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Technical Report No. 1
Contract No. N-onr-404(03)

April 15, 1952 - June 15, 1953
New Jersey Ceramic Research Station
Rutgers University

TECHNICAL REPORT NO. 1

April 15, 1952 - June 15, 1953

OFFICE OF NAVAL RESEARCH
Contract No. N-onr-404(03)

N. J. CERAMIC RESEARCH STATION
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RESTRICTED

TECHNICAL REPORT NO. 1

Contract N-onr-404(03) Project NR 032 348

I. Introduction

It is recognized that half wave sheet radomes offer certain advantages over some other designs on the basis of low reflection for both polarizations over a wide range of angles of incidence. This is particularly true of materials with dielectric constant of 5 or greater. It is also true that to achieve these advantages, one must work to very close thickness tolerances.

II. Object

This project has been initiated to learn how to handle the fabrication of ceramic materials so that radomes could be made with acceptably low transmission losses for all antenna positions.

The first phase of the work has been to develop the precision required to produce large ceramic shapes with thicknesses controlled to one or two thousandths of an inch, maximum variation.

The second phase will then be to fabricate radome shapes which will give satisfactory electromagnetic performance. It is expected that this will be a process combining theory with

experimentation, to make, test, and remake the shapes until the goal is achieved.

II. General Procedure

The four major methods of ceramic fabrication were considered. They were pressing, extruding, slip casting, and jiggering. The contemplated size and shape of the radomes rendered pressing and extruding impractical. The existing body compositions having the optimum electrical properties necessary were not readily adapted to jiggering. Slip casting was thus found to be the most practical method. However, jiggering remains as a possible method of fabricating radomes. This method, however, would necessitate a radical change in body composition in order to introduce substantial amounts of raw clay.

The most practical method of fabricating the contemplated shapes, as mentioned above, was slip casting. The size and contour of these shapes were such that drain casting lends itself to this adaptation readily. The compositions having the proposed electrical properties also lend themselves readily to this process. Slip casting also produced the most uniform structure which appears to be of extreme importance in this application.

The dielectric properties of the material were given

prime consideration in selecting a body composition with which to begin the work. It was indicated that the body should have a dielectric constant of 5 or greater with low loss characteristics. The following dielectric materials were contained in this category: (1) Steatite, (2) Zircon Porcelain, and (3) Wollastonite compositions. Steatite, the most extensively used material of the three mentioned, was selected as the material with which to begin.

In addition, it was decided to use the steatite in previtrified form in order to gain the following advantages:

(1) Accurate shrinkage control.

(2) Maximum Homogeneity of structure and fine body texture.

(3) Lower maturing temperature with minimum firing cycle.

In view of the extremely close dimensional tolerances contemplated for the radome shapes, emphasis was placed upon control of the particle size distribution. An effort was made to correlate the particle size distribution with firing shrinkage.

Work was begun on a laboratory basis using existing equipment. A conventional ceramic process was simulated in order to establish a preliminary technique which could then be applied to a pilot scale process for the production of the large radome shapes. In this manner it was possible to evaluate all of the operations necessary to produce the desired shapes by the slip casting method. An experimental ogive shape

Dwg. No. 404(03)-0, (Fig. B-1), was selected for this phase.

From this preliminary work, a processing system was designed to satisfy the needs for producing the large shapes. This also included the design and construction and/or procurement of equipment necessary to fabricate such shapes.

A general outline of the process devised for the production of the ceramic radomes by the slip casting method was as follows:

1. Preparation of Materials

The previtrified steatite was milled to the desired degree of fineness by wet or dry milling. Various particle size fractions were obtained by dry air classification methods. Selected particle size distributions were obtained by the blending of various fractions.

2. Preparation of Casting Slip

The batch comprised of the milled pre-vitrified steatite, plasticizer, deflocculants and water was milled for a specified length of time.

3. Processing of Casting Slip

The casting slip batch was screened, deaired, ferrofiltered and stored in a tank equipped with an agitator.

Periodic control checks were made on the casting slip. Adjustments were made when necessary with additives to maintain the specific gravity and the viscosity within certain prescribed limits for each property.

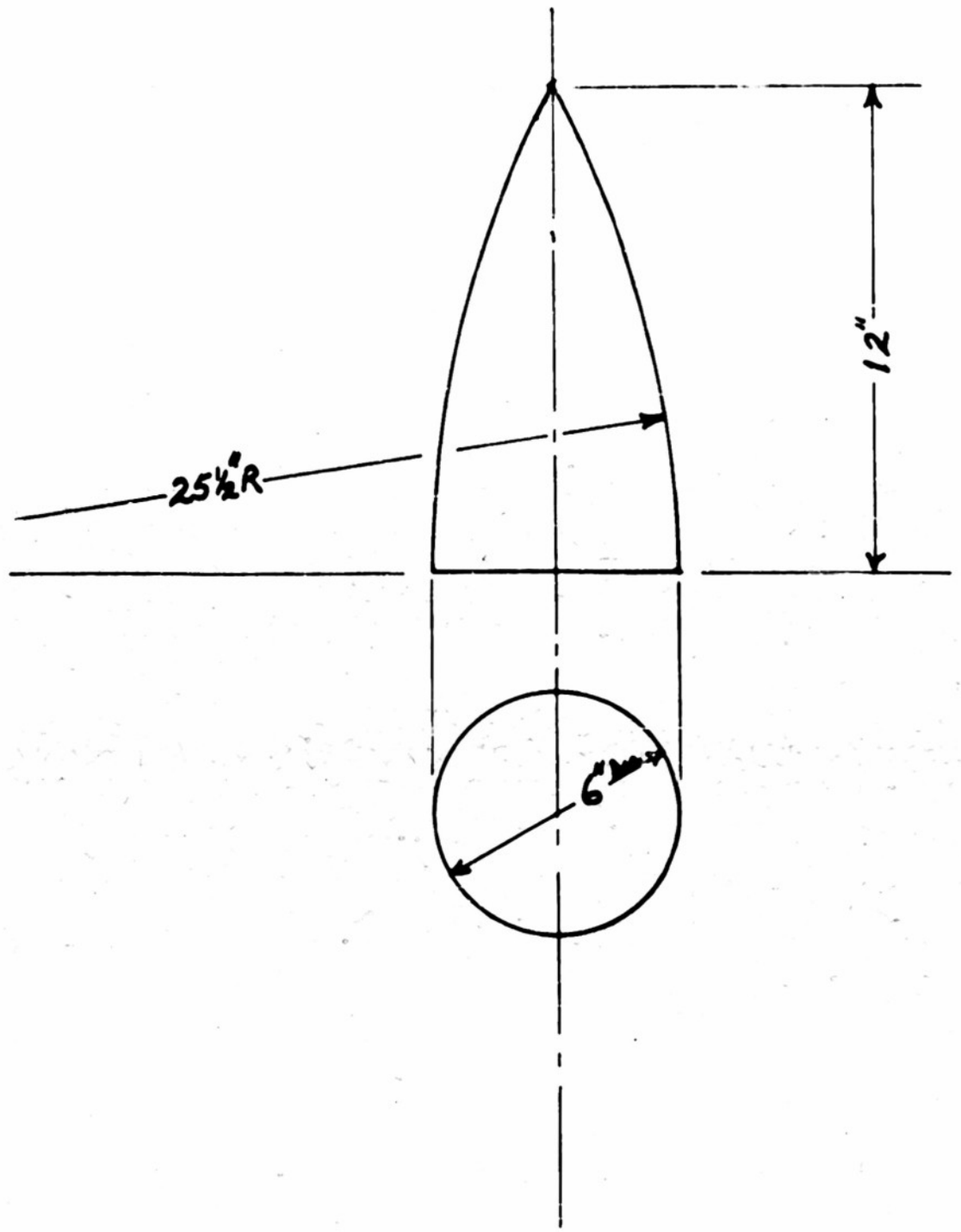


FIG B-1

EXPERIMENTAL SHAPE NO. 1. ALL DIMENSIONS COMPENSATED TO MOLD SIZES.
Scale 3" = 1' DWG. NO. 404(03)-0

4. Casting and Handling

The casts were made and extreme care was exercised to establish the proper timing for each phase of this operation. A technique was established to minimize handling in the green state.

5. Bisque Firing

A bisque firing cycle was developed to obtain optimum handling and machining properties.

6. Machining of Shapes

A complete operation was designed for the machining of radome shapes to satisfy proposed dimensional tolerances

7. Firing

A kiln-setting technique and firing cycle were developed for the radome shapes.

Various testing facilities were investigated for testing and evaluating ceramic radome materials and shapes. It was intended to incorporate the evaluation of the test results obtained in this manner in

(1) improved radome designs

(2) the selection of a suitable ceramic dielectric material

(3) to establish the dimensional tolerances for a specified radome design.

The design of the initial radome shape, Dwg. 404(03)-1, (Fig. B-2), was based upon the following considerations:

1. It had to be adaptable to existing test equipment at the Naval Air Development Center at Johnsville, Pa. After consultation with the group that would do the testing, this indicated about a 10" diameter at the base, capable of taking an 8' antenna.

2. It was advantageous to make it as long as possible since the range of angle of incidence encountered at any point near the nose decreases with the length of the radome. However, this advantage had to be tempered with practical considerations and therefore a 24" length was selected.

3. A pointed rather than a rounded nose was selected to avoid any region of abrupt change in angle of incidence. This would eliminate wide differences in phase shift at different regions of the energy beam.

4. A parabolic curve was chosen which would simulate a logarithmic spiral in that the curvature decreases from the base to the nose of the shape. The definition of this parabolic curve is as follows:

The intersection of the radome outline with a plane through the axis of the radome was a parabola whose axis lies in the base of the radome.

