

Some Physical Properties of Porcelains in the Systems Magnesia-Beryllia-Zirconia and Magnesia-Beryllia-Thoria and Their Phase Relations¹

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The ternary eutectic for the system MgO-BeO-ZrO₂ was located at mole composition 4MgO:5BeO:3ZrO₂ and 1,669° C, and that for the system MgO-BeO-ThO₂ was located at mole composition 5MgO:10BeO:1ThO₂ and 1,797° C. No binary or ternary compounds were found in either of the systems, and an area of solid solution is indicated for each. A number of bodies were found suitable for various high-strength and high-temperature services where thermal shock resistance is not of prime importance. The compressive strength values at room temperature ranged as high as 266,000 lb/in.² for some porcelains in both systems. The maximum values for the strength in bending at room temperatures and at 1,800° F (982° C) were considerably higher for the MgO-BeO-ZrO₂ bodies (39,000 and 32,000 lb/in.², respectively) than for the MgO-BeO-ThO₂ bodies (14,000 and 16,000 lb/in.², respectively), and the modulus of rupture value at 1,800° F (982° C) of one body in the binary system MgO-ZrO₂ was 29,000 lb/in.². The resistance to thermal shock for the MgO-BeO-ZrO₂ porcelains was fair to good, and for the MgO-BeO-ThO₂ porcelains it was poor. All of the MgO-BeO-ZrO₂ bodies can be matured in commercial kilns used currently in the porcelain industry, whereas part of the MgO-BeO-ThO₂ porcelains and none of the MgO-ZrO₂ porcelains can be so matured.

I. Introduction

Under the impetus of the recent war and because of the outstanding advances made in the design and efficiency of such power plants as the gas-turbine and jet-propulsion engines, varied programs are being sponsored by industrial, research, and government laboratories in the realm of high-temperature materials.

In design and, especially, in efficiency the present models of the various heat power-plants are severely restricted by the ability of some of their components to withstand the required high operational temperatures and stresses. The important physical properties of such power-plant elements are dependent to a large extent upon whether the part in question is to be stationary or moving, whether the engine is to be continuously or intermittently operated, or any combination of these conditions [1].²

The allowable metal-blade temperature of the present conventional gas turbine, for example, is limited to approximately 1,500° F (815° C), but conventional fuels could provide inlet-gas temperatures exceeding 3,500° F (1,927° C) [1] and, thereby, greatly increase the efficiency of such an engine. In addition to refractoriness, considerable mechanical strength is required in certain instances as illustrated, for example, by the tensile stresses set up in a turbine blade by centrifugal force [2, 3, 4]. A number of other properties also are required, among which are: high thermal shock resistance, high thermal conductivity, resistance to oxidation and erosion, and ability to maintain exacting dimensions at high temperatures.

Many of the ceramic oxides and combinations of these oxides are well suited for such diversified uses as turbine nozzles, interstage stator blades, rotors and buckets, rocket liners and nozzles, and control vanes because of their high melting points, low density, and potentially low cost and availabil-

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² Figures in brackets indicate the literature references at the end of this paper.

ity. The limitations imposed by the glassy bond of the conventional feldspathic bodies and the earlier developments leading to the formulation of the glass-free oxide porcelains are discussed in detail in a previous report [5]. Excepting the detailed studies of the phase relations of the oxides [6], many reports of the pure ceramic oxide and of the ceramic oxide-metal combination studies are still in the restricted classification.

In order to supply some of the fundamental information upon which the practical work of producing ceramics to meet the future needs can be based, there was undertaken a study of the characteristics of variously heated binary and ternary combinations of the high melting-point oxides. A preliminary report was published in 1941 [7], and a report on some of the porcelain compositions of the systems MgO-BeO-Al₂O₃, CaO-BeO-Al₂O₃, BaO-BeO, BeO-Al₂O₃-ThO₂, and BeO-Al₂O₃-ZrO₂, along with the probable phase relations in these systems, was published in 1946 [5]. A preliminary report on the strength in tension and the creep characteristics of a number of bodies described in the 1946 report also was made in 1946 [2], and the final report was published as NACA Technical Note 1561, April 18, 1949. The present paper reports the results obtained since the 1946 report [5].

It may be of interest to the reader to compare some of the reported physical properties of a few refractory ceramic bodies and of some oxide porcelain bodies. These data are given in table 1.

II. Materials

The oxides used in the preparation of specimens for testing the various physical properties were commercially available high-purity materials, whereas the oxides used in the study of the phase relations were of the highest purity available.

Beryllia (BeO): (1) Commercial grade of nominal 99.7-percent purity. The spectrograms show only traces of copper, iron, and magnesium and very weak lines for silicon.

(2) Fluorescent grade of over 99.9-percent purity.

Magnesia (MgO): (1) Fused, recrystallized material of nominal 97-percent purity.

(2) Especially selected, water-clear crystals of artificial periclase of over 99-percent nominal purity (furnished through the courtesy of The Norton Co.).

Thoria (ThO₂) (1) Calcined material of over 99-percent nominal purity. The spectrograms show only very weak lines for calcium, copper, and silicon.

(2) Calcined material of at least 99.99-percent purity.

Zirconia (ZrO₂): Commercial grade of nominal 99-percent purity. After recalcining at 1,440° C,³ the spectrograms showed medium lines for columbium, titanium, and only very weak lines or traces for calcium, copper, iron, magnesium, lead, and silicon. This material was used for both the porcelain test specimens and for the phase relation studies.

As prepared for use, the materials were, in all instances, sufficiently finely divided to pass the No. 325 U. S. Standard Sieve. In order to obtain this desired fineness, it was necessary to reduce the particle sizes of all of the materials except the fluorescent grade beryllia. The commercial grade of beryllia was reduced to about 95 percent of minus 325 mesh material by wet grinding for 2 hr in a porcelain mill with alumina balls (approximately 2% total mill gain of which about 1½% was gained from the alumina balls). Both grades of magnesia could be reduced in size fairly easily in a mullite mortar, but it was found more advisable for quantity work to mill the less pure material in alcohol for 30 min to 1 hr in a porcelain mill with alumina balls (less than ¼% total mill gain). Both the thoria and the zirconia were dry-ground in a steel rod-mill for 30 hr and then cleaned. A magnetic separator was used first, and the material was then washed alternately with 1:3 hydrochloric acid and distilled water until both the decanted acid and wash water gave a negative iron test using potassium thiocyanate or potassium ferrocyanide or both as indicators.

III. Methods

In order to insure the desired reproducibility of results and to prevent segregation of the batch constituents during specimen preparation, a definite compounding method was followed.

The calculations of weight composition from the desired mole composition were based upon the accepted molecular weights of the oxides [8]. Calculations were made to ±0.005 weight percent,

³ All temperature values are given according to the International Temperature Scale of 1927.

TABLE 1. Some physical properties ^a of various refractory ceramic bodies

[Other compilations of a similar nature, but without literature references, may be found in Ceramic Age **50**, 19 (1947); Materials and Methods **26**, 87 (Dec. 1947); Electronics **17**, 136 (May 1944) and **12**, 33 (April 1939); Ceramic Ind. **39**, 70 (1942); and Rev. Sci. Instr. **12**, 527 (1941).]

Body types	Porosity		Specific gravity		Coefficient of linear thermal expansion			Thermal conductivity ^b			Resistance to thermal shock		Compressive strength		Tensile strength			Modulus of rupture			
	Value	Reference ^c	Value	Reference ^c	Range	Value	Reference ^c	Range	Value	Reference ^c	Value (relative)	Reference ^c	Temperature	Value	Reference ^c	Temperature	Value	Reference ^c	Temperature	Value	Reference ^c
	Percent		g/cm ³		°C	n×10 ⁻⁶ / °C		°C	n×10 ⁻³ watts				°C	n×10 ³ lb/in. ²		°C	n×10 ³ lb/in. ²		°C	n×10 ³ lb/in. ²	
Steatite	0.0 0.0 to 0.08	[1] [2]	2.6 to 2.8 2.5 to 2.7	[1] [2]	20 to 100 25 to 100 25 to 700	6.0 to 9.0 6.9 to 7.7 8.6 to 10.2	[1] [2] [2]	20 to 100 ----- -----	About 29 25	[1] [2]	Good	[1]	-----	117 to 138 65 to 85	[1] [2]	-----	8 to 13 7.5 to 10	[1] [2]	-----	16 to 22 18 to 20 25	[1] [2] [3]
Mullite	3.9 nil	[4] [5]	3.2 2.3 to 2.9 3.03	[4] [5] [6]	20 to 1,320 0 to 1,700 390 to 700 20 to 1,430	4.5 5.3 8.2 5.5	[6] [7] [7] [18]	425 to 870 95 to 1,430 ----- -----	17 to 23 12 32	[5] [6] [8]	do	[5]	-----	60 to 200	[5]	-----	10 to 30	[5]	-----	15 to 25	[5]
Zircon	-----	-----	4.7 4.6	[9] [10]	25 to 1,000 20 to 1,550 0 to 1,500 300 to 700 20 to 1,340 20 to 1,500	4.6 4.2 4.2 to 6.4 4.1 5.1	[5] [6] [7] [18] [19]	425 to 870 200 to 1,000 ----- ----- -----	17 to 23 19 45	[5] [6] [8]	Very good.	[5]	-----	60 to 200	[5]	-----	10 to 30	[5]	-----	15 to 25	[5]
Alumina	0 25. plus 21. plus	[1] [4] [7]	3.24 3.3 to 3.9 3.8 to 4.0 3.67 2.9	[4] [5] [6] [7] [10]	20 to 1,000 0 to 1,580 300 to 700 25 to 1,400 20 to 1,000 20 to 1,700	7.0 7.7 8.2 7.4 8.3 9.3	[6] [7] [7] [10] [19] [19]	425 to 870 1,095 300 350 to 850 400 to 1,200	34 to 44 26 to 29 42 24 31 to 42	[5] [6] [8] [10] [11]	Good	[10]	20 1,200 1,500 -----	^d 410 ^d 69 ^d 14 60 to 200	[1] [1] [1] [5]	20 1,050 1,200 -----	^d 36 ^d 33 ^d 18 10 to 30	[1] [1] [1] [5]	20 20 20 -----	27.6 46.8 55.0 15 to 25	[1] [1] [1] [5]
Silicon carbide	18 to 34 13 to 35	[12] [13]	3.13 to 3.22 3.17 2.1 to 2.6 2.3 to 2.45	[6] [9] [13] [14]	20 to 1,100 0 to 1,700 300 to 700 25 to 1,400	4.5 4.3 4.8 5.-	[6] [7] [7] [13]	1,095 800 to 1,200 400 to 1,200 500 to 1,300	95 159 to 121 159 to 167 243 to 100	[6] [11] [12] [13]	Good do	[13] [14]	-----	12.5 to 14.7	[14]	-----	-----	-----	20 1,350	2.1 0.9 to 2.5	[13] [13]
MgO-BeO-Al ₂ O ₃	0.04 to 0.07	[15]	3 to 5	[16]	20 to 700	7 to 10	[15]	250	^e 4 to 12.5	[15]	-----	-----	25	105 to 286	[15]	-----	-----	-----	-----	-----	-----
BeO-Al ₂ O ₃ -ThO ₂	0.00 to 0.02	[15]	3 to 5	[16]	20 to 700	7 to 10	[15]	250	^e 4 to 25	[15]	do	[17]	25	240 to 279	[15]	870 980	^f 13 ^f 6	[16] [16]	20 980	^f 20 ^f 17	[17] [17]
BeO-Al ₂ O ₃ -ZrO ₂ ^g	0.00 to 0.05	[15]	3	[16]	20 to 700	7 to 8	[15]	250	^e 8 to 10	[15]	do	[17]	25	250	[15]	870 980	20 18	[16] [16]	20 980	25 14	[17] [17]

^a Little evaluation of the test methods and reported results has been made. All literature references are given in footnote c.

