H. A. Huisken

Clay Crucibles for High Temperature Melting

CLAY CRUCIBLES FOR HIGH TEMPERATURE MELTING

BY

HARRY ARNOLD HUISKEN

THESIS

FOR THE

DEGREE OF BACHELOR OF SCIENCE

IN

CERAMIC ENGINEERING

COLLEGE OF ENGINEERING

UNIVERSITY OF ILLINOIS

1920

UNIVERSITY OF ILLINOIS

May 29th 1920

THIS IS TO CERTIFY THAT THE THESIS PREPARED UNDER MY SUPERVISION BY

Harry Arnold Huisken

ENTITLED Clay Crucibles for High Temperature Melting.

IS APPROVED BY ME AS FULFILLING THIS PART OF THE REQUIREMENTS FOR THE

DEGREE OF Bachelor of Science

in Ceramic Engineering.

Ralph K. Hursh
Instructor in Charge

APPROVED: C. W. Washburn

HEAD OF DEPARTMENT OF Ceramic Engineering

CLAY CRUCIBLES FOR HIGH TEMPERATURE MELTING

BY

HARRY ARNOLD HUISKEN

THESIS

FOR THE

DEGREE OF BACHELOR OF SCIENCE

IN

CERAMIC ENGINEERING

COLLEGE OF ENGINEERING

UNIVERSITY OF ILLINOIS

1920

TABLE OF CONTENTS.

--o--

	Page
1. Introduction	1.
2. Requirements of Foundry Crucibles	3.
3. Materials Used in Crucible Mixtures	5.
Bond Clays	5.
Grog	7.
4. Methods of Testing Materials and Mixtures	8.
5. Investigation of Bond Clays	10.
Clays Used	10.
Preparation and Burning	11.
Porosity and Shrinkage Curves	13- 17.
Results of Tests on Bond Clays	18.
6. Investigation of Crucible Mixtures	19.
Size of Grog	19.
Preparation of Mixtures	21.
Testing of Pieces	21.
Sag Test	21.
Results of Load Test	23.
Quenching Test	24.
Results of Quenching Test	25.
Results of Test on Mixtures	26.
7. Test on Crucibles	27.
Preparation and Burning	27.
Slag Test	28.
8. Conclusions	29.

[illegible]

CLAY CRUCIBLES FOR HIGH TEMPERATURE MELTING.

PART 1.

INTRODUCTION

--o--

At present, a large proportion of the metallurgical industries is dependent upon the production of a high grade melting crucible. This is especially true of that part of the industry producing alloys, such as bronzes, brass, and steels alloyed with tungsten, molybdenum, etc. This latter product has greatly developed in recent years due to the great demand for high speed tools and special machine parts required to withstand severe stresses. Although some of these alloys are produced in special melting furnaces, the crucible method, with fireclay ware made in all sizes is the favorite.[1] The great expansion of the metallurgical industry and the greater demand for better quality of alloys requires a corresponding development upon the crucible industry.

At the present time, however, owing to the abnormal conditions, the demand for crucibles is so great that in many cases, even inferior ware is acceptable to the metallurgists, if it can be used in their work. The result is that many crucibles are produced which are of short life and give far from satisfactory service. This, however, is not solely due to the desire of the manufacturers to produce anything which will sell, but due often to ignorance on their part of the qualities of the materials necessary to produce a high grade ware.

The present supply of high grade crucible clays is limited, probably not due so much to the scarcity of crucible clay resources as to the lack of information about deposits and their properties. It is evident that much profitable investigation could be carried out along these lines. Information pertaining to crucible mixtures and the testing of such mixtures is, at present, decidedly inadequate.

There has been some work done in the past in the subject of crucible comp-

[1] Havard - Refractories and Furnaces - Page 231

[illegible]

[illegible]

[illegible]

ositions but this pertains principally to the mixtures themselves, such as graphite crucible mixtures, zirconia crucibles, alumina mixtures, etc. Some work has been done upon the clays used as a bond in the ware, but in this also the data is very limited.

It was the purpose of the present investigation to develop a mixture or mixtures for the production of a clay crucible, suitable for the melting of alloys at high temperatures, and one which could meet, in service and in cost, the competition of the graphite crucibles now on the market. In connection with this work it was necessary, by experimental tests, to determine the comparative value of various refractory clays as bonding materials, the relative effects of different proportions of bond clay and non-plastic and the effect of grog of various sizing on the ability of the body to withstand the conditions of use of such crucibles.

----o----

PART 2.

Requirements of Foundry Crucibles:-

The conditions to which crucibles are subjected govern the requirements they must possess. In general, the crucible containing the charge is placed in a bed of coke, and heated to a temperature of 1500°C, or more. After proper heat treatment, the crucible is removed from the furnace with tongs and the metal poured. A fresh charge is then put in the crucible and the operation is repeated. It is evident from the above operation that stresses are set up in the crucible due to heating and cooling, together with handling at the furnace door. Presumably the crucible is not completely cooled between melts, but in the foundry much of the failure is due to improper and careless handling by the men. The crucibles are often thrown aside after pouring, instead of being returned to the furnace. Under the best conditions, the service is severe, but in ordinary practice, it is extremely so.

In many metallurgical processes, the temperatures used are extremely high, and the crucible must, primarily, be of a very refractory quality. Then, due to the fact that it is removed from the furnace at this temperature to atmospheric conditions, it must have a low coefficient of expansion to resist the stresses set up by sudden cooling. This is made more severe by the addition of a fresh charge of metal, which is comparatively cold.

In some melting operations, fluxes or slags are employed which have a tendency to attack the inside of the crucible by mechanical action and chemical combination. This corrosive action should be reduced to a minimum by making the inner surface as dense as possible. The formation of minute cracks, usually resulting from sudden temperature changes, facilitates corrosion, giving the slag a chance to work its way into the body and this causes many crucibles to

[illegible]

fail after the first or second heating.

The metal charge in the crucible, usually of considerable weight, subjects the body to an appreciable load at the high furnace temperatures. The gripping of the tongs in handling the crucible from the furnace has an intense crushing action. These conditions, together with the stresses set up on sudden cooling and heating of the crucible require that the body possess considerable mechanical strength, both at ordinary temperatures and at high furnace temperatures.

From a purely economic standpoint, the fireclay crucible must be manufactured cheaply enough to compete with the graphite crucible now on the market. On a basis of cost per melt, the clay crucible would have to be as cheap or cheaper than the graphite crucible now being used extensively and, at the same time, give as satisfactory service.

The ware must stand nesting, crating and transportation without much loss due to breakage.

In brief, a crucible should have the following properties:-

1. It must be refractory enough to stand a temperature of 1500°C.
2. It must be of sufficient density to resist the action of corrosive slags.
3. The crucible must be of sufficient strength to hold a full charge, and withstand the gripping of tongs, and rough handling at high temperatures.
4. It must withstand sudden temperature changes without excessive cracking or loss in strength.
5. The cost per melt must be equal to or less than that of the graphite crucibles.
6. The crucibles should be strong enough to bear packing and transportation.

Any crucible which satisfies these conditions would be suitable for average

[illegible]

foundry work and should give good results.

PART 3.

