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INVESTIGATION OF SINTERABLE OXIDE POWDERS AND CERAMICS MADE FROM THEM

C. HYDE
W. H. DUCKWORTH

BATTELLE MEMORIAL INSTITUTE
COLUMBUS, OHIO

JUNE 1961

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AERONAUTICAL SYSTEMS DIVISION

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JUNE 1961

CONTRACT No. AF 33(616)-6238
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AERONAUTICAL SYSTEMS DIVISION
AIR FORCE SYSTEMS COMMAND
UNITED STATES AIR FORCE
WRIGHT-PATTERSON AIR FORCE BASE, OHIO

FOREWORD

This report was prepared by Battelle Memorial Institute under USAF Contract AF 33(616)-6238. This contract was initiated under Project. 7371, "Electrical and Electronic Materials", Task No. 73710, "Electrical and Electronic Inorganic Materials". The work was administered under the direction of the Materials Central, Advanced Systems Technology, Wright Air Development Division, with Lt. J. B. Blandford, Jr. acting as project engineer.

This report covers work conducted from 1 January 1960, through 31 December 1960.

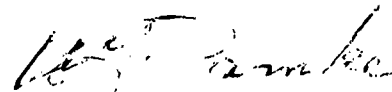
ABSTRACT

This program is concerned with the strength of nonporous monophase ceramics as a function of their microstructure, and with the basic nature of sinterable powders. Sinterable powders provide a convenient way to study a wide variety of microstructural effects without introducing variations in density or purity. MgO powders, prepared by calcining a high-purity basic magnesium carbonate, were studied as representative examples of sinterable powders. The temperature of calcination of the carbonate was found to have a critical effect on the density that could be obtained in sintered compacts of the oxide. The density of compacts of each calcine increased during sintering until a ceiling density was reached after which grain growth occurred without further densification. Ceiling densities of 97 to 98 per cent of theoretical were attained. Sintering in various atmospheres or for extended periods did not affect the attainable density, but sintering in a moisture-free atmosphere reduced the temperature at which the ceiling density was reached. Indications were that sintering atmosphere affected grain growth. The work demonstrated the importance of controlling processing variables to assure uniformly reproducible specimens for strength measurements.

PUBLICATION REVIEW

This report has been reviewed and is approved.

FOR THE COMMANDER:



W. G. RAMKE

Chief, Ceramics and Graphite Branch
Metals and Ceramics Division
Materials Central

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INTRODUCTION

Very broadly, the aim of this continuing research program is to improve capability for producing better refractory ceramics. These materials at their present state of development have strength deficiencies, and it is widely held that their potential strengths have not been realized. Strength properties are dependent on microstructure. Therefore, to realize the **potential strengths of refractory ceramics**, one must know how to control microstructure and how strength is affected by microstructural changes.

With these needs in view, this program is concerned with the strength and microstructure of high-density ceramics made from high-purity sinterable oxide powders, and with the basic nature of enhanced sinterability in these powders. The present report covers progress during the year from January 1 through December 31, 1960. Earlier work in the program was covered in WADD Technical Report 60-108.⁽¹⁾

Past research has shown that higher strength ceramics are possible through the use of powders with increased sinterability.^(1,2) Strength is increased when either porosity or grain size is reduced⁽³⁾, and the combination of low porosity and small grains can be obtained more readily with sinterable powders than with conventional ones. To make the most of this demonstrated approach to stronger ceramics, it is essential to study both sinterable powders and ceramics made from them.

Adjustment of the sinterability of powders used in this program is based on findings at Battelle in 1954⁽⁴⁾ of the marked differences in sinterability of oxides prepared by thermal decomposition. The sinterability depends on the compound calcined (i. e. , whether a carbonate, hydrate, oxalate, etc.), its prior history, and on the calcining temperature. The basic reason(s) for these sinterability differences is not known. No clear correlation has been found with such powder properties as surface area, X-ray parameters, refractive indices, or weight loss on heating. In accord with existing sintering theories, the explanation may lie in a geometry effect, a surface tension effect, or in a self-diffusion effect. As part of the present program, data are being accumulated on the phenomenology of powders prepared at different calcining temperatures to aid in deducing the basic reason(s) for these sinterability differences.

Two processes occur during solid-state sintering - pore removal and grain growth. As indicated above, pore removal strengthens the ceramic and grain growth weakens it. The term "sinterability" as used here refers to the activity of the powder in the pore-removal (shrinkage) process. Compacts of sinterable powders densify at low sintering temperatures to produce high-density ceramics.

According to sintering theory, pores restrain grain growth to a point. By using sinterable powders, porosity is quite low when this point is reached during sintering and, perhaps because it is reached at a low temperature, grain size can be small. If sintering is continued beyond this point, grain growth proceeds with little or no further pore removal. Because this point occurs at a low temperature with sinterable powders, extremely high sintering temperatures are not required to vary grain size over a wide range in ceramics of equivalent high density and high purity. For this reason, sinterable powders are uniquely suited for the experiments on microstructural effects on strength. In addition to sintering time and temperature, other ceramic-processing variables, such as the sintering atmosphere and compacting pressure, are expected to affect ultimate density and microstructure, and are subjects of investigation here.

During the period of this report, principal effort was shifted from BeO to MgO because MgO offers the possibility of improved experimental control. As the work progresses, there are increasing indications that unusually careful control must be exercised in experiments to insure significant data on factors affecting strength and microstructure. The nature of both the process for preparing sinterable oxide powders and of solid-state sintering are such that minor variations in conditions for either could alter significantly the properties of the ceramic. Sintering theory indicates, for example, that the ultimate density obtainable depends on the uniformity of grain size. Incidentally, the low porosity obtained in ceramics made from sinterable powders may be, in part, a consequence of this effect.

It is reasonable to expect that information gained in this program with MgO will be generally, although not necessarily specifically, applicable to BeO and other monophase ceramics.

Because of the finding of higher strength with increased powder sinterability and the fact that wider microstructural variations are possible with a more sinterable powder, effort during the period of this report was directed toward developing as sinterable an MgO powder as possible. Mallinckrodt's AR-Grade basic magnesium carbonate was chosen as the starting material because it was consistent in quality and of high purity, and because preliminary screening experiments indicated that it would yield a highly sinterable MgO powder. In addition to research on producing the sinterable MgO powder, experiments during the year were directed toward establishing sintering and other ceramic-processing conditions to

give dense bodies which had a range of microstructures for subsequent strength studies. Further, as pointed out earlier, in an attempt to understand better the basic reasons for enhanced sinterability, experiments were made to characterize the sintering behavior of powders having different sinterabilities.

