The effect of reheating certain fire brick on pyrometric cone equivalents

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THE EFFECT OF REHEATING CERTAIN FIRE BRICK ON PYROMETRIC CONE EQUIVALENTS

by

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INTRODUCTION

In a recent survey of the refractories field, especially with respect to the softening point of fire brick, it was suggested to the author that the possibility of reheating certain fire brick, according to the specifications given by the American Society for Testing Materials, might produce a difference in pyrometric cone equivalent from that of the original or unreheated sample.

At present, there is considerable lack of knowledge regarding the problem, as evidenced by the utter lack of literature on the softening point resulting from the reheating of fire brick.

It will have to be granted that the results obtained in this experiment might not check or correspond with the results obtained by the manufacturer in any similar test, such as the p.c.e. of a finished ware or product. Undoubtedly it will be impossible to have operating conditions the same in two cases. So, therefore, there may be a great difference in the pyrometric cone equivalent of fire brick as determined by one method with that of another. One manufacturer might not comply with the rigid requirements set forth by
the American Society for Testing Materials while on
the other hand, an accomplished operator may obtain
entirely different results, due to the strict ad-
herence to specifications. So absolute checks on
this type of work are improbable.

Kanolt, in his article on Melting Point of
Refractory Materials, says "the melting point can be
precisely and rationally defined as a temperature at
which a crystalline or anisotropic phase of one comp-
osition and an amorphous or isotropic phase of the
same composition can exist in a state of equilibrium.

When a fire brick is heated or subjected to an
original firing, a certain amount of glass bond is
produced in the ware and the higher the firing tem-
perature, the more glass developed. In practically
all cases, a fired brick is subjected to a much lower
temperature while it is being given its initial fir-
ing than will be experienced by the refractory in
service. In view of this fact, it will be necessary
for the refractories manufacturers to produce a brick
that will not decrease in p.o.e. value when subjected
to service conditions.

In service, fire brick are again and again heated
and cooled and this practice not only affects the
softening point of the fire brick but likewise affects other properties such as density, strength, shrinkage, etc.

In the reheating of fire brick, certain eutectics are formed which tend to lower the p.c.e. value. The more numerous the reheatings or the greater the number of heats, the lower the p.c.e. value will become and finally a point will be reached where the softening point will no longer decrease but will tend to remain constant. This is the chief source for the decrease in p.c.e. value, i.e., the chemical composition and the chemical reactions that take place upon heating, are the cause for reduced softening point.

S.S. Cole has recently been doing very extensive work along this same line and it will be interesting to note his results, as compared with those contained in this study, as soon as the results are published.

MATERIALS

<table>
<thead>
<tr>
<th>Brand</th>
<th>Manufacturer</th>
</tr>
</thead>
<tbody>
<tr>
<td>Laclede King</td>
<td>Laclede-Christy Fire Brick</td>
</tr>
<tr>
<td>Wellsville Hi-heat</td>
<td>Wellsville Fire Brick Co.</td>
</tr>
<tr>
<td>Wellsville Savage</td>
<td>Wellsville Fire Brick Co.</td>
</tr>
<tr>
<td>Empire D.P.</td>
<td>A.P. Green Fire Brick Co.</td>
</tr>
<tr>
<td>Evans-Howard S.M.</td>
<td>General Refractories Co.</td>
</tr>
</tbody>
</table>
PROCEDURE

In this test there were five different brands of fire brick used and of the various brick sent by the refractory companies, one brick of each brand was subjected to the American Society for Testing Materials specifications for reheating: namely, heating uniformly to 1400°C and holding at this temperature for five hours. The brick shall not show more than 2% contraction in original length.

The brick, after being marked for identification and the saturated weight and the suspended weight of each brick determined, were set in a standard Bureau of Mines experiment kiln of the down draft type, along with several dummy brick, so as to have a uniform setting and also to provide for absorption of heat. The door of the kiln was then built with fire brick and the chinks between the brick in the door were plastered with grouting so as to prevent the escape of heat from the kiln. Provision was made in the door of the kiln for the insertion of a platinum-platinum 10% rhodium thermocouple, which was placed in a quartz tube and put into the kiln in the proximity of the test brick. This thermocouple was then connected to
a potentiometer circuit, which was adjusted by a cold and compensator so as to make corrections for atmospheric temperature. Cones were placed in the kiln at two points in order to determine the heat distribution of the kiln. It was by the thermocouple that the temperature was controlled.