Materials used in Crucible Mixtures:-

The materials used in crucible bodies vary considerably with the use to which the crucible is subjected. In general, crucible bodies consist of the plastic or bond material, and the non-plastic or grog portion. In clay crucibles, the bond material is usually a high grade, plastic refractory clay, and the grog consists of ground fire brick of the highest quality or calcined flint clay. In other crucibles, such as alumina, magnesia, zirconia crucibles, etc., the plastic is usually added in the form of a starch, tar or any colloidal organic substance, which holds the non-plastic particles together in the desired shape until it is burned. In the firing, the temporary bond is burned out and the body sinters together by reason of the small amount of fluxes present.

Bond Clays:-[2]

Of the bond clays employed in crucible manufacture, the Klingenberg clay, found in Bavaria, Germany is the most widely known. It possesses excellent plasticity and bonding strength. In addition, it burns to a dense body at 1125°C, with no evidence of softening at 1400°C. The English Stourbridge Clay is also noted for its desirable properties for crucible manufacture. No American clays have been found equal to either of these, but Bleininger[3] advocates the use of a mixture of clays to get the desired effect. Booze[4] found the LaClede-Christy B-291 Clay as being the most suitable clay for graphite crucible manufacture of those which he tested.

Bleininger and Loomis[5] in their work on American Bond Clays, roughly classify

[2]Bleininger - Notes on the Crucible Situation - Jr.In.of Metals -Page 345.
[3]Bleininger - Notes on the Crucible Situation - Jr.In. of Metals-Page 352.
[4]Booze - Some Properties of Bond Clays for Graphite Crucibles-
Jr. Amer. Cer. Society - Page 472.
[5]Bleininger & Loomis, Prop.of Some Amer.Bond Clays-Tr.Am.Cer.Soc.Vol 2-p605

[illegible]

[illegible]

[illegible]

[illegible]

[illegible]

[illegible]

[illegible]

[illegible]

[illegible]

[illegible]

the clays according to their behavior in the raw state and in burning, as follows. In the raw state with a 1:1 sand-clay mixture, a bond clay of class "A" should show a modulus of rupture of 325 lbs. per sq. inch, or more, and those of 225 to 325 lbs. per sq. inch, should be placed in class "B". The ratio of pore to shrinkage water should in no case exceed 1:00 for a class "A" clay. Under burning properties, they divide the clays into three classes: first, those burning dense at 1150°C and showing no evidence of overfiring at 1400°C, as particularly suitable for brass foundry graphite crucibles, provided they possess the other physical properties: second, those burning dense around 1275°C, and not overfiring at 1400°C or higher, as suitable for glass refractories or crucibles for steel melting: third, clays which possess good strength but do not vitrify at low temperatures, becoming dense at 1425°C or higher, are suitable for the glass industry. The unsuitable clays, are those which become dense between 1150°C and 1300°C but possess only a short range between vitrification and the temperature of overfiring. Havard[6] states that clays of the type that become glassy and glazed at high temperatures are unsuitable because of their inability to stand sudden temperature changes, even when mixed with grog. Booze[4] suggests that the structure should be dense at low temperatures, but not completely non-porous so that the body can withstand sudden temperature changes.

The character of the grog used in crucible mixtures is varied and often determines the nature of the bond clay. For instance, graphite demands that the bond clay burn to a dense structure at a low temperature. This is necessary to prevent the graphite from burning out, during firing. The grog used in ordinary fire clay crucibles does not demand this, but, as stated previously, the bond clay should burn to a dense structure at the firing temperature of the ware.

In general, a bond clay of first quality should possess the following

[6] Havard - Refractories and Furnaces - Page 231.
[4] Booze - loc.cit. - page 5.

[illegible]

[illegible]

[illegible]

properties:-

1. A high bonding strength, preferably a modulus of rupture of at least 325 lbs. per sq. inch when mixed with sand in the ratio of 1:1.
2. Must not have excessive shrinkage.
3. Should burn to dense structure around 1200°C.
4. Should have a long vitrification range.
5. Should not show evidence of overburning below 1400°C, and should not have a softening point below Cone 30.
6. Must produce a body of great mechanical strength.
7. Must be able to withstand sudden temperature changes.

Grog:-

The amount and size of grog is an important feature in determining the strength of the ware in the green and burned states. Its behavior under fire is also important, as it affects the porosity to a marked degree. The size and nature of the grog also determines, to some extent, to what degree the ware can withstand sudden temperature changes.

Kirkpatrick[7] found the strongest raw bodies were those having the following size of grog:

20 - 40 mesh	-	25-66.6%
40 - 80 mesh	-	0-25%
80 - Dust	-	33.3-66.6%

In some cases the strongest bodies in the green state were those having a maximum of 80 to dust grog. He further recommends that comparatively large sizes of grog are necessary in case the refractory must stand sudden temperature changes. For greater strength in the burned state, fine grog is desirable. In general, the size of the grog should not be uniform but should be proportioned from coarse to fine. It should also be calcined so that the shrinkage of the body is reduced to a minimum. The grog should, of course, be refractory enough

[7]Kirkpatrick - Effect of Size of Grog in Fireclay Bodies-B.of S. Tech.Paper 104

to stand the temperature at which the crucible is to be used without any signs of deformation or softening. In this respect the grog should be even more refractory than the bond clay, since it forms the skeleton of the body.

Briefly, the grog used in crucible mixtures should be:-

1. Very refractory, to give the ware strength at high temperatures.
2. Grog should be calcined.
3. Should not contain fluxes, which would tend to reduce the refractoriness.
4. Grog should vary in size to give strength and, at the same time, decrease the coefficient of expansion of the body.
5. Grog sizes should be of such nature as to give minimum porosity permissible.

PART 4.

Methods of Testing Materials and Mixtures:-

Although there are no standard methods of testing materials of this type, the general outline followed by other investigators offers an excellent guide, and served as a basis for planning the present work. Probably the most complete work done in regard to the testing of bond clays was that by Bleininger and Loomis[5]. In their investigation the water of plasticity and the pore and shrinkage water were determined: these with the volume shrinkage in drying, and the time for slaking of a clay-flint mixture were used in the comparison of the plastic properties of the clays. The modulus of rupture of the clay alone and of clay-sand mixtures in 1:1 ratio was used as a comparison of the strength and bonding properties in the dry state. The burning test consisted of of firing at a rate of 20° above 800°C and drawing trials, beginning at 1050°C. The temperature-porosity and temperature-volume shrinkage relations were obtained and the temperature of overfiring noted. The softening temperature was also determined.

[5] Bleininger and Loomis - loc. cit. page 5.

Booze[4] in his investigation on graphite crucible mixtures, followed the general outline of the work done by Bleininger and Loomis, so far as it related to the examination of bond clays and also tested the crucible mixes. Although graphite acts differently than other grogs the general methods are applicable in the case of an ordinary grog. The cross breaking strength of the mixes burned to 1150°C and 1425°C was determined after which the trial pieces were subjected to a quenching test. In this, the fired trials were heated to 800°C and plunged into water. This was repeated five times and the modulus of rupture was then determined. This test gave an excellent comparison of the ability of the various mixtures to withstand sudden temperature changes. Kirkpatrick[7] employed quenching tests at 600°C and 1000°C in which he subjected the trial pieces to as many treatments as necessary to cause complete failure of the pieces.