^b These data are given as Watts/cm/cm²/°C. In order to convert to gram-cal/cm/sec/cm²/°C, multiply by 0.2388, and to convert to Btu/in./hr/ft²/°F, multiply by 693.5.

^c References:

[1] R. Russell, Jr., FIAT Final Report No. 617 (Dec. 1945), OTS, Dept. of Commerce, Washington, D. C.

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^d E. Ryschkewitsch data obtained by very rapid loading.

^e These values are reciprocal, multiplied by 100, of the temperature drop through a 0.50-in. height of a specimen approximately ½ in. in diameter.

^f Values given are for a composition in this system containing an addition of 2 weight percent of titanium oxide.

^g Values given are for compositions in this system containing additions of 2 weight percent of calcium oxide.

and there was no correction factor applied for the percentage purity of the material at any time nor for any ignition loss at 1,200° C when that loss was less than 0.01 g of the various 30- to 400-g batches. Each oxide was weighed on a semi-analytical balance to ± 0.005 g. Approximately 1.2 ml of distilled water per gram of batch was added to make a slip similar in consistency to a thick cream and one drop of a 10-percent water solution of Aerosol wetting agent was added for approximately each 10 g of dry batch. The slip was machine-blunged with a chemical laboratory-type variable-speed mixer with plastic stirrers for not less than 15 min, damp-dried under infrared lamps, and completely dried in an oven at 110° C for at least 15 hr. Between 8 and 10 weight percent of a 5-percent soluble starch solution was added as a binder, and the dampened batch was mixed and sieved a number of times through a No. 30 U. S. Standard Sieve. It was then pressed at about three-fourths of the final forming pressure to promote agglomeration, and the resultant briquets were crushed to pass either the No. 20 or the No. 30 sieve. The dampened mixture was tamped and vibrated in the molds before the final pressing. This pressing was done in case-hardened steel, and in oil-hardened, nondeforming, tool-steel molds lightly lubricated with a water-emulsifying oil.

Specimens for the softening-range and fusion tests [5] were ground from $\frac{5}{16}$ by $\frac{1}{2}$ in. diameter plaques pressed at 13,000 lb/in². Those for the preliminary investigation of the maturing range ($\frac{5}{8}$ in. high by $\frac{3}{8}$ in. in diameter) and for the compression tests ($1\frac{1}{4}$ in. high by $\frac{5}{8}$ in. in diameter) were pressed at 9,400 lb/in². The thermal shock and strength test bars were pressed about $\frac{5}{16}$ in. thick in a $\frac{3}{4}$ -in. by $6\frac{1}{8}$ -in. mold at 6,500 lb/in².

The special high-temperature thoria-resistor furnaces used in these studies for determining vitrification ranges, for maturing the compression specimens, and for the phase studies, have been described [7, 9], as have many of the test methods [5]. These are repeated both for the convenience of the reader and to give the details of those procedures that have been revised.

Briefly, the study of the phase relations was accomplished by noting the melting characteristics of small, four-sided pyramidal specimens with an optical pyrometer and by petrographic and X-ray

examinations of the nonquenched heated specimens. It must be kept in mind that equilibrium conditions at the elevated temperatures may not have been established [5] and that the phase relations suggested were based primarily on the fusion behavior observations. Along with the inherent difficulties of petrographic examination and interpretation of powder slides and thin-sections of these nonquenched specimens with high refractive indices (2.08 and 2.15 for ZrO₂ and ThO₂, respectively), there were available only very small specimens for both the petrographic and X-ray examinations.

Absorption determinations were made on specimens boiled in carbon tetrachloride, and the results were converted to equivalent water absorption values. Mature bodies were those having less than one-tenth percent of equivalent water absorption.

The length-to-diameter ratio of all compression test specimens was approximately 2. All specimen ends were ground parallel on a Blanchard grinder using diamond wheels, and the specimens were crushed between parallel-ground, $\frac{5}{8}$ -in. thick, cold-rolled steel blocks at a loading rate of about 31,000 lb/in.² per minute.

Following maturing of the thermal shock and rupture test bars in a high-temperature gas-fired kiln, the specimens were ground on a Blanchard grinder using diamond wheels so that the bars would be approximately $\frac{1}{4}$ -in. thick with parallel faces. Ten cycles of quenching from 1,700° F (931° C) to an air-blast constituted the thermal shock test. The arrangement of the equipment for this test is shown schematically in figure 1 at "A". At "B" is an enlarged view of the specimen holder with six test bars. The bars were placed in the hot furnace (1,700° F $\pm 5^\circ$ F), heated for 30 min (the furnace reached and maintained 1,700° F for about the last 15 min), and were rapidly withdrawn to the air-blast. The orifice of the blower, delivering 625 ft³ of room-temperature air per minute, was 4 in. in diameter and was located 6 in. above the center of the group of specimens. After remaining in the air-blast for 15 min (the specimens were only warm after about 5 min), each bar was examined for failure. The order and position of the test bars were then changed according to a schedule and were replaced in the furnace to begin the next cycle.

The modulus of rupture values in bending were determined at room temperature for both the quenched and nonquenched bars [10].

The modulus of rupture and modulus of elasticity (Young's modulus) values at 1,800° F (982°

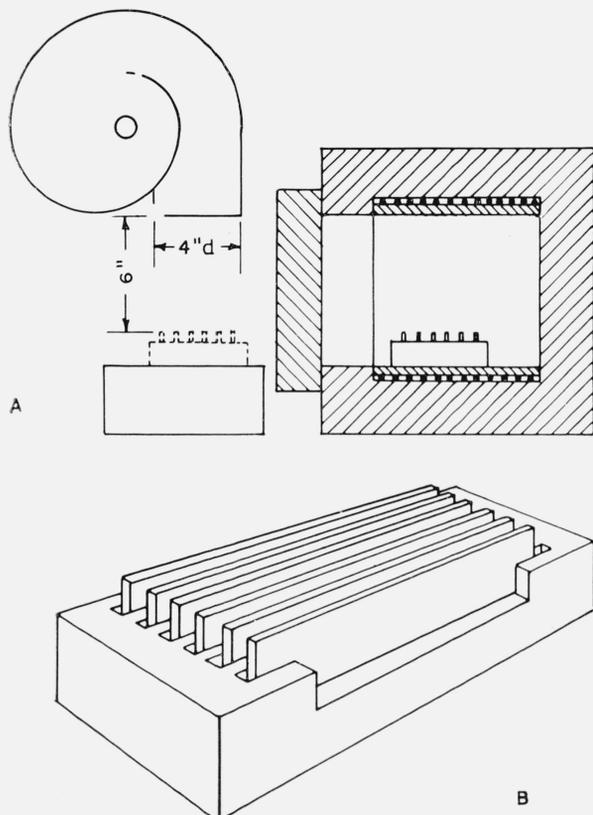


FIGURE 1. Schematic set-up for the thermal-shock test.

A schematic arrangement of the test specimens, furnace, and blower is shown at A, and an enlarged view of the test bars and the light-weight refractory holder is shown at B.

C) for the compositions studied in this investigation were determined by using a furnace designed by M. D. Burdick of this Bureau and heated by means of Globar elements. The furnace is designed to accommodate six heating elements and is intended for use at temperatures up to 2,500° F (1,371° C). The quarter-point loading formulae used for calculating the values for the modulus of rupture (M/R) and the modulus of elasticity (M/E) in bending were:

$$M/R = \frac{3P_1L}{4bd^2}, \quad (1)$$

and

$$M/E = \frac{P_2a(3L^2 - 4a^2)}{4\Delta bd^3}, \quad (2)$$

where P_1 is the total applied load, L is the span, b is the breadth at the breaking point, d is the depth at the breaking point, Δ is the deflection for any load P_2 , and a is one-quarter of the span L .

The interior of the furnace is shown in figure 2. The specimen bar (A) was supported on free-titling refractory knife-edges (B), and the deflection at mid-span was measured with a $\frac{1}{8}$ -in. diameter artificial sapphire rod (C) and a calibrated gage reading to 0.0001 in. In order to eliminate any corrections due to the expansion or movement of the supporting knife-edges during the test, the gage and gage holder, outside of the furnace, were supported with two sapphire rods (D) resting on the specimen at the span points. The four elements with which the furnace was heated for these tests were spaced symmetrically about the specimen,

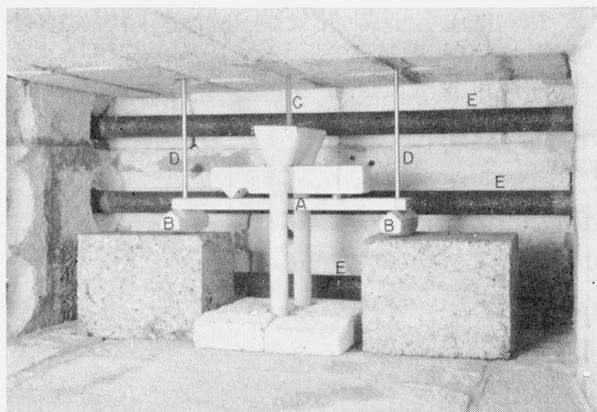


FIGURE 2. Interior view of the furnace for rupture in bending tests.