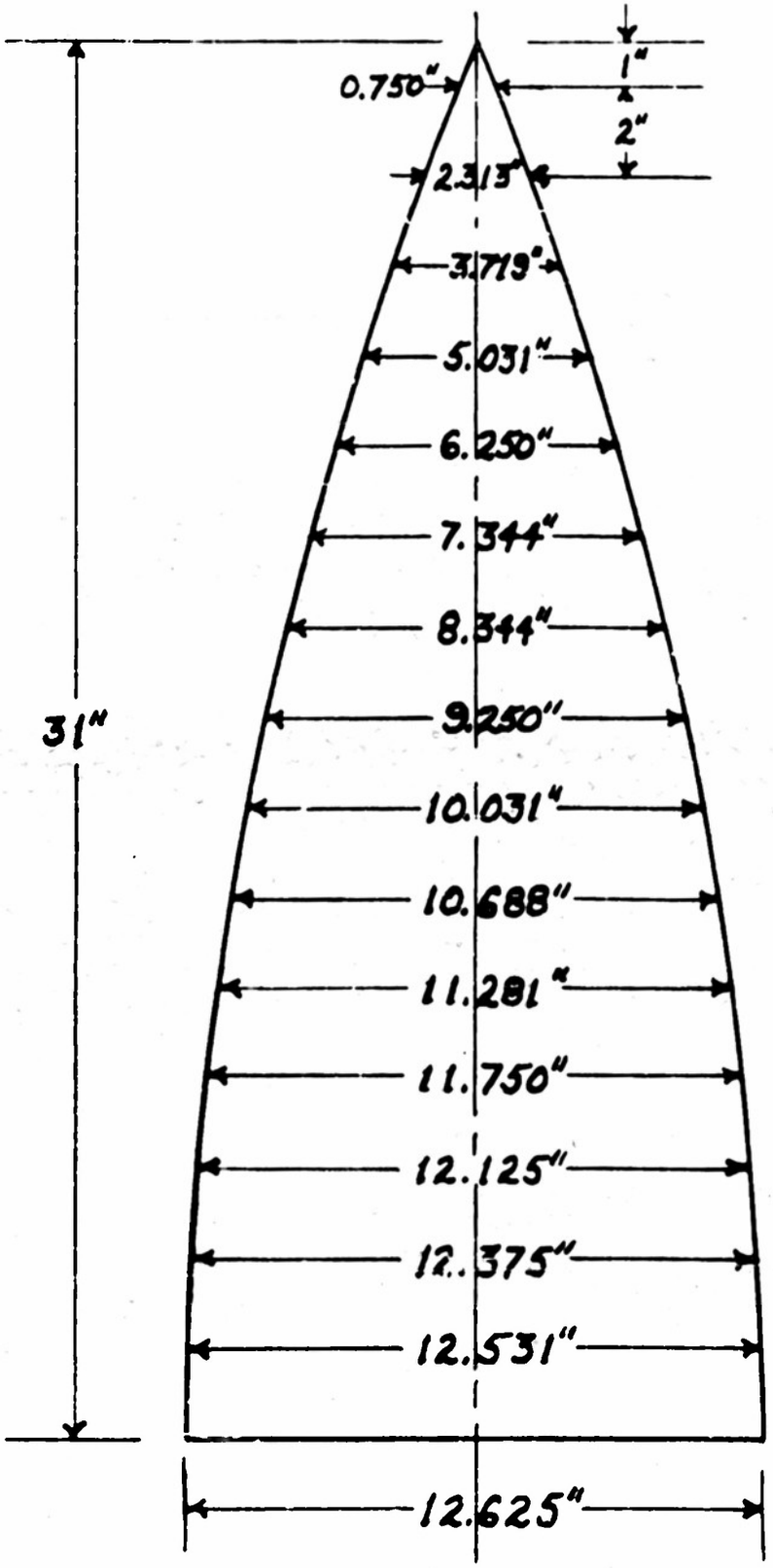


FIG B-2

EXPERIMENTAL TEST SHAPE NO. 1.
ALL DIMENSIONS COMPENSATED
TO MOLD SIZES.
Scale 3" = 1' DWG. NO. 4402-1

IV. Results

A. Operations and Equipment to Produce Radome Shapes

The following flow chart shows the operational sequence and the corresponding equipment procured to produce large radome shapes.

Operation	Equipment
1. Milling of Pre-Vitrified Steatite	A ball mill with inside dimensions of 22" length and 27" diameter was used. It was steel jacketed and porcelain lined. Flint pebbles were used as the grinding media in operations 1 & 5.
2. Drying of the milled Steatite	The wet milled slurry was put into enamelled trays and placed into a drier 42" x 46" x 26". The dryer was electrically heated and controlled.
3. Classification of milled Steatite	An outside source was used for this work. The method used was dry air classification. The work was done by the Reduction Engineering Co., Newark, N. J.
Note (This operation was limited to the dry milled composite, primarily for the purpose of getting a coarse fraction).	

Operation	Equipment
4. Casting Slip Preparation	The materials were batched and placed in the ball mill described in item No. 1 above.
5. Screening of Casting Slip	A forty mesh U. S. laboratory test screen was used to screen the slip.
6. Electro Magnetic Separation of Casting Slip	A Franz ferro filter model No. 41 was used. A Thor Selenium rectifier model No. S-3 was used to supply the D.C. current to the ferro filter.
7. Deairing of Casting Slip	A Binks, 10 gallon, galvanized, glaze pressure tank was used. This tank was equipped with an air driven motor geared to an agitator. It also had a dish type bottom equipped with a discharge opening of 1" diameter. This tank was attached to a Cenco Megavac vacuum pump with a non collapsible hose equipped with a moisture trap.

Operation	Equipment
8. Casting Slip Storage	A 50 gallon stainless steel tank equipped with a gear reduced agitator (30 R.P.M.) was used. This tank also had a dish type bottom with a 2" outlet at the center. A quick opening valve was incorporated on this opening with detachable piping of sufficient length to pour directly into the mold.
9. Casting	Two piece molds were used. The tops were held in place by a steel ring clamped with rods to a steel band secured to the body of the mold. The molds were handled during the draining and removal periods by a yoke and hoist assembly shown in Fig. No. B-3.
10. Drying of Shapes	Air drying was sufficient for this purpose.

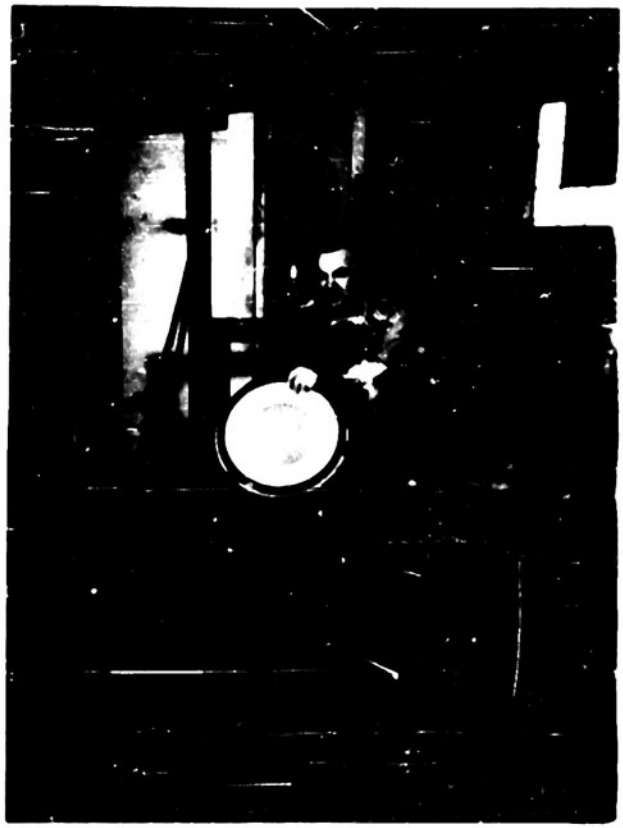
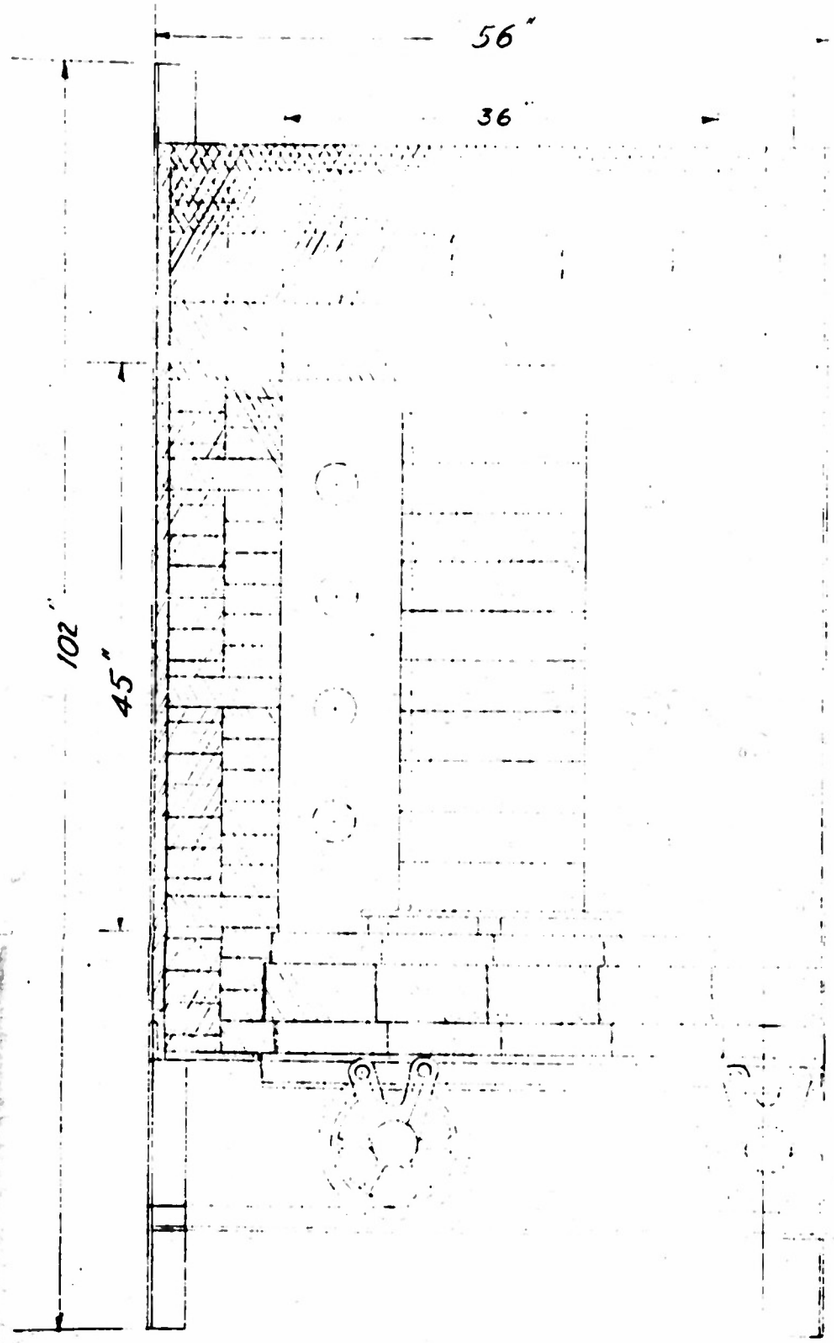