The ability of a refractory to stand up under a definite load at a high temperature is very important in many instances, especially in the case of crucibles. In previous investigations carried out in crucible mixtures this has apparently been neglected. Load tests[8] on fire brick during heating have been made, giving conclusive results as to a basis of comparison. Warpage tests have been used as a standard method in testing whiteware bodies. A test similar to the above, in which trials of definite length, breadth and thickness, supported at one or both ends, so as to subject the trial to a definite stress should be of value for testing crucible mixtures. This test was adopted in the present work, to compare the strength under load at high temperatures of the mixtures used.

Some work has been done on the testing of the finished ware. Orton Jr. and Henderson[9] tested the corrosive effect of assay slags upon commercial and experimental crucibles. In their work, assay slags were heated in the crucibles to a

[4]Booze - loc. cit. Page 5
[7]Kirkpatrick - loc. cit. Page 7
[8]Bleininger and Brown - Testing of Clay Refractories. Tech. Pap.7-B.of S.
[9]Orton Jr. and Henderson - Tests on Assay Crucibles - Trans. Am. Cer. Soc. Volume 10 - 1908 - Page 484

temperature of 1150°C for thirty minutes. The loss in weight of the crucible was determined after each treatment. Stull[10] tested a number of graphite crucibles in the foundry proper, in which the life of the crucibles were noted when used in melting various alloys and metals. It is highly desirable to make such tests upon finished crucibles under conditions which are similar to those to which they must be subjected in actual foundry practice, in order to determine exactly how they act.

On the whole, the investigation carried out on refractory mixes and clays would seem to be rather abundant, but on close observation it seems that the field is so broad that only a relatively small amount has been accomplished. From these investigations, however, a suitable plan could be obtained for this work, by selecting the desirable tests and methods of making such tests from what has been done by other investigators. Until some standard tests are devised, these will suffice for similar work done in the future.

PART 5.

DESCRIPTION OF TESTS ON BOND CLAYS.

Clays Used:-

The following high grade domestic refractory clays and mixtures were selected for the tests on bond clays:-

1. Hickory Ball Clay.
2. Hickory Ball Clay and 10% raw flint clay.
3. Hickory Ball Clay and 20% raw flint clay.
4. Highland Fire Clay.
5. Highland Fire Clay and 10% raw flint clay.
6. Highland Fire Clay and 20% raw flint clay.
7. LaClede-Christy Diaspore Clay.
8. Southern Illinois Bond Clay (Illinois Kaolin Co.)
9. Kentucky Ball Clay - #8400

[10] Stull-Behavior under Brass Foundry practice of Crucible containing Graphite - Jr.Amer.Cer.Soc. Vol.2.-Mar.1919.

Preparation and Burning:-

In preparing the mixtures, the flint clay was ground to pass a 20-mesh screen, the fire clay added in the above proportions by weight, and the mixes were ground for one hour in porcelain lined ball mills with the same charge of pebbles in each grinding. The single clays were simply put thru a jaw-crusher and rolls. Sufficient water was then added to give the best working consistency, the clay was thoroughly pugged and ten 1"x1"x4" trial pieces were made from each batch.

The trial pieces were dried at room temperature for two days, then for 12 hours at 100°C. They were then allowed to stand for 18 hours in kerosene and the volumes were determined by means of a volumeter of the Seger type. They were dried thoroughly at room temperature, then for 12 hours at a temperature of 100°C. The test pieces were then burned in a small gas-fired test kiln at the rate of 35° per hour, draws being made every 25° between the temperatures of 1200°C and 1500°C. The changes within this temperature range were those thought really necessary for the work in question. On removal from the furnace, the pieces were covered with quartz sand to avoid shattering, due to sudden cooling, but even with this precaution, a few of the trials were found unfit for further test, either shattering in the furnace or after drawing.

After cooling, the pieces were weighed, and then boiled for six hours in water, and the volumes again determined. The percent loss in volume in terms of dry volume was calculated for each piece by the following formula:-

$$\frac{V_1 - V_2}{V_1} \times 100 = \text{percent loss in volume.}$$

V_1 = original volume.
V_2 = volume after burning.

The surface was removed by means of a damp cloth, the trials were again weighed, and the percent porosity for each piece was determined by the following formula:-

$$\frac{W_d - W_w}{V_1} \times 100 = \text{percent porosity.}$$

W_d = dry weight.

W_w = wet weight.

V_1 = volume after burning.

Curves were then plotted showing the relation between percent loss in volume and temperature, and percent porosity and temperature.

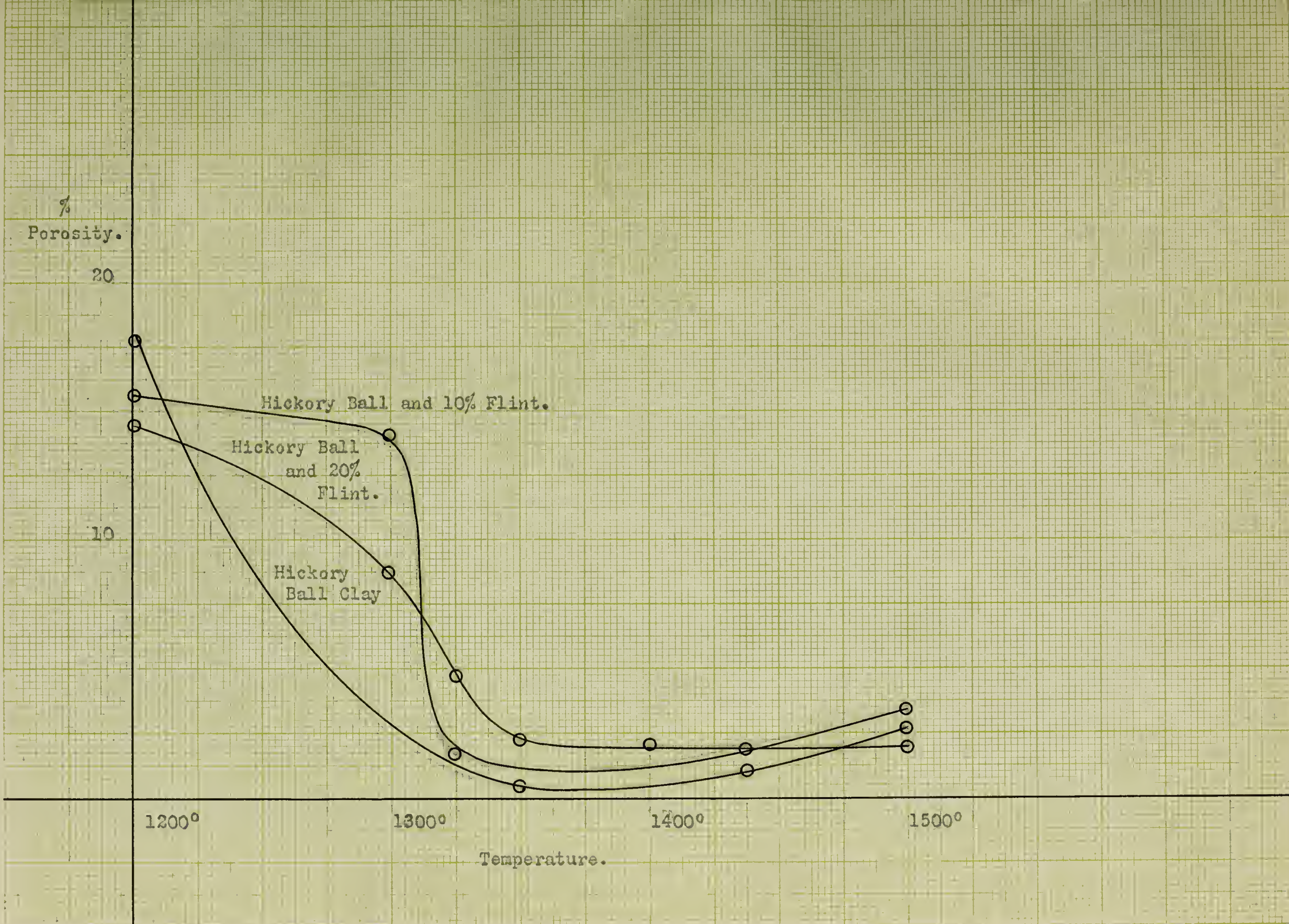

%
Porosity.
20
10
Hickory Ball and 10% Flint.
Hickory Ball
and 20%
Flint.
Hickory
Ball Clay
1200°
1300°
1400°
1500°
Temperature.