The preliminary heating of the kiln was done with gas until the kiln had attained sufficient temperature to ignite the oil, which was forced from a storage tank under a pressure of 50 pounds gage. The oil was fed into the kiln in an atomized spray in a Denver Fire Clay Company burner.

The gas was turned on at three p.m. and at five p.m. the oil was turned on and both oil and gas were then used until the temperature of the kiln was sufficient to ignite the oil alone. The oil was fed into the kiln as rapidly as it was possible to without creating reducing conditions.

The kiln was heated at a rate of 200°C per hour until a temperature of 1400°C was obtained, after which, the temperature was held at 1400°C for five hours.

After the kiln had been fired and allowed to
cool, the test brick were taken from the kiln and the saturated weight and suspended weight again determined. The cone plaques were also examined and found to be practically the same, indicating that fairly uniform heating was obtained. With the weights of the various brick determined, both before reheating and after reheating, the percent linear shrinkage was calculated.

PREPARATION OF SAMPLES

Both the reheated and non-reheated brick were put through a primary crusher of the Blake type, the jaws and receiving pan having previously been thoroughly cleaned so as to prevent contamination of samples.

After having been crushed to a diameter of one-half inch or more, the various samples were then put through a set of Stueveant laboratory rolls, having eight inch crushing rolls and traveling at a speed of 150 R.P.M. In putting the samples through the above operations, care was shown in cleaning the primary crusher and rolls after each sample was introduced. The samples were then, after being reduced to one-quarter inch diameter by the rolls, quartered and the amount reduced to 50 grams. A magnet was repeatedly
passed through the samples so as to free them of any metallic iron they might possess. This portion of each sample was then ground in an agate mortar to such a fineness that all the samples passed a No. 65 mesh standard Tyler sieve, the diameter of wire being .183 m.m. (0.0072") and the screen openings being .208 m.m. (0.0082"). In order to prevent excessive reduction of fines, the fines were thrown on the sieve from time to time and the grinding of the coarser particles continued until all the various samples had passed the sieve.

Preparation of TEST CONES

The samples were thoroughly mixed and a 10% solution of dextrine added as an organic bond. This dextrine was prepared by using 50 grams of dextrine and 500 grams of distilled water. This mixture was then heated just below boiling in order to aid the solution of the mixture or rather of the dextrine. The solution was then cooled and about 2 c.c. of formaldehyde added as a preservative.

The test cones were molded in a metal mold in the shape of tetrahedrons, eight m.m. (5/16") on sides at the base and 25 m.m. (1") in height. Ten test cones were made from each sample, that is, both of reheated and non-reheated brick. It was not necessary to sinter the test cones as they possesses considerable strength or rigidity in the dry state.
MOUNTING OF TEST CONES

The test cones were mounted in a refractory plaque containing eight holes or places for the insertion of cones. These holes were filled with a refractory mixture of 15% kaolin and 85% fused alumina, made into a soft mud consistency. The test and standard cones were then set in this refractory material, imbedded to approximately to 1\(\frac{1}{10}\) and inclined at an angle of 32° with the horizontal.

The arrangement with respect to Orton standard cones was as follows:

![Figure 1](image)

**Figure 1**

HEATING

The heating was done in a Fulton-Coursen granular carbon resistance electric furnace, details of which are shown in the blue prints accompanying this report.
This furnace is run on single phase, 110 volt A.C., current supplied from a synchronous convertor, which transforms or converts from three phase, which is the current normally supplied to the panel boards, to one phase, which is supplied to the furnace. The rate of heating for this experiment is the same as specified by A.S.T.M. and can be secured from the graphs on rate of heating of the samples, which are included in the back of this report.

The furnace is started from room temperature by applying about 15 volts to the terminals of the furnace for about one hour. At the end of this time, the voltage is increased to 25-27 volts by manipulation of the panel board. This voltage, at the end of one hour, should have the furnace to dull red heat, about 1500°F, from whence the temperature is increased according to the A.S.T.M. rate given by the graphs.

