AD 419255

DEVELOPMENT OF REFRACTORY CERAMICS THAT CAN BE PROCESSED AT TEMPERATURES CONSIDERABLY LOWER THAN THEIR MAXIMUM USE TEMPERATURE

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PREFACE

The undersigned constitute the planning committee for this project. Professor E. J. Smoke is project leader. The experimental work has been carried out by Mr. Peter Fleischner and Mr. Chester Ringel.

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Abstract

Cordierite compositions were prepared utilizing the fritted composition as the basic raw material. Density, moisture absorption, X-ray determinations, thermal expansion, and transverse strength results are shown.

PART II - SINTERING STUDIES

Abstract

A study of the densification of six compositions in the alumina magnesia system was carried out in vacuum, dry hydrogen and dry helium. The compositions range from pure alumina at one end to a 2.0% magnesia content at the other end. Density and moisture absorptions are shown as a function of the composition and firing atmosphere.

I. PRESINTERING STUDIES

A. Introduction

This phase of the program is devoted to the application of the devitrification technique of the prereacted material procedure for lowering the firing temperature of ceramic materials yet retaining all the desired engineering properties. This approach is best suited for radome materials to be used at 2200°F and lower.

In the devitrification process, the whole composition is melted and poured into water to retain the glass or randomly oriented structure. This now results in the mixing of the composition on an atomic basis. The quenched glass is reduced to a controlled particle size distribution and fabricated. During the second firing a crystalline phase devitrifies from the glass and the specimen fires to the highest bulk density stage. Values of bulk density of 99% of the true density of compositions have been realized.

The present effort is devoted to the development of a ceramic material of a low thermal expansion, possessing a high density, which can be fabricated easily into radome shapes. The fabrication which lends itself to this type of body is the utilization of prereacted raw materials.

Since this process lends itself to lesser refractory compositions, cordierite was chosen as the initial material in this study. Cordierite is a magnesium alumino-silicate composition that possesses a very low thermal expansion.

A ceramic material with a low thermal expansion may possess lower strength properties under normal conditions, however, if thermal shock is introduced the low expansion body can possess greater strength properties than a stronger high expansion body in which thermal shock has been introduced.

B. Procedure

The compositions were selected as identified in the tri-axial diagram Figure 1 on page 3 and tabulated in Table 1 on page 4. All the compositions were weighed and mixed for one hour in a V-blender. Each batch was melted at approximately 2700°F in fire clay crusibles using a pot furnace, then quenched by pouring into cold water. The fritted material was ground in an alumina mill with alumina grinding media for 24 hours and passed through a 200 mesh screen specimens were prepared from all compositions by dry pressing in a steel die. Draw trials were made in order to establish the maturing range and the specimens were analyized for crystalline phases present.

C. Compositions

All compositions chosen are shown in Table 1. The bodies were prepared in 2000 gram batch using Alcoa A-14 alumina, Pennsylvania Glass Sand Company, "Supersil" silica and Magnesium