Fig. 1.

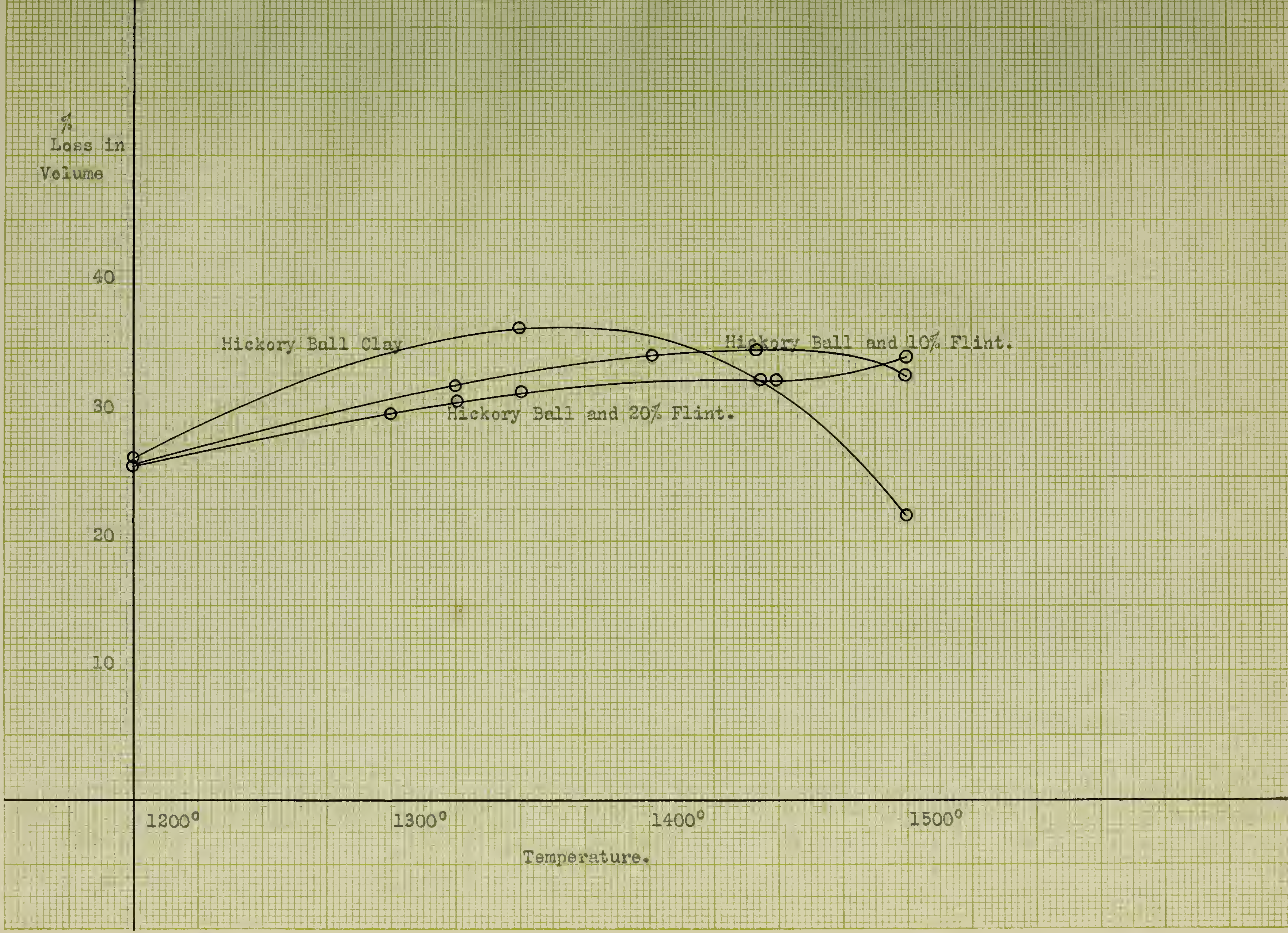

% Loss in Volume
40
30
20
10
Hickory Ball Clay
Hickory Ball and 10% Flint.
Hickory Ball and 20% Flint.
1200°
1300°
1400°
1500°
Temperature.

Fig. 2.

