technique. These data indicate that density may even tend to decrease as pressures are increased. Density and shrinkage figures on other separately fired pieces show that shapes cast in a Hydrostone core steel shelled mold tend to have higher densities than pieces cast in the organic coated plaster mold. Further experiments would be necessary to verify this observation.

A cast beryllium oxide radome blank was sectioned and portions selected for strength and density uniformity determinations. The results as shown in Figure 12 indicate extremely uniform density of 2.81 g/cc. Strength data were quite good and fairly uniform considering test bars for the modulus of rupture test were not rectangular but somewhat curved due to the shape of the piece from which they were cut.

Typical dimensions of the radome blanks as cast in the molds described and fired to a density of 2.80 - 2.82 g/cc with no machining whatsoever are shown in Figure 13. The piece illustrated was cast with the apex end downward in a coreless mold and shows a 10% thicker wall near the apex than at the base. Coreless casting with the base end down can reverse this trend. The tilting table apparatus can be employed to eliminate this tendency by reversing the position at some point about midway through the cast.

FIGURE 11

CAST BeO RADOMES

TABLE IX

COMPARATIVE DATA ON BeO RADOMES CAST AT VARIOUS PRESSURES
AND FIRED SIMULTANEOUSLY (3300°F)

Casting Pressure psi	Shrinkage %	Density g/cc	Percent of Theor. Density	Water Absorption %
none	19.0	2.80	93.1	0.07
15	21.5	2.81	93.4	0.00
20	19.3	2.57	85.4	0.3
30	---	2.68	89.1	2.6

1.625

D = 2.81
S = 23,500

1.625

D = 2.81
S = 26,000

180°

1.625

D = 2.81
S = 23,500

D = 2.81
S = 19,000

D = 2.81
S = 24,500

.625

180°

D = 2.80
S = 23,000
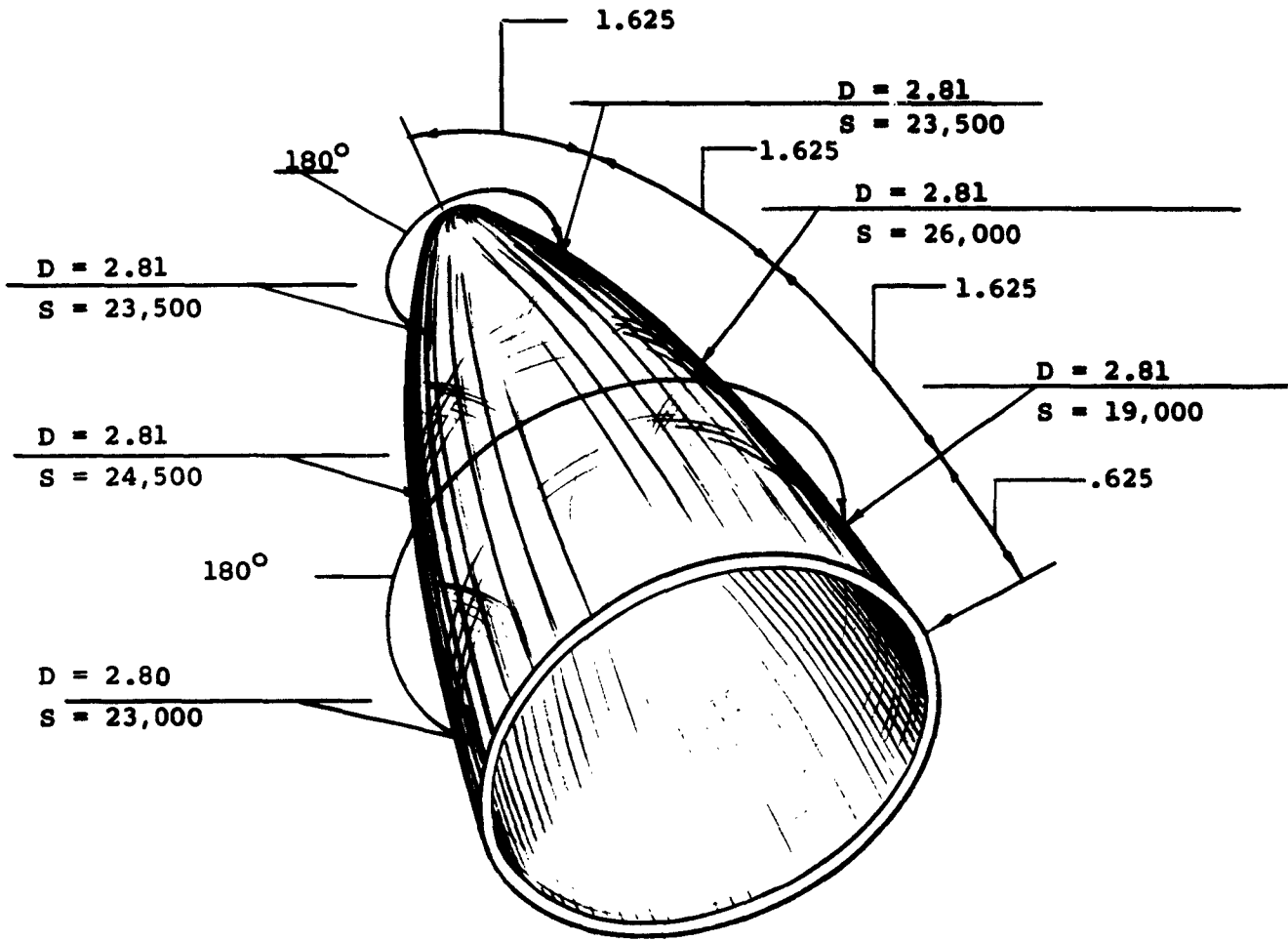
NOTE:

    1 - D = DENSITY - G/CC

        S = STRENGTH - PSI

FIGURE 12
DENSITY AND STRENGTH
UNIFORMITY IN CAST
BeO RADOME

1B

.195

60° 60°

.195

2B .194

6B

SECTION B-B

1C

.203

60° 60°

.199 .197

2C 6C

SECTION C-C
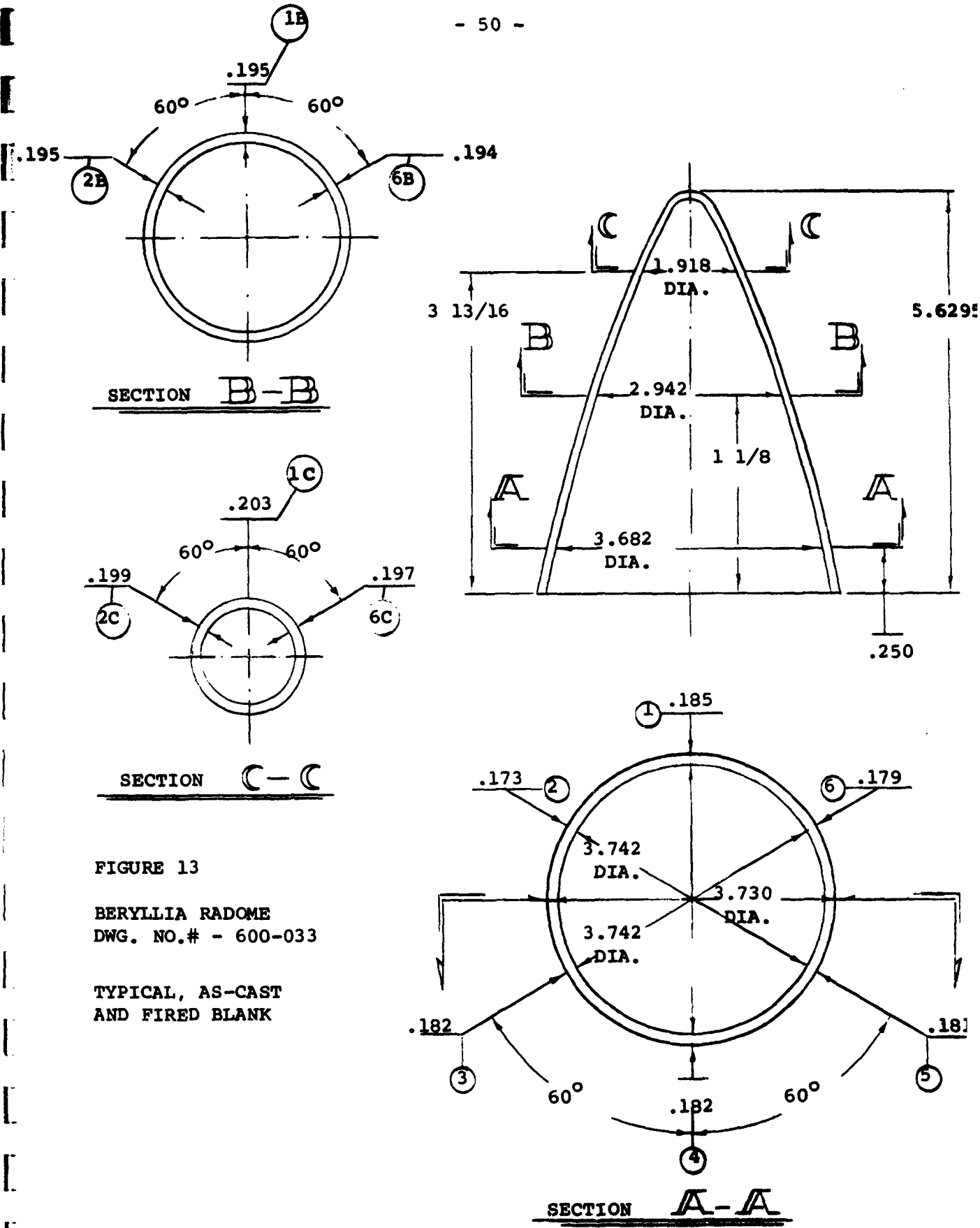
FIGURE 13

BERYLLIA RADOME
DWG. NO.# - 600-033

TYPICAL, AS-CAST
AND FIRED BLANK

C C

1.918
DIA.

3 13/16

5.6295

B B

2.942
DIA.

1 1/8

A A

3.682
DIA.

.250

1 .185

.173 2 6 .179

3.742
DIA. 3.730
DIA.

3.742
DIA.

.182 .18

3 5

60° 60°

.182

4

SECTION A-A

(2.4) EVALUATION AND PROPERTY MEASUREMENT

The important physical properties to be considered critical in evaluation of materials for aircraft and missile radomes are:

(a) Mechanical Strength

Tensile strength as measured under flexural loading; modulus of rupture

(b) Impact and Erosion Resistance

Modulus of Elasticity

Hardness

(c) Density

(d) Thermal Stress Resistance

Thermal Conductivity

Thermal Expansion

Poissons Ratio

(e) Dielectric Properties

Dielectric Constant

Loss Tangent

These properties may all be categorized as structure-sensitive and as such, are dependent upon the starting materials and method of fabrication. While a great deal of data is available in the literature on various forms of beryllia, in the present program actual values are being generated for the bodies used in specimens and radomes for greater reliability in future design requirements.

For example, modulus of rupture values for BeO have been reported as low as 15,000 psi and as high as 45,000 psi. A typical value under production conditions is 20,000 psi, although under close control, average values of 30,000 psi can be reproducibly obtained. The dependence of the strength of BeO on crystal size has been published by several investigators, and the general indication is that a grain size in the fired body of 10-20 microns produces optimum mechanical strength assuming the density is high; that is, 95% of theoretical or better.

The modulus of rupture values obtained on cold-pressed BeO radome compositions using different firing techniques are shown in Table X.