This photograph of the interior of the bending strength test furnace shows the test bar, A, in position for quarter-point loading. At B are shown the supporting knife-edges and the deflection at mid-span as measured with the sapphire rod, C, and a calibrated gage. This gage and the gage holder, outside of the furnace, are supported with two sapphire rods, D, resting on the specimen at the span points. Only the rear "Globar" heating elements, E, are shown.

and the temperatures were obtained with two chromel-alumel thermocouples; one located above and the other below the mid-span of the specimen. After having attained approximately equilibrium conditions in the furnace, the test bars were stressed in increments of 1,500 lb/in.² every 2 min, and gage deflection readings were taken every minute. When it was noted that the deflection following loading was practically constant by the end of the first minute, it was assumed that a permanent deformation, or "plastic flow", had not occurred.

IV. The System MgO-BeO-ZrO₂

1. The System MgO-BeO, Fields of Stability

The binary eutectic between MgO and BeO has been reported as being between 1,670° and 1,955° C and 50 and 48 percent of BeO⁴ [11, 12, 13]. A more recent investigation [5] has shown the binary eutectic to be at 1,855° C and 48.2 percent of BeO. No compounds or mixed crystals were found.

2. The System BeO-ZrO₂, Fields of Stability

In the system BeO-ZrO₂ a simple eutectic was first reported at about 2,180° C and 65 percent of ZrO₂ [14]. The compound 3BeO·2ZrO₂ and two eutectics at about 2,100° C and 65 and 85 percent of ZrO₂ were given in 1930 [15], but a year later a single eutectic at about 75 percent of ZrO₂ and 2,240° C was reported [16]. In the work at this Bureau [5] no evidence was found of a compound between BeO and ZrO₂.

3. The System MgO-ZrO₂

(a) Fields of Stability

The phase relations in this system are apparently complex, and many conflicting data have been given in the literature. Ruff and Ebert [17] reported two eutectics at about 26 and 80 mole percent of ZrO₂ and 1,600° and 1,550° C and the compound MgO·ZrO₂ melting at about 2,150° C. In addition, they indicated that a series of cubic mixed crystals exists between about 60 and 100 mole percent of zirconia. The first investigations of von Wartenburg and Werth produced similar results, but in a later report [15] they gave only one eutectic at about 50 mole percent of zirconia and 2,160° C and no compound formation. Ebert and Cohn [18] corroborated these later data, and in 1939 Zhirnova [19] further defined the

⁴ All composition values are given in weight percentage, unless otherwise noted.

simple eutectic at 50 mole percent (75.3 weight percent) of ZrO₂ and 2,070° C.

In the present investigation no attempt was made to redetermine the binary eutectic, but a considerable number of X-ray studies of various mixtures were made in the Constitution and Microstructure Section of this Bureau. In general, the results of this work indicate that the conclusions of Ruff and Ebert are correct in so far as the cubic isomorphous series between 60 and 100 mole percent (82 to 100%) of zirconia is concerned; but no compound of zirconia and magnesia was identified, which supports the work of Ebert and Cohn.

(b) Ceramic Bodies

Rieke and Ungewiss [20] made a number of ceramic bodies of MgO-ZrO₂ compositions, mainly for dielectric testing, and found that only one mixture (72.5% of MgO) in the range from 14 to 100 percent of magnesia showed a porosity of more than 0.1 percent at maturity. Shrinkage values were rather high, from 20 to 28 percent. The imperviousness of magnesia-zirconia compositions matured at high temperatures, 1,700° to 1,800° C, was noted also in a previous study of the thermal length changes of zirconia [9].

In this investigation only a few properties of selected body mixtures, ranging at about 10-percent intervals from 38 to 92 percent of zirconia, were determined, and these are given in table 2. All compositions between 50 and 92 percent of ZrO₂ could be matured to imperviousness at temperatures between 1,700° and 1,800° C. The 38 percent of zirconia mixture was overfired at 1,800° C and was not completely matured at 1,750° C (0.15% absorption). A complete study was not carried out because of the relatively high density of these bodies, although rather high values (29,000 to 30,000 lb/in.²) were obtained for the modulus of rupture in bending at 1,800° F (982° C).

TABLE 2. Some physical properties of compositions in the system MgO-ZrO₂

Composition				Maturing range	Properties at maturity (less than 0.1 percent of water absorption)									
Mole		Weight			Properties at maximum density					Compressive strength (±5,000 lb/in. ²)	Shrinkage ^a	Absorption ^a	Strength in bending at 1,800° F (982° C)	
MgO	ZrO ₂	MgO	ZrO ₂		Test No.	Temperature	Shrinkage	Absorption	Density				M/R	M/E
		%	%	° C	° C	%	%	g/cm ³	lb/in. ²	%	%	lb/in. ² × 10 ³	lb/in. ² × 10 ³	
5	1	62.06	37.94	1,800	423-L	1,750	12.5	0.15	---	---	---	---	---	
3	1	49.54	50.46	1,700	494-L	1,700	14.9	.01	4.23	166,000	17.93	0.08	28.8	30.8
2	1	39.56	60.44	1,700 to 1,800	417-L	1,750	13.2	.00	---	---	---	---	---	
3	2	32.92	67.08	1,700 to 1,800	492-L	1,700	13.5	.00	4.64	208,000	16.45	.00	---	---
1	1	24.65	75.35	1,700 to 1,800	451-L	1,800	15.1	.03	---	---	---	---	---	
1	2	14.07	85.93	1,750 to 1,800	495-L	1,750	13.8	.00	4.96	229,000	16.60	.03	29.8	23.0
1	4	7.56	92.44	1,800	422-L	1,800	12.5	.00	---	---	---	---	---	

^a These values were determined for the strength in bending test bars.

^b The composition was overfired at 1,800° C and was not completely matured at 1,750° C.

^c These specimens were matured at 1,750° C for 1 hour in an oxy-gas furnace by the Linde Air Products Co.

4. The System MgO-BeO-ZrO₂

(a) Fields of Stability

The results of the fusion-point and softening-range determinations and of the petrographic and X-ray examinations of some of the 61 mixtures investigated are summarized in table 3. Only those considered essential to the establishment of the probable phase relations are reported. The recorded data for each mixture were obtained for the specimen that had been heated at or near the estimated liquidus or at the highest temperature when the liquidus was not reached. It must be emphasized that equilibrium may not have been attained and that the slow cooling (about 200° C per hour) in the furnace may have destroyed any evidence of the phases present at the higher temperatures.

Figure 3 is a graphic representation of the system as derived from the available data. This system is believed to be a simple one with the ternary eutectic at or near mole composition 4M:5B:3Z⁵ and 1,669° ± 3° C. The primary phase boundaries (short-dash lines) are assumed to be essentially straight lines joining the various binary eutectics and the ternary eutectic. No evidence of compound formation was observed either with the fusion-point and softening-range determinations or with the petrographic and X-ray examinations.

The intimately mixed oxides were used without presintering for the initial observations of the melting characteristics in this system.

⁵ This short form of indicating mole composition mixtures in the ternary systems is used throughout the text. The code is M=MgO, B=BeO, Z=ZrO₂, and T=ThO₂. The short form, i. e., 4M:5B:3Z, then reads 4 moles of MgO plus 5 moles of BeO plus 3 moles of ZrO₂ and does not indicate a compound.

TABLE 3. Results of fusion-point and softening-range observations and of petrographic and X-ray examinations of some compositions in the system MgO-BeO-ZrO₂