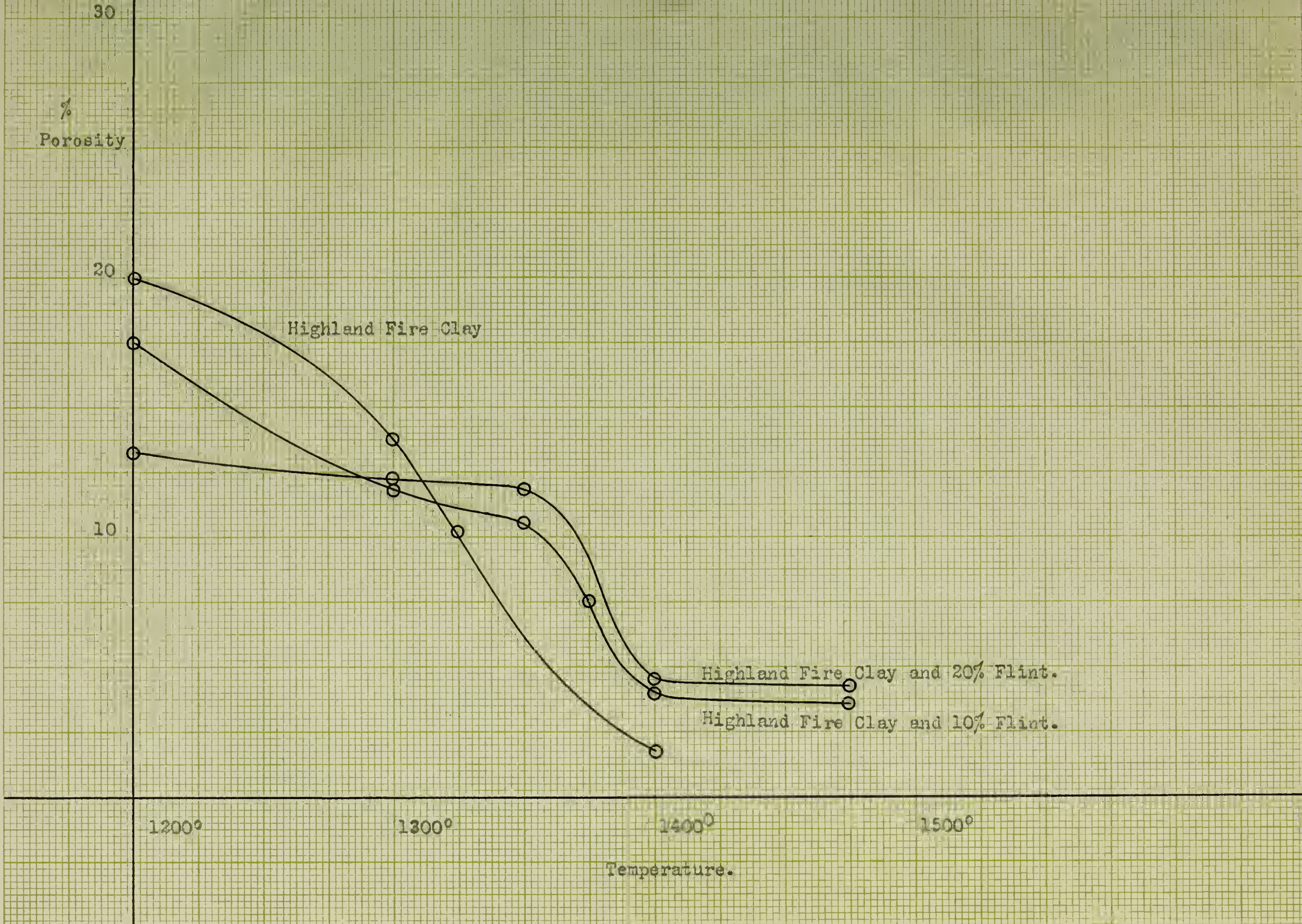
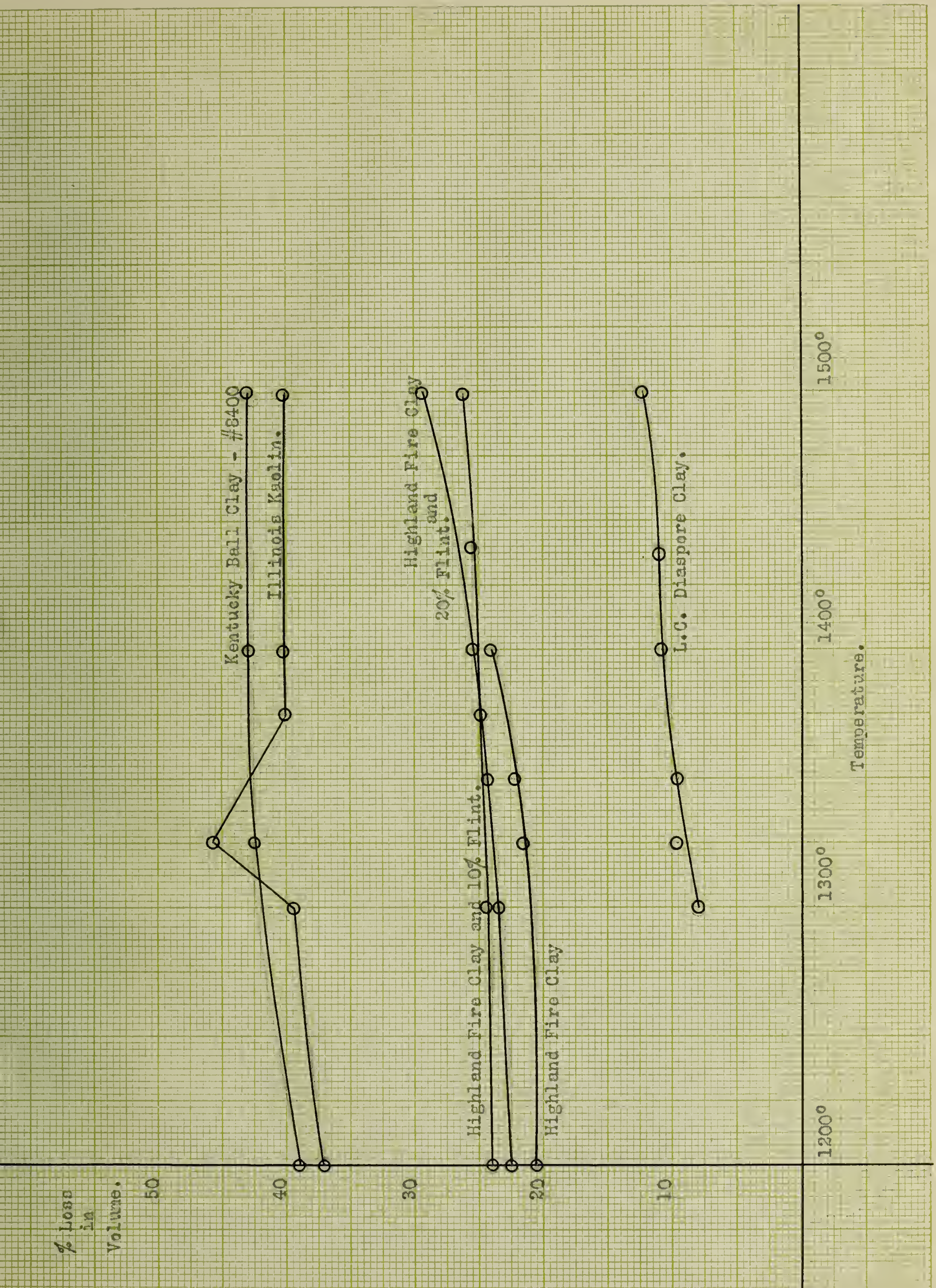

30
20
10
%
Porosity
Highland Fire Clay
Highland Fire Clay and 20% Flint.
Highland Fire Clay and 10% Flint.
1200°
1300°
1400°
1500°
Temperature.

Fig. 3.



% Loss in Volume.
50
40
30
20
10
1200°
1300°
1400°
1500°
Temperature.
Kentucky Ball Clay - #6400
Illinois Kaolin.
Highland Fire Clay and 20% Flint.
Highland Fire Clay and 10% Flint.
Highland Fire Clay
L.C. Diaspore Clay.

Fig. 4.

