

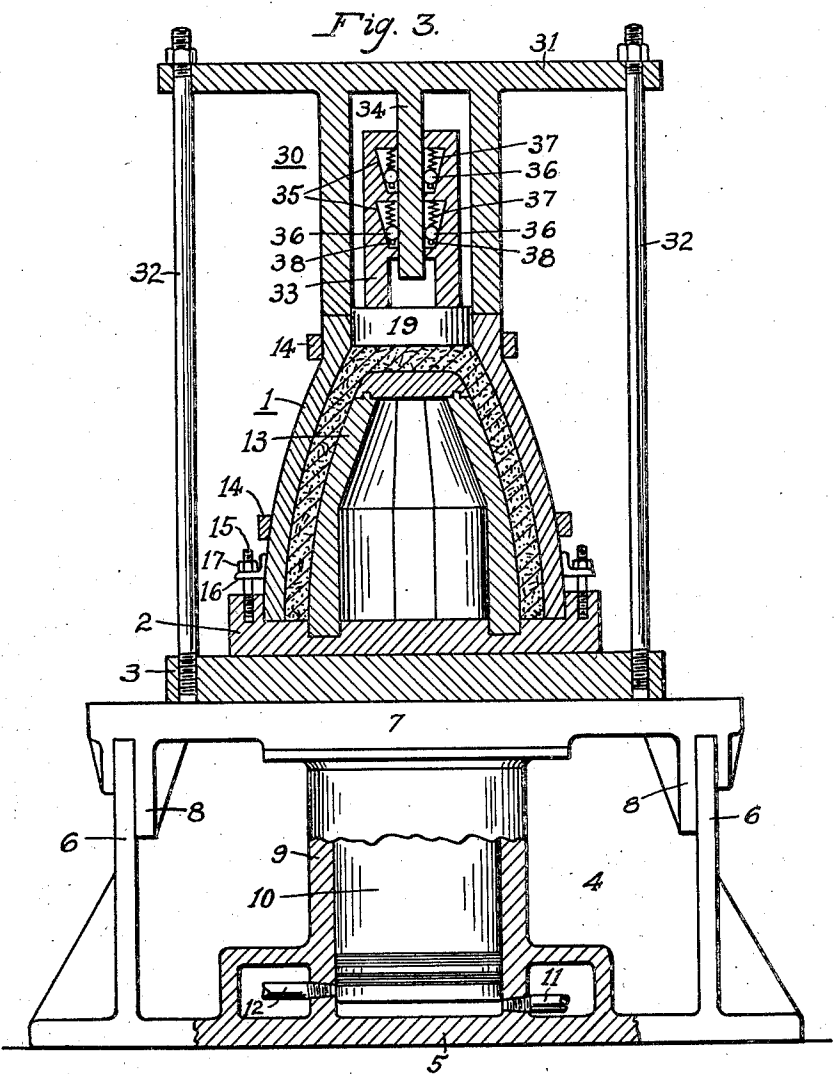
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FORMING REFRACTORY ARTICLES

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FORMING REFRACTORY ARTICLES

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The present invention relates to the forming of refractory articles, and is especially applicable to crucibles and other hollow articles having walls which are utilized for the conduction of heat, and to their manufacture. The present application is a division of my copending application, Serial No. 450,160 filed May 6, 1930. The latter application is a continuation in part of my copending application Serial No. 210,293 filed August 3, 1927.

It is desirable in some types of refractory articles, such as crucibles or the like, that the material be as densely compacted as possible, and that the particles of the material rather than being pressed together in a haphazard or random arrangement, be so arranged or oriented that they occupy the greatest possible proportion of available space. The high density thus obtained not only renders the article more impermeable to gases and slags, but also increases its strength. There are also instances where it is advantageous to control the thermal conductivity of the article in a certain preferred direction. In the case of a crucible, it is desirable that the transmission of heat through the wall is as great as possible, but the conduction of heat toward the top or lip portion of the crucible should be minimized, since the useful portion of the heat flow is limited to that passing through the wall of the crucible to the charge. The optimum condition is represented by a crucible in which the thermal conductivity through the wall greatly exceeds that in the vertical direction axially of the crucible.