Figure 1

COMPOSITIONS CHOSEN IN
$MgO-Al_2O_3-SiO_2$ PHASE DIAGRAM

C-1

60 SiO_2
15 MgO
25 Al_2O_3

C-2

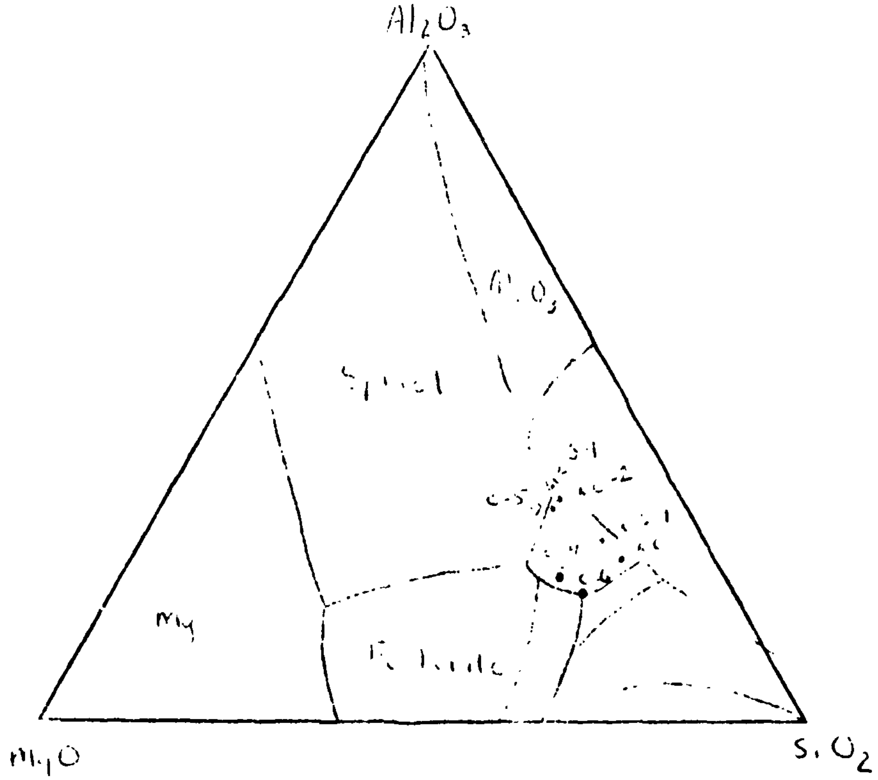
50 SiO_2
17 MgO
33 Al_2O_3

C-3

64% SiO_2
11% MgO
25% Al_2O_3

S-1

48% SiO_2
17% MGO
35% Al_2O_3



C-4

58% SiO_2
19% Al_2O_3
23% MgO

C-5

63% SiO_2
20% Al_2O_3
17% MgO

C-6

61.4% SiO_2
18.3% Al_2O_3
20.3% MgO

TABLE I

Compositions and Properties of C Series

	C-1	C-2	C-3	S-1
	60 SiO_2 25 Al_2O_3 15 MgO	50 S 33 A 17 M	64 S 25 A 11 M	48 S 35 A 17 M
Max. D.[1]	2.45	2.08	2.22	2.25
Moisture Abs.[2]	0.10	7.43	3.35	3.50
Temp. (°F)[3]	2450	2450	2450	2450
X-ray[4]	Cord.	Cord.	Cord.	Cord.

				Commercial Kiln Firing
	C-4	C-5	C-6	C-6
	58 S 19 A 23 M	63 S 20 A 17 M	61.4 S 18.3 A 20.3 M	61.4 S 18.3 A 20.3 M
Max. D.	2.587	2.587	2.457	2.441
Moisture Abs.	0.1	0.2	0.4	0.0
Temp. (°F)	2400	2400	2350	2290
X-ray	Cord.	Cord.	Cord.	Cord.
Trans. S.[5]	----	----	----	10,500 psi

	CB-7	CB-8	CB-9	CB-10
	58 S 19 A 23 B	58 S 19 A 11.5 M 11.5 B	58 S 19 A 7.67 M 7.67 B 7.67 C	58 S 19 A 5.75 B 5.75 S 5.75 C 5.75 M
Max. D.	2.813	2.828	2.738	2.655
Moisture Abs.	1.4	0.8	0.0	0.0
Temp. (°F)	2200	2200	1900	1900
T. E.[6]	----	----	8.72×10^{-6}	8.31×10^{-6}

1. Maximum Density Attained
2. Moisture Absorption (%)
3. **Firing Temperature (°F)**
4. Crystalline Phases shown by X-ray Analysis
5. Transverse Strength
6. Thermal Expansion

Carbonate-Mallincrodt Chemical Works. The compositions are shown in Figure 2. The phase diagrams of the $MgO-Al_2O_3-SiO_2$ system.

D. Results

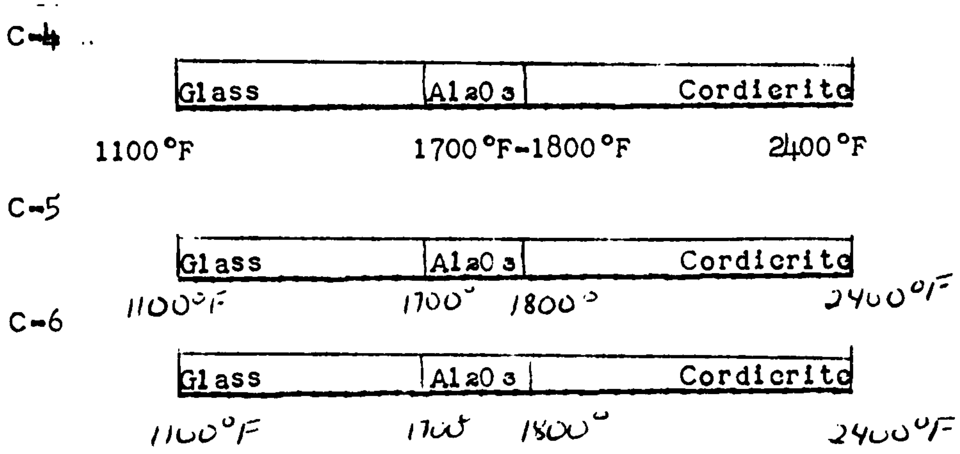
All results are shown in Table I on page 4.

Compositions C-1, C-2, C-3, C-4, C-5, C-6, and S-1 from the draw trial procedure were X-rayed to determine the crystalline phases present at various temperatures in the firing operation. The samples removed from the kiln in the draw trials were used for the X-ray analysis.