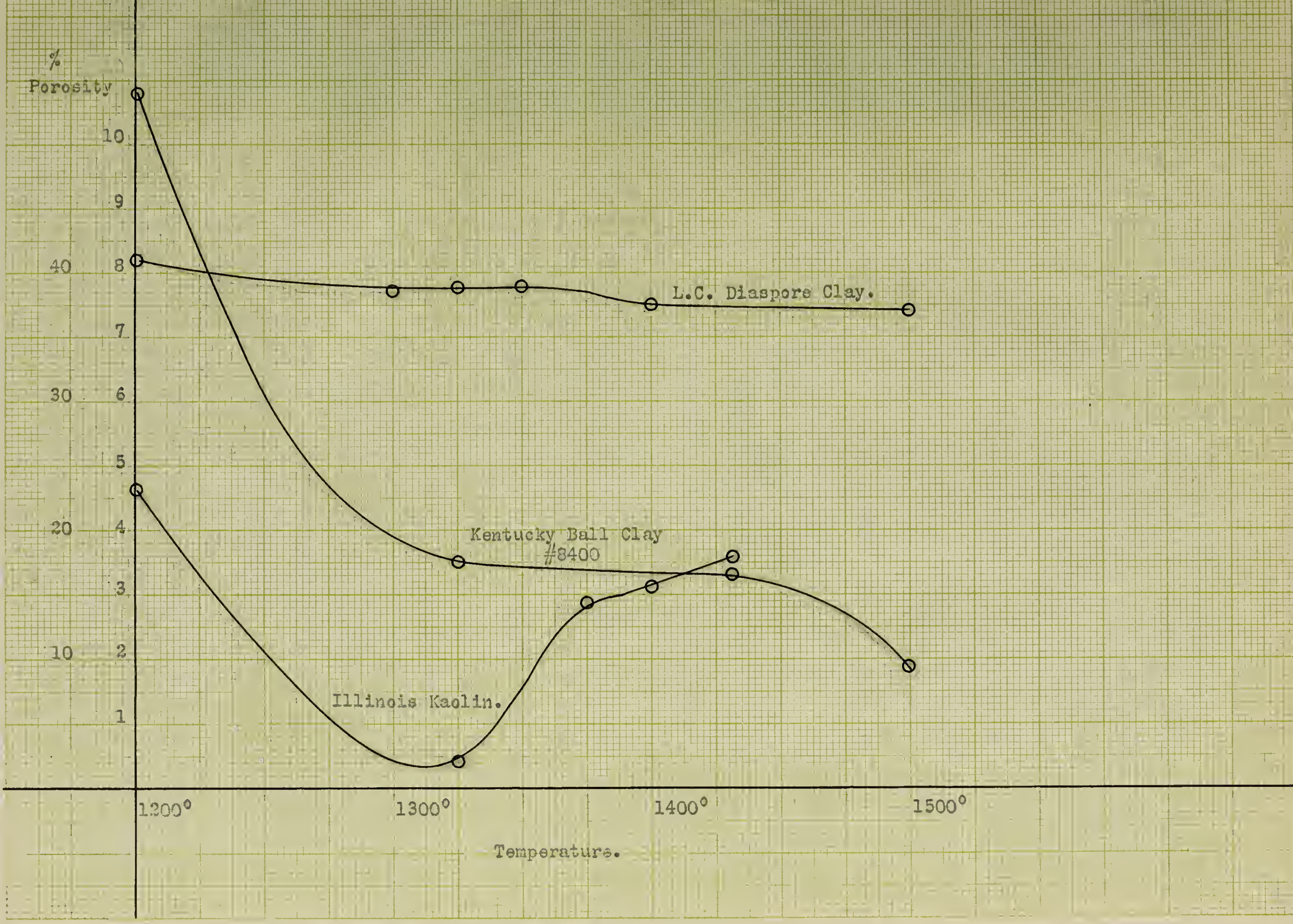

%
Porosity
10
9
8
7
6
5
4
3
2
1
40
30
20
10
L.C. Diaspore Clay.
Kentucky Ball Clay
#8400
Illinois Kaolin.
1200°
1300°
1400°
1500°
Temperature.

Fig. 5.

RESULTS OF TESTS ON BOND CLAYS.

1. Hickory Ball Clay (Fig.1) shows a rapid decrease in porosity between 1200°C and 1350°C, with a comparatively short range of vitrification. Above 1400°C, this clay has a tendency to overburn. Due to its short vitrification range and the low overburning temperature, this clay is not considered suitable for a crucible bond clay. The addition of flint clay holds up the porosity for a considerable range of temperature and shows a sudden fluxing action at 1300°C. The volume shrinkage (Fig.2) indicates overburning of the Hickory Clay alone at 1350°C by a sudden decrease in volume shrinkage. The addition of flint, as before noted, tends to decrease this tendency and increases the range of vitrification.

2. Highland Fire Clay (Fig.3) shows a steady drop in porosity to 1400°C at which temperature the porosity is 0.1%. Due to shattering of trials, the behavior above this point is not shown. The addition of flint clay as in the preceding case, causes a slower decrease in porosity and a sudden fluxing action between 1350°C and 1400°C. Beyond this temperature the porosity remains constant, which probably indicates a comparatively short vitrification range. The shattering of the trials above 1400°C indicates that this clay lacks resistance to sudden temperature changes, which would be a serious defect in its use for crucibles.

3. The L. C. Diaspore Clay (Fig.5) shows a high porosity (41.0%) and has not started to vitrify at 1500°C. The temperature of vitrification is far above this temperature, as might be expected of a highly aluminous, non-plastic clay of this character. It shows little change in volume (Fig.4), within this range of temperature and for this reason it was selected as a clay suitable for a non-plastic in crucible bodies.

4. The Illinois Bond Clay (Fig.5) has a low vitrification temperature (1310° to 1320°C) with sudden overburning at 1325°C. Due to its short vitrification range and sudden change in volume (Fig.5), it is of little value for high temperature crucibles.

5. The Kentucky Ball Clay - No.8400 shows a gradual drop in porosity from 1200°to 1300°C and up to 1425°C no further decrease is apparent. Beyond 1425°C the porosity again drops, reaching 2% at 1500° with no evidence of overburning. It is evident that the vitrification temperature is above 1500°C. It shows only a slight shrinkage between the temperatures of 1200°C and 1500°C and is suitable as a bond clay for crucibles. Due to its comparatively low porosity 1300° and 1500°C it is suitable as a bond clay for glass refractories and crucibles for steel melting.[5] Its long range of safe working temperature up to 1500°C, is a highly important advantage for this clay.

In general, the Kentucky Ball Clay No.8400 is the most suitable clay, because of its lack of marked volume changes throughout this temperature range, and low porosity between 1300° and 1500°. The L. C. Diaspore Clay, also, due to its refractoriness and constant volume would be suitable. Although not primarily a bond clay, if mixed with a bond clay having good properties, it might produce a mixture far better than either of the two clays.

PART 6.

MIXTURES TESTED AND DESCRIPTION OF TESTS.

Size of Grog:-

After selecting a suitable bond clay, the size of grog was the next factor to be considered. The results obtained by previous investigators on this subject were of assistance in this selection, namely that a body containing comparatively large sizes of grog withstood sudden temperature changes better than a body containing fine sizes, and the bodies that showed greater strength in the burned state contained fine grog sizes. For these reasons the following grog sizes were selected and varied in amounts as is shown in the table, to show their effects on certain body mixtures.

[5]Bleininger and Loomis - loc. cit. Page 5.

TABLE 1.

Size of Grog.	Percent Sizes in Mixtures. Sample 1.	Sample 2.	Sample 3.
10-20 mesh	50	75	100
20-dust	50	25	0

Calcined Missouri flint clay (LaClede-Christy) was used for grog. It was ground in a small jaw crusher and rolls and screened to the above sizes. The body mixtures were made up of the bond clay, raw non-plastic clay and the grog. The two raw non-plastic clays used were LaClede Christy Diaspore Clay and Flint Fire Clay. Each clay was crushed in a small jaw crusher and ground in the rolls to pass 20 mesh.

The following batch mixtures were used:-

SERIES 1.