Crucibles are usually manufactured by a spinning process, and although there may be considerable orientation of the flake-like particles in the crucible mix during the spinning operation, the structure tends to be laminated radially and the orientation of the particles is exactly the opposite from that necessary to give an increased heat conductivity through the crucible wall. The orientation of the flake-like particles lying with their planes parallel to that of the wall instead of increasing the thermal conductivity in a direction through the wall, may actually act as a detriment as far as the thermal properties of the crucible are concerned.

In the process described in the present application, the refractory mix is consolidated by jolting in a direction parallel to the vertical axis of the crucible. If the mix used in making a crucible by

this process contains particles of a flaky or plate-like character, these particles so orient themselves during the jolting that they lie generally in planes which are approximately perpendicular to the direction of the jolt. The introduction of materials in which the flaky characteristic is pronounced has been found to give very unusual properties to the resulting crucible. As an example, if a material such as flake graphite is incorporated into the mix and the mix consolidated by the jolting process as herein described, a crucible is produced in which the thermal conductivity radially through the wall greatly exceeds that axially of the crucible, i. e., in the vertical direction.

The extent to which the directional thermal conductivity may be controlled depends upon the properties of the individual particles used in the mix. In the case of natural flake graphite, which combines both flakiness and comparatively high thermal conductivity, heat can flow more readily in a direction parallel to the planes of the flakes than in other directions. Measurements made with mixes composed principally of flake graphite have shown a thermal conductivity at right angles to the direction of jolting several times that parallel to the direction of jolting, and even as high as five and six times as great. Even when the amount of flake graphite is decreased to the extent where it constitutes a minor but material portion of the mix, the directional thermal conductivity effect is still pronounced. The use of flaky graphite greatly increases the directional thermal conductivity effect over that which can be obtained when granular or non-flaky particles are used.

The orientation of the flaky particles in an approximately horizontal position produces a crucible which differs from the usual spun product in properties other than thermal conductivity. The orientation of the flakes at right angles to the wall surface exposes only their edges at the surface, whereas with the reverse orientation obtained in the spinning process the entire surface of the flake is exposed. The material in which the flakes are horizontally oriented therefore does not flake or scale off as readily under abrasion. In a crucible where the glaze is worn off in use, the rate of oxidation will also be effected to a certain degree by the orientation of the particles

and the ease with which the protective scale will flake off.

The electrical properties of an article made from a mix containing flake graphite show pronounced directional effects when the mix is consolidated by the process herein described. The magnitude of the effect may be controlled through a wide range by varying the amount of comparatively non-conducting material incorporated into the mix. For example, the ratio of the electrical conductivity in a direction perpendicular to that of jolting in comparison with that parallel to the direction of jolting is approximately 2 to 1 in the case of a mix composed of flaky particles of graphite and a carbonaceous binder such as tar or pitch which on carbonization becomes a fairly good electrical conductor. In a mix containing 70% flake natural graphite and 30% clay, where the bonding material is comparatively non-conducting, the ratio was approximately 6 to 1. If the nature of the mix is known, the measurement of the electrical conductivity of the article in different directions will also give some idea of the relative thermal conductivities in these directions.

In order to obtain the maximum degree of orientation and at the same time secure the densest possible consolidation of the mix, it is preferable to jolt the mix in combination with a follower which will confine the mix to the minimum volume which it reaches during the jolting operation. This volume can be retained by the application of hydraulic pressure to the mix in combination with an instantaneously acting check valve to prevent recession of the follower, or by other quick acting means, as will be hereinafter described. The absolute prevention of the rebound of the follower and mix during jolting accentuates the properties obtainable through the orientation of the flakes by jolting.

The accompanying drawings illustrate a preferred embodiment of the apparatus which may be used in jolting an article illustrated as a crucible.

