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DRY PRESSING REFRACTORY INSULATING BRICK

BY

FRANK CLARENCE STEINKE

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
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Approved by... *Paul H. Herold*

Assistant Professor of Ceramics

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INTRODUCTION

In recent years the trend has been toward the production of alloy steels, non corrosive metals, etc. This trend has necessitated the use of higher firing temperatures which in turn has put a tremendous load on the refractory manufacturers for better and better products.

One of the more recent developements in the refractory field has been the insulating brick. The general accredited advantages of the refractory insulating brick are (1) saving in heat storage, and (2) reduced time in bringing the furnace to temperature. These two points lead to the same economic advantage namely, reducing the fuel consumption of the furnace. To be sure there are other minor advantages such as reducing the size of the furnace walls which is important in small portable furnaces and cutting down the initial construction cost.⁽¹⁾ Some estimates

(1) "Insulating Refractories", W.M. Hepburn. Journal American Ceramic Society, V18 , 1935 pp 18.

have been as high as 60 to 70% reduction of heat losses by radiation and conduction through furnace walls by the use of insulating brick.

The chief limitations to the use of insulating brick in modern furnaces is the lack of resistance to slags at high temperatures. Other disadvantages are low resistance to abrasion, low spalling resistance, and poor mechanical strength.

None of the porous brick now made can be used in direct contact with slags, or where they will be exposed to abrasion, or flame impingement. Where abrasive combustion cannot be avoided the insulating brick may be faced with a course of refractory brick which will stand the direct action of flame, slag or abrasion.

Due to the very low thermal conductivity of insulators a nine inch wall has the same heat resistance as a twenty-three inch wall of fire brick. This fact makes a much smaller grate area possible and at the same time production is speeded up.⁽²⁾

(2) " Wood, T.E. " Light Weight Cellulor Brick Made For Furnace Installation". Ceramic Age, V26, Sept. 1935 pp 96-98.

Listed in the order of their importance, the characteristics of brick we would desire are as follows:

- (1) Uniformity
- (2) Shrinkage
- (3) Spalling
- (4) Physical strength
- (5) Insulating values
- (6) Thermal capacities

There have been four general methods used for the production of insulating refractories:

- (1) Formation of bubbles in the soft mix.
- (2) Addition of porous material to the clay body.
- (3) Burning out particles of organic matter.
- (4) Sublimation of solid particles in the mass. (3)

One other method is used for the manufacture of low temperature insulators. That is brick made out of diatomaceous earth. The material itself was formed by the skeletons of tiny marine animals settling to the bottom of the ocean over long periods of time and

(3) Thesis: Tyrell, "Manufacture of Insulating Refractories". Missouri School of Mines, 1937, p3.

forming huge masses of porous material which later were lifted out of the ocean. Due to their low refractoriness no more discussion will be given about them.

The method used to produce bubbles consists essentially of adding some carbonate to the brick in the plastic state and then adding an acid which causes the evolution of carbon dioxide throughout the brick to form a porous structure. The difficulty with this method is controlling the evolution of the carbon dioxide in order to prevent the rupturing of the brick by the carbon dioxide.
(4)

M.E. Tyrell on work done in this line produced a froth of soap in a clay slip and held the soap in a frothy condition by the addition of molten paraffin. The main difficulty in this process was that the brick cracked during the drying process. There was also some difficulty with the brick sticking to the bottom of the mold.
(3)

(3) Thesis: M.E. Tyrell, "Manufacture of Insulating Refractories." Missouri School of Mines, 1937.

(4) Thesis: E.L. Dudley, "Development of Refractory Insulating Brick." Missouri School of Mines. 1935

The most common method used in this country for producing insulating brick is the introduction of some combustible material such as sawdust, cork, coal, coke or peat to the plastic mass of clay which burns out during firing and leaves open spaces in the body. This method tends to make the brick less refractory due to the fluxing action of the ash left behind.