FIG. B-3

FIG. B-4



SIDE SECTION BB

B & W K-23 BRICK

B & W K-30 BRICK

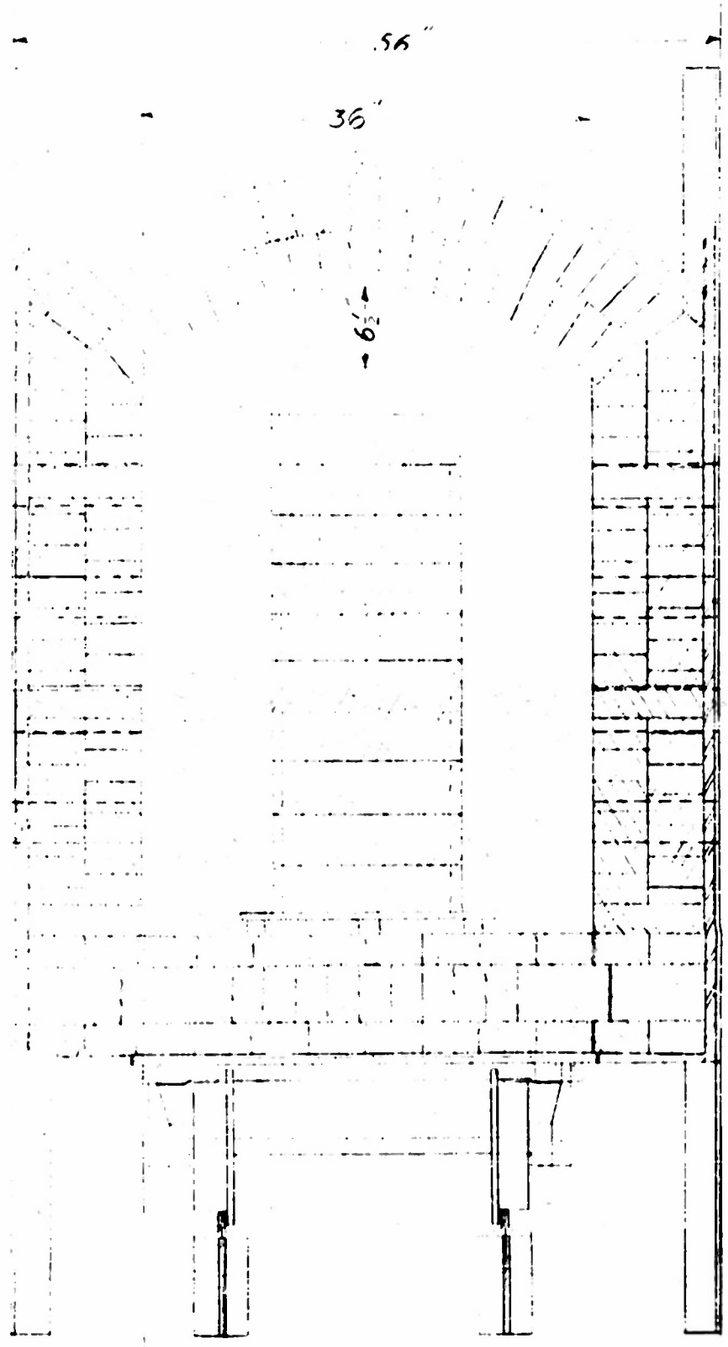
FIRECLAY BRICK

CARBORUNDUM SETTER RINGS

FLY ASH

JOHNS-MANVILLE 'SUPEREX'

SCALE : $\frac{1}{8}$" = 1"



FRONT SECTION AA

PROPOSED REFRACTORY LAYOUT FOR CAR BOTTOM TYPE PERIODIC KILN MUFFLE CAPACITY : 43" HEIGHT & 15" DIAMETER BASE SETTING

DWG. NO. 4040-3

Operation	Equipment
11. Bisque Firing	A car-bottom hearth kiln with a firing chamber 3' x 3' x 4' was used. See Dwg. 404(03)-3, Fig. B-11 and Fig. B-4. It was gas fired and equipped with an electronically controlled temperature recorder.
12. Machining	The inside and outside machining of the shape is being accomplished by a lathe turning operation. A Monarch lathe with a 16" survey and a 14' bed is being used for this purpose. Special chucking fixtures for the above mentioned machining oporations are being designed. See Figures B-5 and B-6.
13. Final Firing	Same equipment as No. 11 above.
14. Cut off	The same lathe fixturing will be used as in No. 12 above with the addition of a Dumore grinding head equipped with a diamond impregnated cut off wheel.

FIG. B-5

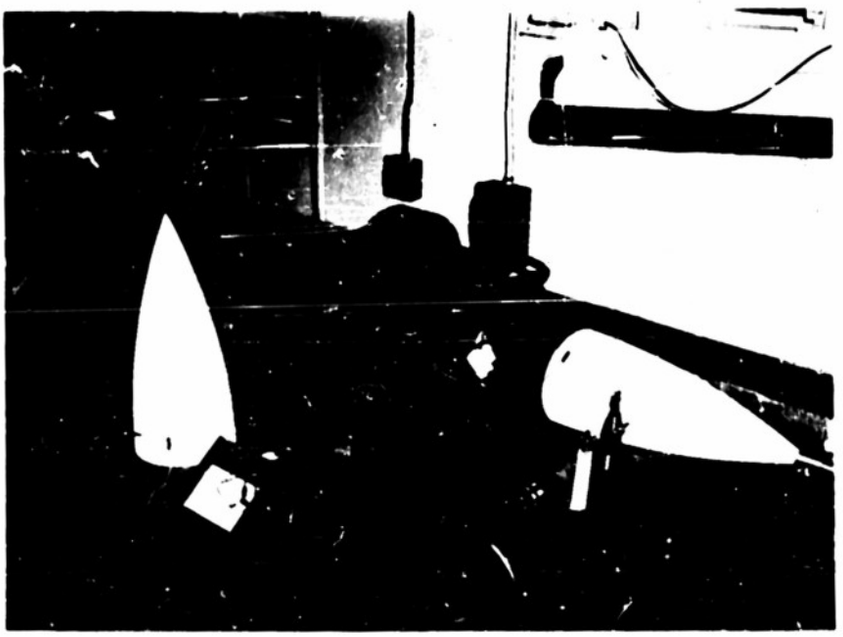


FIG. B-6

B. Techniques Developed for Producing Radome Shapes

1. Milling of Pre Vitrified Steatite Scrap

(a) A wet milling method was used for the procurement of R-1 material with a particle size range from 3μ to 0.4μ. The mill charge consisted of the following:

1) 50# of pre-vitrified steatite in thin walled tubular form or some other easily reducible form.

2) 15 gallons of water.

3) Pebble charge of approximately 1/3 the volume of the mill.

This was milled for a minimum of 18 hours. The supernatent slurry then syphoned off and dried.

(b) A dry milling method was used for procurement of R-2 material with a particle size range from 40μ to 12μ. The mill charge consisted of the following:

1) 50# of pre-vitrified steatite in thin-walled tubular form or some other easily reducible form.

2) Pebble charge of approximately 1/3 the volume of the mill.

This was milled for 6 hours. The material was then screened through a 325 mesh screen. The minus 325 mesh material was air classified.

2. Preparation of Casting Slip

A 200 pound batch with the following composition was charged into the above described ball mill.

	Material	% by Wt.	% by Volume only where indicated
Base Composition	Milled Steatite	99.0	
	Bentonite	1.0	
Additives	"N" Brand Sodium Silicate		0.132 (Vol.)
	Sodium Hexametaphosphate		3.3 (Vol.)
	(Conc. 24.55 gs./500 ml.)		
	Superloid Gel		1.0 (Vol.)
	(Conc. 10 gs./500 ml.)		
	Ammonium Hydroxide		0.2 (Vol.)
	(Conc. 26-28%)		
	Water		44.6 (Vol.)

This batch was milled for 3 hrs. The slip was then screened out of the mill through a 40 mesh screen in order to get the pebbles out of the slip.

The slip was de-aired in increments of 10 gals. in order to eliminate entrapped air. This reduced the holes in the cast piece to a minimum. A more homogeneous and dense structure

resulted giving more uniform electrical performance.