Batch	Clay Portion		Parts	Parts	Batch Weight.		
	Kentucky Ball Clay	Diaspore Clay	Clay	Grog	Kentucky Ball Clay	Diaspore Clay	Grog.
A	100	0	50	50	50	0	50
B	90	10	50	50	45	5	50
C	80	20	50	50	40	10	50
D	80	20	60	40	48	12	40
E	80	20	70	30	56	14	30
F	70	30	60	40	42	18	40
G	70	30	70	30	49	21	30
H	70	30	80	20	56	24	20

For Series 2 batches, the same weights were used, but in these, raw flint clay was substituted forDiaspore Clay. Each batch mixture was made up with grog of three different ranges of size, as shown in Table 1, making in all, 24 mixtures

[illegible]

[illegible]

[illegible]

[illegible]

[illegible]

in each series. For example, Batch "A" was made up in three different mixtures, A_1 with grog consisting of grog mixture 1, A_2 with grog mixture 2, and A_3 with grog mixture 3.

A third series was made up with carborundum as a non-plastic. This was fine material, all of which passed a 20 mesh screen. The mixtures used in this series consisted of the following:-

Batch	Percent Kentucky Ball Clay	Percent Carborundum
SC_1	40	60
SC_2	60	40

Preparation of the mixtures:-

The Kentucky Ball Clay was crushed and ground in rolls to a suitable size for mixing. The various batch compositions were weighed out and mixed thoroughly in the dry state. Each mixture was then worked with water to a consistency that would permit molding. The batches, in this condition, were allowed to age in a humidor for forty-eight hours. At the end of this time they were removed and again thoroughly wedged by hand before molding into trial pieces. These were made up by pressing the clay by hand in iron molds 12"x1"x1", care being taken to prevent laminations. The test pieces were dried in the open room for two days, then in a steam drier. None of the pieces showed signs of cracking at this stage. They were then burned to Cone 8 in thirty-six hours in a coal fired kiln in order to make them strong enough to withstand handling for setting in the load test, and to determine the approximate weight after being burned.

TESTING OF THE PIECES.

Sag Test:-

A modified load test, or sag test was applied to the test bars with the purpose of determining the mixture or mixtures, which would show the least

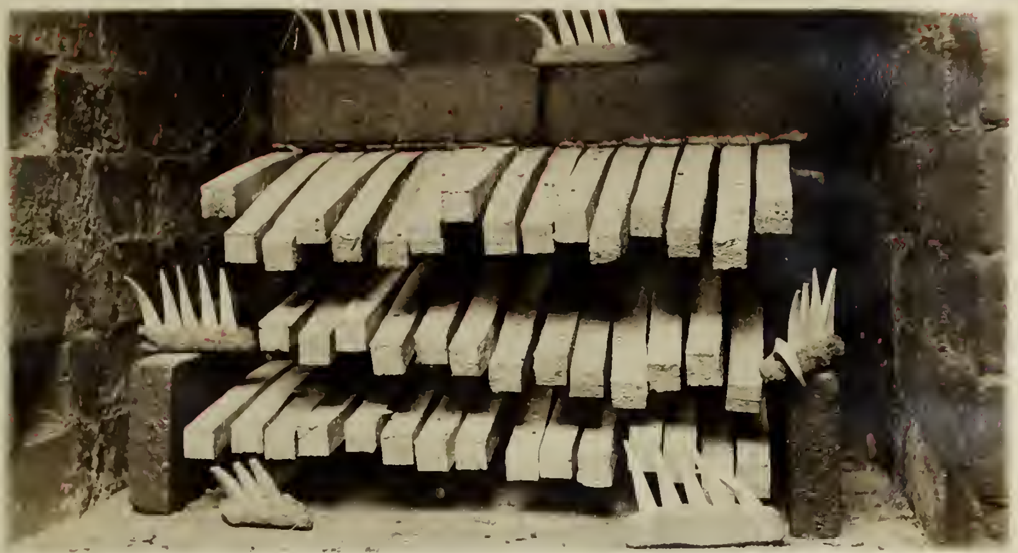
sag or deformation under a definite load at a high temperature, namely 1430°C for four hours. The load, to which a No. 10 crucible would be subjected in actual practice was calculated to be approximately 15 lbs. per sq. inch., so the bars were subjected to 20 lbs. per sq. inch in order to cover any additional stress that may arise. The method of applying the load was by supporting the bars at one end only, each acting as an individual cantilever beam. The bars were set to project beyond the supports to give the proper stress in lbs. per sq. inch in formula 3, below.

1. $M = \frac{1}{2}Wl$ for a cantilever beam.
2. $M = S\frac{I}{c}$
3. $S = \frac{1}{2}Wl \cdot \frac{c}{I}$

W = Total weight of the bar in pounds.
l = Length of bar beyond supports in inches.
S = Stress in lbs. per square inch.
I = Moment of Inertia.
c = Distance in inches of the remotest fiber from the central axis.

The average length of the bars was eleven inches, and the unsupported length was nine inches. The bars were supported in fire brick laid on edge and were built up in three tiers as is shown in the photograph. Cone pats were placed in different parts of the kiln to indicate any differences in heat treatment. The burning was done in an oil-fired, load-test kiln and a temperature of 1430°C was reached in twelve hours. At this point no sag was apparent, so this temperature was maintained for four hours, at the end of which time, some of the bars showed considerable deformation (as is shown in the photograph on the next page) and the firing was stopped. The amount of deflection for each bar was then measured carefully. Those mixtures, showing a deflection of more than one inch were discarded as being unsuitable. The load actually applied on the others was recalculated upon the basis of the weights and lengths of the bars after this heat treatment.

RESULTS OF LOAD TEST.



Batch No.	Series	Deflection in Inches	Stress in lbs. per square inch.
G_1	Series 1	3/4"	26.2
G_2	Series 1	15/16"	20.3
C_2	Series 2	15/16"	20.0
D_1	Series 2	7/8"	19.8
H_2	Series 2	3/4"	24.6
SC_1	Series 3	5/8"	19.7

Some interesting results were obtained, namely that all the batches containing Grog mixture 3,(100% -10-20 mesh) did not stand the treatment as well as the batches containing Grog mixtures 1, (50% -10-20 mesh and 50% -20 to dust) and 2, (75% -10-20 mesh and 25% -20 to dust).

Another feature brought out by this test is that all mixtures containing more than 80 parts in the clay portion of Kentucky Ball Clay, did not stand the required test.

Of Series 3, batch SC_1 shows the least deflection, indicating that 60% or more of carborundum in the batch is necessary to compare with the clay mixtures.

Little can be said about the respective series as compared to each other, for the above results show samples of each series coming within the selected limits.

Quenching Test:-

The test bars used for the previous test were employed in the quenching test which was carried out in the following manner:-

The test pieces were placedin a gas fired test kiln, and heated to 1000°C in eight hours. The bars were then removed separately from the furnace, quenched in water, remaining there until they cooled to the water temperature. The pieces were then dried and returned to the hot furnace, remaining there for an hour or until they reached the furnace temperature. They were then removed and quenched again, and this operation was repeated until the bars broke. All the trials stood three quenchings, and those which did not stand six such treatments were discarded as unsuitable.

This test was more severe than the ordinary quench test in that the pieces were not thoroughly dried before replacing them in the furnace for reheating. Care was taken, however, so that the same treatment was received by each trial piece so that the results obtained are comparable. The trials showing six or more quenches before breaking are recorded in the following table:-

RESULTS OF QUENCHING TEST.