In all the methods described the bricks are hand formed using very soft plastic clay masses with from 20 to 30% water. In the drying and firing process the brick becomes mis-shaped and necessitates grinding of the brick to the required shape. The machining to size of the brick is the most expensive part of the production and elimination of this feature is very desirable.

If the brick could be dry pressed a much larger production in a given time could be accomplished and due to the small water content, (8%) perhaps the grinding operation eliminated. The dry pressing of the brick is very difficult due to the resiliency of most of the materials now used as combustibles. Cork and wood when put under pressure will compress but not deform except at extremely high pressures. When the

pressure is removed the material will expand during drying and crack the ware. Coke, coal and peat have this objection but not to as great an extent as the former materials mentioned.

PURPOSE

The expensive process of grinding the insulating brick and the slow production methods of hand forming led to the possibilities of dry pressing the brick. The use of the dry press would increase production, give greater uniformity to the brick and materially reduce grinding if not eliminating it entirely.

Since present day combustives were either impractical or else impossible to use, some new material must be found. Three materials presented themselves, naphthalene, sulfur and petroleum coke. The former for its low melting temperature and the latter two for their complete volatility at high temperatures and low if not negligible resiliency.

Due to the high fire hazard of the naphthalene in the dryer it was dropped as a possibility after preliminary investigations.

Materials Used

The materials used were clays. "Empire" fire clay from A.P. Green Firebrick Company in Mexico, Mo.; "Dark Barbee Sagger Clay" from the Kentucky-Tennessee clay mining company, Marfield, Kentucky; "Wyodate" bentonite from Wyoming and grog made from A.P. Green high heat duty firebrick.

The sulfur used was of the roll sulfur type and cost \$2.85 per hundred pounds. The coke was a petroleum coke which is made by the Shell Petroleum Co. No information on the cost was available as the material used was sent as a sample to the Missouri School of Mines.

Sizing of Materials

The primary crushing was performed in a jaw crusher and the lumps reduced to a maximum of one inch. Then the secondary crushing was done by dry panning in a three foot, convertible, wet and dry pan, running at a speed of 60 rpm. The openings in the screen plates of the dry pan were one-eighth inches in width and five inches in length. The milled material was screened through the desired mesh on a Great Western Manufacturing Company Gyratory Riddle. The mixing and tempering

of the materials was done by hand.

Forming

A hydraulic dry press made by the Hydraulic Press Manufacturing Company of Mount Gilead, Ohio, was used for forming all the brick.

The operating specifications of this press are as follows: Total pressure obtainable equivalent to 6000 psi: mold box dimensions, 20" x 9 $\frac{1}{4}$ " x 4 $\frac{1}{4}$ " : lower ram travel, 22 inches: mold box travel, 1 $\frac{1}{4}$ " : a gage in the compression line between the press proper and the electrical plunger pump indicated the pressure at all times. By manipulating the valves the desired pressure could be reached and held for any length of time.

A small scoop was used to fill the mold box to prevent segregation. A weighed amount of the tempered mix was used for each brick. Kerosene was used to lubricate the mold. The pressure used was varied until the right pressure was obtained: approximately 400 psi.

Method of Investigation

Trial # 1

The body used had the following composition:

50% Empire thru # 10 on # 20

10% Empire thru # 20

30% Grog thru # 20 on # 40

5% Sagger Clay thru # 10 on # 20

5% Sagger Clay thru # 20

The combustible to be used was sulfur, the amount and grain size being varied in order to find the correct proportions. Since sulphur melts at 110° C. it was believed that the sulphur could be melted out and recovered in the drier.

The grain sizes of sulphur used were thru # 50, thru # 40 on # 80 and thru # 10 on 40. 10%, 15% and 20% of each grain size were added to the clay body.

The sulphur was added to the clay, 8% water added and the brick formed using 500 psi pressure. The formed bricks were placed in the dryer and the temperature raised and held at 110° C.

The sulphur in burning out cracked the brick during drying. This was probably due to the fact

that the temperature of the dryer could not be controlled close enough to allow the sulfur to melt out but volatilized it inside the brick with an accompanying increase in volume which due to the inelasticity of the brick at this stage caused it to crack.