The slip was discharged from the deairing tank through the ferro filter in order to remove tramp iron particles picked up during processing. This improved the electrical properties and appearance of the radome shapes.

The slip was stored in the stainless steel storage tank with continous agitation 24 hours a day.

3. Casting the Radome Shapes

(a) The casting slip was tested for specific gravity and viscosity prior to use. Limits on these properties have been established as follows:

Specific Gravity-1.84-1.86

Viscosity -40-70

Note

The viscosity units given above were direct scale readings using the Brookfield Viscosimeter employing the No. 3 Spindle at 12 R.P.M. and the 500 scale.

(b) The mold surface was sponged with clean water.

Note

This operation may be discontinued when the mold is properly "tempered" after daily use.

(c) The tapered side of the mold top was coated with an impervious plastic cement. The flat side of the mold top which forms the "lip" extension of the radome shape was water tempered and lightly dusted with 200 mesh feldspar. The mold top was secured to the mold casing with a ring clamp.

(d) The casting slip was poured into the mold in such a manner that a minimum of air was entrapped in the casting slip.

(e) The hoist and yoke was positioned on the mold ring and the mold was raised off the floor.

(f) The casting slip was allowed to remain in the mold from 40 to 60 minutes, depending on the ultimate thickness desired.

(g) The mold was then inverted and the excess slip drained. The mold was tipped at various angles from the vertical to insure complete drainage. A total of 30 minutes draining time was allowed.

(h) The mold was then uprighted and suspended slightly off the floor and the cast radome shape was allowed to shrink away from the mold.

(i) The mold top was removed and the cast shape was allowed to remain in the mold for 24 hours, minimum or 48 hours maximum time.

(j) The setter bisqued at 1500°F, was placed on the base of the cast shape with a 3/8" rubber-tube gasket between them. A silicon carbide slab was placed over the setter resting on shims which were placed on the mold casing. The mold top retaining ring was clamped over the silicon carbide slab and the mold inverted.

(k) The mold was lowered to the floor carefully while in the inverted position. The mold clamp was removed and the mold casing was hoisted slowly from around the cast shape.

(l) The cast radome shape was allowed to remain in this position until thoroughly air dried.

(m) The dried shape was carried to the kiln car on its setter and slab. The slab was placed on setter blocks using three-point suspension.

(n) The rubber-tube gasket was replaced with cotton wads and setting sand introduced alternately. This was accomplished in the following manner: A portion of the rubber-tube gasket (2 inches) was rolled out and a cotton wad was inserted. The intervening space was then filled with setting sand. This operation was repeated until the entire gasket was replaced with cotton wads and setting sand.

(o) The shape was muffled with silicon carbide rings and finally covered with a slab. (See fig. B-7)

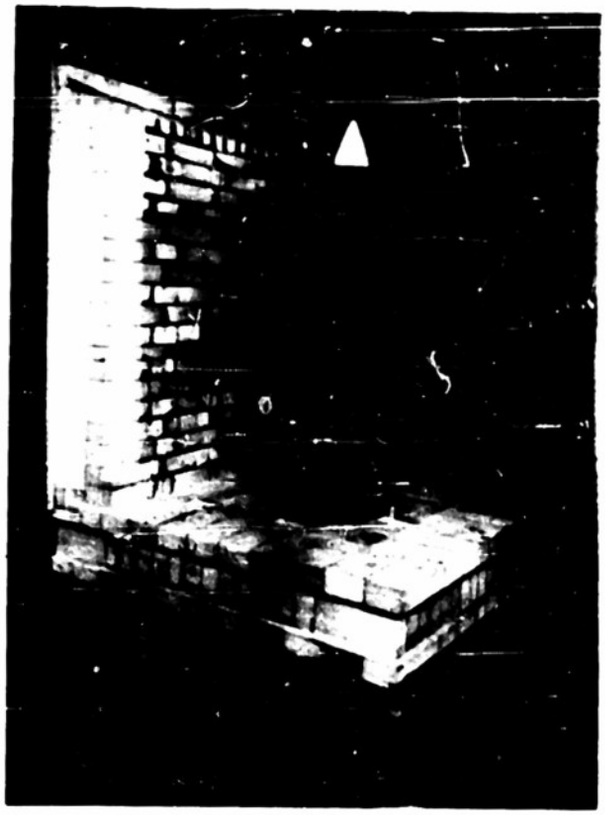


FIG. B-7

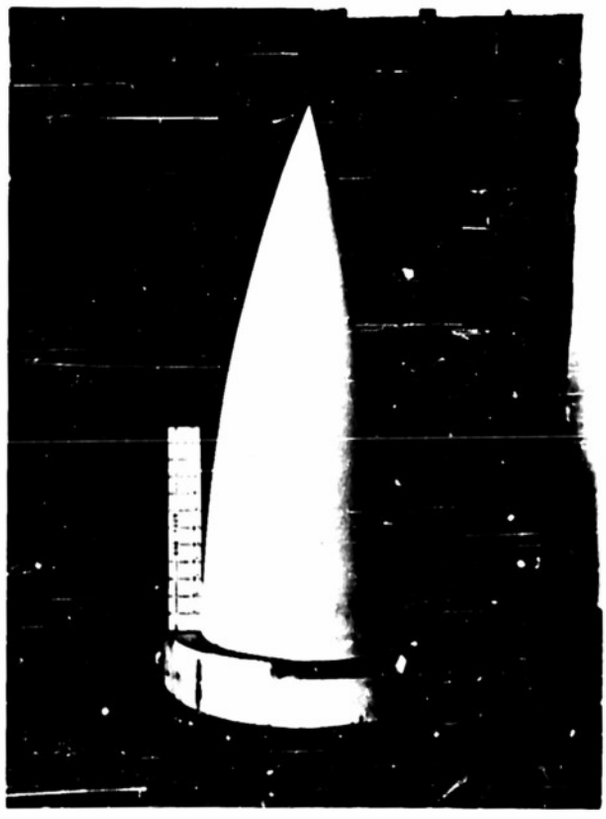


FIG. B-10

(p) The shape was bisque fired at 1800°F for a one hour period. (Cone 010 down). This temperature and soaking time was found through actual trial to be the optimum firing cycle to give the best machining qualities.

(q) All major machining and finishing operations, except that of cutting off the "lip" extension at the base, were performed on the radome shape while in the bisque state. This included the machining of both the inside and outside surfaces to obtain uniform concentricity and thickness over all. The machining dimensions were compensated for firing shrinkage to give the desired final size and thickness.

(r) The finished shape was reset on the kiln car upon its setter and slab with placing sand between each. It was muffled as indicated in "o" above.

(s) The shape was fired to 2200°F with a one hour soaking period in order to obtain the desired degree of vitrification. This was established by a standard moisture absorption test run on a small sample of the material fired with the shape. The desired degree of vitrification was considered to have .02% of water absorption or less.

(t) The fired shape was given a final inspection for physical and dimensional properties.

(u) The "lip" extension at the base of the shape was cut off to length by using a diamond saw.

(v) The base of the shape was finished by lapping with very fine abrasive material.

C. Dimensional "runout" experienced with molds in use up to the present time.

After the techniques for producing large radomes had been established and tried, consecutive casts were made from one mold, Dwg. 404(03)-1, (Fig. B-2), in order to investigate the reproducibility of the shapes from a standpoint of eccentricity. The machining operations to be performed on the radomes, specifically to maintain the thickness tolerances, were directly dependent upon the degree of concentricity of the shape.

The shape was set up in the lathe using the fixtures designed for this purpose. The degree of concentricity of the shape was then determined by an Ames dial reading to one-thousandth of an inch. This was repeated at one inch intervals along the entire length of each shape.

In the course of collecting this data certain inconsistancies indicated a common runout condition on all three shapes. Therefore, this led to an examination for eccentricity of the mold used to fabricate the three shapes in question.

This examination indicated that there were inaccuracies in the mold. The degree of eccentricity found in the mold

was the cause of the largest portion of the "runout" found in the radomes.

Upon further investigation to find the cause of this runout in the mold, it was found that inaccuracies in the model from which the mold was made, were the origin of the difficulty. To eliminate a recurrence of this condition in the future, it was planned to use an accurately machined aluminum model in place of a plaster model.

This method is being used to make the models for shapes, Dwg. No. 404(03)-5 and No. 404(03)-7. (See Figs. No. B-8 and No. B-9).

D. Testing of Radome Materials and Shapes

Wave guide samples were prepared and submitted to both the Naval Air Development Center at Johnsville, Pa. and the Raytheon Manufacturing Company at Waltham, Mass. The results obtained from Johnsville, Pa. indicated that the material had the following properties:

Dielectric Constant	6.13
Loss Tangent	0.0045

Tested at a frequency of 9375 megacycles.