SUMMARY

An exceptionally sinterable MgO powder was prepared from a high-purity basic magnesium carbonate. Compacts of the powder sintered to 97 per cent of theoretical density in dried oxygen or nitrogen at a temperature as low as 2200 F.

The temperature at which the basic carbonate was calcined to oxide significantly affected the sinterability of the resultant MgO powder. Under the conditions of calcining used, the most sinterable powder was obtained by calcining at 1100 F.

The density of sintered compacts increased with sintering temperature until some ultimate density was reached. The sintering temperature at which this ultimate density was attained depended on the temperature at which the basic carbonate was calcined. The 1100 F calcine reached its ultimate density of 97 to 98 per cent at the lowest sintering temperature. The ultimate density appeared to be highest when the 1100 F calcine was used; however, powders obtained by calcining at 100 degrees or so below or above 1100 F when sintered at higher temperatures also gave ultimate densities of the order of 97 per cent, so a firm conclusion cannot be drawn from the data on this point.

Compacts of 800, 1100, or 2400 F calcines were sintered in air, vacuum, dried oxygen, hydrogen, moistened or dried nitrogen, nitrogen containing small amounts of chlorine, or the atmosphere of a gas-fired kiln. Moisture in the atmosphere impeded densification of the 800 F and 1100 F compacts. Otherwise, sintering atmosphere had little or no effect on compacts of these calcines. The 1100 F calcine sintered to a density of 97 per cent of theoretical at 2200 F in dried oxygen or nitrogen, while a sintering temperature of 2400 F was required to achieve this density in normal air. The dried atmosphere did not raise the ultimate density of 97 to 98 per cent attainable with this calcine.

When the basic carbonate was calcined at 2400 F (overcalcined), the resultant MgO powder was affected differently by these sintering-atmosphere variations. Chlorine in the atmosphere promoted its densification, as did the use of a vacuum atmosphere at sintering temperatures above 2200 F; further, moisture did not retard densification.

When sintered at 2400 F or 2750 F in air, soaking for 50 hours at temperature did not increase the densities of specimens of the 800 F or 1100 F calcines over those obtained with a 1-hour soak. When sintered at 2000 F or 2200 F, soaking for 50 hours gave densities a per cent or so higher. At these lower sintering temperatures, the ceiling density of the compacts was not reached with the 1-hour soak, but was in the case of the higher sintering temperatures.

Soaking compacts of the 2400 F calcine for 50 hours caused much greater densification than did a 1-hour soak, especially at the higher sintering temperatures used, 2400 and 2750 F. However, when soaked for 1 hour, these sintered compacts were much less dense than were corresponding sintered compacts of the 800 F and 1100 F calcines.

After compacts of the 800 F or 1100 F calcines reached their ultimate densities during sintering, crystal growth proceeded with further time or temperature of heating. Indications were that this crystal growth proceeded less rapidly in nitrogen and more rapidly in vacuum than in an air or oxygen. Indications also were obtained that crystals remain small, of the order of a micron, in these compacts during the sintering process as long as densification is taking place.

Dilatometer measurements showed that shrinkage of compacts of the 1100 F calcine starts during sintering at about 700 F and is most rapid in the range of 1100 to 2000 F. Shrinkage of the 800 F calcine started at a lower temperature, 600 F, and followed the same pattern as the 1100 F calcine to about 1900 F where it almost ceased. In contrast, shrinkage of the 2400 F calcine did not start until 2000 F to 2100 F.

To obtain desired strength data, because of the increased numbers and sizes of MgO specimens required, it is first necessary to develop well-controlled laboratory procedures in scaled-up operations. Effort was started toward this goal. Cracking of specimens, both in green state and during sintering, was encountered in initial efforts to obtain strength data; progress has been made toward eliminating this trouble.

EXPERIMENTAL WORK*

As discussed in the Introduction, Mallinckrodt's AR-Grade basic magnesium carbonate was selected for use in the experiments. According to the supplier, the maximum impurities in this material are as follows:

*The original data are in Battelle Laboratory Records Books Nos. 16835, pp 1 through 100, 17539 pp 1 through 60.

	<u>Per Cent</u>
Calcium (Ca)	0.02
Chloride (Cl)	0.002
Heavy metals (as Pb)	0.001
Iron (Fe)	0.002
Nitrate (NO ₃)	0.003
Soluble salts	0.40
Sulfate and sulfite (as SO ₄)	0.005

In the experiments, the effects of calcining and sintering temperatures on ceramic density were investigated in the effort to produce as sinterable an MgO powder as possible. To characterize powders having different sinterabilities in the effort to learn basic reasons for the sinterability differences and to establish conditions for preparing dense specimens covering a range of microstructures for strength determinations, investigations were made of sintering atmosphere and time on density and grain size.

In calcining, 500-gram lots of the basic carbonate were placed in covered fire-clay crucibles, and heated for 1 hour at the selected calcining temperature in a gas-fired furnace. Calcined powders were stored in sealed glass jars until needed.

To prepare ceramic specimens, the MgO calcine was screened through a 40-mesh sieve, placed in a surgical rubber tube, deaired, and the tubes sealed. Compacts about 1/4 inch in diameter and 1/4 inch long were obtained by isostatic pressing at 50,000 or 100,000 psi.

Sintering was done in Globar-heated furnaces equipped with temperature controllers. The temperature was raised at a rate of 400 F per hour, and specimens were held at the desired sintering temperature for periods specified. After being allowed to cool with the furnace, the specimens were stored in a desiccator.

Density was measured by xylene displacement. Grain size was measured by direct count at 250X magnification on photographs of sintered compacts after polishing and etching with 20 per cent HCl in ethanol, or by petrographic methods.

Effect of Calcining and Sintering Temperature on Density

The basic magnesium carbonate was calcined for 1 hour at temperatures ranging from 800 to 1600 F in 100 F increments, and compacts of each calcine pressed at 50,000 psi were sintered in air by heating for 1 hour at temperatures ranging from 1900 F to 2900 F.

Table 1 summarizes the data, and Figures 1 and 2 illustrate the effect of calcining and sintering temperatures on density.