Composition						Maximum temperature	Effect of heating ^a	Results of petrographic and X-ray examinations (Specimens examined were not quenched, and the phases observed may not be those in equilibrium at the maximum temperature of heating or at the liquidus)	Test No.
Mole			Weight						
MgO	BeO	ZrO ₂	MgO	BeO	ZrO ₂				
			%	%	%	° C	(All temperatures in ° C)		
1	1	1	21.39	13.27	65.34	1,710	Fusion began at 1,688°, completed at less than 1,710°.	Cubic zirconia is the principal phase. Beryllia occurs as small angular crystals.	558
1	2	1	18.88	23.43	57.69	1,704	Fusion began at 1,689°, almost completed at 1,704°.	Beryllia occurs as blocky grains, zirconia as round grains usually cloudy from small intermixed magnesia particles.	583
1	3	1	16.90	31.46	51.64	1,765	Fusion began at 1,690°, completed at 1,765°.	Beryllia is the primary phase. Magnesia and cubic zirconia are present. X-ray: MgO, BeO, and ZrO ₂ —9/10 cubic.	540
1	4	2	10.42	25.87	63.71	1,886	Fusion began at 1,731°, nearly completed at 1,886°.	Beryllia is the primary phase, 40 microns and blocky, in a matrix of partially cloudy zirconia.	545
1	4	4	6.37	15.80	77.83	1,850	Fusion began at 1,770°, no noticeable change at 1,850°.	Large and small blocky beryllia crystals in a matrix of cubic and monoclinic zirconia. The ZrO ₂ is cloudy from small particles, an exsolution texture. X-ray: weak MgO, BeO, and ZrO ₂ —9/10 cubic.	566
1	4	8	3.58	8.89	87.53	1,890	No noticeable fusion up to 1,890°.	Zirconia appears to be the primary phase.	527-L
1	5	1	13.97	43.34	42.69	1,825	Fusion began at 1,687°, completed at 1,825°.	The principal phase is beryllia. Very small inclusions in the zirconia are probably due to exsolution. X-ray: MgO, BeO, and ZrO ₂ —9/10 cubic.	542
1	6	3	7.20	26.80	66.00	1,850	Fusion began at 1,819°, no noticeable change at 1,850°.	Beryllia appears to be the primary phase. The zirconia has small inclusions probably having an exsolution texture.	533
1	9	2	7.88	43.99	48.13	1,925	Partial fusion at 1,925°-----	Beryllia is the primary phase, occurring as large as 100 microns, with cubic and monoclinic interstitial zirconia.	554-L
1	10	1	9.75	60.47	29.78	1,925	Advanced fusion at 1,925°----	Large, blocky beryllia crystals occur with small interstitial zirconia grains. X-ray: MgO, BeO, and ZrO ₂ —2/3 cubic.	554-L
1	12	4	4.84	36.03	59.13	1,890	No noticeable fusion up to 1,890°.	-----	528-L
1	24	1	5.28	78.59	16.13	1,890	Fusion began at 1694°, no noticeable change at 1,890°.	Principal phase is beryllia, 70 microns and blocky, with interstitial zirconia having very small inclusions. X-ray: very weak MgO, BeO, and cubic ZrO ₂ .	548
2	1	1	35.23	10.93	53.84	1,731	Fusion began at 1681°, completed at 1731°.	Magnesia is the primary phase occurring as flaky crystals. Zirconia crystals are cloudy and a few small beryllia crystals are seen.	565
2	5	1	24.52	38.03	37.45	1,715	Fusion began at 1,687°, almost completed at 1715°.	The principal phase is beryllia. Magnesia occurs as round grains. Interstitial zirconia occurs as small rod-shaped or as dendritic growths.	568
3	1	1	44.94	9.29	45.77	1,745	Fusion began at 1691°, almost completed at 1,745°.	Magnesia is the principal phase. Zirconia is all cubic.	571
3	5	0.4	40.96	42.36	16.68	1,800	Fusion began at 1689°, completed at 1,800°.	The principal phases are beryllia and magnesia with interstitial cubic zirconia.	559
3	5	1	32.76	33.88	33.36	1,715	Fusion began at 1689°, completed at 1715°.	Similar to composition 3MgO:5BeO:0.4ZrO ₂ .	504-L
3	5	2	24.56	25.40	50.04	1,693	Fusion began at 1688°, completed at 1,693°.	Large magnesia and beryllia crystals occur in parts in a rosette matrix of dendritic zirconia and magnesia. In other parts a three-phase mosaic structure occurs. Near the eutectic but with an excess of zirconia.	552
3	5	3	19.65	20.32	60.03	1,695	Fusion began at 1690°, completed at 1695°.	The principal phases are beryllia and zirconia.	567
3	5	4	16.37	16.93	66.70	1,761	Fusion began at 1688°, completed at 1,761°.	The principal phase is zirconia. It occurs as both clear and cloudy cubic and monoclinic round crystals and as small amoeboid rods, and dendritic crystals with intermixed similarly shaped magnesia crystals.	563
3	5	8	9.82	10.16	80.02	1,890	Fusion began at 1688°, completed at 1,890°.	Zirconia occurs as the principal phase. The large round grains have an anisotropic center with the cubic form surrounding it and occasionally passing through as a band. X-ray: BeO and cubic ZrO ₂ .	544
3	10	1	24.47	50.61	24.92	1,777	Fusion began at 1692°, completed at 1777°.	Beryllia and magnesia occur with interstitial cubic zirconia. Beryllia appears to be the primary phase.	535
3	15	1	19.53	60.58	19.89	1,800	Fusion began at 1689°, completed at 1800°.	Beryllia is the primary phase with small interstitial magnesia and zirconia crystals.	543
3	24	1	14.32	71.09	14.59	1,915	Fusion began at 1,688°, nearly completed at 1915°.	Beryllia is the primary phase-----	550

See footnote at end of table.

TABLE 3. Results of fusion-point and softening-range observations and of petrographic and X-ray examinations of some compositions in the system MgO-BeO-ZrO₂—Continued

Composition						Maximum temperature	Effect of heating ^a	Results of petrographic and X-ray examinations (Specimens examined were not quenched, and the phases observed may not be those in equilibrium at the maximum temperature of heating or at the liquidus)	Test No.
Mole			Weight						
MgO	BeO	ZrO ₂	MgO	BeO	ZrO ₂				
			%	%	%	° C	(All temperatures in ° C)		
3	40	1	9.72	80.38	9.90	1,850	Fusion began at 1,689°, partial fusion up to 1850°.	Beryllia is the primary phase. X-ray: MgO, BeO, and cubic ZrO ₂ .	594
4	5	2	30.27	23.48	46.25	1,697	Fusion began at 1,690°, completed at 1,697°.	The principal phases are magnesia and beryllia with small intergrowth crystals of magnesia and zirconia.	577
4	5	3	24.59	19.07	56.34	1,693	Fusion began at 1,689°, completed at 1,693°.	Magnesia, beryllia, and zirconia occur as about 30 micron single crystals in a matrix of small, 5 micron and less, intermixed crystals of the same three phases. This is at or near the eutectic composition.	555
5	5	3	28.95	17.97	53.08	1,697	Fusion began at 1,690°, almost completed at 1,697°.	Magnesia occurs in parts in a rosette matrix of the three constituents. In other parts a three phase mosaic structure is present, near the eutectic but with an excess of magnesia.	585
5	24	1	21.79	64.89	13.32	1,911	Fusion began at less than 1,693°, partial fusion up to 1,911°.	Beryllia is the primary phase.....	572
5	48	1	13.22	78.705	8.075	1,855	Fusion began at 1,692°, partial fusion up to 1,855°.	Similar to composition 5MgO:24BeO:1ZrO ₂	573
6	5	1	49.35	25.52	25.13	1,783	Fusion began at 1,690°, partial fusion up to 1,783°.	Magnesia and beryllia are the principal phases with rare interstitial small crystals of zirconia.	569
6	10	3	28.08	29.03	42.89	1,704	Fusion began at 1,689°, completed at 1,704°.	Round magnesia and blocky beryllia crystals occur in a matrix of small zirconia crystals intermixed with similar magnesia and beryllia crystals.	580
6	10	5	21.83	22.58	55.59	1,695	Fusion began at 1,688°, nearly completed at 1,695°.	Beryllia is the principal phase in a matrix of small (5 microns and less) intermixed magnesia, beryllia, and ironia crystals, near the eutectic and with an excess of beryllia.	575
15	5	1	70.89	14.67	14.44	1,860	Fusion began at 1,690°, partial fusion up to 1,860°.	Magnesia is the primary phase.....	570
0.28	10	1	3.00	65.00	32.00	1,925	No noticeable fusion up to 1,925°.	Beryllia occurs with interstitial cloudy zirconia grains. X-ray: very weak MgO, BeO, and ZrO ₂ —9/10 cubic.	554-L
1	16	3	5.00	50.00	45.00	1,925	No noticeable fusion up to 1,925°.	Very similar to weight composition 3MgO+65BeO+32ZrO ₂	554-L
3	5	2	24.56	25.40	50.04	^b 1,691	Fusion began at 1,679°, completed at 1,691°. ^b	Beryllia is the primary phase in a matrix of very small intermixed magnesia and zirconia crystals. X-ray: MgO, BeO, and cubic ZrO ₂ .	602 ^b
4	5	3	24.59	19.07	56.34	^b 1,683	Fusion began at 1,669°, completed at 1,683°. ^b	Same as composition 4MgO:5BeO:3ZrO ₂ (Test No. 555)...	615 ^b

^a In most cases, the beginning of fusion as indicated is for the test of the specimen described and is usually within 2° C of the average value obtained for three or more determinations of the same composition. The term "complete fusion" means that the pyramidal specimen had fused sufficiently to flow into a flat button shape and does not necessarily connote that the mixture had completely liquefied.

^b These compositions were presintered at 1,670° C for 30 min, crushed, and reformed before the fusion-point and softening-range determinations were made.

An attempt to identify the ternary eutectic solely by microscopic examination and subsequent interpretation was only partially successful. The most that could be done was to identify an area in which the specimens examined contained no distinguishable primary phases and which were composed mainly of a regular mosaic type structure. The area (fig. 3) so defined is bounded by the compositions 3M:5B:2Z, 1M:2B:1Z, 3M:5B:4Z, 1M:1B:1Z, and 5M:5B:3Z.

In order to determine the ternary eutectic more accurately, a number of compositions were presintered at 1,670° C for 30 min and ground in

mullite and boron carbide mortars to pass the No. 325 U. S. Standard Sieve. This finely divided material was then pressed, shaped, and tested according to the procedures given previously. The results of tests on two of these presintered compositions are included at the end of table 3. It was noted that the temperature at which fusion began for the presintered ternary eutectic mixture was 20° C lower than that for the unsintered mixture.

An area of possible solid solution is suggested for this system extending along the BeO-ZrO₂ join from about 5 percent to about 85 percent of BeO

and bounded by, but excluding, compositions 1M:24B:1Z, 1M:10B:1Z, 1M:5B:1Z, 3M:5B:4Z, and 3M:5B:8Z. This area is shown on the diagram enclosed by a long-dash line. In this area there may be at least two phases containing limited amounts of the end members in solid solution. The ends of the line approximate the limits of solid solubility. Although the differences in the refractive index values between MgO and BeO and ZrO₂ are appreciable, petrographic examination failed to reveal any changes in these values, and only occasionally were the indices of the cubic form of ZrO₂ [9] found. In the compositions 1M:6B:3Z and 1M:4B:4Z, however, a very definite exsolution texture was seen, although the crystals of lower refractive index than zirconia were too small to be sufficiently resolved by the microscope for complete optical identification.

The appearance of this exsolution texture is similar to that seen in specimens of the MgO-BeO-ThO₂ system, but the texture is of much finer grain size. The appearance of lines for cubic ZrO₂ in the X-ray patterns is not necessarily an indication of ternary solid solution, but rather the expected form of ZrO₂ in the presence of magnesia [9, 19].

(b) Ceramic Bodies

The only available literature information on ceramic bodies in this system [21, 22] is concerned mainly with modifying the thermal expansion characteristics of zirconia by the addition of small amounts of the other oxides.