This increase is accomplished by either cutting in or cutting out resistance as the case may be, namely, cutting out resistance raises the voltage and consequently increases the temperature of the furnace, while cutting in resistance decreases the voltage and likewise lowers the temperature of the furnace.
It is not necessary to cool the furnace down to room temperature after every run, as the plaque can easily be removed at a temperature of 1500°F and a new plaque inserted and the heating up again started. This practice saves a great deal of time as the furnace can be cooled from 3200°F (cone 34) to 1300°F in just a little more than an hour, whereas, it would probably take at least five or six hours to cool the furnace from 3200°F down to room temperature after every run. So, therefore, considerable time is saved by cooling the furnace to 1300°F and inserting a new plaque. This procedure was followed in operating the furnace.

It was very evident, while operating the furnace, that if great care was not taken in controlling operating conditions, "hot spots" would develop or be set up in the furnace. This is not only harmful to the actual testing or p.c.e. of the test sample but it is also severe on the alundum muffle, both inner and outer muffles. This condition causes one part of the muffle to be heated to a much higher temperature than that of another part of the muffle, consequently on cooling, stresses are set up which cause the alundum muffles to crack badly, due to unequal expansion and
contraction. This, besides the other aforementioned items, is also undesirable from the standpoint of cost, as the alundum muffs are costly and are difficult to replace, as the furnace must be dismantled and then, of course, rebuilt according to the blue prints accompanying this report.

In order to avoid hot spots in this type of furnace, it is necessary to loosen the graphite or carbon in such a manner as to thoroughly mix it. This should be done after every run. Another necessary item is to have the inner muffle centrally spaced with regard to the outer muffle, so there is an equal amount of graphite on all sides of the inner muffle. If these few suggestions are followed, the tendency for having hot spots in the furnace will be reduced to a minimum.

Care should be taken to always have oxidizing conditions in the furnace. Excessive reducing conditions should be avoided as much as possible. On a few occasions, very small particles of graphite inside the inner muffle were the cause of slight reducing conditions, however, this condition did not last any appreciable time as the graphite soon burned out. This effect of reducing conditions also made it difficult to obtain the correct temperatures of the furnace.
A Leeds and Northrup optical pyrometer was used in reading the temperature of the furnace and this instrument was adjusted for zero point after every run. This was done by first removing the connecting plug from the socket of the milli-ammeter. To check the zero of the ammeter, throw the lever towards position B, making sure that the lever has gone as far as possible to the left. This movement will expose a hole in the case through which the zero adjusting screw can be seen. Adjustment should be then made so the pointer reads on the first line of the scale, if it does not already rest there. The lever is then pushed to position A, making sure that it goes as far as possible to the right and over the small bump. The connecting plug is then inserted into the socket, making sure that a good contact is made between the plug and the socket. The meter should then indicate a flow of not over 300 milli-amperes.

After the instrument has been properly adjusted, the temperature of the furnace is read by pointing the telescope directly at the hot body and the rheostat regulated in such a manner that the tip of the lamp filament just dissapears against the hot body background. Black body conditions should be avoided at all times.
DETERMINATION OF SOFTENING POINT

The softening point of the test cone was indicated by the top bending over and assuming a position of the tip of the cone touching the top of the plaque. There was no evidence of squatting or blunting in any of the runs of this experiment. The softening point of the unknown cone was reported in terms of standard Orton cones and also was reported as that standard cone which more nearly corresponded in time of softening with the test cone or unknown cone.

RESULTS

The results are given in the following table:

<table>
<thead>
<tr>
<th>Brand of fire brick</th>
<th>Pyrometric cone equivalent</th>
<th>Non-reheated sample</th>
<th>Reheated sample</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>Cone 34</td>
<td>cone 33</td>
<td></td>
</tr>
<tr>
<td>B</td>
<td>Cone 33</td>
<td>Cone 33</td>
<td></td>
</tr>
<tr>
<td>C</td>
<td>Cone 32</td>
<td>Cone 32</td>
<td></td>
</tr>
<tr>
<td>D</td>
<td>Cone 33</td>
<td>Cone 33</td>
<td></td>
</tr>
<tr>
<td>E</td>
<td>Cone 33</td>
<td>Cone 32-33</td>
<td></td>
</tr>
</tbody>
</table>

All of the above samples were run at least twice, and if the results did not check then, another run was made and this continued until checking results were obtained. However, it was not necessary to run most of the samples more than twice, as checking results
were usually obtained in two runs. Nevertheless, samples B (reheated) and C (reheated) required three runs in order to have checking results.