All these compositions devitrified at 1700°F into alumina, and at all higher temperatures cordierite was the only phase present. This is shown in Table II.

TABLE II

Crystalline Phases vs. Firing Temperature as Determined From Draw Trials



Thermal expansion determinations were performed using an interferometer on samples CB-9, and CB-10, cordierite compositions in which a series of alkaline earths were substituted for the magnesia. The thermal expansion of these compositions was desired since they attain a non-porous state as low as 1600°F. The compositions possessed relatively high thermal expansions 8.72×10^{-6} and 8.31×10^{-6} for bodies CB-9 and CB-10 respectively.

The thermal expansion of the other cordierite compositions will be shown in the following report.

Compositions C-1, C-4, C-5, and C-6, all cordierite bodies, attained maturity in the temperature range of 2350-2450°F. The lowest firing composition, C-6, was fired in an industrial kiln at approximately 2290°F and was found to attain maturity under these conditions. Compositions C-2, C-3, and S-1 did not approach maturity as high as 2450°F. The maximum densities and lowest moisture absorption at the corresponding temperatures are shown in Table III.

Compositions CB-7, CB-8, CB-9, and CB-10 are a cordierite composition in which the magnesia has been replaced by a series of alkaline earths. Compositions CB-9 and CB-10 were found to be non-porous at 1600°F. These compositions attained their highest density at 1900°F and began to melt at 2200°F. These results are shown in Table III.

TABLE III

	Density	Moisture Absorption (%)
C-4		
2350 °F	2.541	0.11
2400 °F	2.587	0.13
2450 °F	2.456	0.30
C-5		
2350 °F	2.407	0.09
2400 °F	2.487	.20
2450 °F	2.216	2.2
C-6		
2350 °F	2.457	0.74
2400 °F	2.443	0.44
2450 °F	2.190	0.36
Commercial Kiln		
2290 °F	2.441	0.20
CB-9		
1600	2.593	0.00
1700	2.666	0.00
1800	2.738	0.00
1900	2.738	0.00
2000	2.726	0.00
2100	2.717	0.00
2200	2.523	0.00

TABLE III (Cont.)

CB-10

1600	2.495	0.00
1700	2.601	0.00
1800	2.646	0.00
1900	2.655	0.00
2000	2.648	0.00
2100	2.643	0.00
2200	Melted	

E. Summary

The devitrification technique of the pre-reacted materials procedure has been utilized to prepare a series of compositions whose principal crystalline phase is cordierite a magnesium aluminosilicate which exhibit low thermal expansion. Of 10 compositions prepared and partially evaluated, one in particular shows considerable promise. It matures to 0.00% moisture absorption when fired to approximately 2300°F in a laboratory kiln and in an inducted thermal kiln. Its transverse strength is 10,500 psi. The bulk density is high, however, its percentage of true density has not been determined as yet, nor has its thermal expansion. These properties will be reported in the next report.

In an effort to reduce the firing temperature of a cordierite composition a series of alkaline earth oxides were substituted for the magnesia. This resulted in maturity at 1700°F, however, addition of these oxides has resulted in rather high thermal expansions.

II. SINTERING STUDIES

A. Introduction

An investigation of densification in vacuum of pure alumina and alumina containing 0.25% and 2.00% MgO was completed and reported. In order to determine the range of solid solution and densification characteristics three additional compositions were fabricated between the 0.25% and 2.00% MgO compositions. Six compositions were fabricated and fired in vacuum, dry hydrogen and helium. The effects of compositional change and atmosphere change on densification are compared in this report. An understanding of the manner in which firing atmospheres and additives affect the sintering of pure aluminum oxide will enable better control of the sintering process. This in turn will reduce the firing temperature of high alumina ceramics and maintain the refractoriness of these compositions.

B. Theoretical Considerations and Methods of Approach

The six compositions investigated range in composition from 100% Al_2O_3-0% MgO to 98% Al_2O_3-2.00% Mgo. As seen from the vacuum firings described in previous reports there seems to be a different mechanism operative in the sintering in the 0, 0.25 and 2.00% MgO compositions. It was postulated that this difference might be attributed to the formation of a solid solutionof magnesia in pure alumina for the 1-4% MgO composition and the formation of a solid solution and a second phase, namely spinel, for the 2.00% MgO composition, X-ray determinations will be made on the six compositions investigated. In order to make very precise measurements utilizing X-ray diffraction, reflection of X-rays will be made at high Bragg angles. Since the d spacing is a function of Sm θ^{-1} at high values of θ the precision of the value of d determined by x-ray diffraction increases. Any change in d spacing as MgO is added to pure alumina indicates the formation of a solid solution of magnesia in alumina. When no further change of d spacing occurs as more MgO as added to alumina the limit of substitutional solid solution has been reached. In this way the number of vacancies introduced to the alumina matrix can be calculated. The phase relationship between solid solution and formation of a second phase will be evaluated as a function of composition, temperature, and firing atmosphere. In addition a comparison of the densification characteristics of these six compositions will be compared as a function of temperature and atmosphere.