Impervious bodies may be made from compositions in approximately one-half of the ternary diagram area. The compositions of these bodies

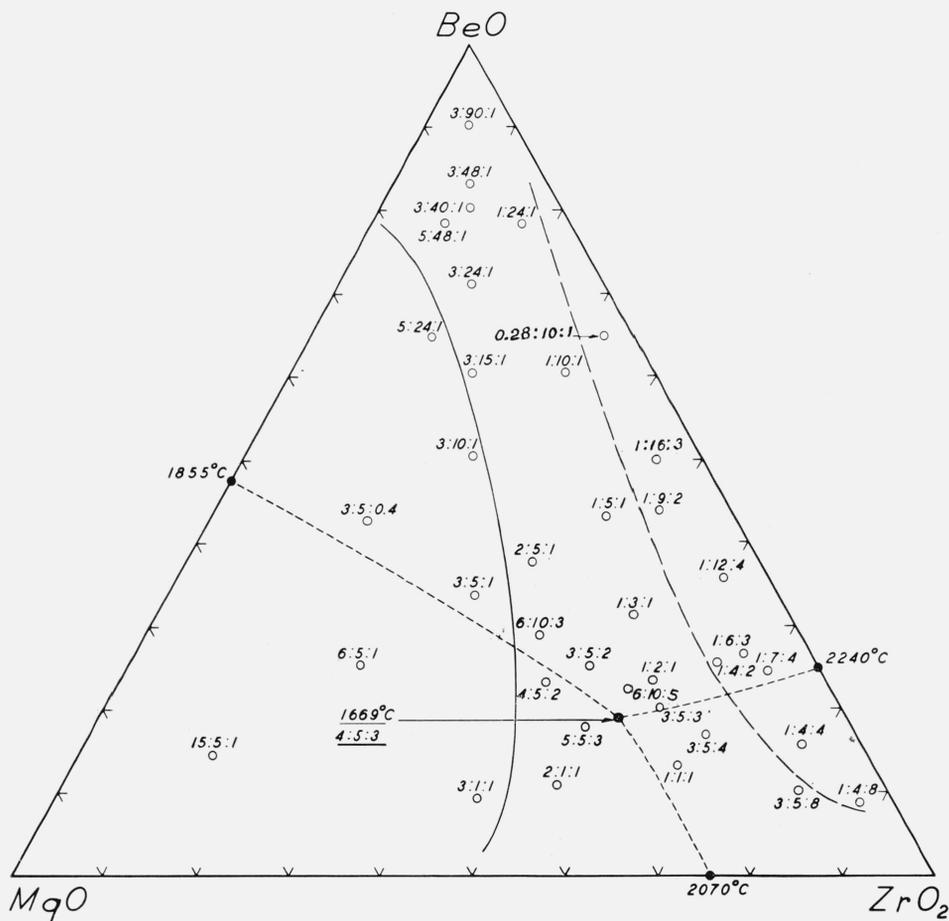


FIGURE 3. The system MgO-BeO-ZrO₂.

This figure gives the location of the primary phase boundaries (short-dash lines), a suggested area of solid solution (long-dash lines), and the range of compositions from which impervious porcelain bodies may be made (to the right of the solid line). Compositions are indicated in molar ratios but are graphically placed according to weight percentages. Twenty-two additional mixtures, not reported, were investigated in the eutectic area.

lie to the right of the solid line shown in figure 3 and could usually be matured between 1,450° and 1,600° C. Table 4 gives the results of the maturing range studies and the values of porosity and shrinkage at maximum density. Those bodies that could be matured to a practically impervious condition over a range of 50° C or more have absorption values averaging about 0.02 percent and shrinkage values ranging from 15 to 21 percent.

Compressive strength values (table 4) ranged from 164,000 to 264,000 lb/in.² for these porcelains, and the outstanding body in these tests was 1M:24B:1Z (264,000 lb/in.²). The maturing range of this porcelain was considerable (1,450° to 1,600° C), and its density (3.25 g/cm³) was one of the lowest observed. The table gives some comparative compression results for three body compositions similarly fabricated but matured in a gas-fired furnace.

TABLE 4. *Maturing range studies and compressive strength results of some compositions in the system MgO-BeO-ZrO₂*

Composition						Maturing ^a b range	Properties ^b				Properties of compressive strength specimens ^d						
Mole			Weight				Test No.	Temperature	Shrinkage	Absorption	Test No.	Temperature	Shrinkage	Absorption	Density	Height/diameter ^e	Strength ^a at room temp. (±5,000 lb./in. ²)
M	B	Z	MgO	BeO	ZrO ₂												
			%	%	%	° C								g/cm ³		n x10 ³	
1	1	1	21.39	13.27	65.34	1,500 to 1,550	519-L	1,500	18.03	0.03	549-L	1,500	14.42	0.01	4.46	1.93	225
1	3	1	16.90	31.46	51.64	1,450 to 1,600	470-L	1,600	18.07	.00	507-L	1,600	14.61	.02	3.87	1.97	204
1	4	2	10.42	25.87	63.71	1,450 to 1,600	471-L	1,550	17.59	.00	508-L	1,550	17.94	.00	4.48	1.96	259
1	4	4	6.37	15.80	77.83	1,500 to 1,600	471-L	1,550	16.96	.00	509-L	1,550	16.98	.01	4.81	1.98	234
1	5	1	13.97	43.34	42.69	1,450 to 1,600	470-L	1,600	18.86	.00	510-L	1,600	18.42	.03	3.9	1.83	231
1	6	3	7.20	26.80	66.00	1,500 to 1,600	516-L	1,600	17.56	.01	530-L	1,600	16.35	.00	4.37	1.94	249
1	6	3	7.20	26.80	66.00	1,500 to 1,600	-----	-----	-----	-----	6/16/44	1,600	-----	.01	4.37	1.08	^f 243
1	6	3	7.20	26.80	66.00	1,500 to 1,600	-----	-----	-----	-----	7/25/44	1,550	-----	.00	4.30	1.93	^f 262
1	7	4	5.69	24.73	69.58	-----	-----	-----	-----	-----	7/15/44	1,600	-----	.00	4.49	1.92	^f 207
1	7	4	5.69	24.73	69.58	-----	-----	-----	-----	-----	7/25/44	1,550	-----	.05	4.22	1.90	^f 241
1	10	1	9.75	60.47	29.78	1,450 to 1,600	473-L	1,500	19.46	.01	512-L	1,500	18.91	.04	3.47	1.97	220
1	24	1	5.28	78.59	16.13	1,450 to 1,600	-----	1,500 to 1,600	19.4	.00	513-L	1,550	19.55	.01	3.25	1.91	264
2	1	1	35.23	10.93	53.84	1,550	517-L	1,550	17.24	.05	531-L	1,550	15.03	.01	4.40	1.86	206
2	5	1	24.52	38.03	37.45	1,500 to 1,550	519-L	1,500	18.19	.07	532-L	1,500	16.29	.02	3.73	1.95	215
3	5	2	24.56	25.40	50.04	1,500	519-L	1,500	17.56	.02	526-L	1,500	15.60	.02	4.05	1.96	208
3	5	3	19.65	20.32	60.03	1,500 to 1,600	313-L	1,600	15.2	.01	514-L	1,600	16.18	.01	4.38	1.92	202
3	5	8	9.82	10.16	80.02	1,550	475-L	1,550	15.56	.01	515-L	1,550	15.17	.01	4.80	1.92	220
3	5	8	9.82	10.16	80.02	1,550	-----	-----	-----	-----	7/25/44	1,550	-----	.00	4.81	1.89	^f 238
3	15	1	19.53	60.58	19.89	1,500 to 1,600	319-L	1,550	19.1	.05	502-L	1,550	17.38	.05	3.31	1.97	164
3	24	1	14.32	71.09	14.59	1,450	491-L	1,450	17.4	.06	545-L	1,450	16.50	.05	3.23	1.95	242
3	40	1	9.72	80.38	9.90	1,500 to 1,600	313-L	1,600	20.6	.03	522-L	1,600	19.01	.01	3.18	2.00	222
3	48	1	8.37	83.10	8.53	1,450 to 1,550	493-L	1,500	19.15	--	550-L	1,500	18.26	.04	3.07	2.02	229
3	90	1	4.74	90.32	4.94	1,450 to 1,550	499-L	1,550	19.5	.02	524-L	1,550	19.07	.02	2.97	2.08	172
5	48	1	13.22	78.705	8.075	1,450 to 1,550	491-L	1,450	19.62	.05	-----	---	---	---	---	---	-----

^a Overfiring and underfiring occurred at 50 deg C above and at 50 deg C below the temperature ranges given.

^b Values obtained for specimens matured in an electric furnace for 1 hour at the temperature specified.

^c Specimens about 1 in. high by ½ in. in diameter.

^d Unless otherwise indicated, the values given are the averages for three test specimens.

^e Compression testing was done at a constant loading rate of 31,250 lb/in.² per min, using cold-rolled steel contact blocks.

^f Values obtained for specimens matured in a gas-fired furnace.

^g Value given is for one specimen only.

Nine body compositions were selected to represent the relative resistance to thermal shock, and strengths in bending at room and at elevated temperature, of the refractory porcelains in the system. Table 5 gives the data for these properties and also includes some values of the modulus of rupture and Young's modulus for a few bodies tested at 1,900° F (1,038° C) and 2,000° F (1,093° C). As expected, the high-beryllia bodies were the most resistant to thermal quenching, and the high-zirconia bodies either did not survive the heat-shock test or were very easily broken by finger pressure alone after testing. Generally, the rupture values of the high-beryllia bodies were slightly higher after shocking than those obtained

for unshocked specimens. The values for the modulus of rupture in bending at room temperature for the high-beryllia bodies ranged from 13,000 to 20,000 lb/in.², and from 23,000 to 39,000 lb/in.² for the high-zirconia bodies. Modulus of rupture and modulus of elasticity (Young's modulus) values in bending at 1,800° F for the high-beryllia bodies ranged from 13,000 to 18,000 lb/in.² and 38,000,000 to 34,000,000 lb/in.², respectively, and for the high-zirconia bodies the values were from 21,000 to 35,000 lb/in.² and from 31,000,000 to 15,000,000 lb/in.², respectively. It is interesting to note that, at 1,800° F, the Young's moduli values decrease as the moduli of rupture values increase.