TABLE 1. EFFECT OF CALCINING AND SINTERING TEMPERATURE ON DENSITY

Calcining Temperature, F	Density, per cent of theoretical, When Sintered at Indicated Temperature									
	1900 F	2000 F	2100 F	2200 F	2300 F	2400 F	2500 F	2600 F	2750 F	2900 F
800	79.6	81.0	82.6	85.2	85.8	88.0	88.5	89.2	90.4	89.2
900	85.8	86.9	87.7	89.6	90.5	92.5	91.9	93.6	93.4	92.5
1000	89.9	93.3	93.4	95.0	95.3	97.2	96.6	97.3	97.8	97.4
1100	89.4	94.3	94.9	95.5	96.4	97.5	97.2	97.5	97.8	97.5
1200	--	88.3	92.0	93.6	95.5	96.4	97.2	97.1	98.1	96.6
1300	87.4	89.7	92.0	94.8	94.7	95.8	97.2	97.5	97.3	96.8
1400	80.4	84.1	87.3	92.3	91.1	94.1	95.8	96.9	96.7	95.8
1500	75.4	79.1	81.8	87.3	89.1	93.6	96.6	96.9	96.6	95.8
1600	--	--	76.3	81.6	84.4	88.0	95.8	96.5	96.4	94.6

Notes: Average of two specimens.

Bulk densities were determined by the xylene displacement and absorption method. Theoretical density of MgO was taken as 3.58 g/cm³.

The 1100 F calcine had exceptional sinterability. Extremely severe sintering conditions (above 3000 F) are usually necessary to achieve densities of 95 per cent or above in MgO ceramics. Using the 1100 F calcine, this density was obtained with sintering temperatures as low as 2100 F. Use of the powder provides the desired capability to investigate strength of high-purity dense bodies as a function of a range of microstructural features. For example, the grain size in the sintered specimens was about 5 microns for those sintered at 2400 F and about 30 microns for those sintered at 2900 F.

As shown in Table 1, within the range of forming and sintering conditions used, the maximum density obtained was 97 to 98 per cent of theoretical. This density resulted from several combinations of calcining and sintering temperatures. At the higher sintering temperatures used, i. e., above 2600 F, it was obtained by calcining at 1000 F, 1100 F, 1200 F, or 1300 F. Calcining temperature was more important at lower sintering temperatures; only the 1000 F and 1100 F calcines gave densities over 97 per cent when the sintering temperature was 2400 F.