The program described above will afford a greater degree of control of the sintering of aluminum oxide. In this way control of grain size and bulk density can be obtained for near 100% Al_2O_3 compositions that do not contain a glassy phase. In addition, the maturing temperature of these compositions can be considerably reduced and the matured body will have more desirable engineering properties than those maturing within this temperature region.

C. Compositions

The compositions to be used in this investigation are:

1. 100% Al_2O_3 - 0.00% MgO
2. 99.75% Al_2O_3 - 0.25% MgO
3. 98% Al_2O_3 - 2.00% MgO

In addition to the above mentioned compositions three other compositions were prepared for X-ray studies. Density and microstructure of these additional compositions will be compared at the five hour soaking time to the three major compositions.

These compositions are as follows:

1. 99.5% Al_2O_3 - 0.50% MgO
2. 99% Al_2O_3 - 1.00% MgO
3. 98.5% Al_2O_3 - 1.50% MgO

D. Fabrication

One hundred gram batches were prepared of the six compositions under investigation. Alumina (Linde "A") was weighed on an analytical balance and placed in a glass jar. Magnesium nitrate solution, in the proper proportion was added to the batch. Five hundred mullilities of distilled water was added to form a slurry. The glass jar was sealed and agitated for half an hour, and the slurry poured into a pan covered with aluminum foil. The residual slurry was removed from the glass jar by repeated washings with distilled water and added to the pan. Next, the slurry was dried and placed into a jar. The residual material sticking to the aluminum foil was carefully scrapped off and placed into the jar making sure that no agglomerations had occurred. The dried material was agitated in the glass jar for 30 minutes and then removed and placed in an alumina mortar andpestle where sufficient distilled water to prepare a powdered compact was added. The batch was remixed and ground. In order to avoid the sticking of the batch to the die, one piece of paper was peaced on each punch face. Discs of the raw material were prepared in hydraulic press with a 5/8" die, pressed to 50,000 psi and then removed and stored in a desication.

E. Firings

Firings of the six compositions were carried out at 1550, 1650, 1700 and 1750°C, for a five hour soaking period, in atmospheres of dry hydrogen, dry helium and vacuum.

The furnace used for these firings is a high temperature vacuum furnace, utilizing resistance heating. The heating element is a cylindrical sheet of molybdenum twelve inches long by 0.005" thick. The diameter is approximately two inches. Electrical contact in the furnac was maintained by press fitting the molybdenum heating element between an upper contact cone at the top of the heating element and a lower contact cone at the bottom of the heating element. These cones rest on an upper and lower seat respectively. A further detailed description of the furnace can be found in progress report No. 3.

Difficulty was encountered in maintaining electrical contact in the furnace when dry hydrogen was used. In order to remedy this situation the following was done.

1. Holes were drilled and topped into both the top contact cone and the top seat. Screws were then put in thereby stopping the top contact cone from rising and breaking electrical contact.

2. Two pins were soldered into the bottom seat and two keyways were cut into the bottom contact cone. When a torque was applied to the bottom contact cone the pins were able to slip into the keyway thereby stopping the bottom contact cone from rising and breaking electrical contact.

A problem of oxidation developed with the use of a helium atmosphere. The molybdenum heating element was found to oxidize. In order to eliminate this during a firing dry hydrogen gas was introduced until 1000°C at which time it was discontinued. It was found that by doing this the oxidation of the heating element was nearly completely stopped.

It is speculated that the source of oxygen is due to the residual air in the furnace and the air absorbed on the surface of the molybdenum heating element.

F. Results

Bulk densities and moisture absorption results for the six compositions investigated as a function of temperature and firing atmosphere are summerized in Table 1 on page .

1. Moisture Absorption

It can be seen that in vacuum only the 0.00% MgO composition does not attain 0.00% moisture absorption at the two lowest temperatures investigated. In hydrogen it can be seen that the 0.00% MgO composition doesn't have a zero moisture absorption at 1550°C. The moisture absorption of the 2.00% composition is much lower than the 0.00% composition. In the case of the helium firings it can be seen in Table I that all the compositions do not have zero moisture absorptions at 1550°C except for the 0.50% MgO composition. In addition the moisture absorption for the 0.00 and 1.50% compositions are the highest and the 0.25 and 2.00% MgO compositions are the lowest. In the case of hydrogen and helium there is no moisture absorption above 1550°C.