TABLE 5. Resistance to thermal shock and rupture and elastic strength values of some compositions in the system MgO-BeO-ZrO₂

Composition						Maturing data ^a			Resistance to thermal shock ^b (2 bars each composition)	Strength in bending values ^c						
Mole			Weight			Test No.	Temperature	Absorption		M/R at room temperature ^d (±2,000 lb/in. ²)	M/R at room temperature after shocking ^e (±2,000 lb/in. ²)	M/R at elevated temperatures ^f (±2,000 lb/in. ²)		Young's Modulus at elevated temperatures ^g (±3,000,000 lb/in. ²)		
M	B	Z	MgO	BeO	ZrO ₂							Temperature	Value	Temperature	Value	Temperature
			%	%	%		° C	%		n×10 ³ lb/in. ²	n×10 ³ lb/in. ²	° F	n×10 ³ lb/in. ²	° F	n×10 ⁶ lb/in. ²	
1	5	1	13.97	43.34	42.69	22-R	1,625	0.03	Cracked on 3d and 4th cycle.	23.1	-----	1,800	20.7	1,800	31.3	
1	6	3	7.20	26.80	66.00	-----	1,600	h,00	Broke on 3d and 4th cycle. ^b	h 38.6	-----	1,800	h ^k 30.9	1,800	h ^k 22.7	
1	6	3	7.20	26.80	66.00	-----	1,600	h,00	-----	-----	-----	1,900	h ^j 34.1	1,900	h ^j 10.6	
1	6	3	7.20	26.80	66.00	-----	1,600	h,00	-----	-----	-----	2,000	h ^k 32.8	2,000	h ^k 11.8	
1	7	4	5.69	24.73	69.58	-----	1,600	h,03	{ One of five test specimens ^h broke on 8th cycle.-----	h ^j 28.2	-----	1,800	h ^k 24.5	1,800	h ^k 19.7	
1	7	4	5.69	24.73	69.58	-----	1,600	h,03		-----	-----	-----	2,000	h ^j 28.9	2,000	h ^j 13.2
1	10	1	9.75	60.47	29.78	8-R	≈ 1,600	.02	-----	21.7	23.3	1,800	18.9	1,800	33.8	
3	5	3	19.65	20.32	60.03	-----	1,600	h,02	{ Two of ten test specimens were easily broken after 10th cycle. ^h	h 26.3	h ⁱ 29.2	1,800	h ^j 31.8	1,800	h ^j 16.9	
3	5	3	19.65	20.32	60.03	-----	1,600	h,01		-----	-----	-----	1,900	h ^j 24.0	1,900	h ^j 9.7
3	5	8	9.82	10.16	80.02	-----	1,600	h,03	{ Two of eleven test specimens were easily broken after 10th cycle. ^h	h 24.5	h ^j 25.6	1,800	h ² 25.8	1,800	h ¹⁹ 6	
3	5	8	9.82	10.16	80.02	-----	1,600	h,00		-----	-----	-----	1,900	h ^j 33.8	1,900	h ¹⁴ 4
3	5	8	9.82	10.16	80.02	-----	1,600	h,00		-----	-----	-----	2,000	h ^j 32.7	2,000	h ^j 5.4
3	24	1	14.32	71.09	14.59	14-R	1,550	.15	No failure.-----	20.1	20.4	1,800	18.4	1,800	34.1	
3	40	1	9.72	80.38	9.90	13-R	≈ 1,650	.4	do.-----	12.6	13.2	1,800	13.1	1,800	38.0	
3	90	1	4.74	90.32	4.94	25-R	{ 1,625 1,600	1.0	do.-----	15.3	14.3	1,800	12.6	1,800	38.4	

^a All test specimens were matured in a gas-fired furnace at the temperature indicated for 1 hour unless otherwise specified.

^b Ten cycles of test from 1,700° F to room temperature air blast for two test specimens, about 5½ in. by ¾ in. by ¼ in., of each composition.

^c M/R is the modulus of rupture.

^d Two specimens, ca. 5½ in. by ¾ in. by ¼ in., of each composition were tested.

^e Only those specimens that survived 10 cycles of heat shocking were tested.

^f Three specimens, ca. 5½ in. by ¾ in. by ¼ in., of each composition were tested unless otherwise specified.

^g Matured 2 hours.

^h For preliminary report see reference [2].

ⁱ Quenched from 1,500° F.

^j One specimen tested.

^k Two specimens tested.

^l Matured for 1 hour at each of these temperatures.

V. The System MgO-BeO-ThO₂

1. The System MgO-ThO₂, Fields of Stability

Von Wartenburg and Prophet [11] describe the system MgO-ThO₂ as having no melting below 2,500° C.

2. The System BeO-ThO₂, Fields of Stability

A simple system of one eutectic at about 20 percent of BeO and 2,200° C was reported by von Wartenburg and coworkers [13].

3. The System MgO-BeO-ThO₂

(a) Fields of Stability

The results of the fusion-point and softening-range determinations and of the petrographic and X-ray examinations of some of the mixtures investigated are summarized in table 6. Only those considered essential to the establishment of the probable phase relations are reported. The recorded data for each mixture were obtained for the specimen that had been heated at or near

the estimated liquidus or at the highest temperature when the liquidus was not reached. It will bear repeating that equilibrium may not have been attained and that the slow cooling (about 200° C per hour) in the furnace may have destroyed any evidence of the phases present at the higher temperatures.

Figure 4 is a graphic representation of the system as derived from the available data. This system is believed to be a simple one with the ternary eutectic at or near mole composition 5M:10B:1T and 1,797° ± 5° C. The primary phase boundaries (short dash lines) are assumed to be essentially straight lines joining the various binary eutectics and the ternary eutectic. The boundary line between the ternary eutectic and the MgO-ThO₂ eutectic is shown partially drawn, approximating the results of this investigation. No evidence of compound formation was observed either with the fusion-point and softening-range determinations or with petrographic and X-ray examinations.

TABLE 6. Results of fusion-point and softening-range observations and of petrographic and X-ray examinations of some compositions in the system MgO-BeO-ThO₂

Composition						Maximum temperature	Effect of heating ^a (All temperatures in ° C)	Results of petrographic and X-ray examinations ^b (Specimens examined were not quenched and the phases observed may not be those in equilibrium at the maximum temperature of heating or at the liquidus)	Test No.
Mole			Weight						
MgO	BeO	ThO ₂	MgO	BeO	ThO ₂				
			%	%	%	° C			
2	7	1	15.51	33.69	50.80	1,832	Fusion began at 1,801°, completed at 1,832°.	Beryllia is the primary phase and occurs with interstitial cubic and rounded magnesia and thoria. Some thoria occurs as exsolution from beryllia. X-ray: weak MgO and BeO and very strong ThO ₂ .	653
2	10	1	13.56	42.05	44.39	1,871	Fusion began at 1,798°, advanced fusion at 1,871°.	Similar to composition 2MgO:7BeO:1ThO ₂ , except that the X-ray shows medium strong ThO ₂ .	654
2	15	1	11.20	52.12	36.68	1,870	Fusion began at 1,790°, slight fusion at 1,870°.	Similar to composition 2MgO:7BeO:1ThO ₂ , except for relative amount of beryllia by petrographic examination.	658
2	25	1	8.31	64.47	27.22	1,910	Fusion began at 1,801°, slight fusion at 1,910°.	Beryllia occurs as round grains with thoria as exsolution in major amounts in a restricted portion of the grain. Thoria also occurs interstitially. X-ray: medium strong ThO ₂ , strong BeO, and very weak MgO.	677
3	28	4	6.44	37.31	56.25	1,900	Fusion began at about 1,795° to 1,800°, no further fusion up to 1,900°.	An exsolution texture of thoria from beryllia in practically all beryllia crystals. X-ray: strong ThO ₂ , weak BeO, and very weak MgO.	659
5	10	1	28.16	34.95	36.89	1,814	Fusion began at 1,797°, completed at 1,814°.	Beryllia crystals contain both magnesia and thoria inclusions as spheres and dendritic crystals mostly as an intergrowth structure. This is near the ternary eutectic. X-ray: strong ThO ₂ and weak MgO and BeO.	671
6	4	1	39.92	16.51	43.57	1,839	Fusion began at 1,813°, completed at less than 1,839°.	Magnesia is the primary phase. Thoria occurs as dendritic inclusions in very large, 300 micron, beryllia crystals.	645
6	9	2	24.31	22.62	53.07	1,818	Fusion began at 1,802°, almost completed at 1,815°.	Thoria appears to be the principal phase. It also occurs occasionally as an exsolution from beryllia. See photomicrograph of a selected section of this specimen in figure 5.	635

See footnotes at end of table.

TABLE 6. Results of fusion-point and softening-range observations and of petrographic and X-ray examinations of some compositions in the system MgO-BeO-ThO₂—Continued

Composition						Maximum temperature	Effect of heating ^a (All temperatures in °C)	Results of petrographic and X-ray examinations ^b (Specimens examined were not quenched and the phases observed may not be those in equilibrium at the maximum temperature of heating or at the liquidus)	Test No.
Mole			Weight						
MgO	BeO	ThO ₂	MgO	BeO	ThO ₂				
			%	%	%	°C			
6	10	1	31.99	33.09	34.92	1,822	Fusion began at 1,803°, almost completed at 1,822°.	The primary phase could not be determined. Beryllia occurs as large crystals, some with intergrowth of dendritic thoria, magnesia as round or cube-shaped crystals, and thoria as small spheres and dendritic crystals.	646
6	10	3	18.83	19.48	61.69	1,819	Fusion began at 1,805°, completed at about 1,819°.	The primary phase, thoria, occurs as spheres and as dendritic rods in a matrix of beryllia.	636
6	20	1	24.04	49.72	26.24	1,814	Fusion began at 1,803°, completed at 1,814°.	Beryllia appears to be the primary phase. The interstitial material consists of dendritic thoria in an intergrowth texture with small magnesia and beryllia crystals.	638
6	42	1	15.54	67.50	16.96	1,916	Fusion began at 1,790°, nearly completed at 1,916°.	Beryllia is the primary phase and occurs with thoria inclusions clustered in one area of each crystal as an exsolution texture (fig. 5). X-ray: strong BeO, medium strong ThO ₂ , and weak MgO.	657
6	78	1	9.84	79.41	10.75	1,902	Fusion began at about 1,792°, slight fusion at 1,902°.	Similar to composition 6MgO:42BeO:1ThO ₂ . The beryllia crystals are larger. X-ray: very weak MgO, others are the same as above.	675
7	10	1	35.43	31.41	33.16	1,823	Fusion begun at 1,799°, almost completed at 1,823°.	Magnesia appears to be the primary phase.....	638-L
12	10	1	48.47	25.07	26.46	1,826	Fusion began at 1,797°, completed at 1,826°.	Magnesia is the primary phase. The crystal sizes of the three components average about 40 microns.	666
12	42	1	26.90	58.42	14.68	1,820	Fusion began at 1,797°, completed at 1,820°.	Beryllia is the primary phase and contains an intergrowth of dendritic thoria. Magnesia appears interstitially and as inclusions in some beryllia crystals. X-ray: strong ThO ₂ , medium strong BeO, and very weak MgO.	669
12	78	1	17.93	72.29	9.78	1,860	Fusion nearly completed at 1,860°.	The principal phase is beryllia occurring as round grains having exsolution thoria inclusions in selected areas of the crystal. X-ray: medium strong BeO and weak MgO and ThO ₂ .	663
30	42	1	47.91	41.63	10.46	1,848	Fusion began at 1,802°, completed at 1,848°.	Magnesia occurs as large cubes and as striations across and through some beryllia crystals indicating proximity to a phase boundary with magnesia as the probable principal phase.	667
30	120	1	27.02	67.08	5.90	1,842	Fusion began at 1,801°, completed at 1,842°.	Beryllia is the principal phase and occurs as large round and blocky crystals usually having exsolution thoria inclusions. X-ray: medium strong MgO and BeO and weak ThO ₂ .	674

^a In most cases the beginning of fusion as indicated is for the test of the specimen described and is usually within 2° C of the average value obtained for three or more determinations of the same composition. The term "complete fusion" means that the pyramidal specimens had fused sufficiently to flow into a flat button shape and does not necessarily connote that the mixture had completely liquefied.