While it is really premature to discuss performance of any shapes made to date, some data have been obtained from two fired shapes taken to the Naval Air Development Center

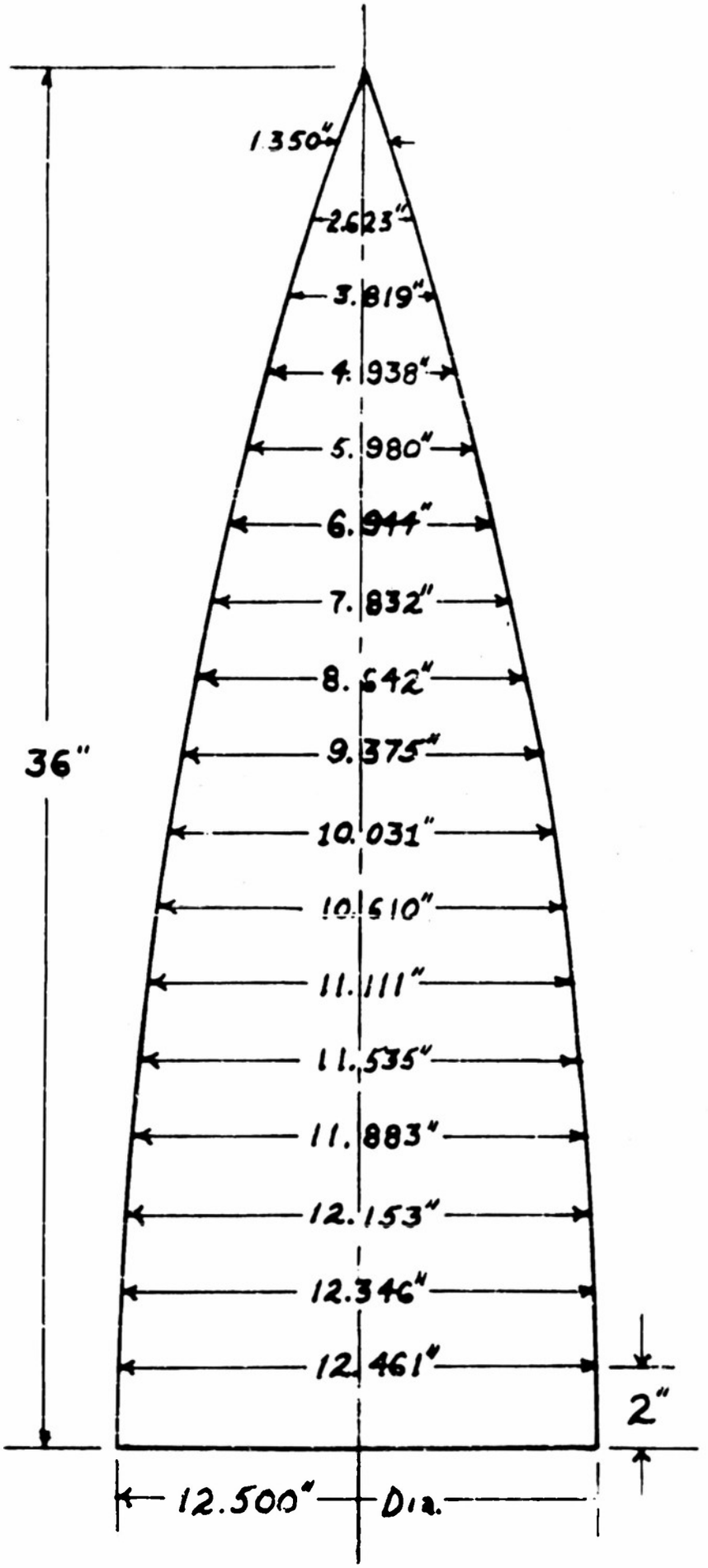


FIG 8-8

EXPERIMENTAL TEST SHAPE NO. 2.
ALL DIMENSIONS COMPENSATED TO MOLD SIZES.
Scale 3" = 1'. DWG. No. 404(03)-5

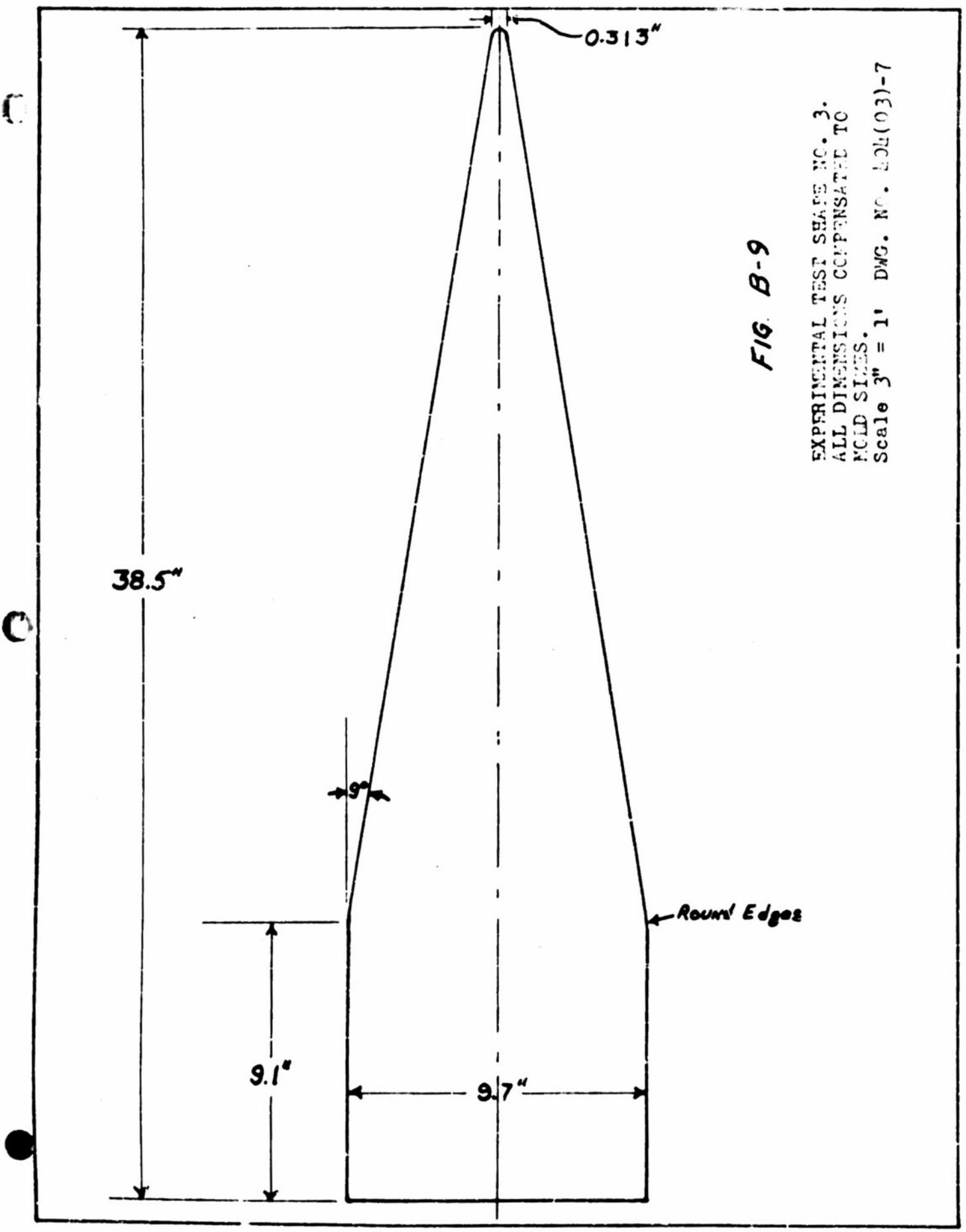


FIG. B-9

EXPERIMENTAL TEST SHAPE NO. 3.
ALL DIMENSIONS COMPENSATED TO MOLD SIZES.
Scale 3" = 1' DWG. NO. 404(03)-7

at Johnsville, Pa. Although these had not been machined in any way and no effort had been made to accomplish any exact thickness, the laboratory at Johnsville have been kind enough to give these shapes the usual test for boresight errors.

These were pointed ogives of the shapes shown in Dwg. No. 404(03)-1, (Figs. B-2 and B-10). The main difference between the two shapes was in their thickness. Shape No. 1 measured 0.237" in thickness close to the base and shape No. 2 measured 0.193". In all probability this thickness increased toward the nose. Plans were made to determine the thickness variation by cutting up these shapes after all useful data have been obtained

The thicknesses of both pieces tested (measured close to the base) were considerably lower than the optimum thickness for low reflection. This was calculated to be approximately 0.260".

Shape No. 1 showed a maximum deflection of 12-16 mils for an antenna rotation of about 14-16°. The maximum rate of change of beam deflection was about .06 degree per degree.

The percent power transmission varied from 77% for 0° to 94% for 20° azimuth angle for horizontal polarization and from 75-1/3% for 0° to 56% for 20° azimuth angle for vertical polarization.

Shape No. 2 which was, in thickness, somewhat farther from the optimum for low reflection gave slightly greater deviations and considerably lower power transmission.

Sketchy though these results are, it seems worthwhile to attempt to get the maximum amount of information from them. Accordingly, it is planned to cut up these shapes and measure thickness as a function of position. It is felt that all of this information will be useful in specifying shapes and thicknesses as soon as it is possible to control the manufacturing processes to the tolerances expected.

E. Correlation of Particle Size Distribution with Firing Shrinkage In a Ceramic Whiteware Composition.

An investigation was made to correlate particle size distribution with firing shrinkage. The correlation was established using classified fractions of pre-vitrified ceramic whiteware material. A two component system was selected with a ratio of 0.01 between the smallest, 0.4 micron, and the largest, 44 micron, fraction. The firing shrinkage on cast samples was reduced by approximately 50% when a ratio of 65% large to 35% small fraction was used. This comparison was based upon the firing shrinkage obtained when 100% of the small fraction was used. (See Appendix)

V. Future Work

In general an effort will be made to improve each phase of the operations necessary to produce radome shapes within specified tolerances.

The following specific modifications to the described procedure will be incorporated to achieve this result.

1. New molds will be constructed using precision machined metal models in order to establish a condition for minimum eccentricity of the shape.

2. The present machining procedures will be supplemented with the development of the techniques necessary to machine the inside as well as the outside of the shape.

3. The test equipment, necessary to check the uniformity and reproducibility of the dielectric properties of the material under similated operational conditions, will be procured and installed.

The reason for the procurement of this equipment was based on the following considerations:

(a) In the performance of radomes it is just as important to control dielectric constant of the material from which the radome is made, or, more precisely, the square root of the dielectric constant must meet the same percentage tolerances as the thickness, if d/λ is to be held to some predetermined value.