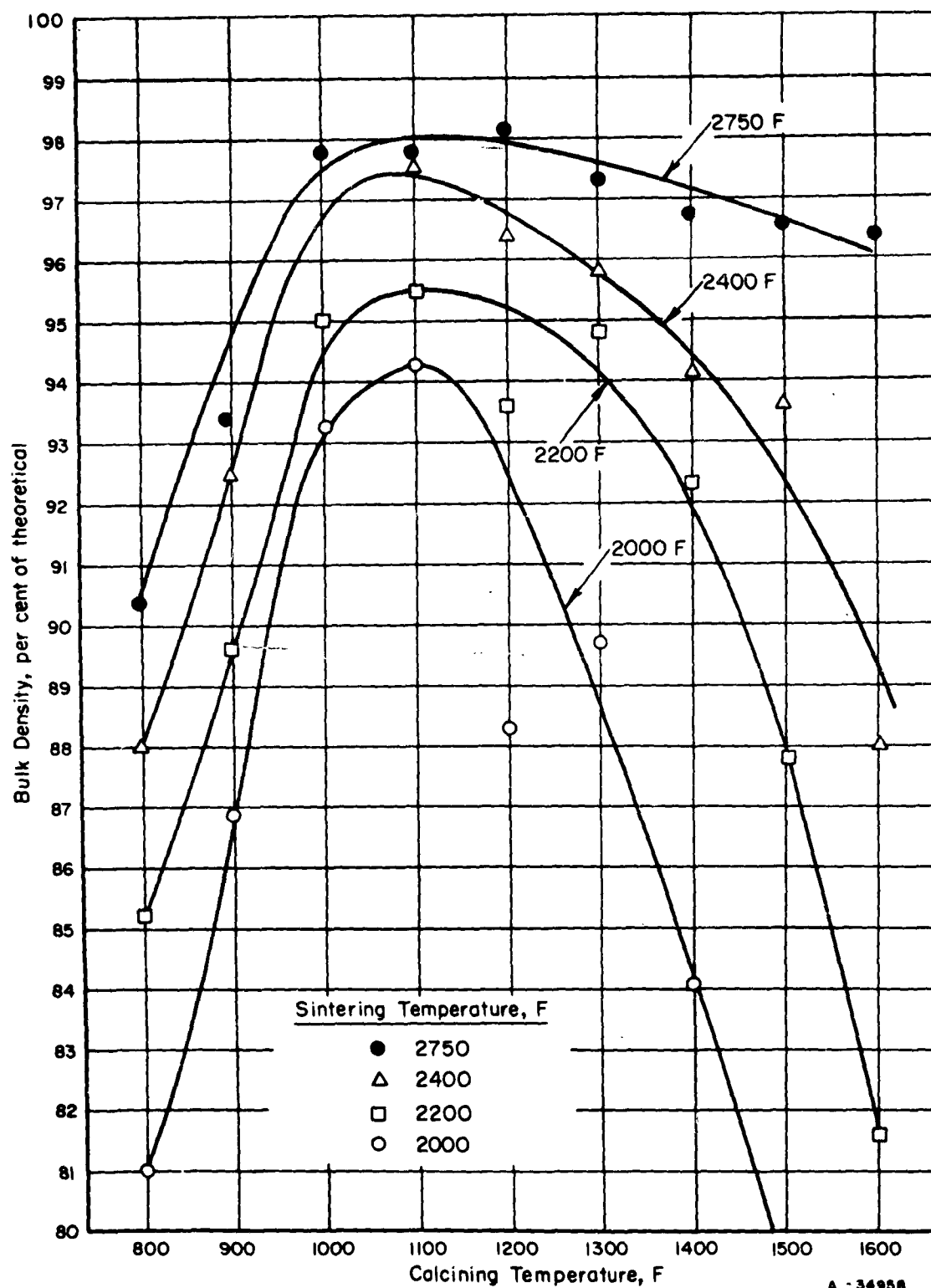


FIGURE 1. SINTERED DENSITY OF MAGNESIA COMPACTS PREPARED FROM DIFFERENT CALCINES

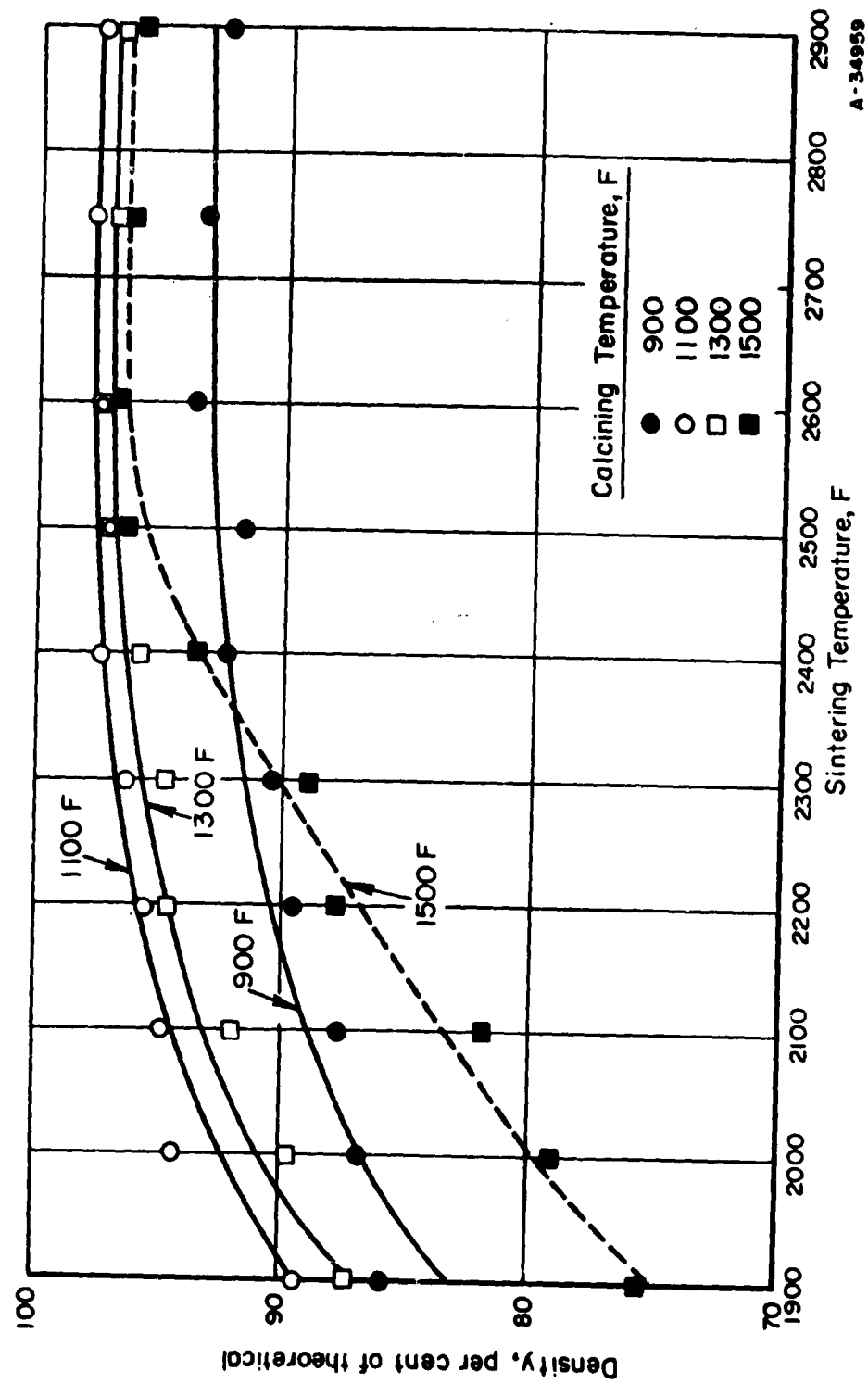


FIGURE 2. DENSITY OF MAGNESIA COMPACTS SINTERED AT DIFFERENT TEMPERATURES

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These data showed that the pore-removal process ceases as sintering proceeds, in accord with sintering theory. A point is reached during sintering where pores no longer restrain grain growth and, when this occurs pores rapidly separate from grain boundaries thus essentially stopping pore removal. The data showed that the density at which pore removal ceases (or ultimate density is obtained) is sensitive to calcining temperature. Calcining much above or below 1100 F decreased the ultimate density, as well as requiring a higher sintering temperature to achieve ultimate density.

Effect of Sintering Atmosphere on Density

In the experiment described in the foregoing section, the highest density obtained was 97 to 98 per cent of theoretical. This density apparently represents the stage in sintering where the pores are isolated from grain boundaries and are of large enough diameter that gas pressure in the pore is equal to the solid surface tension. On the basis of this reasoning if the gas in the pore were more soluble and diffusible or had a lower pressure, the ceramic should be denser. However, a change in the sintering atmosphere could, in several possible ways, affect the shrinkage process occurring prior to this late sintering stage where pores become relatively stable. Present knowledge of sintering mechanism and kinetics, particularly with powders of the type being investigated here, is inadequate to postulate reasonably well the effect of atmosphere on the earlier stages of sintering.

To investigate the possibility of adjusting sintering atmosphere to obtain higher density and to characterize sinterable powders further for a basic understanding of their behavior, an experiment was undertaken to determine the effects of sintering atmosphere.

For this experiment, the basic carbonate was calcined as previously described at 800 F ("undercalcined"), 1100 F ("optimum calcined"), or 2400 F ("overcalcined"). Compacts were made by isostatic pressing at 100,000 psi and were sintered for 1 hour at temperatures ranging from 2000 F to 3100 F in still air, vacuum, the atmosphere of a gas-fired furnace, or in flowing atmospheres of dried oxygen, dried nitrogen, moistened nitrogen, or dried nitrogen containing a small amount of chlorine. For the flowing atmospheres, 1 liter per minute of the gas was passed through the 1-1/2-inch-diameter-tubular furnace chamber. Gases were dried by passing them through a tower containing anhydrous CaSO_4 or were moistened by bubbling them through water maintained at 180 F.