Another objectional feature of sulphur is its high specific gravity, (2.6). In order to obtain an insulating brick with the desired pore space as high as 80% sulphur would have to be used. This would materially increase the cost of the brick if it were possible to make one. Due to these facts no farther investigations using sulphur were made.

The body used did not have enough bond in the dry state. This was due to two facts. First, there was not enough plastic clay present and second the water did not wet the sulphur which caused a lack of bond between the clay and sulphur. During the drying of these bricks, those containing the finer grain sizes of sulphur cracked the worst, so the conclusion was drawn that the coarser grain size for the combustible was better.

Trial # 2

Due to the need for more bond in the brick the body was changed to the following composition:

40% Empire clay thru # 10 on # 20 mesh

10% Empire clay thru # 20 mesh

30% Grog thru # 20 mesh

10% Sagger clay thru # 10 on # 20

10% Sagger clay thru # 20

The combustible used was petroleum coke which is one of the by products of petroleum refineries. This coke as received was very moist, being saturated with the heavier oils. By placing the coke in a drier the oils could be volatilized off leaving a dry, coke body. The grain size of the coke used was thru # 10 and thru # 20 mesh screens. The percentages of grain sizes tried were 40, 50, 60, and 70% coke. The body was mixed and tempered with 8% water with little if any increase in the plasticity of the body. At this point in order to provide sufficient bond in the dry state, 2% Wyoming Bentonite was added. This is an extremely plastic clay and small amounts will impart considerable plasticity to a clay body. The bricks were formed using 400 psi pressure.

The bricks were air dried and then dried for two days at 110° C in an electric dryer. After drying the bricks were fired to cone 12 in ten hours and fifteen minutes in an electric glo-bar furnace.

The results obtained were rather encouraging as none of the bricks cracked. Due to the speed of firing and the lack of proper oxidizing conditions in the kiln, however, the carbon did not entirely burn out but remained in the center portion of the brick as a black core.

Those bricks containing the 40 and 50% carbon were good on the outside, they showed negligible shrinkage and were fairly strong. The bricks containing the 60 and 70% coke had no mechanical strength and crumbled between the fingers when picked up.

The open pores of the brick containing the coke thru # 10 mesh seemed to be too large. The brick containing the coke thru # 20 mesh seemed about right.

Trial # 3

After careful study it was decided that in order to successfully remove the carbon one of two things must be provided. Either a very long firing period with a long oxidation period or

supply air to the kiln for oxidizing the carbon. At that particular time the former idea was impractical as we could not run a three or four day firing schedule and attend our classes. The latter method was adopted. A silica tube 1" in diameter was placed upright in one end of the kiln in such a manner as to direct a stream of air on to the floor of the kiln. One end was attached to a line containing compressed air. A mercury manometer was used to measure the pressure of the air coming into the kiln.

The body used was:

50% Empire clay thru # 20

20% Saggar clay thru " 20

30% Grog clay thru # 20

20% Bentonite clay thru # 100

Two bricks were made using coke thru # 20 mesh and containing 40 and 50% coke. The bricks were mixed using 8% water and formed under a 500psi pressure. The bricks were air dried and then dried in an electric drier for 48 hours at 110° C.

The bricks were set in the kiln, the air started and the kiln fired at a rather rapid rate.

At about 700° C. cracks were noted running longitudinally down the $9\frac{1}{2} \times 2\frac{1}{2}$ " face. The kiln was shut down and the following observations made. The forced draft caused the formation of CO_2 which due to the small grain size of the carbon could not be carried away as fast as it was being formed. The result was an expansion of the brick from the inside which caused a long crack down the longitudinal axis of the brick. The brick was also fired too fast which caused a more rapid evolution of the CO_2 .