^b Many of the petrographic descriptions given are illustrated in the photomicrographs of figure 5.

All compositions for the fusion-point and softening-range determinations reported in this system were presintered at 1,750° C for 30 min and ground in mullite and boron carbide mortars to pass the No. 325 U. S. Standard Sieve. This finely divided material was then pressed, shaped, and tested according to the procedures previously described.

The location and temperature of the ternary eutectic of this system were based almost entirely upon the data obtained from the fusion-point and softening-range observations, because petrographic examinations failed to reveal even an area sur-

rounding the probable ternary eutectic as was found for the zirconia system. In addition, many of the specimen compositions in all sections of the diagram showed both a mosaic and a dendritic type structure as seen in the photomicrographs, A and B of figure 5. The dendritic structure practically always occurred at an unmeasured depth in those surfaces exposed to the furnace atmosphere, and the mosaic structure usually constituted the remainder of the thin-section specimen. The photomicrographs also give an indication of the relative difficulty in distinguishing small or large crystals of either magnesia or beryllia without polarized light.

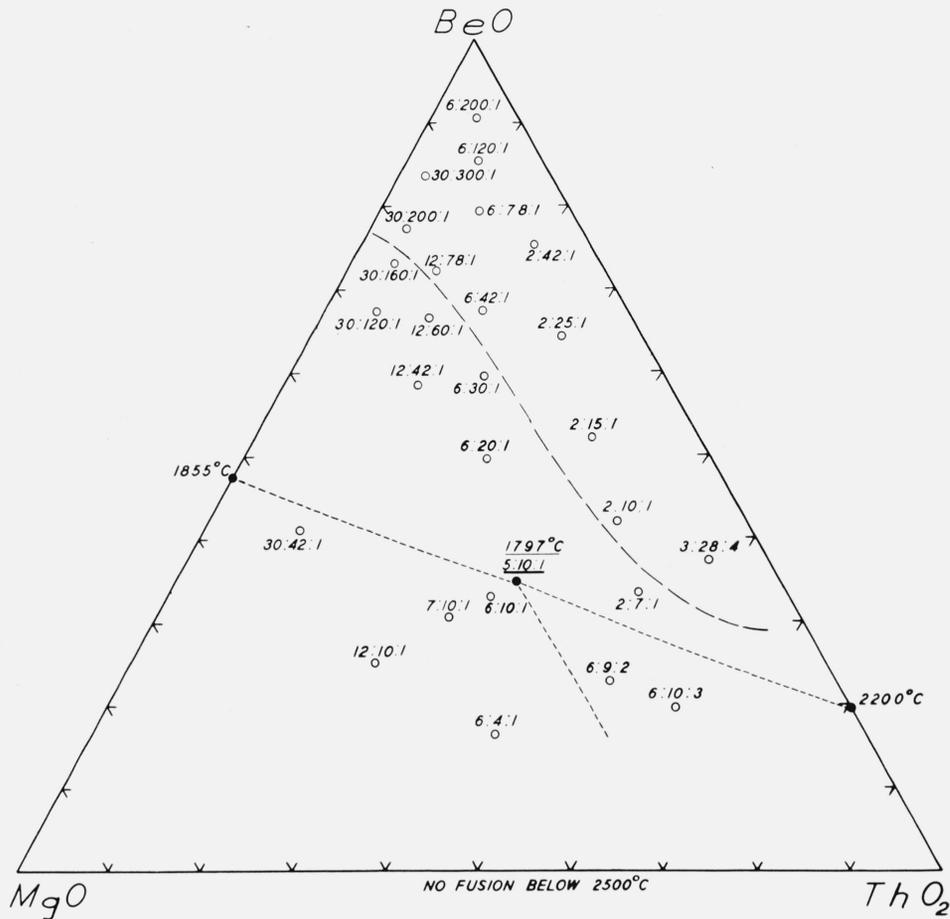
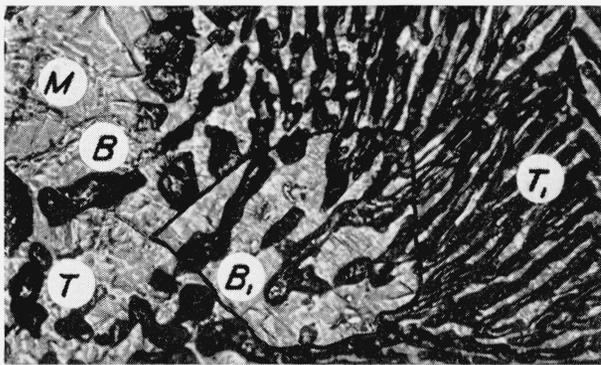
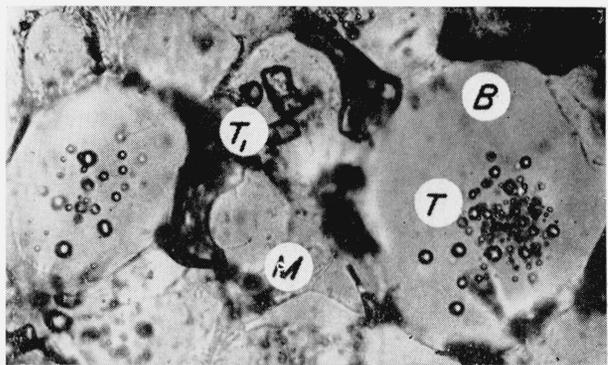


FIGURE 4. The system MgO-BeO-ThO₂.

This figure gives the location of the primary phase boundaries (short-dash lines), a suggested area of solid solution (long-dash lines enclosing the BeO apex area), and the range of compositions from which impervious bodies may be made (similar to the solid solution area). Compositions are indicated on the figure in molar ratios but are graphically placed according to weight percentages. Eight additional mixtures, not reported, were investigated in the eutectic area.



A



B

FIGURE 5. Thin-section photomicrographs of two fused compositions of the system MgO-BeO-ThO₂.

Photomicrograph A of mole composition 6MgO:9BeO:2ThO₂, ×300, Test No. 635—1,818° C, shows typical thoria crystals at T, the high relief dendritic thoria at T₁, a typical beryllia crystal at B, the dendritic thoria inclusion structure in a large beryllia crystal at B₁ (outlined), and the typical appearance of magnesia at M. In photomicrograph B of mole composition 6MgO:42BeO:1ThO₂, ×340, Test No. 657—1,916° C, the high relief material T is a typical thoria exsolution texture from beryllia (the large, almost clear round crystal B). Shown also are a magnesia crystal M and a few high relief thoria crystals T₁.

Because of the apparent highly viscous nature of compositions in this system at high temperatures, there still remains considerable doubt whether the ternary eutectic may be located more accurately than in the area (fig. 4) bounded by the compositions 7M:10B:1T, 5M:10B:1T, and 6M:9B:2T. The fusion-point and softening-range tests, however, indicate it to be near mole composition 5M:10B:1T and $1,797^{\circ} \pm 5^{\circ} \text{C}$.

An area of possible solid solution is suggested for this system to be bounded by and to include compositions 12M:78B:1T, 6M:42B:1T, 2M:10B:1T, and 3M:28B:4T. This area is shown on the diagram enclosed by a long-dash line. The X-ray examinations failed to reveal any shift in the *d*-spacing values, and the petrographic examinations failed to reveal any differences in refractive index values of the component oxides, although a definite exsolution texture (photomicrograph *B* of fig. 5) was noted in all compositions within the area. Examination of the softening-range observations (not all of which are included in table 6) shows that the liquidus temperatures of the compositions located in the central portion of the diagram are between $1,814^{\circ}$ and $1,840^{\circ} \text{C}$ and that, at the proposed solid solution boundary, there occurs a large increase in these temperatures. Within the solid solution area, the mixtures of the compositions 12M:78B:1T, 6M:42B:1T, 2M:15B:1T, 2M:10B:1T, and 3M:28B:4T show only very limited melting in the temperature range from $1,870^{\circ}$ to $1,915^{\circ} \text{C}$, whereas those mixtures just outside of the area, 30M:120B:1T, 12M:42B:1T, 6M:30B:1T, 6M:20B:1T, and 2M:7B:1T, are completely melted in the temperature range from $1,812^{\circ}$ to $1,842^{\circ} \text{C}$.

It is believed that the very viscous nature at high temperatures of the mixtures in this region has obscured to an appreciable extent the expected increase in the solidus temperatures associated with the solid solution phenomena. All of the available data seems to indicate that the suggested solid solution area is one that exists only at high temperatures, and that the solid solution is not present after slow cooling, which apparently destroys all evidence of the existence in the specimens of the high-temperature form.

It is possible to produce impervious porcelains from compositions in approximately one-half of the beryllia field of this system. These bodies, whose compositions are confined to the solid solution area, could usually be matured between $1,500^{\circ}$ and $1,750^{\circ} \text{C}$. Table 7 gives the results of the maturing range studies and the values of absorption and shrinkage at maximum density. Those porcelains that could be matured to a practically impervious condition over a range of 50°C or more have shrinkage values ranging from 18 to 21 percent and absorption values averaging about 0.02 percent.

Compressive strength values (table 7) ranged from 165,000 to 265,000 lb/in.² for these bodies. The outstanding porcelain from the standpoint of compressive strength was 3M:28B:4T (265,700 lb/in.²), but its maturing range is relatively short ($1,600^{\circ}$ to $1,650^{\circ} \text{C}$) and its density (4.91g/cm^3) was the highest observed.

It had been found previously [5] that thoria-containing bodies were extremely brittle and usually possessed rather poor heat-shock resistance. For practical purposes it was, therefore, considered sufficient to determine the relative thermal-shock resistance and the strength in bending at room and at elevated temperatures for only a few body compositions selected to represent the maturable area of the system.