The results are given in Table 2. These data show that moisture in the sintering atmosphere was detrimental to sintering in the case of the under- and optimum-calcined powders, the 800 F and 1100 F calcines, respectively. In the case of these calcines, densities of compacts sintered in the moistened gas (nitrogen) were always lower than were those of

TABLE 2. EFFECT OF SINTERING ATMOSPHERE ON DENSITY

Atmosphere	Density, per cent of theoretical		
	800 F Calcine	1100 F Calcine	2400 F Calcine
Unsintered	57.3	59.4	68.7
<u>Sintered at 2000 F for 1 Hour</u>			
Air	88.0	94.7	68.7
Vacuum	82.7	93.9	67.9
Dried oxygen(a)	89.7	95.0	68.4
Dried nitrogen(a)	90.5	95.8	68.2
Moistened nitrogen(b)	80.4	86.3	68.7
Dried nitrogen with chlorine(c)	89.4	95.0	70.9
<u>Sintered at 2200 F for 1 Hour</u>			
Air	89.9	95.5	70.1
Vacuum	91.1	96.9	70.1
Dried oxygen(a)	91.3	97.2	69.6
Dried nitrogen(a)	91.9	97.2	69.3
Moistened nitrogen(b)	86.9	89.9	70.1
Dried nitrogen with chlorine(c)	90.5	94.4	82.1
<u>Sintered at 2400 F for 1 Hour</u>			
Air	91.9	97.5	71.3
Vacuum	91.9	97.2	89.1
Dried oxygen(a)	91.9	97.2	72.6
Dried nitrogen(a)	91.6	97.2	72.3
Moistened nitrogen(b)	88.5	94.4	72.6
Hydrogen	92.2	98.0	72.3
Gas-fired kiln	89.2	94.1	72.3
<u>Sintered at 2750 F for 1 Hour</u>			
Air	92.7	98.0	82.5
Vacuum	91.9	96.9	92.7
Hydrogen	93.0	98.3	83.8
Gas-fired kiln	92.5	97.5	81.3
<u>Sintered at 3100 F for 1 Hour</u>			
Hydrogen	93.6	97.5	91.9
Gas-fired kiln	93.3	96.9	93.6

Notes: (1) Specimens were sintered in an electrically heated porcelain-tube furnace with a flowing-gas atmosphere; forced gas flow was not used in air heating. Heating in vacuum was done in an induction furnace with a tantalum susceptor under a 1-micron vacuum.

(2) During sintering, specimens were heated at about 400 F per hour to temperature.

(3) Density was measured by xylene displacement. Theoretical density of MgO was taken as 3.58 g/cm³. Average of two specimens.

(a) Gases were dried by passing them through a tower containing anhydrous CaSO₄.

(b) Gases were bubbled through water at 180 F.

(c) Dried nitrogen containing about 1 ppm of chlorine was passed through the 1-1/2-inch-diameter furnace tube at 3 liters per minute.

compacts sintered in other atmospheres under the same temperature conditions. The ceiling densities of 97 to 98 per cent of theoretical for the 1100 F calcine and 92 to 93 per cent for the 800 F calcine were not raised by drying the atmosphere, but the ceiling densities were attained at sintering temperatures in the dried atmospheres. The 1100 F calcine sintered to 97.2 per cent of theoretical at 2200 F in dried oxygen or nitrogen, while a sintering temperature of 2400 F was required to achieve this density with a normal air atmosphere. Except for moisture retarding densification and, perhaps, a retarding effect of vacuum sintering the 800 F calcine at 2000 F, the atmosphere variations investigated had little or no other effect on sintered density.

The behavior of the 2400 F calcine, the overcalcined powder, was quite different. However, it should be pointed out that the shrinkage processes were considerably activated in the other powders even at the lowest sintering temperature used, 2000 F, and were, at most, moderately activated in the 2400 F calcine. Unlike the case of the 800 F and 1100 F calcines, chlorine in the atmosphere promoted sintering of the 2400 F calcine. When sintered at 2200 F in the chlorine-containing atmosphere (1 ppm of chlorine in dried nitrogen), compacts of the 2400 F calcine had a density of 82.1 per cent, while in the other atmospheres the densities were all about 70 per cent. The retarding effect of moisture noted on densification of the other calcines was absent in the case of the 2400 F calcine. Also, the use of a vacuum at the higher sintering temperatures caused greater densification of the 2400 F calcine, while it had either no effect or an adverse effect on densification of the other calcines.

Because chlorine promoted sintering of the overcalcined powder, future work should include further investigation of the effect of chlorine in the sintering atmosphere, particularly on powders calcined between 1100 F and 2400 F with sintering temperatures that give densities above 95 per cent.

The effect of "double sintering" on densification was investigated. In the experiment, specimens were sintered in oxygen at atmospheric pressure and then heated in vacuum, or were sintered in vacuum and then heated in oxygen under normal atmospheric pressure. The purpose was to determine if either of these treatments would increase density. The basis of reasoning is this: in one case, the subsequent vacuum treatment would provide a pressure gradient for oxygen diffusion from the pores and surface tension would tend to close them; in the other case, external pressure acting on pores formed in a vacuum would decrease their size. All heat treatments were at 2400 F for 1 hour, and the processing of the powders, preparation of compacts, and density measurements were as given in the notes in Table 1. Results were as follows:

	Density, per cent of theoretical		
	800 F Calcine	1100 F Calcine	2400 F Calcine
Initial sinter in vacuum	91.1	96.4	89.7
Subsequent heat treatment at normal pressure in oxygen	91.3	96.6	90.8
Initial sinter in oxygen	90.8	95.0	74.6
Subsequent heat treatment in vacuum	90.5	94.9	80.0

An increase in density was not observed in this experiment, perhaps because 2400 F and 1 hour were insufficient temperature or time for the densification mechanisms to act during the subsequent heat treatments, or because an insufficient number of pores were closed in the initially sintered compacts. The experiment should be continued with higher temperatures and longer soaking periods.

