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The bloating of clays

Everett Walter Sharp

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Department: Materials Science and Engineering

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THE BLOATING OF CLAYS

BY

EVERETT W. SHARP

A

THESIS

submitted to the faculty of the
SCHOOL OF MINES AND METALLURGY OF THE UNIVERSITY OF MISSOURI
in partial fulfillment of the work required for the
Degree of
BACHELOR OF SCIENCE IN CERAMIC ENGINEERING
Rolla, Missouri
1940

Approved by

Professor of Ceramic Engineering

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The Bloating of Clays

Introduction:

The effect of firing on clays in relation to volume is either a shrinkage, bloating, or no change whatsoever. The reason for the shrinkage and the constant volume is well known, but the cause of the bloating has not as yet been determined.

In some instances, as the use of fire clay brick in steel ladles, this property of bloating is desirable to a certain extent because this packs the brick lining in the ladle tightly after the first heat is poured in, and prevents the brick lining from floating out when the metal is poured.

There is little or no literature available on the causes of this bloating. Therefore this investigation was carried out to determine the causes of bloating.

Bloating of Ladle Brick:

In ladles used for pouring metal 9" straights are used, with the 9 x 2-1/2" side exposed. After heating and cooling, the brick are found to be bloated, or take up a permanent expansion.
Ladle Brick: ¹

Most present day ladle brick have a P. C. E. value of cone 23. Generally they are made of rather siliceous clays. Typical chemical analysis of a ladle brick is:

- \(66.0\% \quad \text{SiO}_2\)
- \(25.0\% \quad \text{Al}_2\text{O}_3\)
- \(4.5\% \quad \text{iron and titanium oxides}\)
- \(.4\% \quad \text{CaO}\)

The remainder being mostly magnesium, sodium and potassium oxides. The maximum firing temperature is probably about cone 5. The firing treatment is closely controlled and progresses at a very slow rate, being of more than usual importance in producing satisfactory ware. The fired brick are quite hard and dense, having a water absorption of 7 to 9%. However, the brick do not appear to be highly vitrified. These properties are essential in preventing penetration and fluxing action of slag and steel.

Firing Behavior Volume Test: ²

Size and shape of Test Pieces.---Test pieces shall be made approximately 1-1/8 x 1-1/8 x 1-7/8 inches.

---

Preparation of the Clay.—The clay shall be thoroughly dried at a temperature above $64^\circ$ C, but under $76^\circ$ C and crushed to pass a standard No. 20 sieve. (Tentative Standard Series for Testing Sieves.) It shall then be made up with water to a soft plastic consistency and thoroughly wedged and kneaded by hand.

Making the Test Pieces.—The test pieces shall be made in a suitable metal mold measuring approximately 1-1/8 x 1-1/8 inches by any desired length. After making, the pieces shall be cut into 1-7/8 inch lengths. The mold shall be thinly and evenly oiled with kerosene or light machine oil only as frequently as is necessary to keep the clay from sticking. The test piece shall be formed by taking in the hand a lump of clay, somewhat larger than required to fill the mold, and kneading it into a roll approximately the length of the mold. It shall then be placed in the mold and forced into the corners by blows with the cheek of the fist. The excess shall then be struck off with a wire and the piece slicked with a spatula and appropriately marked or numbered for identification. Care should be taken by the operator to keep his hands free from
The test piece shall be removed from the mold immediately and transferred to a smooth, straight pallet, care being taken not to distort it. If preferred, the test piece may be made in a piston plunger press fitted with a die 1-1/8 inches square. In either event the corners shall be slightly rounded.

Drying.—The test pieces shall be allowed to dry at room temperature until air-dry. They shall then be dried at a temperature between 64 and 76°C for at least five hours and finally at 110°C to approximately constant weight. They shall not be allowed to cool to room temperatures between these operations unless they be placed in a desiccator. After the final drying treatment they shall be placed in a desiccator to cool and remain there until the test is continued.