(b) Such equipment would prove helpful in predetermining the effects of temperature changes on the dielectric properties of the radome material. The actual testing

conditions would be made to simulate actual operating conditions through the atmosphere. A microwave Dielectrometer is being considered for this purpose.

4. New Shapes Dwg. No. 404(03)-5 and Dwg. No. 404(03)-7 as shown in Figs. B-8 and B-9 respectively will be fabricated for testing and evaluating, incorporating the new techniques described above.

5. The testing of the new shapes will be conducted with the cooperation of various organizations equipped to make the tests. The evaluation of the test results will be made by the personnel assigned to this group.

(a) The first objective will be to attain maximum transmission.

(b) When maximum transmission has been attained other parameters will be adjusted to minimize beam deflection.

Contract N-onr-404(03) Project NR 032 348

APPENDIX

TO

TECHNICAL REPORT NO. I

CORRELATION OF PARTICLE SIZE DISTRIBUTION WITH FIRING SHRINKAGE IN A CERAMIC WHITEWARE COMPOSITION[1]

William A. Contardi and George J. Hund

Abstract

An investigation was made to correlate particle size distribution with firing shrinkage. The correlation was established using classified fractions of pre-vitrified ceramic whiteware material. A two-component system was selected with a ratio of 0.01 between the smallest, 0.4-micron, and the largest, 44-micron, fraction. The firing shrinkage on cast samples was reduced by approximately 50% when a ratio of 65% large to 35% small fraction was used. This comparison was based upon the firing shrinkage obtained when 100% of the small fraction was used.

Introduction

The extent and variations of firing shrinkage of ceramic compositions have long been a matter of concern in the ceramic whitewares industry. The condition has been intensified in recent years, because of the ever increasing demand by industry and science for closer dimensional tolerances.

In an attempt to minimize the effect of firing shrinkage, a pre-vitrified ceramic material was selected as the basic material in a composition. However, the initial results

[1] Paper presented at the 55th Annual Meeting of the American Ceramic Society, Whiteware Division, April 29, 1953, New York City.

using a pre-vitrified material indicated firing shrinkages similar to those experienced with equivalent raw materials. Therefore, it was decided to investigate and attempt to correlate particle size distribution with firing shrinkage using the conventional slip casting process.

Literature Survey

A survey of the literature indicated that the particle size distribution greatly influences the particle packing. Westman[1] showed that particles of uniform shape and size will pack to a minimum of 38% voids, stating, "The closeness of packing of very large volumes of particles of uniform shape and size is independent of the size of particles, provided electrostatic and air-film effects are negligible." However, when a system contains various sized particles, the smaller ones will pack in the voids created by the larger ones. This, obviously, reduces the percentage of voids in the system.

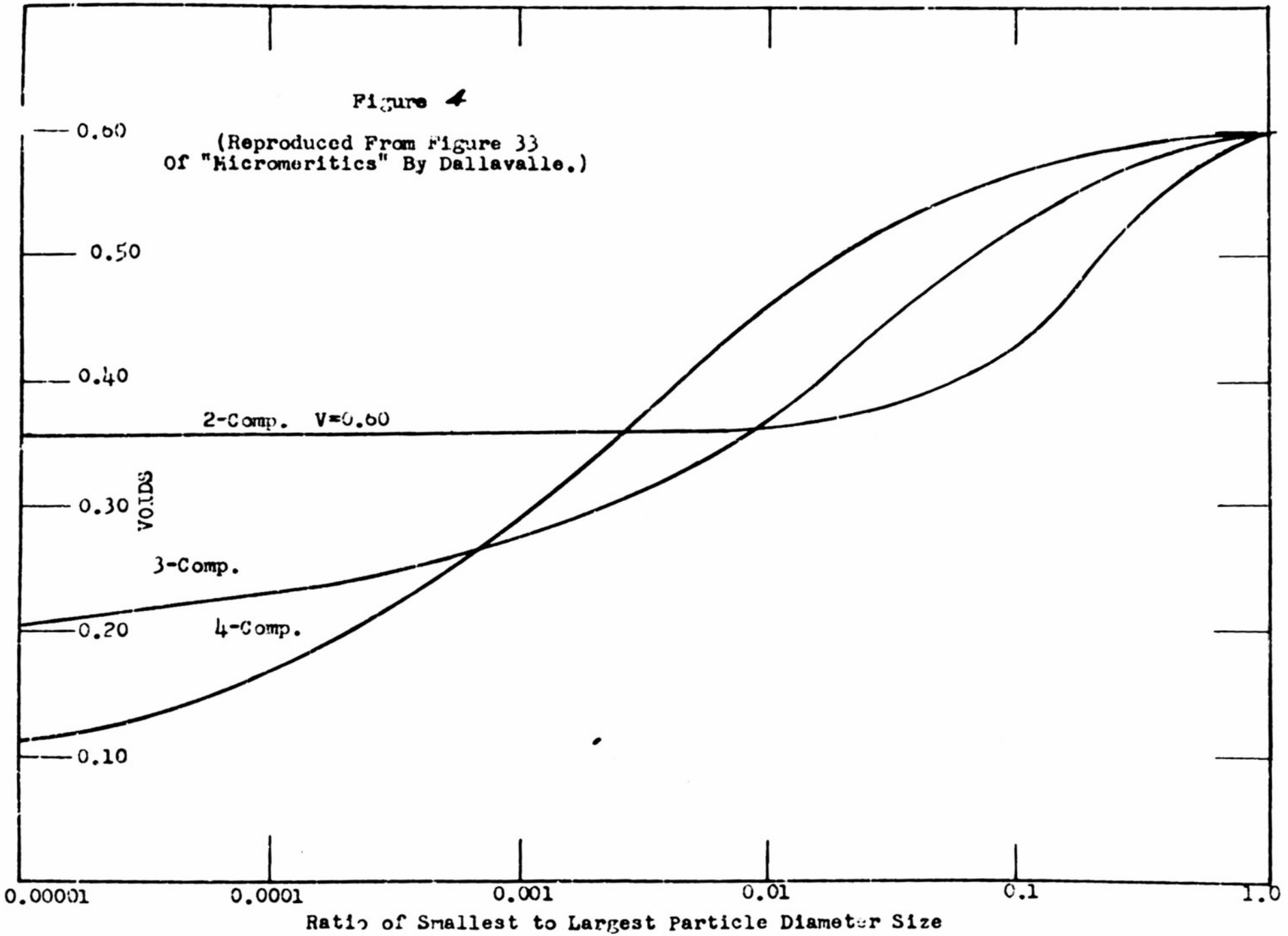
Furnas[2] pointed out that two, three or four component systems may be used to reduce the percentage of voids. He devised a system whereby the ratio between the particle size diameters determined the number of components needed to minimize the

[1] A.E.R. Westman and H.R. Hugill, "The Packing of Particles," J.A.C.S. (13), 767, 1930.

[2] C.C. Furnas, "Grading Aggregates," Ind. Eng. Chemistry 23, 1052-1058, 1931.

Figure 4

(Reproduced From Figure 33
Of "Micromeritics" By Dallavalle.)



percentage of voids in a system. See Figure 4.[3]

Discussion

The basic material used in this investigation was a pre-vitrified ceramic whiteware material, which was ground wet in a conventional ball mill. After a prescribed milling time, the suspended portion of the charge was syphoned off and dried. The remaining residue was analyzed. This analysis indicated little particle size distribution between 44 microns (325 mesh) and 3 microns. The major distribution was confined to a range of 3 to 0.4 microns. See figure 1 and Table 1.

It was decided to use Furnas's findings as a basis for this work. Because of practical considerations, a two component system was selected with a ratio of 0.01 between the smallest and largest particles. Forty-four microns (325 mesh) was selected as the upper limit because particles larger than this would prove difficult to suspend. Four tenths of a micron was selected as the lower limit of particle size because of grinding limitations.

Procedure

In order to obtain the two components needed for the system selected, wet and dry grinding were found necessary.

[3] Reproduced from J.M. Dallavalle, "Micromeritics" Fig. 33, p. 137 Pitman Publishing Corp., New York 1948, 555 Reprinted by permission.

This produced two composites, from which the classifications were made. The fine (0.4 micron) range was obtained from the wet milled composite and the coarse (44 microns) range from the dry milled composite. This produced a maximum yield in each case.

The wet milled composite was produced by the aforementioned method. The dry milled composite was produced by milling the pre-vitrified ceramic material dry, in the same mill for six hours and screening the residue through a 325 mesh screen. That material which remained on a 325 mesh screen was remilled, while the material which passed through a 325 mesh screen was used as the dry milled composite.

Two methods of classification were considered by which the wet and dry milled composites could be separated into their component fractions. They were (1) elutriation by water and (2) classification by air.

The major disadvantage of the first method was the predominating tendency for the formation of agglomerates due to drying which were not easily broken down. This condition was in evidence in the wet milled composite. See Figure 1. Therefore classification by air was adapted in this work.

The individual fractions were then analyzed by using the Casagrande hydrometer method,[4] with the long arm centrifuge

[4] F. H. Norton and S. Speil, "The Measurement of Particle Sizes in Clays" J.A.C.S. (21), 3, 89, 1938.

adaptation.

In this method, the amount of suspended material is determined at any one time by the specific gravity of the suspension as measured by a special hydrometer bulb. A special centrifuge has been developed and adapted to the method to accelerate the settling rate of particles below 1 micron in diameter.

Using this method, accurate measurements are possible down to 0.05 micron.

Figures 1 and 2 and Table 1 illustrate the particle size distribution of each individual fraction of both composites.