Batch Number	Series	Number of quenches before breaking.
A_3	Series 1	8
B_3	Series 1	8
C_2	Series 1	6
D_3	Series 1	7
D_2	Series 1	8
G_1	Series 1	7
F_3	Series 1	7
H_3	Series 1	7
B_2	Series 2	6
C_3	Series 2	6
E_2	Series 2	6
E_3	Series 2	7
H_2	Series 2	8
H_3	Series 2	6
SC_1	Series 3	8
SC_2	Series 3	7

The results bear out those obtained by previous investigators, namely, the bodies with large grog sizes withstood sudden temperature changes better than thosewith fine sizes.

The batches containing Grog mixture 3 (100% 10-20 mesh) showed the best ability to withstand sudden temperature changes. Only one mixture containing 50%-10-20 mesh, batch number "G_1" compares favorably with the other samples under this treatment.

Series 1 and 2, taken as a whole, show little variation, but the two samples in Series 3, containing 40% and 60% carborundum show good ability to withstand sudden temperature changes.

RESULTS OF TESTS ON MIXTURES.

Upon comparing the results of the sag test and quenching test, three batches seem to give better results than any of the others. These are the following:-

Batch Number	Series	Parts in percent by weight. Kentucky Ball Clay	Diaspore Clay	Flint Fire Clay	Carbor-undum	Grog. 10-20 mesh	20-dust
G_1	Series 1	49	21	--	--	15.0	15.0
H_2	Series 2	56	--	24	--	15.0	5.0
SC_1	Series 3	40	--	--	60	--	--

Batch Number - G_1 - Series 1 stood seven quenching before failing and sustained a load of 26.2 lbs. per sq. inch with a deformation of 3/4" at a temperature of 1430°C. for four hours.

Batch Number - H_2 - Series 2 stood eight quenchings before failing and sustained a load of 24.6 lbs. per sq. inch with a deformation 3/4" at a temperature of 1430°C for four hours.

Batch Number - SC_1 - Series 3 stood eight quenchings before failing and had only a deformation of 5/8" under a stress of 19.7 lbs. per square inch in the sag test.

These mixtures, therefore, were selected from the three series for making up into crucibles, as they showed the best strength when subjected to a load at a high temperature and, in addition, showed good results when subjected to rapid temperature changes. The other mixtures that stood the quenchingtest failed under the sag test and vice versa.

PART 7.

TEST ON CRUCIBLES.

Preparation and Burning:-

The three crucible mixtures were made up in the same manner as the mixtures for the sag test. Crucibles of the following dimensions were jiggered in plaster molds and were allowed to remain in the molds for three hours.

Height - - - - - - - -	$12\frac{1}{2}$ inches.
Diameter at top (outside) - - - -	$5\frac{3}{4}$ inches.
Diameter at bottom (outside) - - - -	$4\frac{3}{4}$ inches.
Thickness - - - - - - - -	5/8 inches.

The crucibles were then removed and placed in a closed cupboard for 18 hours were slow uniform drying was permitted to take place. They were then placed in a steam drier until thoroughly dried. On removal, they were free from hair line cracks and showed a smooth inner surface.

The crucibles were burned in an oil-fired, load test kiln to Cone 15 in sixteen hours. The kiln was held for water smoking for three hours and the temperature was then raised at a uniform rate to the finish. The rate of burning was regulated by a pyrometer which registered 1470°C at the end of the burn with Cone 15 down.

The burned crucibles did not show the perfect smooth surface of the dried ware due to the shrinkage of the bond clay which allowed the shapes of the surface grog particles to become discernable. This was not apparent in the carborundum crucible which showed the same uniform surface in the burned state as was apparent in the dry state.

Slag Test:-

A slag test was used to determine the relative corrosive effect of a slag or flux on the bodies selected from previous tests. The slag used was made up with raw materials to approximate the composition of one used by Prof. S. W. Parr in melting his high temperature alloy, Illium. Parr's flux consists of powdered window glass with an addition of borax to soften it somewhat. The following batch was used in the present test, cobalt oxide being added to color the glass and better show the penetration into the clay body:

Whiting - -	200 parts
Soda Ash - -	212 parts
Borax - -	240 parts
Flint - -	720 parts
Cobalt Oxide -	3 parts
	1375 parts.

The crucibles were completely filled with the batch mixture, heated in a gas-fired, test kiln to 1150°C in five hours and held at this temperature for six hours. The crucibles were then removed from the furnace and the slag was poured out. After cooling they were broken for examination. No apparent penetration in any of the bodies was evident at 1150°C. The same pieces were then heated to a temperature of 1500°C. In heating to this temperature, the slag had penetrated into the minute cracks which had formed in the bodies G_1 and H_2, but no penetration was discernable in the case of crucible of composition SC_1.

PART 8

CONCLUSIONS.

In this investigation, it was endeavored to select the best crucible bodies for high temperature foundry service from a group of materials, which were available at this time. Probably the most important component of such a mixture is the bond clay. This must possess good strength in the green state, no excessive drying shrinkage and should be plastic so as to carry a good proportion of grog or non-plastic in the mix. In theburned state, it should be dense in structure, should be refractory and should have good strength at furnace temperatures. During burning the shrinkage should take place at a uniform rate. Vitrification should take place at a fairly low temperature and extend over as long a range as possible, with no overburning below 1450°C. It is apparent that no one clay is likely to possess all these desirable properties, but a mixture of two or more clays might be prepared which would possess these characteristics and which could be used advantageously.

The crucible mixture should be able to stand rapid temperature changes, to sustain sufficient load at high temperatures, to resist corrosion and to withstand the general rough usage to which the ware is subjected in actual practice. The character of the grog mixture is important in determining these properties. It should contain sufficient coarse material to withstand sudden temperature changes and fine sizes to give the body the necessary strength and the all important ability to withstand load.

The Kentucky Ball Clay - No. 8400 showed the best results of the bond clays tested. It had excellent plasticity and strength in the dry state; the drying shrinkage was not excessive and there was no tendency toward warping and cracking. It showed a long vitrification range (approximately 1300° to 1500°C) with a low porosity and gave no evidence of overburning at 1500°C

The beneficial effects of mixtures of flint clay with bond clays as a means

of increasing their refractoriness and overburning temperature, and the refractoriness of the diaspore clay warranted the use of such mixtures as the clay portion of the crucible batches. The following body compositions gave the best results in the tests:

Percent by weight.

G_1			H_2		
Kentucky Ball Clay	-	49.0	Kentucky Ball Clay	-	56.0
Diaspore Clay		21.0	Flint Clay		24.0
Grog	10-20 mesh	15.0	Grog	10-20 mesh	15.0
	20-Dust	15.0		20-Dust	5.0

SC_1

Kentucky Ball Clay	40
Carborundum	60.0

The results confirm the conclusions drawn by other investigators as to grog sizes, namely, that large particles alone give to the body, the property of withstanding sudden temperature changes, but did not give the ware sufficient strength to stand the load required. A mixture of sizes from the large to the fine gave the best results in each test.

Although some interesting results were obtained in this work, it is evident much can yet be done in crucible compositions for this field of work. More bond clays can be tested, various mixtures of clays tried and undoubtedly valuable information would be obtained. A wider variation of grog sizes could be used and the field enlarged to cover a greater range in proportions of mixtures.

------0------