In the drawings:

Figure 1 is a vertical section through a crucible forming mold mounted on a jolting machine; Figure 2 is a horizontal section on the line II—II of Figure 1; and

Figure 3 is a vertical section through a modified form of the apparatus, employing a mechanical clutch to check the rebound of the jolt.

In making a crucible, a suitable composition, consisting principally of or containing a material proportion of flaky material, is thoroughly mixed and placed in the mold which is mounted on the machine used for jolting. The usual "plumbago" mixes may be used, which consist of flaky graphite and clay binder, or mixes may be used consisting of natural flake graphite, silicon carbide, a mineral fluxing material, and a bituminous binder. In case a bituminous binder such as tar or pitch is used, the mix is heated in a steam jacketed kettle or other suitable heating apparatus to a temperature where the binder is sufficiently fluid to be thoroughly incorporated in the mix and to coat all of the particles.

The mixture, which is plastic, is preferably fed into the mold while the latter is jolted because by so doing the successive additions of mix are caused to conform to the mold cavity and the compacting process takes place more effectively than when the entire amount of mix is added before the jolting action is commenced.

Referring to the embodiment of the invention illustrated in Figures 1 and 2, reference numeral

1 indicates generally a crucible forming mold, made preferably of metal and constructed in sections. The mold rests on a bottom plate 2. The mold and bottom plate are carried by a base plate 3 adapted to be placed on a jolting machine, indicated generally by reference numeral 4. The jolting machine 4 comprises a base 5 having sides 6 and a table 7 provided with downwardly extending guides 8 which telescopically receive the upper ends of the sides 6. The base is provided with a cylinder 9 which receives a piston 10 attached to the table 7. For raising the table fluid under pressure is supplied to the cylinder through a pipe 11, and to permit the table to fall and jolt the mold the fluid is exhausted from the cylinder through a pipe 12. While the jolting machine above described is illustrated, it is to be understood that any other suitable type of jolting machine may be employed.

Supported within the mold 1 is a sectional core 13 shaped to give proper form and size to the interior of the crucible. The core 13 is assembled on the bottom plate 2 and the mold sections 1 are put in place and retaining rings 14 are forced around the outside. The mold sections 1 are clamped to the bottom plate 2 by means of bolts 15 engaging lugs 16 on the mold sections and provided with nuts 17.

After the mold is assembled the crucible forming mix 18 is fed into the open top of the mold, which is jolted during the introduction of the mix. The upper part of the mold forms a cylindrical chamber to receive the uncompacted mix, and after a sufficient quantity of mix has been added, a weight or follower 19 is introduced into this cylinder. The follower functions to confine the mix and to act as an aid in its consolidation.

After the follower 19 has been inserted, a hydraulic head, indicated generally by reference numeral 20, is clamped to the mold by means of bolts 21 extending from the base plate 3 and provided with clamping nuts 22. A hydraulic pressure is then applied to the top of the follower or ram 19 by introducing water, oil, or other liquid under pressure through a pipe connection 23, and the air is removed through a pipe 24 until the space 25 of the cylinder above the follower 19 is filled with liquid. The valve 26 is then closed and the mix is jolted until consolidation is completed.

A high hydraulic pressure is not required, and the pressure available from the usual city supply means under, say, a pressure of fifty to one hundred pounds per square inch, is sufficient. The liquid, which may be of water or oil, is supplied under hydraulic pressure through the pipe 23, which is provided with a check valve 27, which permits the liquid to flow toward the chamber 25 but not in the reverse direction. It is found that a ball check valve of the type illustrated operates satisfactorily and is practically instantaneous in action, allowing the water to flow in one direction but instantaneously checking any tendency to flow in the reverse direction.

In the jolting operation the principal problem consists in the prevention of the rebound of both the follower and the mix. The jolting action takes place with considerable violence, and as the downward velocity of the mold, the contained mix and follower is checked by the jolt, the particles are for an instant compacted into a smaller volume, but owing to the tendency of the weight and the mix to rebound, this minimum volume is not ordinarily retained unless some instantaneously acting means be employed to check

the rebound of the follower and mix. The hydraulic arrangement of the type illustrated has been found to be a satisfactory means for instantaneously checking the tendency to rebound.