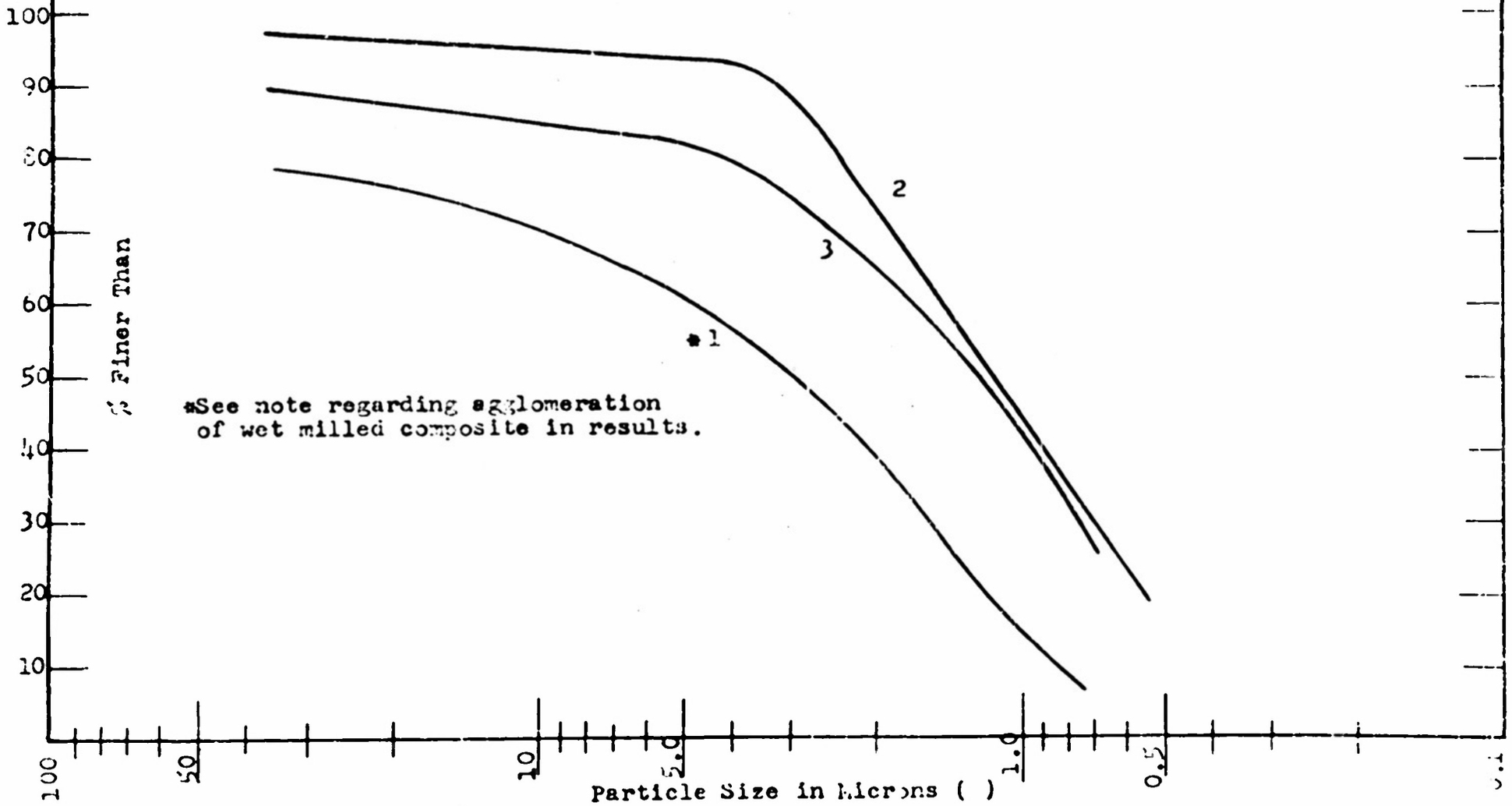
Individual batches of casting slip were prepared from each of the composites and from each of their classified fractions. The base composition (weight basis) consisted of 99% of the nonplastic pre-vitrified material and 1% of bentonite. The water and other additives were kept constant in all cases, in order to get a true shrinkage comparison. The casting slip made from the coarse fraction of the dry milled composite was designated "A". The casting slip made from the fine fraction of the wet milled composite was designated "B". Casting slips "A" and "B" were then blended together in varying proportions based on the weight of the individual slips.

Round test bars 3/4" in diameter and 4" long were cast from the casting slips prepared from each of the composites,

Figure 1

PARTICLE SIZE DISTRIBUTION CURVES FOR

(1) WET MILLED COMPOSITE
(2) WET MILLED FINE FRACTION
(3) WET MILLED COARSE FRACTION



*See note regarding agglomeration of wet milled composite in results.

Figure 2

PARTICLE SIZE DISTRIBUTION CURVES FOR
(1) DRY MILLED COMPOSITE
(2) DRY MILLED FINE FRACTION
(3) DRY MILLED COARSE FRACTION

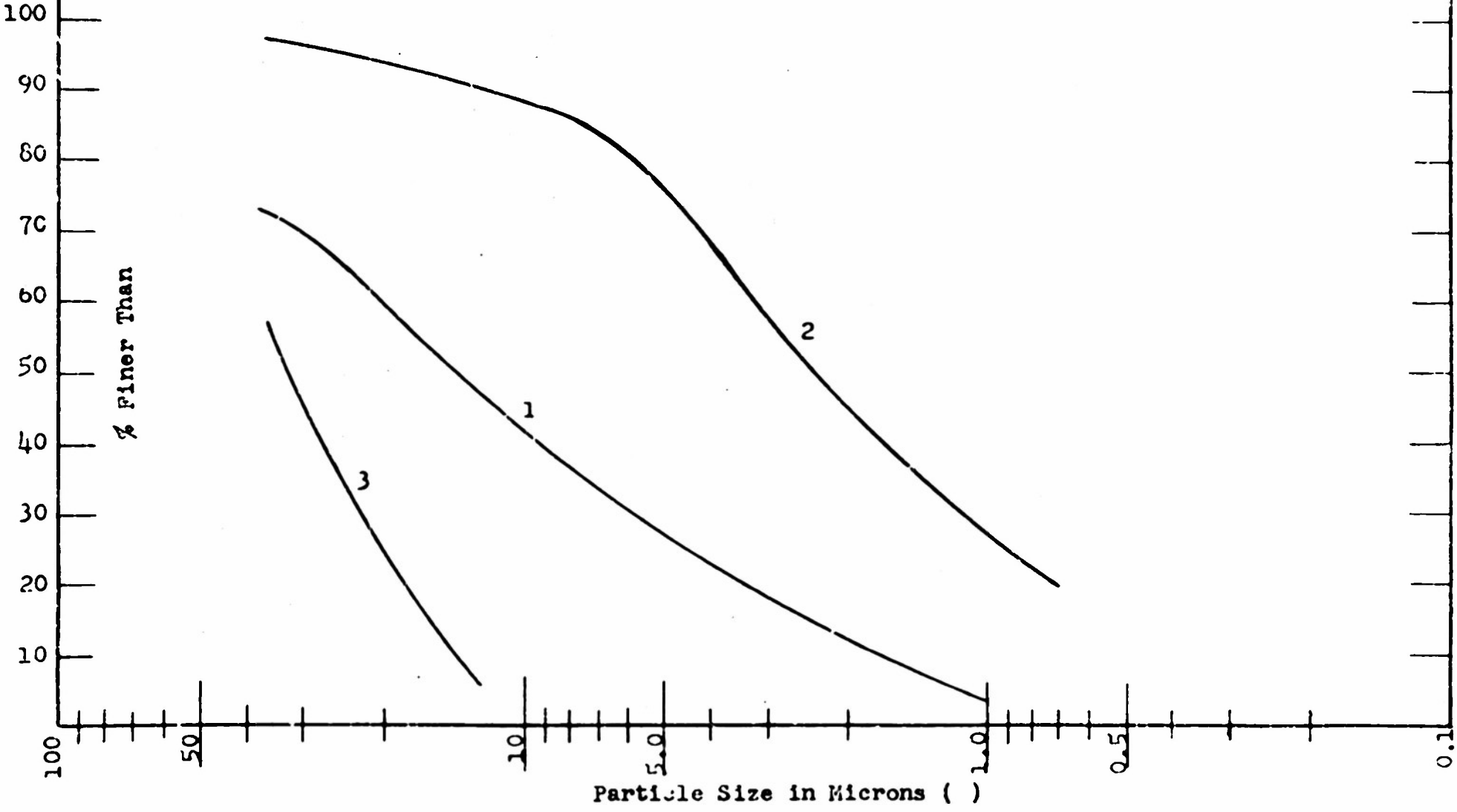


TABLE I

PER CENT (%) FINER THAN

Particle Size Range in Microns (μ)	Wet Milled Composite (R-1)*	Wet Milled Fine Fraction	Wet Milled Coarse Fraction	Dry Milled Composite	Dry Milled Fine Fraction	Dry Milled Coarse Fraction
37.5	78.5	97.6	90.1	73.8	97.5	57.1
21.5	76.2	95.7	86.2	63.5	95.3	31.8
17.0	73.0	94.4	83.9	53.2	90.5	18.3
12.0	71.5	96.0	83.5	46.0	87.3	5.55
8.5	69.8	93.6	83.5	38.9	85.7	--
6.8	63.5	96.0	83.5	31.8	85.7	--
4.8	60.4	94.0	82.6	27.8	69.7	--
3.4	52.0	92.8	76.6	23.0	59.9	--
1.9	35.0	70.6	62.4	13.5	48.4	--
1.45	26.2	57.9	54.8	7.9	44.4	--
1.20	20.7	46.8	46.8	7.9	35.7	--
0.94	12.7	38.8	38.9	4.0	26.2	--
0.68	7.15	23.0	25.4	--	19.1	--
0.50	--	19.1	--	--	--	--

* Not truly representative because of agglomeration.

fractions, and blends. Small cones were drain cast from the casting slips which had the better casting properties. The test specimens were dried and fired to vitrification in a small gas fired periodic kiln. The degree of vitrification was determined by the standard moisture absorption test. The specimens having .02% or less moisture absorption were considered dense.