Dry Volume.—The dry volume shall be determined by a volumeter of the Sager type. The dry test pieces shall be allowed to soak in kerosene of the same specific gravity as that used in the volumeter for at least twelve hours before determining the volume. Kerosene with a specific gravity of about 0.8 shall be used as the measuring fluid. The volume shall be read to the nearest 0.1 cc.
Firing.—After again drying, the test pieces shall be placed in a refractory muffle or sagger as thin-walled as possible, which is set in the test kiln. The test pieces shall not be stacked together too compactly. The test kiln shall be of such a size that the effective space available is not less than 1-1/2 cu. ft. The heating may be accomplished by any convenient means but the temperature distribution should be thoroughly explored during the first few firings. A set of pyrometric cones of the Seger formulae as made by Professor Edward Orton, Jr., shall be placed as closely to the stacked-up pieces as possible; but temperature control by means of a platinum platinum-rhodium thermocouple is required also. The temperature shall therefore be reported in terms of cones and pyrometric readings in which correction has been made for the cold junction temperature.

The range of cones used shall cover the firing range of the clay, which must be judged by the operator. Generally the cones to be placed on the kiln are 014 to 3 for impure, common brick clays; 012 to 5 for shales and better types of ferruginous clay; and 02 to 15 for clays of the fire-clay type. Oxidizing conditions must be maintained during firing.
The heating shall be preferably at the rate of 45 deg. C. per hour from the start of the firing until the heat treatment is reached, corresponding to the softening point of the third consecutive cone below that at which the first trial is to be drawn; and 20 deg. C. per hour from that point until the end of the firing. In no case shall the rate of heating be greater than 60 deg. C. per hour nor less than 30 deg. C. per hour for the first period, or greater than 25 deg. C. per hour nor less than 15 deg. C. per hour for the last period.

Drawing the Test Pieces.—According to the nature of the clay, test pieces are first drawn at cone 010 for impure, common brick clays, cone 08 for shales and the better grade of ferruginous clays; cone 2 for clays of the fire-clay type. A test piece is then drawn from the kiln at each interval of two cones, and immediately before drawing the temperature indicated by the pyrometer shall be recorded. The removal of the test pieces from the kiln shall be done as quickly as possible to avoid cooling down the kiln too much. It will be necessary as a rule to increase the application of heat a little after each draw.
Upon removal from the test kiln the test pieces shall be (a) immediately covered with hot sand or (b) placed in a small supplementary furnace kept at a dark-red heat and large enough to hold all of the test pieces to be drawn. Upon completion of the firing, the auxiliary furnace shall be allowed to cool at a natural rate.

Saturation.---The fired test pieces shall be placed in distilled water in a suitable vessel and boiled for two hours, then allowed to cool to room temperatures, while still immersed in the water. During boiling, the test pieces shall not be in contact with the heated bottom of the container.

Fired Volume.---The fired volume shall be determined in a volumeter of the Seger type, using distilled water as the measuring fluid. The test pieces shall be introduced into and removed from the volumeter in substantially the same manner so as not to appreciably change the volume of the water in the instrument.

Volume Change.---The volume change shall be determined by the relation:

$$b_1 = \frac{(V_d - V_f) \times 100}{V_d}$$

in which $b_1$ = per cent volume change;

$V_d$ = volume of the dry test piece, cc;

$V_f$ = volume of the fired test piece, cc.
Plotting Results.---When the results are plotted in graphical form (and this is advisable) heat treatment is preferable expressed in cone numbers. Equal distances on the abscissa and ordinate shall represent two cones and 5 per cent volume change, respectively.

Procedure and Data:

The percentage volume change was run by the standard progressive firing method as explained on the preceding pages. Draw trials were made from the oil-fired test kiln at the following cone equivalents which are given below with their corresponding temperatures.

<table>
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<th>Temperature °C</th>
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<td>1180</td>
<td>2156</td>
</tr>
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<td>11</td>
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<td>2345</td>
</tr>
<tr>
<td>13</td>
<td>1350</td>
<td>2462</td>
</tr>
<tr>
<td>15</td>
<td>1410</td>
<td>2570*</td>
</tr>
</tbody>
</table>

*Most of the clays were fired to only cone 13.
A P.C.E. test was made on each of the clays tested and a chemical analysis was made. The chemical analysis was converted into the mineral composition (assuming the alkali lies to be all muscovite) and the volume change in percent was plotted against the cone equivalent.