Effect of Sintering Time on Density

An experiment was made to measure the effect of sintering time on densification. Previously, all specimens were held for 1 hour at the sintering temperature. In this experiment, the specimens were soaked for 50 hours. A normal air atmosphere was used. The results were as follows:

Sintering Conditions		Density, per cent of theoretical		
Maximum Temperature, F	Soaking Period, hours	800 F Calcine	1100 F Calcine	2400 F Calcine
Unsintered		57.3	59.4	68.7
1200	50	64.8	69.0	68.7
1600	50	79.9	86.0	68.7
2000	1	88.0	94.7	68.7
2000	50	88.5	95.5	69.8
2200	1	89.9	95.5	70.1
2200	50	91.3	96.6	73.5
2400	1	91.9	97.5	71.3
2400	50	91.6	97.8	79.9
2750	1	92.7	98.0	82.5
2750	50	93.3	98.0	95.5

These data indicate that sintering time did not have a pronounced effect on densification of compacts made from under- and optimum-calcined powders. Longer soaking did not raise the ultimate density; the 1100 F calcine, for example, densified to 97.5 per cent in 1 hour at 2400 F and, with a 50-hour soak, the density (97.8 per cent) was essentially unchanged. At lower sintering temperatures (temperatures below those at which the ceiling density was reached in 1 hour), the longer sintering time increased the density a per cent or so. At still lower sintering temperatures below those investigated, the effect of sintering time on density might be more pronounced. However, no investigation of this was made since major interest was on high-density compacts.

As was the case with the effects of sintering atmosphere, sintering time had a much different effect on densification of the 2400 F (overcalcined) powder than it did on the 800 F and 1100 F calcines. Again, however, it must be remembered that the shrinkage processes had progressed much further in the 800 F and 1100 F calcines than in the 2400 F calcine in the sintering-temperature range investigated. Unlike the 800 F and 1200 F calcines, compacts of 2400 F calcine at the higher sintering temperatures had appreciably higher densities when sintered for 50 hours than when sintered for 1 hour. In the case of the 2400 F calcine, the data show that shrinkage was time dependent, as expected.

It is interesting to note from the data that, whereas sintering at 2400 F for 50 hours removed about one-third of the porosity present in compacts of the 2400 F calcine, sintering at only 1600 F for 50 hours removed about two-thirds of the porosity present in compacts of the 1100 F calcine.

Microscopic Examination of Sintered Compacts

Microscopic examination of polished and etched surfaces of sintered compacts were made primarily for the purpose of selecting processing conditions to prepare strength specimens having different microstructures. Polished surfaces were etched with 20 per cent HCl in C_2H_5OH and examined at 250X. This technique defines crystal boundaries as long as the crystal size is above about 5 microns; it does not give sufficient definition to permit interpretation of differences in pore or grain-boundary character. Petrographic techniques also were used to measure crystal size in compacts in which crystals were smaller than about 5 microns.

Knowledge of crystal growth has been authoritatively presented by Burke. Of particular interest is the phenomenon of crystal growth after the impeding effect of pores has been overcome, i. e., the ultimate density is reached. In sintering, a point is reached at which crystal boundaries move over pores, further densification proceeds with difficulty, and larger crystals

start to grow at the expense of small ones. According to Burke, the crystal size, D_1 , at which this occurs is approximated by

$$D_1 = \frac{S}{P},$$

where S is the pore size and P is the volume fraction of pores. Prior to reaching this point in isothermal sintering, the crystal size, D , is theoretically given by

$$D = Kt^{1/2} + D_0,$$

where

K is a temperature-dependent constant

t is time

D_0 is the crystal size at $t = 0$.

After reaching the size D_1 in sintering, it would appear that crystal growth would cease if all crystals were the same size. If they were not the same size, crystal growth would occur, as pointed out previously, by large crystals growing at the expense of small ones. Thus, in interpreting effects on crystal size, it appears important to correlate size data with effects on density.

For the data at hand, let us consider first crystal growth after densification stops. In the case of compacts of the 800 F calcine, this occurred at a density of about 92 per cent and, for the 1100 F calcine, at about 97.5 per cent. Data at hand are insufficient to indicate where it occurs for the 2400 F calcine.

At these densities, the following effects of sintering time and temperature were recorded.

Sintering Conditions ^(a)		Average Crystal Size, microns	
Temperature, F	Soaking Period, hours	800 F Calcine	1100 F Calcine
2400	1	6.7	6.0
2400	50	13.0	7.9
2750	1	14.5	33.0
2750	50	44.0	60.0

(a) Sintered in normal air atmosphere.

These data show that crystal growth does occur in these compacts after densification ceases and that the growth is a function of both time and temperature. Photomicrographs of these specimens are in Figure 3.

The following effects of sintering atmospheres were recorded:

<u>Atmosphere</u>	<u>Average Crystal Size, microns</u>	
	<u>800 F Calcine</u>	<u>1100 F Calcine</u>
<u>Sintered at 2400 F for 1 Hour</u>		
Air	6.7	6.0
Vacuum	9.8	18.3
Dried oxygen	6.1	6.2
Dried nitrogen	--	2.2
<u>Sintered at 2750 F for 1 Hour</u>		
Air	14.5	33.0
Vacuum	23.5	26.2

Indications are that dried nitrogen inhibits crystal growth and a vacuum accelerates it after the ceiling density is reached, but further data are required before a firm conclusion can be drawn.

A few data were obtained on crystal size in less dense compacts in which sintering had not reached the stage at which densification essentially stops. The data were all from compacts of the 1100 F calcine sintered for 1 hour, as follows:

<u>Sintering Conditions</u>			
<u>Maximum Temperature, F</u>	<u>Atmosphere</u>	<u>Density, per cent of theoretical</u>	<u>Average Crystal Size, microns</u>
2200	Air	95.5	1.6
2000	Dried nitrogen	95.8	0.8
2000	Moistened nitrogen	86.3	0.6
2200	Moistened nitrogen	89.9	0.8
2400	Moistened nitrogen	94.4	1.1

These data indicate that with this powder crystal size remains small during the latter stages of densification. While the density increased from 86.3 to 94.4 per cent, the crystal size increased from 0.6 micron to 1.1 microns. This is encouraging from the standpoint of fracture strength, as both small crystal size and high density are known to favor high strength. The inhibiting effect of dried nitrogen on crystal growth again is indicated.