2. Bulk Density

Figures 1, 2, and 3 are plots of bulk density versus firing temperature, in a particular atmosphere, for the six compositions investigated. These data are shown in tabular form in Table 1 on page .

Figure 1 is a plot of bulk density in vacuum, as can be seen from this figure the 0.00% MgO composition has the lowest density of all six compositions. The 0.50 and 0.25% have the highest density with the 1% composition lower but very close to the 0.25% MgO composition. The 1-1/2 and 2% composition have intermediate densities but these densities diverge at the higher temperatures while the 1/2, 1/4 and 1% compositions tend to converge at the higher temperatures. It is seen from these graphs that the 1/4% and 1/2% MgO compositions approach theoretical density at 1700 and 1750 °C.

Figure two is a plot of Bulk Density versus Firing Temperature in dry hydrogen. It can be seen that theoretical density (3.987) is very closely approached by the 1/2, 1/4, and 1% MgO compositions. The 2% and the 1-1/2% compositions are again of intermediate density with the density of the 2% composition dropping off at 1750 °C. The pure alumina composition has the lowest density.

Figure three is a plot of Bulk Density Versus Firing Temperatures in dry helium. It is interesting to note that after 1650 °C the densities of all compositions remain the same or drop. The densities of the 1/4% and 1/2% MgO compositions are the highest and are fairly close but they do not approach theoretical density as in the case of vacuum or dry hydrogen. The same general relationship of composition and density holds in this atmosphere as in the others.

G. Discussion

1. Compositional Density Variations

It was found that with all the atmospheres investigated that the 1/4, 1/2 and 1% MgO composition had the highest degree of densification. The 1/4% MgO composition was found to have the highest density while the 1/2% MgO composition had the same or very slightly lower density. The 1% MgO composition always had the lowest density of the three. From this it appears that the limit of solid solution is somewhere in between the 1/4% and 1% magnesia. This would mean that there would be an increase in vacancy concentration due to substitutional solid solution and therefore an increase in the sintering rate for all three compositions. In the case of the 1% composition some of the MgO might also go in as a second phase and thereby inhibit sintering and counteract some of the beneficial effect on sintering of the MgO in solid solution. This effect is further increased with the further addition of magnesia, thereby yielding lower densities for the 1.5 and 2% MgO compositions. The data show that the 1.5% composition is lower than the aforementioned three compositions and the 2% MgO composition is still lower. The pure alumina composition has the lowest density of all. This indicates the beneficial effect magnesia has on densification, even if some of the Magnesia forms a second phase. The proposed mechanisms mentioned above can be investigated by X-ray diffraction at high Bragg Angles.

2. Firing Atmosphere - Density Variations

In all three atmospheres the effect of composition on density is the same. It can be seen that both the 1/4% and 1/2% MgO compositions approach theoretical density in both dry hydrogen and vacuum. They do not approach very near theoretical density in a helium atmosphere. This effect may be attributed to the following reasons:

1. In vacuum the total gas pressure is reduced and therefore the surface tension forces can collapse the pores. In addition vacuum itself may add in sintering by vaporizing Al_2O_3 and redepositing it as a reduced form of alumina, (the vapor of Al_2O_3 is the AlO or Al_2O species), creating vacancies.

2. In hydrogen the gaseous hydrogen atom or diatomic molecular can react with alumina to form a suboxide and thereby increase the vacancy concentration. In addition atomic or ionized hydrogen may be formed at the alumina surface at these temperatures and, since these species are dimentionally small and reactive with alumina, diffuse to the grain boundary and sink there. In this way the gases in the pores are removed and the pore can collapse due to surface energy considerations.

3. In helium we are dealing with a nonreactive atmosphere with atoms having a diffusity similar to hydrogen. The only major difference between the two atmospheres is the chemical reactive difference between them. It does seem likely that helium gas can diffuse through the alumina structure. This is substantiated by the results which show that theoretical density cannot be achieved with helium and overfiring is noted with nearly all compositions above 1650°C. It is felt that in this final stage of sintering alumina to theoretical density the helium gas is trapped within the pores and the pores are trapped

within the grians, due to exaggerated grain growth, or are closed off. As the temperature increases the pressure within the pore is increased and the pore expands thereby decreasing the density.

H. Future Work

The compositions discussed above will be analyzed with report to structure, grain size, voids, etc.