The liquid under pressure follows up the downward movement of the follower or ram 19 at the moment of impact, but because of the check valve 27 it instantaneously acts to prevent rebound after the jolting impact.

The rebound of the follower would affect not only the density of the resulting article, but also the degree of orientation of the flaky particles. The degree to which the particles orient themselves in a given time depends upon both the violence of the jolt and the freedom of movement of the particles in the mix. If hydraulic pressure is used in combination with an instantaneously acting check valve, the hydraulic pressure need not be high, and the freedom of movement of the particles will be correspondingly greater than when extremely high consolidating pressures are used; at the same time the decrease in volume produced with each re-arrangement of the particles under pressure is preserved by the positive check of the rebound. By positively checking the rebound and by preserving the minimum volume which the mix attains with each individual jolt, a product having an exceptional density and a high degree of orientation of the flaky particles is made possible.

Although the hydraulic head of the check valve is preferred for checking the rebound, mechanical devices may be employed which will permit the follower to follow up the mix during consolidation, but which will instantaneously check rebound thereof. Such a mechanical device is illustrated in Figure 3. In Figure 3 the mold and jolting table are similar to that illustrated in Figure 1. A mechanical clutch of the so-called "Horton" type is indicated generally by reference numeral 30. The clutch 30 is carried by a head 31 held by rods 32 to the base plate 3. As illustrated, the clutch consists of a head 33 carried by the follower 19 and receiving a bar 34. The bar 34 projects downwardly from the yoke 31. The bar 34 is of hardened steel and is of square or rectangular cross-section having smooth sides. The head 33 has integrally inclined hardened wedge faces 35. Between the faces of the bar 34 and the inclined wedge faces 35 are placed hardened steel rollers 36. The rollers will be naturally held downward by gravity, although springs 37 may be used to additionally urge them downward. Fingers 38 may be provided for holding the rollers 36 in released position when desired.

This type of clutch is commonly known as the "Horton" clutch, and its operation is as follows:

The wedging rollers 36 permit the head 33 to move downwardly with respect to the bar 34, but instantaneously prevent upward movement with respect thereto. When the downward movement of the mold and its attached parts is suddenly arrested the mix which is being consolidated and the plunger or follower 19 continue to move downwardly because of their inertia. The inertia of the rollers 36 will also tend to move them downwardly into tight wedging engagement so that they will instantaneously lock the head 33 and prevent any upward rebound.

The instantaneously acting hydraulic check or Horton clutch as applied to a jolting machine is described and claimed in the copending application of Simeon M. McAnulty, Serial No. 390,949 filed September 7, 1929, which has become Patent No. 1,818,234.

The jolting is continued until the desired consolidation of the mix is attained, after which the hydraulic follower or Horton clutch is released and removed, the mold disassembled and the crucible removed and fired in the usual way. When a thermo-plastic mix is employed, such as that having a tar or pitch binder, the mold may be heated during the jolting operation and then preferably cooled before the crucible is removed.

The crucible is preferably jolted in an inverted position, as illustrated. When formed in such inverted position the lip or upper portions of the crucible wall, which are lowermost in the inverted jolted position, are given an increased density and strength which is desired in the lip portion of the crucible.

In order to obtain the pronounced directional heat conductivity and other advantages, such as diminution of scaling, the crucible mix should consist principally of or contain a material portion of particles of a pronounced flaky character. Natural graphite is such a material as it is made up of flakes or laminae. A crucible made of the usual plumbago mixes in which the flake natural graphite is incorporated with from 25% to 50% clay, shows these desirable characteristics to a pronounced degree.