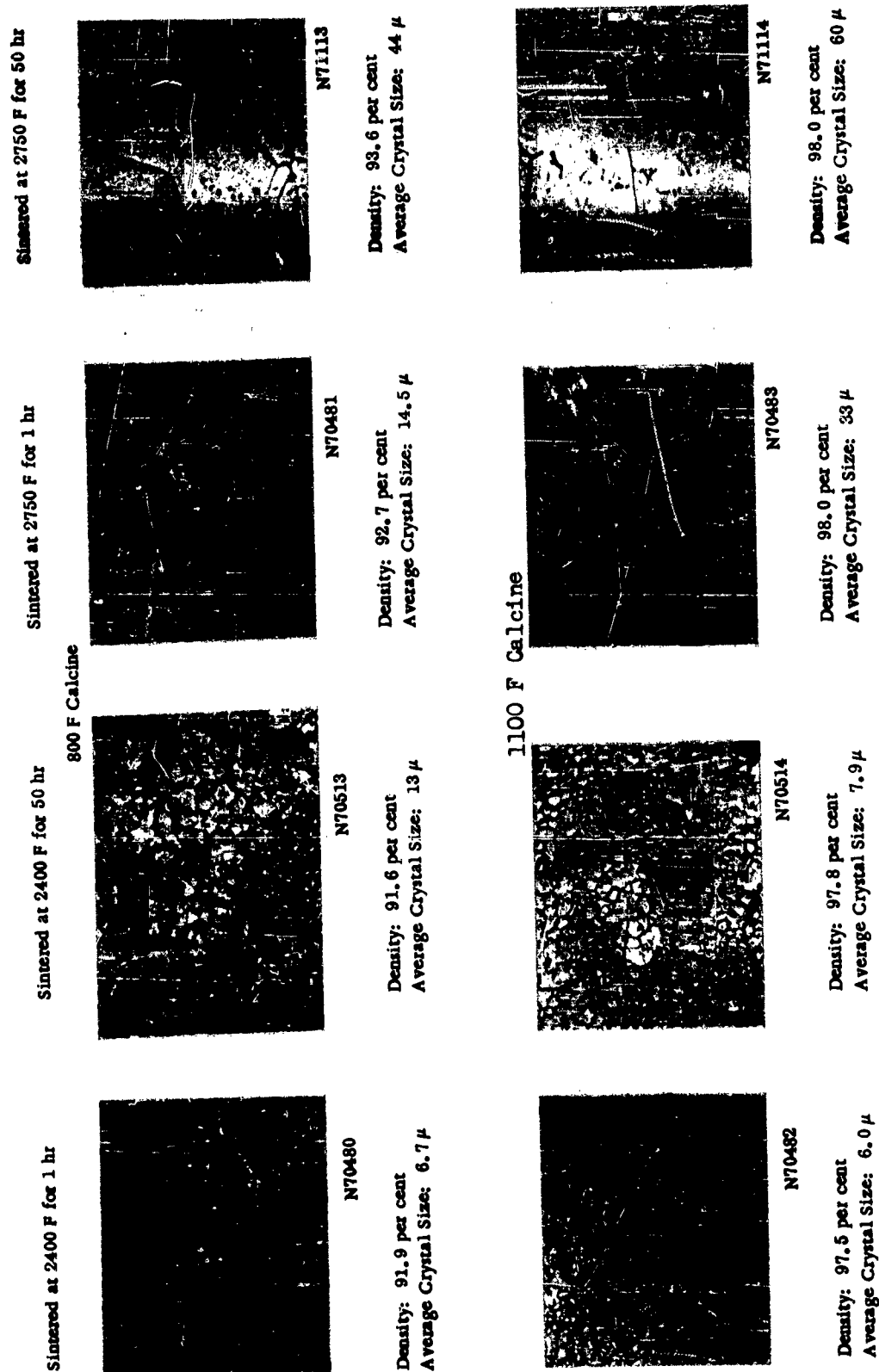


FIGURE 3. VARIATIONS IN MICROSTRUCTURE WITH SINTERING TIME AND TEMPERATURE IN AIR ATMOSPHERE

Magnification: 250X.

Progress of Shrinkage During Sintering

Using the same heating schedule, 400 F per hour, followed in sintering the compacts discussed in previous sections, dilatometer measurements were made to obtain information on shrinkage behavior. This information will be useful in selecting a safe sintering schedule for the preparation of specimens for the determination of strength, since the possibility of shrinkage cracks exists if specimens are heated rapidly in temperature ranges where high shrinkage occurs. Also, the data were designed to gain further insight into differences among different calcines.

Briefly, in making the measurements, a compact about 1-1/2 inches high and 3/8 inch in diameter was placed with a thermocouple in the bottom of a closed-end, sintered alumina tube. An alumina rod was held on top of the specimen and extended above the open end of the tube. An Ames dial was mounted to measure length changes in the specimen as transmitted by the rod. The assembly was inserted vertically in a Globar-heated furnace. The apparatus was not calibrated, so the shrinkage data given in Figure 4 are comparative rather than absolute.

As shown in Figure 4, the compact of the 2400 F (overcalcined) powder did not begin to shrink until the temperature reached 2000 F to 2100 F. In contrast, the compact of the 1100 F calcine started shrinking at about 700 F, and that of the 800 F calcine, somewhat surprisingly, at about 600 F. It would appear that the temperature at which shrinkage starts is an inverse function of the calcining temperature and, on this basis, the more sinterable powder might be expected to be the one calcined at the lowest temperature. The reason that this is not the case, that the more sinterable powder is obtained at an intermediate calcining temperature, lies in the fact that the density at which shrinkage ceases also depends on the calcining temperature. Shrinkage ceases at a lower density with the 800 F calcine, apparently because of an increased rate of grain-boundary migration at lower temperatures.

Shrinkage of 1100 F calcine was most rapid between 1100 F and 2000 F, and that of the 800 F calcine was most rapid between 1000 F and 1900 F. Shrinkage of the 800 F calcine slowed drastically at 1900 F, but the 1100 F calcine did not exhibit this behavior in the temperature range used. This slowdown of shrinkage in the 800 F calcine accounts partially for it producing a less dense ceramic than does the 1100 F calcine, even though it shrinks more prior to this slowdown. Also, green compacts of the 800 F calcine are less dense than those of the 1100 F calcine, so more shrinkage is required to reach a given density.