Trial # 4

This time it was decided to open up the body using grog thru # 10 and to use coke thru # 10 mesh. The same method of forced draft was used in the kiln with a lower draft and a slower firing schedule. The body used had the following composition:

- 40% Empire clay thru # 10 on # 20 mesh
- 10% Empire clay thru # 20 mesh
- 30% Grog thru # 10 mesh
- 10% Sagger clay thru # 10 and # 20 mesh
- 10% Sagger clay thru # 20 mesh
- 2% Bentonite clay.

The bricks were formed using 40 and 50% coke, 8% water and a pressure of 400 psi. They were

dried in the same manner as the other samples and then placed in the kiln. The fire was started using a very small amount of forced draft. At about 600° C. cracks again appeared running down the longitudinal axis. At this point the idea of forced draft was abandoned. It was also thought that perhaps some of the cracking was due to pressure cracking from forming the brick under too high a pressure. Due to the difficulty in keeping the gas-bar furnace heating at a slow rate of speed under 600° C. it was decided to fire the next trial in an oil fired kiln.

Trial # 5

In this trial the following body composition was used.

50% Empire Clay thru # 20 mesh

30% Saggar clay thru # 20 mesh

30% Grog thru " 40 mesh

~~5%~~ Bentonite thru # 100 mesh

The coke used was screened thru # 14 mesh and on # 60 mesh. Only one brick was made which contained 50% coke which was thought to be the best

mixture. The brick was formed in the same manner with the exception that only a pressure of ²⁵⁰ psi was used. After drying in the usual manner the brick was fired in a four burner oil kiln to cone 12 in 48 hours.

The brick again cracked but this time in fine cracks which suggested too rapid firing. The carbon at this schedule was still not completely burned out and the brick had no mechanical strength at all, crumbling under touch.

Trial 6

At this time the school purchased a G.E. control using a Leeds & Northrup indicating potentiometer which was calibrated to read temperature directly which could be connected to a wire wound furnace and the temperature held constant at any heat for the desired length of time. It was decided to form a brick and fire it at a very slow schedule in a wire wound furnace up to 1000° C. After this the brick could be cooled and fired at the rather rapid schedule of the gas-bar furnace.

It was also thought that a better bond could be had by forming a slip with the bentonite and tempering water. The theory being that the bentonite

would coat each individual clay grain and thus produce a better bond. The body used for this trial was:

50% Empire clay thru # 20
25% Sagger clay thru # 20
25% Grog thru # 40
2% Bentonite thru # 100

The brick was tempered by adding the bentonite and the water in the form of a slip. A pressure of 250 psi was used for forming the brick and it was dried in the usual manner.

The brick was fired in the electric furnace up to 1000° C. in 6 days using the G.E. controller. After cooling the brick was placed in the glo-bar furnace and fired to cone 12 in 9 hours.

The results were very good. The brick showed no cracking or black-coring and was strong enough to be handled with a reasonable amount of caution. The brick had the following physical properties:

Plastic length = 9.75"

Plastic width = 4.75"

Plastic weight = 2000 grams

Fired length = 9.45 "

Fired width = 4.60 "

Fired Depth = 1.75"

Fired Wt. = 2.2#

$$\% \text{ total linear shrinkage} = \frac{\text{dry length} - \text{fired length}}{\text{dry length}} \times 100$$

$$\frac{9.75 - 9.45}{9.75} \times 100 = \frac{.35}{9.75} \times 100 = 3.6\% \text{ total shrinkage}$$

$$\text{Bulk density} = \frac{\text{weight}}{\text{volume}} = \frac{9.45 \times 1728}{9.45 \times 4.60 \times 1.75} = 45.3 \frac{\#}{\text{cu. ft.}}$$

The shrinkage was very low which would probably eliminate the grinding process. The bulk density compares very favorable with other commercial brands which have bulk densities as high as 51.3 #/ cu. ft.

CONCLUSION

From the material obtained during the experimentation it would seem very evident that insulating brick can be successfully dry pressed with the use of petroleum coke as the combustible. The data showed that the total shrinkage was low enough to probably eliminate or else materially reduce the grinding to size the brick.

It is the writers firm belief that within the next few months that a commercial dry press method for the manufacture of insulating bricks may be developed.

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