However, other crucible mixes may be employed, as for example, mixes which contain graphite, a granular refractory, a flux and a plastic carbonaceous binder. An example of such a mix is one containing

	Per cent
Natural flake graphite	21
Crushed silicon carbide grain	45
Flint	11
Borax as a mineral flux	5
Tar as a plastic carbonaceous binder	18

The silicon carbide may be the ordinary crushed variety which contains both flakes and irregular particles as formed by the ordinary crushing operation. A silicon carbide, however, which consists principally of flaky or plate-like particles may be obtained by subjecting the ordinary crushed silicon carbide to an air separation process, which separates the markedly flaky particles from the rest of the material. By incorporating such air-winnowed silicon carbide consisting principally of the flaky or plate-like particles, the selective heat conductivity of the crucible can be further increased.

Another suitable mix consists of about 58% of silicon carbide ground and screened through a screen of forty meshes to the inch, about 21% natural crystalline flake graphite, about 5% of mineral flux such as a low melting clay, borax or feldspar, and about 16% of a plastic carbonaceous or bituminous binder such as a batch preferably having a density of about 17½ Bé. at 150° centigrade. When mixes containing such thermoplastic binders are used they are intimately mixed and heated, as for example, in a steam jacketed mixer, to a temperature such that the binder is fluid enough to be thoroughly incorporated with and coat all of the particles before the mixture is introduced to the jolting mold. Preferably the mold is also heated before and during the jolting operation. After the jolting operation the mold is preferably cooled before it is disassembled and the crucible removed therefrom.

The high density of crucibles as made by the present process is shown by a determination of the ratio of the apparent density to the true spe-

cific gravity of mix, from which the percentage of voids or porosity may be calculated. In the following table the density of crucibles made by the present jolting process is compared with that of two makes of crucibles made by the spinning process. The spun crucibles were of two well-known American makes and were a high grade product.

	Apparent density	True specific gravity	Percent porosity
Jolted crucible.....	1.99	2.67	25.3
Spun crucible make #1.....	1.83	2.69	31.9
Spun crucible make #2.....	1.71	2.65	35.5

The above results were obtained with ordinary crucible mixes in order that the values would be comparative with crucibles made by other processes. If a judicious selection of particle sizes is made and the various proportions of fine, medium and coarse particles so adjusted that the solid material fills the greatest possible proportion of available space, the porosity can be decreased to between 10 and 15% when the present process of jolting is employed.

The pronounced directional thermal conductivity through the wall of the crucible in comparison with that in a vertical direction, is shown by the following measurements with a crucible made of an ordinary plumbago mix containing 70% of natural flake graphite bonded with clay.

	Thermal conductivity Cal/cm. ² / °C./sec.
Through the wall (perpendicular to direction of jolting).....	.0464
Parallel to the wall (parallel to direction of jolting).....	.0107

The thermal conductivity through the wall is between four and five times that in a vertical direction.

While the present process is especially adapted for use with mixes containing flaky graphite, it will be understood that other particles of pronouncedly flaky form may be substituted for the graphite, and that a directional thermal conductivity effect may be obtained by the use of particles of this character other than by graphite flakes. Examples of flaky particles which may be used in refractory mixes are flaky fused aluminum oxide, flake anthracite, flaky silicon carbide, or flaky materials which occur in nature, such as mica, some kinds of gypsum, talc, slate, etc. By a flaky material is meant a material in which the particles have two dimensions considerably greater than the third dimension, and hence may be classified as flakes or plates in contrast with the irregular shaped particles produced by the ordinary crushing operations on a substance such as coke or a non-flaky mineral.

It is possible to separate the flaky constituents from materials in which the particles are generally irregular but not all of the flaky form. For example, if fused aluminum oxide is subjected to an air stream, the air will tend to remove the thinner or more flaky or plate-like particles from the particles which are generally irregularly shaped, but which are not flaky. Similarly, flaky silicon carbide may be obtained by air separation. Such flaky fused aluminum oxide or flaky silicon carbide differs from the usual crushed fused aluminum oxide or silicon carbide

in that the particles are nearly all of a flaky or plate-like form having a thickness considerably less than the length or width of the plate or flakes.