**TABLE II**

<table>
<thead>
<tr>
<th></th>
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<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Num.</td>
<td>wt.</td>
<td>wt. c.c.</td>
<td>wt.</td>
<td>wt. c.c.</td>
<td>Shr.</td>
<td>Shr.</td>
</tr>
<tr>
<td>A</td>
<td>3835</td>
<td>2120</td>
<td>1715</td>
<td>3813</td>
<td>2109</td>
<td>1704</td>
</tr>
<tr>
<td>B</td>
<td>3854</td>
<td>2144</td>
<td>1710</td>
<td>3832</td>
<td>2128</td>
<td>1710</td>
</tr>
<tr>
<td>C</td>
<td>3880</td>
<td>2112</td>
<td>1738</td>
<td>3845</td>
<td>2090</td>
<td>1755</td>
</tr>
<tr>
<td>D</td>
<td>3844</td>
<td>2184</td>
<td>1680</td>
<td>3835</td>
<td>2185</td>
<td>1651</td>
</tr>
<tr>
<td>E</td>
<td>3790</td>
<td>2071</td>
<td>1719</td>
<td>3763</td>
<td>2060</td>
<td>1703</td>
</tr>
</tbody>
</table>

Sample Calculations

\[ \% \text{Linear shrinkage} = \frac{a}{b} \times 100 \]

\[ a = 1.005 - 1 \]

\[ b = \% \text{volume shrinkage} \]

Sample A: volume = Saturated weight-suspended wt. / Specific gravity of Liquid.

\[ = \frac{3835 - 2120}{1} = 1715 \text{ c.c.} \]

Percent volume shrinkage = \[ \frac{1715 - 1704}{1715} \times 100 = 0.65\% \]

Using the above formula:

\[ a = (1.005 - 1) \times 100 = 0.5\% = \% \text{percent linear shrinkage} \]

DISCUSSION OF RESULTS

From the results obtained in this test, it would be practically impossible to state any law regarding the
effect of reheating on the pyrometric cone equivalent or softening point of fire brick, due to the limited number of samples tested. In order to declare any such rigid law or statement, a great many different brands of fire brick would have to be tested, in order to more nearly include the entire field of refractories. However, there is some fire brick that show no effects on reheating, that is, there is some evidence of the fact that reheating does not alter or change fire brick with regard to softening point. Even this statement is not true in all cases, as can be seen by inspection of the test data.

Sample A for instance. After running both the reheated and the non-reheated sample twice, and checks obtained on the third run, there is slight evidence of a decrease in softening point of the reheated sample over the non-reheated sample. However, there is a possibility that this change may be due to non uniformity of furnace conditions, even though checks were obtained.

This was only evident in samples A and B of the reheated samples. However, in all other cases, the effect of reheating showed no definite relation to the softening point.
Still there is the instance, in case of samples A and B, where the reheated sample gave a lower softening point than the non-reheated sample and whether or not this fact can be attributed to the composition of these two samples remains to be further investigated.

CONCLUSIONS

In endeavoring to account for the results obtained it can be most logically based upon the fact that a given fire brick will stand only a given amount of heat treatment and any heating after the original heating has a tendency to more or less stabilize the softening point. There is necessarily a limit to which the softening point of a refractory may decrease and this becomes the limit that would be difficult to determine, in the case of the reheated sample.

However, it is my belief that there is really a difference in softening point of the reheated sample and that of the non-reheated sample and this difference is that the reheated sample is slightly lower in pyrometric cone equivalent value that the non-reheated sample.

Although no great definite data was found supporting this statement, it may be justified by the fact that there are changes in chemical properties, also physical properties, of the reheated specimen over the
non-reheated sample. It is only natural to believe that certain eutectics are formed in the reheat test and the more advanced these reactions, the less resistant the brick becomes to softening. From this, it is fair to assume that the eutectics formed by reheating are combined in such a manner as to give a lower softening point than the constituents of the non-reheated sample.

RECOMMENDATIONS FOR FURTHER RESEARCH

It might be worthy to note at this point, that if the temperature of 1400°C was maintained for a somewhat longer time or even if the reheat test was conducted at a higher temperature, a greater decrease in pyrometric cone equivalent value would probably be brought about.