Dielectric constant measurements on 99.5% purity BeO have been made at three microwave frequencies and are expressed as follows as a function of density:

| | | Dielectric Constant | |
| | (MIT) | (MIT) | (NADC) |
Density g/cc	3.14 KMC	8.5 KMC	33.4 KMC
3.00	---	6.62	---
2.85	6.59	6.57	5.87
2.70	---	5.97	---
2.20	---	---	---
1.96	---	---	3.73
1.86	---	---	3.66
1.33	---	---	2.46

The dielectric loss tangent measured by MIT at 3.14 KMC showed a value of .00044 at room temperature and varied with temperature as shown in Table XI.

TABLE X

Gas-Firing 3400°F

(A)	Body Composition	Modulus of Rupture, psi
	RX 1	19,300 - 20,600
	RX 2	19,800 - 20,400
	RX2A	18,500 - 15,500
	RX 3	18,300 - 21,800
	RX3A	18,600 - 18,800
	RX 4	17,500 - 18,500
	RX4A	18,600 - 19,700
	RX 5	18,600 - 20,800
	RX5A	18,500 - 19,100
	RX3A (fired and Steel-milled)	14,600 - 17,600

Electric-Firing 2950°F

(B)	Body Composition	Modulus of Rupture, psi
	RX3E	24,300 - 28,500
	RX3C	25,000 - 26,000
	RX3BU	20,300 - 22,500
	RX3BU-1	24,800 - 26,300

*Values were measured as the average bending strength of six pieces 1/4" X 1/4" square broken on a 2" span.

TABLE XI

Dielectric Properties of 99.5% BeO at 3.14 to 4.87 GC
(Density 2.85 g/cc)

Temperature

^{o}C	^{o}F	Dielectric Constant*	Loss Tangent
RT	77	6.59	.00044
200	392	6.70	.00040
400	752	6.91	.00049
600	1112	7.13	.00089
800	1472	7.43	.00156
1000	1832	7.78	.00253
1100	2010	8.00	.00370
1150	2102	8.15	.00643
1200	2192	8.33	.01040

*Corrected for thermal expansion

(Data Courtesy Sylvania Electric Products, Inc. and M.I.T.)

(3.0) CONCLUSIONS

Based on the research and development work conducted during the 9 month period of the contract the following general conclusions can be drawn:

(1) Slip casting both with and without pressure can be utilized for the fabrication of high purity beryllium oxide radomes.

(2) Based on sinterability and densification tests, GC grade raw material was selected as the best compromise between economics and technical characteristics for radome fabrication.

(3) Beryllium oxide powders were prepared as casting slip in three different conditions: (1) Using as-received powder, the specific gravity range of slips achieved was between 1.30 and 1.43 g/cc. This represents a nominal solids content of 33 to 45 weight percent. (2) Slips made using calcined powders ranged from 1.43 to 1.60 g/cc or 45 to 55 weight percent solids. (3) Slips prepared using pre-processed and milled powders showed specific gravity values of 1.60 g/cc or greater with a corresponding increase in solids content.

(4) Particle size measurements made on beryllia slip materials indicated that as-received powders were

better than 90% finer than 10 microns in agglomerate
size although the ultimate particle size was less
than 1 micron. Fired powders indicated an agglomerate
size range of in some cases up to 40 microns.

(5) Experiments utilizing pressed and sintered test
compositions indicated that BeO of 99.6% purity
could be fired to a density of 2.87 reproducibly at
$3000^{\circ}F$.

(6) A series of pressure casting devices was designed and
constructed for casting experiments conducted both
with and without pressurized slip. In all cases
unidirectional casting was utilized both with and
without a core. A general conclusion based on this
work is that the resultant physical properties of
beryllia radomes cast with and without pressure are
essentially identical. With further refinement of the
equipment it is probable that the major advantage
would be a speed up of the casting cycle under production
conditions.