TABLE I

DENSITY AND MOISTURE ABSORPTION FOR THE 6 COMPOSITIONS AS A FUNCTION OF TEMPERATURE AND ATMOSPHERE AT A CONSTANT SOAKING TIME OF FIVE HOURS

	Temperature 1550°C		Temperature 1650°C		Temperature 1700°C		Temperature 1750°C	
Composition	B. D.	M.A.%	B. D.	M.A.%	B. D.	M.A.%	B. D.	M.A.%
VACUUM								
0.00% MgO	3.716	1.50	3.857	1.17	3.889	0.00	3.906	0.00
0.25% MgO	3.888	0.00	3.976	0.00	3.979	0.00	3.981	0.00
0.50% MgO	3.942	0.00	3.978	0.00	3.984	0.00	3.981	0.00
1.00% MgO	3.934	0.00	3.975	0.00	3.975	0.00	3.976	0.00
1.50% MgO	3.918	0.00	3.956	0.00	3.965	0.00	3.971	0.00
2.00% MgO	3.916	0.00	3.953	0.00	3.958	0.00	3.958	0.00
DRY HYDROGEN								
0.00% MgO	3.791	0.87	3.862	0.00	3.886	0.00	3.885	0.00
0.25% MgO	3.919	0.00	3.957	0.00	3.971	0.00	3.984	0.00
0.50% MgO	3.924	0.00	3.970	0.00	3.974	0.00	3.980	0.00
1.00% MgO	3.934	0.00	3.964	0.00	3.969	0.00	3.979	0.00
1.50% MgO	3.911	0.00	3.939	0.00	3.960	0.00	3.963	0.00
2.00% MgO	3.872	0.14	3.943	0.00	3.948	0.00	3.933	0.00
DRY HELIUM								
0.00% MgO	3.746	1.10	3.875	0.00	3.888	0.00	3.881	0.00
0.25% MgO	3.911	0.13	3.973	0.00	3.975	0.00	3.965	0.00
0.50% MgO	3.902	0.00	3.977	0.00	3.971	0.00	3.964	0.00
1.00% MgO	3.859	0.30	3.969	0.00	3.955	0.00	3.956	0.00
1.50% MgO	3.700	1.22	3.957	0.00	3.944	0.00	3.943	0.00
2.00% MgO	3.885	0.13	3.950	0.00	3.922	0.00	3.921	0.00