Another possible point of attack in which additional data on this subject of reheating fire brick could be obtained, might be the effect of different rates of heating of both reheated and non-reheated samples. This is suggested as a possible subject for more work on this subject.

APPLICATION TO THE REFRactories INDUSTRy

In as far as applying this test data to the refractories industry, it would be altogether impractical. As a matter of fact, in the first place it
would be undesirable for a manufacturer to lower the
refractoriness of a brand of his fire brick by re-
heating, as in practically all instances it is just
the opposite that is true, namely, increasing the
softening point instead of decreasing it.

Then this practice of reheating fire brick would
be undesirable from another standpoint, and that is
the added expense involved in reheating the fire brick
would not offset the possible increase in good qual-
ities in the brick enough to be profitable to the
manufacturer. Therefore, this test data could not be
applied to the refractories industry.
The melting point of a refractory can be precisely and rationally defined as the temperature at which a crystalline or anisotropic phase and an amorphous or isotropic phase of the same composition can exist in equilibrium. This melting point may be either above or below the apparent melting point or softening point. Some substances, which possess perfectly definite true melting points, undergo changes resembling fusion at lower temperatures. Solids, in general, become softer when heated; they may become so soft as to yield to their own surface tension, with the result that sharp corners are rounded off.

The true melting point of silica is about 1600°C. However, fused silica possesses such extreme viscosity near the melting point that it does not flow or change shape distinctly until higher temperatures are reached.

The melting point of a fire brick is not only a property to be determined in order to determine its fitness for a given purpose. Its cold crushing strength, its behavior under load when heated, and its
resistance to fluxes may all be important.

Bleininger and Brown have made a large number of tests of fire brick under loads at 1300°C, this work being carried on at the Pittsburgh Testing Station of the Bureau of Mines or rather Bureau of Standards. The chemical composition, cold crushing strength, porosity, and density of these samples are given by Bleininger and Brown. They also determined the Seger cone numbers corresponding to the softening point of the bricks.²

Kenolt determined the melting points of a large number of samples of fire brick. The experiments were conducted in an Arsen graphite-resistance vacuum furnace. As a matter of precaution the samples were protected by a refractory tube. The refractory tube was made of 72% kaolin and 38% alumina, these being in the proportions for the formation of sillimanite (Al₂O₃ SiO₃). According to Shepard and Rankin⁵, sillimanite melts at 1611°C. The chrome and magnesia brick were melted in graphite crucibles without protecting tubes.

The temperatures were determined by means of a Horace optical pyrometer of the Wolburn-Kurlbaum type which was sighted downward through the furnace window.

Table I

ON FOLLOWING PAGE.
<table>
<thead>
<tr>
<th>Materials</th>
<th>No. of samples</th>
<th>Temp. of visible flow</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fire clay brick</td>
<td>41</td>
<td>1535-1735 (mean 1649)</td>
</tr>
<tr>
<td>Bauxite brick</td>
<td>8</td>
<td>1535-1735</td>
</tr>
<tr>
<td>Silica brick</td>
<td>3</td>
<td>1700-1705</td>
</tr>
<tr>
<td>Chromite brick</td>
<td>1</td>
<td>2050</td>
</tr>
<tr>
<td>Magnesia brick</td>
<td>1</td>
<td>2155</td>
</tr>
<tr>
<td>Kaolin brick</td>
<td>1</td>
<td>1735-1740</td>
</tr>
<tr>
<td>Bauxite clay</td>
<td>1</td>
<td>1820</td>
</tr>
<tr>
<td>Chromite</td>
<td>1</td>
<td>2180</td>
</tr>
</tbody>
</table>

The Arsem furnace can be used for work upon oxides, filling the furnace with a non-oxidizing gas, at atmospheric pressure, when the vaporization of the oxide prevents the use of a vacuum.

Magnesia can be melted in graphite crucibles without contamination. Some other oxides such as lime, readily form carbides when heated in graphite; and still others, such as chromia oxide, are reduced too rapidly to metal. Tungsten has been found to be a suitable crucible material for such of these oxides as have been tried.

REFERENCES

Bureau of standards technologic paper 7 (1912)
See also Hofman and Demond, Trans. Am. Inst. Mining Eng., 25, p. 42 (1894)\textsuperscript{3}