(7) Beryllia radomes slip cast using the described techniques
showed very good uniformity of density in the range of
2.80 to 2.82 g/cc and nominal strength of 25,000
lbs/sq. in. in flexure.

(8) Dielectric measurements on samples of 99.5% BeO indicated a loss tangent of .0004 and a dielectric constant of 6.57 in the microwave range.

(9) Although the radomes fabricated in this program were limited to a 7" green length no inherent limitations were observed in the size of radomes which could be produced by this process. It is anticipated that larger pieces would have a tendency to distort in firing although this could very likely be overcome by using a reduced firing temperature in electric firing and proper design of various support devices.

(4.0) RECOMMENDATIONS FOR FUTURE WORK

Future effort conducted on this program should emphasize refinement of the basic casting equipment to assure maximum compatibility of mold components for optimum control of dimensions. Future study should be conducted of core materials with particular emphasis on the mold release problem.

Additional research on the beryllia body itself should be done toward reduction of shrinkage both in the drying and firing stages of processing. This would, in addition to reducing dimensional variations, assist in the mold release and allow more ease in the molding operation. Development work should be conducted on a non-aqueous molding system of high solid content. By eliminating the water removable aspect of the molding operation and by taking advantage of heat and pressure, it is felt that higher green densities could be achieved than were possible in the present facility.

Future effort is required on the aspects of reproducibility and the control of dimensions in the cast blanks. Based on this initial study there is every indication that close dimensions could be held and that grinding procedures could be greatly simplified once a reproducible and systematic process was put into operation

Radome blanks of 99.5% beryllium oxide fabricated under this program should be subjected to physical and dielectric testing including thermal cycling and shock tests and measurements of transmission efficiency.

(5.0) REFERENCES

1. Darwin, G. E. and Buddery, J. H. Beryllium. New York, Academic Press (1960).

2. Ryshkewitch, E. Oxide Ceramics. New York, Academic Press (1960).

3. S. C. Carniglia and J. E. Hove, "Fabrication and Properties of Dense Beryllium Oxide". NAA-SR-MEMO 5572.

4. R. E. Taylor, "Thermal Conductivity and Thermal Expansion of BeO at Elevated Temperatures". NAA-SR-4905, July 30, 1960.

5. E. A. Aitken, "Initial Sintering Kinetics of Beryllium Oxide" J. Am. Ceram. Soc. 43, 627 (1960).

6. J. Lillie, "Some Properties of Beryllium Oxide", UCRL-6457 (May 19, 1961).

7. M. L. Stehsel, R. M. Hale, C. E. Waller, "Modulus of Rupture Measurements on Beryllium Oxide At elevated Temperatures". Lawrence Radiation Laboratory P. O. 3951607, Report No. 1777 (Final) (March 1960).

8. A. J. Rothman, "Beryllium Oxide - Its Properties and Application In Nuclear Reactors". UCRL-6502-T. A report presented at Alfred University June 28, 1961.

9. Armour Research Foundation of Illinois Institute of Technology, "Thermal and Mechanical Properties of Beryllia Ceramics," GA-1906 - Final Report (August 1960).

10. R. D. Reeve and E. J. Ramm, A COMPARATIVE STUDY OF TWO GRADES BeO, Australian Atomic Energy Commission, Research Establishment Lucas Heights, Report AAEC/E80, Sidney, Australia, November 1961.

11. DEVELOPMENT OF FABRICATION TECHNIQUES FOR BERYLLIUM OXIDE RADOME MATERIALS, Contract NOw-60-0057-c, National Beryllia Corporation, Haskell, New Jersey, June 1961.

12. E. J. Smoke, J. F. Ward, W. J. Alford and A. L. Clayton, "Technical Report No. 5, Final Report, June 30, 1958", Contract No. N-onr-404(03).

13. E. J. Smoke, W. J. Alford, V. F. Ward, E. K. Steindal and G. Himich, "Development of Manufacturing Methods for The Production of Alumina Radomes", Final Report, Contract No. NOas-58-872-c.