As had been expected, none of the bodies tested (table 8) were satisfactory in their resistance to thermal shocking (all were either cracked or could easily be broken after testing) nor could their strengths in bending at room temperature ($9,000$ to $14,000 \text{lb/in.}^2$) be considered as more than fair to poor. Modulus of rupture and modulus of elasticity (Young's modulus) values in bending at $1,800^{\circ} \text{F}$ (982°C), ranged from $13,000$ to $16,000 \text{lb/in.}^2$ and from $40,000,000$ to $32,000,000 \text{lb/in.}^2$, respectively. The higher values for the tests at $1,800^{\circ} \text{F}$ as compared to those obtained for the room-temperature tests may have been due to the incomplete maturing of all but one of the test bars. The values obtained for body composition 2M:42B:1T, which was completely matured, were slightly lower for the $1,800^{\circ} \text{F}$ test.

TABLE 7. *Maturing range studies and compressive strength results of some compositions in the system MgO-BeO-ThO₂*

Composition						Maturing ^a b range	Properties ^b			Properties of compressive strength specimens ^{b c}							
Mole			Weight				Test No.	Temperature	Shrinkage	Absorption	Test No.	Temperature	Shrinkage	Absorption	Density	Height/diameter ^d	Strength ^e at room temperature (±5,000 lb/in. ²)
M	B	Z	MgO	BeO	ZrO ₂												
			%	%	%	° C		° C	%	%	° C	%	%	g/cm ³		n×10 ³ lb/in. ²	
2	10	1	13.56	42.05	44.39	1,650 to 1,750	323-L	1,700	18.7	0.02	626-L	1,700	18.47	0.05	4.42	1.91	203
2	15	1	11.20	52.12	36.68	1,600 to 1,750	329-L	1,750	19.8	.00	618-L	1,750	18.25	.06	3.96	1.96	187
2	25	1	8.31	64.47	27.22	1,500 to 1,700	487-L	1,600	19.46	.01	610-L	1,625	19.10	.01	3.66	1.97	253
2	42	1	5.78	75.30	18.92	1,500 to 1,650	584-L	1,600	19.36	.02	616-L	1,625	19.42	.01	3.40	1.94	185
3	28	4	6.44	37.31	56.25	1,600 to 1,650	585-L	1,650	17.28	.01	611-L	1,675	17.99	.01	4.91	1.88	266
6	42	1	15.54	67.50	16.96	1,550 to 1,700	487-L	1,600	19.30	.01	609-L	1,625	19.3	.01	3.43	2.00	230
6	78	1	9.84	79.41	10.75	1,550 to 1,750	323-L	1,700	21.1	.02	613-L	1,700	19.90	.01	3.25	1.97	250
6	120	1	6.89	85.58	7.53	1,650 to 1,750	-----	1,700 to 1,750	20.6	.00	615-L	1,725	19.6	.01	3.15	1.99	235
6	200	1	4.39	90.82	4.79	1,450 to 1,700	575-L	1,600	19.20	.01	619-L	1,625	19.8	.01	3.03	1.99	251
12	78	1	17.93	72.29	9.78	1,550 to 1,700	578-L	1,600	17.92	.02	-----	-----	-----	-----	-----	-----	-----
30	200	1	18.67	77.25	4.08	1,550 to 1,700	578-L	1,600	18.08	.02	614-L	1,625	19.5	.03	3.10	1.95	166
30	300	1	13.47	83.59	2.94	1,500 to 1,700	571-L	1,650	18.40	.01	612-L	1,675	19.8	.01	3.07	1.93	242

^a Overfiring and underfiring occurred at 50 deg C above and 50 deg C below the temperature ranges given.

^b Values obtained for specimens matured in an electric furnace for one hour at the temperature given.

^c The values given are the averages for three test specimens.

^d Specimens about 1 in. high by ½ in. in diameter.

^e Compression testing was done at a constant loading rate of 31,250 lb/in.² per min until failure, using cold-rolled steel contact blocks.

TABLE 8. *Resistance to thermal shock and rupture and elastic strength values of some compositions in the system MgO-BeO-ThO₂*

Composition						Maturing data ^a			Resistance to thermal shock ^b (2 bars each composition)	Strength in bending values ^c			
Mole			Weight			Test No.	Temperature	Absorption		M/R at room temperature ^d (±2,000 lb/in. ²)	M/R at room temperature after shocking (±2,000 lb/in. ²)	M/R at 1,800° F ^e (±3,000 lb/in. ²)	Young's Modulus at 1,800° F ^e (±3,000,000 lb/in. ²)
M	B	T	MgO	BeO	ThO ₂								
2	15	1	%	%	%	21-R	1,800	0.40	Cracked on 2d cycle.....	n×10 ³ lb/in. ²	n×10 ³ lb/in. ²	n×10 ³ lb/in. ²	n×10 ⁶ lb/in. ²
2	42	1	11.20	52.12	36.68	23-R	1,700	.07	Cracked on 3rd cycle.....	11.7	-----	15.6	31.9
6	78	1	5.78	75.30	18.92	---	Cone 31	≅ 5.9	-----	-----	≅ 17.3	≅ 15.4	
6	120	1	9.84	79.41	10.75	21-R	1,800	.54	Cracked on 5th cycle.....	9.1	-----	15.0	40.0
6	120	1	6.89	85.58	7.53	---	1,600	≅ 6.0	Survived 10 cycles from 1,500° F to still air.≅	-----	f ≅ 11.0	-----	-----
30	200	1	18.67	77.25	4.08	23-R	1,700	.79	Cracked on 5th cycle.....	12.6	-----	14.8	36.7

^a All specimens were matured in a gas-fired furnace at the temperature indicated for 1 hour.

^b Ten cycles of test from 1,700° F to room temperature air blast for two test specimens, about 5½ in. by ¾ in. by ¼ in., of each composition.

^c M/R is the modulus of rupture.

^d Two specimens, about 5½ in. by ¾ in. by ¼ in., of each composition were tested.

^e Three specimens, about 5½ in. by ¾ in. by ¼ in., of each composition were tested unless otherwise specified.

^f See column on resistance to thermal shock.

≅ For preliminary report see reference [2].

^h One specimen tested.

VI. Conclusions

No outstanding porcelain-type bodies were found in either of the systems studied, which combine all of the desirable high-temperature properties required for their use as components of heat

engines, although a number of bodies are now available for various high-strength and high-temperature services where thermal-shock conditions are not of prime importance.

The limited study of the phase equilibrium relations suggests that the system MgO-BeO-ZrO₂

be considered as a simple one with the ternary eutectic at composition $4\text{MgO}:5\text{BeO}-3\text{ZrO}_2$ and $1,669^\circ \pm 3^\circ \text{C}$. No evidence of binary or ternary compound formation was observed, and an area of solid solution is suggested to extend along the $\text{BeO}-\text{ZrO}_2$ join. Ceramic bodies in this system may be matured at temperatures ranging from $1,450^\circ$ to $1,600^\circ \text{C}$ in approximately one-half of the ternary diagram area. The values of the physical properties of these bodies are summarized in table 9.

The summary table of these physical properties is included in order that the general properties of these porcelain bodies may be readily compared with the properties of other refractory bodies, such as are given in table 1.

TABLE 9. Summary table of the general physical properties of some matured two- and three-oxide porcelain bodies^a

Physical properties	Two-oxide bodies	Three-oxide bodies	
	$\text{MgO}-\text{ZrO}_2$ ^f	$\text{MgO}-\text{BeO}-\text{ZrO}_2$	$\text{MgO}-\text{BeO}-\text{ThO}_2$
Maturing range, °C.	1,700 to 1,800	1,450 to 1,600	1,500 to 1,750
Density, g/cm ³	4 to 5.5	2.9 to 4.8	3.0 to 4.9
Shrinkage, percent.....	12.5 to 17.9	15.2 to 20.6	17.2 to 21.1
Compressive strength ^b at room temperature, $n \times 10^3$ lb/in. ²	166 to 229	163 to 263	166 to 266
Modulus of rupture at room temperature, $n \times 10^3$ lb/in. ²	-----	13 to 39	e 9 to 14
Modulus of rupture at 1,800° F (982° C), $n \times 10^3$ lb/in. ²	29	13 to 32	e 13 to 16
Modulus of elasticity at 1,800° F (982° C), $n \times 10^6$ lb/in. ²	31 to 23	38 to 31	e 40 to 32
Thermal shock resistance. ^c	-----	Fair to Good	Poor
Modulus of rupture at room temperature after heat shocking, ^d $n \times 10^3$ lb/in. ²	-----	13 to 23	-----

^a Matured bodies are those having less than 0.1 percent water absorption.

^b Crushed at a length-to-diameter ratio of 2 between cold-rolled steel blocks, $\frac{1}{2}$ in. thick, at about 31,000 lb/in.² per min.

^c Relative values of 10 cycle test, $1,700^\circ \text{F} \rightleftharpoons$ room temperature air blast. Failure before five cycles is considered as being poor.

^d Tests made only on those specimens surviving 10 cycles of thermal shocking without cracking or spalling.

^e Some test bars were not completely matured.

^f Data based on a very limited number of tests.

Some limited data were determined for a few porcelains in the binary system $\text{MgO}-\text{ZrO}_2$, and these are included in the summary table.

The phase relations and the location of the primary phase boundaries in the system $\text{MgO}-\text{BeO}-\text{ThO}_2$ are similar to those in the previous system, although this is a distinctly high-temperature one with the ternary eutectic at about mole composition $5\text{MgO}:10\text{BeO}:1\text{ThO}_2$ and $1,797^\circ \pm 5^\circ \text{C}$. No evidence of compound formation was observed; and an area of solid solution is suggested, bounded by an S-shaped curve extending from about 25 percent of MgO on the $\text{MgO}-\text{BeO}$ join along the line of 20 percent of MgO to about 70 percent of ThO_2 on the $\text{BeO}-\text{ThO}_2$ join. Ceramic bodies in this system may be matured at temperatures ranging from $1,500^\circ$ to $1,750^\circ \text{C}$ in approximately one-half of the beryllia field of the system bounded by the same line describing the suggested area of solid solution. The range of values determined for the various physical properties of these matured bodies is summarized in table 9.

It is of interest to note that the maturing ranges of the porcelains of each of the systems are such that all of those studied in the system $\text{MgO}-\text{BeO}-\text{ZrO}_2$, some of those studied in the system $\text{MgO}-\text{BeO}-\text{ThO}_2$, and none of those studied in the system $\text{MgO}-\text{ZrO}_2$ can be matured in the commercial kilns currently being used in the porcelain industry.

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