It was seen that a relationship between the cone that bloating starts at or the degree of bloating and the chemical composition should be established if the desired results were to be obtained. This was attempted by plotting the various mineral constituents against the cone at which bloating starts and against the degree of bloating.

The following tables give the average of two trials in the percentage of volume change at each cone that the trials were fired to, and also gives the chemical analysis, cone equivalent, and mineral composition.
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<td>MgO</td>
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<td>TiO₂</td>
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<td>Alkalies</td>
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<p>| Chemical Anal. | -  | -  | -  | -  | -  | -  |
| % Composition  | -  | -  | -  | -  | -  | -  |
| SiO₂          | 65.96 | 65.06 | 63.06 | 77.66 | 69.42 | 66.62 |
| Al₂O₃         | 19.54 | 18.57 | 20.19 | 11.17 | 17.50 | 19.45 |
| Fe₂O₃         | 3.15  | 3.66  | 2.98  | 2.80  | 2.45  | 2.70  |
| CaO           | .79   | .64   | .69   | .49   | .64   | .99   |
| MgO           | .87   | 1.03  | 1.34  | .99   | 1.12  | .98   |
| Alkalis       | 1.48  | 1.23  | 1.68  | .72   | 1.08  | 1.19  |
| Ignition loss | 7.13  | 8.38  | 9.38  | 5.25  | 7.01  | 7.35  |
| P. C. E.      | 18+   | 17    | 19-   | 16-   | 19+   | 17    |</p>
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RELATION OF VOLUME CHANGE TO FIRING TEMPERATURE

GRAPH #1

CONE EQUIVALENT

GRAPH #2

CONE EQUIVALENT
Relation of Volume Change To Firing Temperature

Graph #3

% Volume Change

Cone Equivalent

Graph #4

% Volume Change

Cone Equivalent
Relation of Volume Change to Firing Temperature

Graph 5

% Volume Change

Cone Equivalent

Graph 6

% Volume Change

Cone Equivalent
Relation of Volume Change to Firing Temperature

Graph #7

% Volume Change

Cone Equivalent

Graph #8

% Volume Change

Cone Equivalent
Relation of Volume Change to Firing Temperature

Graph #9

Cone Equivalent

Graph #10

Cone Equivalent
Relation of Volume Change to Firing Temperature

Graph II

% Volume Change

0 5 10 15 20 25 30

08 06 04 02 1 3 5 7 9 11 13

Cone Equivalent
Discussion of Data:

The preceding graphs were plotted to show clearly the cone at which bloating started and the degree of bloat. Most of the clays tested were found to bloat, while some of them didn't bloat. This gave a good variety for correlation. Below is a summary of the cone at which the clays bloated.

<table>
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<th>Cone of Bloating</th>
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<td>Graph No.</td>
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<td>Cone of Bloating</td>
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Many graphs were plotted attempting to relate the cone at which bloating started against the constituents of each clay, that is, the muscovite, clay(kaolinite), ferric oxide ($\text{Fe}_2\text{O}_3$), titanium oxide ($\text{TiO}_2$), silica, calcium carbonate, magnesium carbonate, and free carbon.

Graphs were plotted attempting to relate the degree of bloating and the constituents of each clay.

These graphs showed no relation and hence they are not included here.

Conclusions:

It can be noticed from the preceding tables that, in general, the clays that bloated at low temperatures were high in clay, low in silica, and high in magnesium carbonate. This was not true in all cases though.
Hoffman found that alumina, when bonded with Lumnite cement and fired to cone 15 produced a bloating, in some cases, that was proportional to the amount of cement and the grain size of the alumina. This cement is very high in calcium oxide and relatively high in ferrous oxide (Fe\text{O}).

**Suggestions for Further Work:**

Some of these clays might be mixed with grog and flint clay to produce a fire brick that would be suitable for use in steel ladles.

Some of the minerals in the clay might be added in different portions to kaolinite to determine if the mineral by itself would make the clay bloat.

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Acknowledgment:

The writer wishes to express his thanks to Dr. Paul G. Herold of the Ceramic Department of the Missouri School of Mines and Metallurgy for his cooperation and his helpful suggestions in carrying out this research.

Appreciation is also extended to the Missouri Geological Survey for their help and cooperation in the running of the chemical analysis on the clays.
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