FIGURE 1

THE PLOT OF BULK DENSITY IN VACUUM

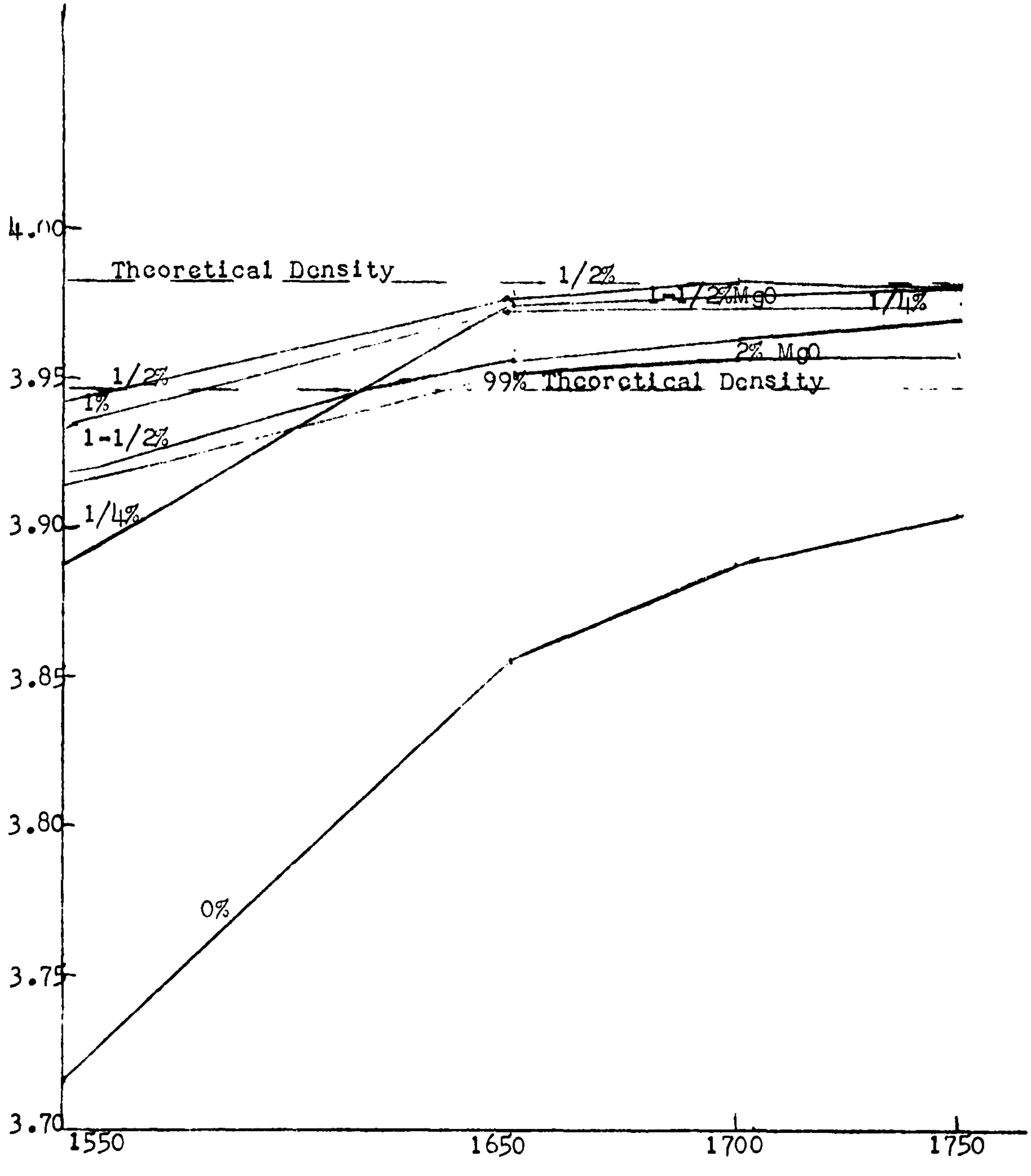


FIGURE 2

BULK DENSITY VERSUS FIRING TEMPERATURE IN DRY HYDROGEN

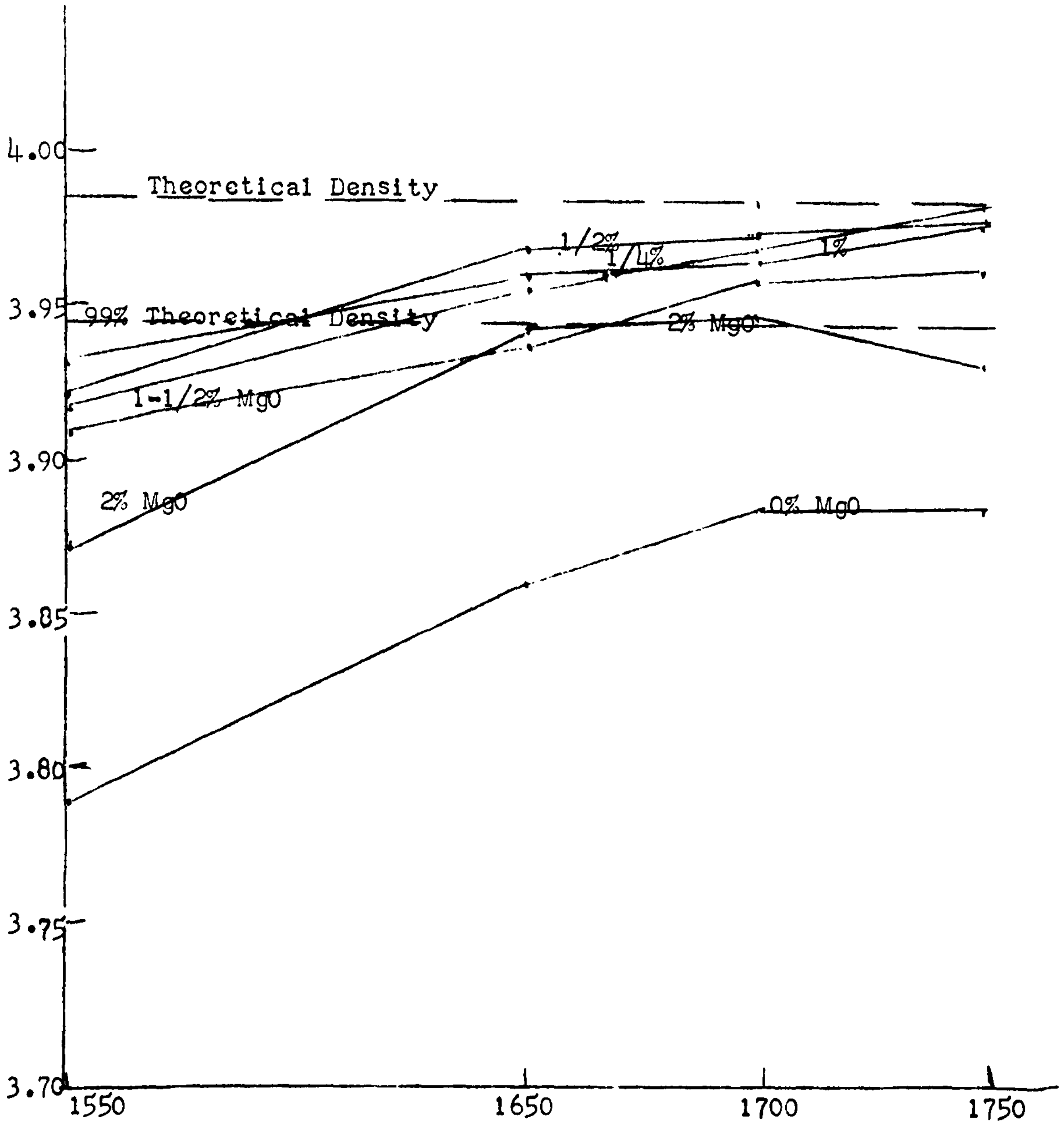
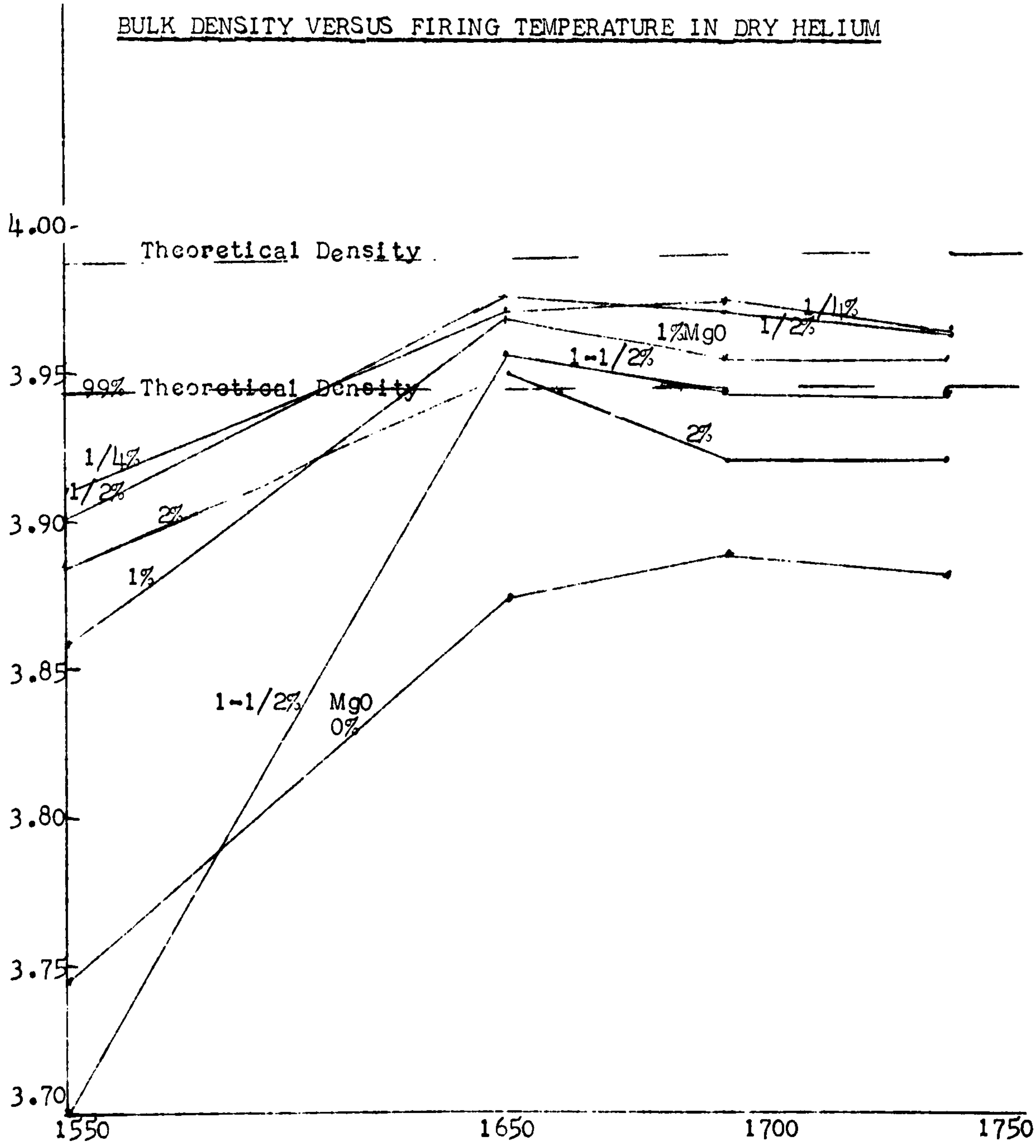


FIGURE 3


BULK DENSITY VERSUS FIRING TEMPERATURE IN DRY HELIUM
4.00
Theoretical Density
3.95
99% Theor~~etical~~ ~~Density~~
3.90
3.85
3.80
3.75
3.70
1550
1650
1700
1750
1/4%
1/2%
2%
1%
1-1/2%
Mg0
0%
1%Mg0
1/2%
1/4%
1-1/2%
2%

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