The finding that extensive shrinkage occurs in compacts of the 800 F and 1100 F calcines below 1800 F indicates that special care will be needed to prepare crack-free ceramics. Little or no plastic flow would be expected

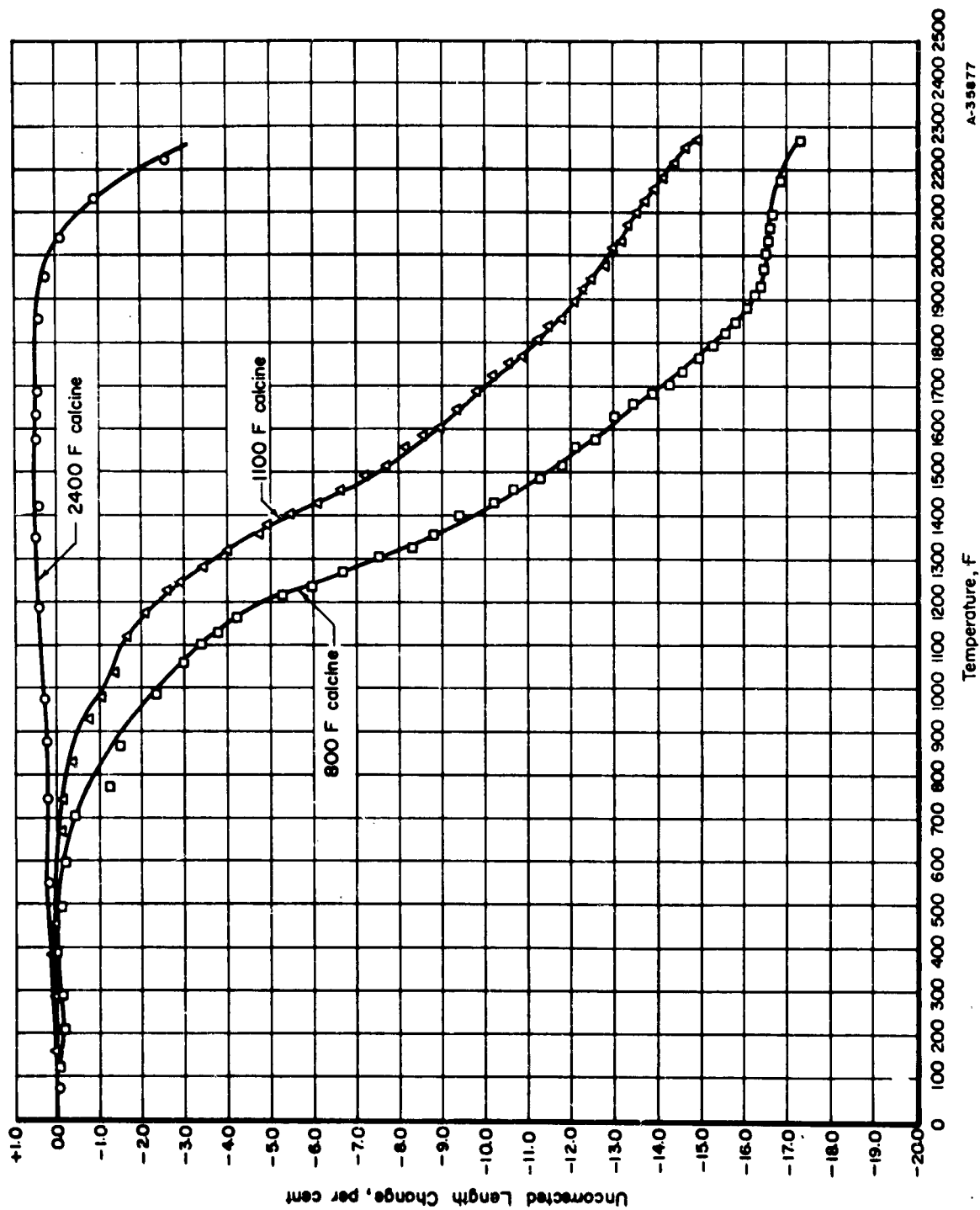


FIGURE 4. SINTERING SHRINKAGE-TEMPERATURE RELATIONSHIPS

in magnesia at these low temperatures, so stresses from differential shrinkage from temperature differences in a piece during sintering would be relieved by cracking rather than plastic deformation.

Preparation of Strength Specimens

The specimens used in the experiments described in the previous sections of this report were small, 1/4 inch in diameter and 1/4 inch long, and the nature of the experiments was such that few specimens were required for each determination. This situation changes in strength experiments. In the interest of experimental control, transverse strength (modulus of rupture) tests are preferred, and these tests require rod or bar specimens of the order of 10 diameters long. Also, statistically significant strength data are expected to require that several nominally identical specimens be broken.

Accompanying the requirements for the larger size and greater number of specimens for each strength determination is the requirement for reproducibility of powder batches and ceramic specimens. This requirement necessitates studies of the control of the powder and ceramic processing in scaled-up laboratory operations.

As indicated in the Introduction, another reason to be concerned over processing control is that the attainable density of the ceramic might be raised by using a more uniform powder or by having more uniform conditions within the compact during forming and sintering.

Effort on developing the well-controlled scaled-up processing conditions preparatory to experiments on strength behavior were started during the period of this report, but have not yet been extensively pursued.

Strength specimens prepared early in the research often cracked shortly after pressing but prior to sintering. This cracking did not occur in the smaller specimens used in other experiments. The cracks were circumferential indicating stresses due to an expansion of the interior relative to the surface, or a surface contraction. It was theorized that an exothermic reaction, possibly hydration or carbonation, within the isostatically formed compacts resulted in thermal stresses which cracked the specimens. The specimens were noted to become warm to the touch upon removal from the isostatic-pressing chamber, in support of this theory. In attempts to prevent exothermic reactions, the MgO powders were handled in a dry box and the formed specimens were stored under xylene. These measures were beneficial, but the problem is not considered to be solved as yet.

Very recently, Davies, et al. (7), of NASA have reported on a comprehensive study of the spontaneous cracking of unfired MgO powder compacts isostatically pressed without binder. They concluded that it is caused by a combination of hydration and carbonation, and suggested methods to prevent it. Future efforts at Battelle to overcome spontaneous cracking will be guided by their suggestions.

In other instances, cracking of the transverse-strength test specimens was not noticeable until after the specimens were sintered. The cracking during sintering probably was caused by differential shrinkage at temperatures below those at which relief from plastic flow would be expected, as discussed in the previous section of this report. Development of a sintering technique so that heating is sufficiently uniform over the temperature range within which extensive shrinkage occurs is expected to eliminate this source of cracks.

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