Results

See Table 2 for the characteristics of the sample batches in the various stages of fabrication.

1. Casting Slip Properties

Generally, the blending of casting slips "A" and "B" produced better casting slip properties than either of the end points alone. For example, the casting slips which were prepared with a high concentration of fine particles, such as the wet milled fine fraction, were characterized by a gelling tendency upon standing. On the other hand, the casting slips prepared with a high concentration of large particles, such as the Dry Milled coarse fraction, exhibited a slight settling tendency. When blended together the detrimental slip properties of end points "A" and "B" were completely eliminated indicating a complementary action of one for the other.

2. Drying Properties

The samples prepared from the trial casting slips dried satisfactorily. Some differences were observed in the drying rates of the samples cast from the various slips. For example, samples prepared from the 70% coarse and 30% fine particle sizes dried at a slower rate than the samples cast from the other slips. This condition is believed to be caused by dense particle packing, reducing to a minimum the capillaries through which the water might travel to the surface. The slips with high concentrations of large particles (example: 76.4% A + 23.6% B) had low drying shrinkage and were difficult to release from the molds. The slips with the high concentration of fine particles (example: wet milled composite and wet milled fine) had high drying shrinkage and released easily from the molds.

3. Handling Properties in the Dry State

The specimens cast from the slips with high concentrations of small particles (example: wet milled composite, wet milled fine, dry milled fine fraction) were difficult to machine. The test samples chipped and fractured when cut by an abrasive wheel. The same samples could be ground easily on a felt pad. The specimens prepared from the blended casting slips handled satisfactorily and indicated good machining properties.

4. Fired Shrinkage

The firing shrinkage changed substantially with varying proportions of fine to coarse material, see Figure 6. Generally, the shrinkage was reduced by increases of the coarse fraction at the expense of the fine fraction. However, a limit was established at 30% fine and 70% coarse blend. Larger percentages of the coarse fraction resulted in increased firing shrinkage. This substantiated the findings of Furnas and Westman.

The discrepancies in the firing shrinkage of the wet milled composite is believed to be the result of agglomerates formed in drying the wet milled composite slurry. It is evident that the agglomerates do not break down in the casting slip, but remain as such with excessive voids. During the firing, however, the fine particles comprising the agglomerates act individually and break down causing increased shrinkage such as is shown in the table (see Table 2).

5. Firing Range

In general, the firing range is increased by the fineness of the particle. The specimens which contained large percentages of fine particles (example: wet milled composite and wet milled fine fraction) became dense at a lower maturing temperature than those containing the larger particles. However, the upper limit of temperature remained constant for

Figure 6

% SHRINKAGE VS PARTICLE SIZE DISTRIBUTION
(MOLD SIZE TO FIRED SIZE)

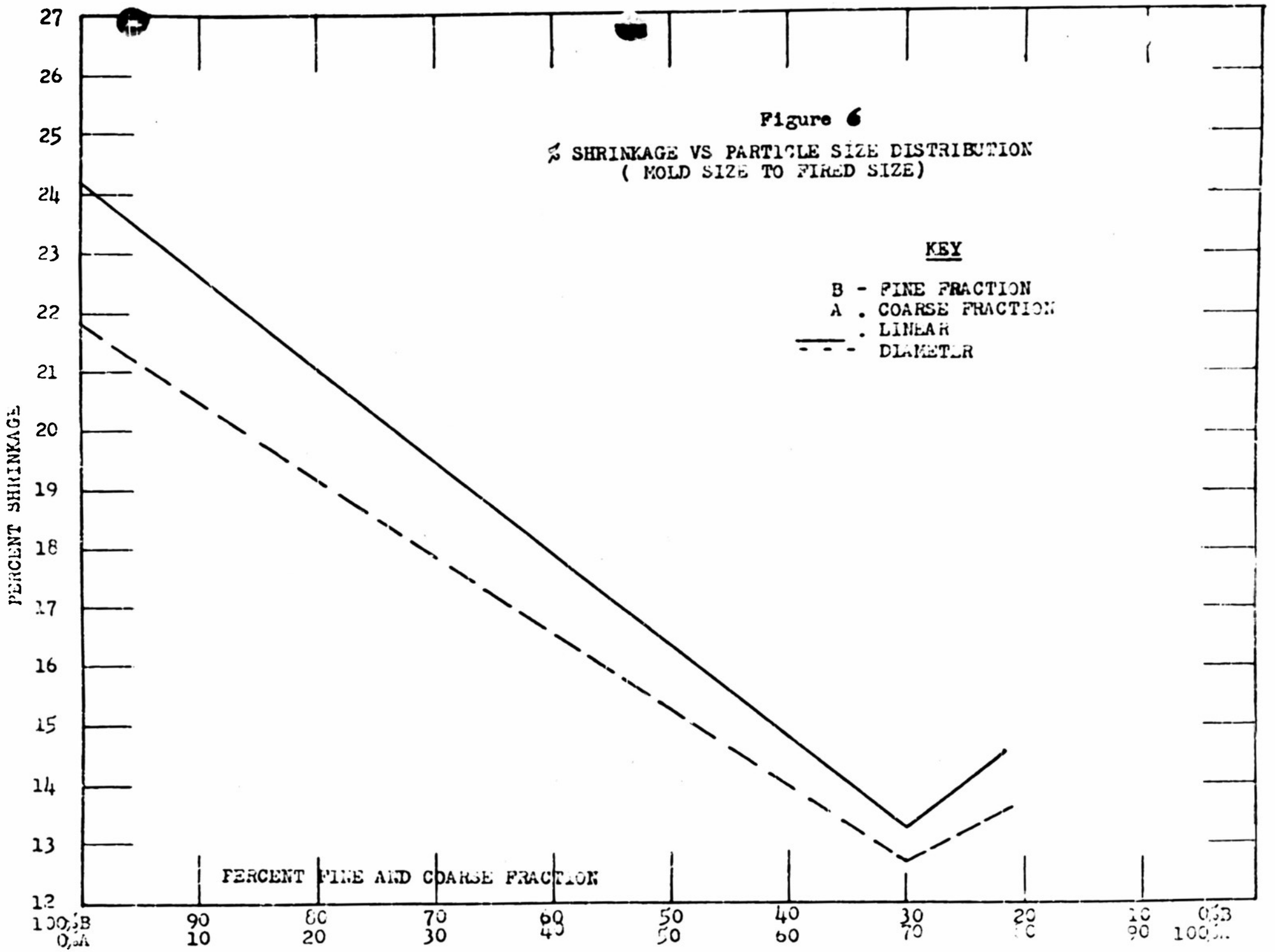


TABLE 2

Material	Slip and Casting Properties	Drying Properties	Handling Properties in Dry State	Firing Shrinkage Diam.	Firing Shrinkage Length	Texture
Wet Milled Composite (C-1)	Exceptionally good	Good	Brittle, little strength. Machined poorly.	26.65	22.05	Exceptionally smooth. Almost glassy.
Wet Milled Fine (B)	Needed frequent water adjustments. Fair	Excessive shrinkage	Same as wet milled composite	24.2	21.8	Exceptionally smooth.
Wet Milled Coarse	Needed water adjustments. Fair	Excessive shrinkage	Same as wet milled composite	24.85	20.2	Smooth
Dry Milled Composite	Good	Good	Fair strength Fair machining properties	16.6	15.1	Fine
Dry Milled Fine	Needed frequent water adjustments	Excessive shrinkage	Brittle, fractured on machining. Poor handling properties.	21.6	21.15	Smooth
Dry Milled Coarse (A)	Difficult to suspend. Unable to cast.	--	--	--	--	--
Blend 73.4% B 26.6% A	Good	Good	Fair strength. Substantial improvement on machining over C-1	19.7	18.2	Exceptionally smooth. Slight gloss.

TABLE 2 Cont'd.

Material	Slip Casting Properties	Drying Properties	Handling Properties in Dry State	Firing Shrinkage Diam.	Firing Shrinkage Length	Texture
Blend 48.0% B 52.0% A	Exceptionally good	Good	Good strength. Machined well.	16.2	15.2	Fine
Blend 40% B 60% A	Exceptionally good	Low shrinkage. Exceptionally good	Good strength. Machined exceptionally well.	14.85	14.0	Fine
Blend 38.2% B 61.8% A	Exceptionally good	Low shrinkage. Exceptionally good.	Same as blend 40% B 60% A	14.7	13.9	Fine
Blend 30% B 70% A	Good	Low shrinkage. Slow drying.	Same as blend 40% B 60% A	13.3	12.7	Exceptionally fine.
Blend 23.6% B 76.4% A	Exceptionally poor	Exceptionally low shrinkage. Cracked in molds.	Fair. Indicates tendency to break on machining.	14.55	13.5	Indication of roughness.

all samples. All of the test specimens indicated tendencies to overfire at 2300°F with 1/2 hour soak when fired in a laboratory gas fired kiln.

6. Texture

In general the finer the particle, the smoother the texture of the fired specimen. However, smooth fired texture was obtained also when optimum particle packing was achieved (example: 70% A and 30% B blends).

7. Appearance

Electromagnetic separation beneficiated the appearance of the samples in that it removed tramp iron which entered the casting slip at various stages in the processing of the pre-vitrified material. Visual improvements in appearance were evident especially in those bodies containing larger amounts of coarse material.

Conclusions

The results prove that there is a correlation between firing shrinkage and particle size distribution. A reduction of 50% in firing shrinkage was realized by using a two component system of particles whose diameters bear the ratio of 0.01, the smallest to the largest.

In carrying out this work certain information, important and relative to the process, has been gained in addition to establishing the correlation. This includes the effect of

part [illegible] on:

a. Firing Range

b. Machining properties

c. Texture

d. Drying properties

Acknowledgment

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