There are also a number of substances having a characteristic splinter or needle-like form which can be oriented by the above described process so that the particles are oriented in a preferred direction, and which, when so oriented as by the jolting operation, can impart the pronounced directional thermal conductivity effect.

The orientation of flaky particles and particularly flaky graphite by the process here described, has also been found advantageous in the case of articles other than crucibles, as, for example, muffles, where it is desired to have maximum heat conductivity through the wall.

While I have specifically described the preferred embodiments of my invention, it is to be understood that the invention is not so limited, but may be otherwise embodied and practiced within the scope of the following claims.

I claim:

1. The process of forming a refractory article, which comprises forming a refractory mix containing particles of pronounced flaky character, placing the mix in a mold and jolting it so as to consolidate the mix and orient the flaky particles in a direction at right angles to the direction of jolting.

2. The process of forming refractory articles which comprises forming a refractory mix containing natural flake graphite, placing the mix in a mold and jolting it so as to consolidate the mix and orient the graphite flakes in a direction at right angles to the direction of jolting.

3. The process of making hollow articles such as crucibles or the like, which comprises forming a mix containing particles of pronounced flaky character, placing the mix in a mold and jolting it so as to consolidate the mix and orient the flaky particles in a direction at right angles to the direction of jolting.

4. The process of molding a crucible, which comprises forming a crucible mix containing particles of pronounced flaky character, placing the mix in a mold and jolting it axially of the crucible to be formed so as to cause the flaky particles to lie in planes approximately at right angles to the side walls of the crucible.

5. In a method of making crucibles, the steps of filling a mold, representing an inverted crucible, with loose crucible material so that crucible material before compacting extends above the portion of the mold forming the bottom of the inverted crucible, applying pressure to the upper end of said material, and simultaneously jarring said mold and contents to compact the crucible material.

6. In a method of making crucibles the steps of assembling a core within a mold casing, filling the chamber of the casing with a carbonaceous crucible material bonded with a partially volatile hydrocarbon binder, said crucible material being loose and lacking in free flowing properties, and jarring the casing and its contents while applying pressure to said contents to compact the material to the desired density about the core.

7. A method of making crucibles, including filling a mold, representing an inverted crucible, with loose crucible material including a carbonaceous material bonded with a partially volatile hydrocarbon binder, said crucible material extending above the portion of the mold forming the bottom of the inverted crucible, applying a

weight to the top of said material, and simultaneously jarring said mold and contents to compact said crucible material, and heating the crucible to harden the same.

5 8. The process of molding a refractory crucible comprising filling a crucible mold with an intimate plastic mix containing graphite and a carbonaceous binder, and applying a weight to the top of the mixture and jolting the filled mold in a direction parallel to the axis of the crucible to compact the mixture, the portion of the mold cavity forming the lip of the crucible being lowermost during the jolting operation.

10 9. The process of molding a refractory crucible, comprising filling a crucible mold with an intimate plastic mixture containing graphite and a carbonaceous binder, and applying a weight to the top of the mixture and jolting the filled mold in a direction such that the inertia of the particles of the mixture and the inertia of the weight when the falling movement of the mold is arrested during jolting, will tend to compact the mixture toward the lowermost part of the mold cavity, the crucible mold being inverted during the jolting so that the portion of the mold cavity forming the lip of the crucible is lowermost during the jolting operation.

25 10. The process of molding a refractory crucible having increased density, mechanical strength and thermal conductivity, comprising filling a

crucible mold with an intimate plastic mixture containing graphite and a carbonaceous binder, and applying a weight to the top of the mixture and jolting the filled mold to compact the mixture and render it dense, the portion of the mold cavity forming the lip of the crucible being lowermost during the jolting operation. 5

11. The process of molding a refractory crucible comprising filling a crucible mold with a loose plastic mixture of crucible material, and applying a weight to the top of the mixture and jolting the filled mold in a direction such that the inertia of the particles of the mixture and the inertia of the weight when the falling movement of the mold is arrested during jolting, will tend to compact the mixture toward the lowermost part of the mold cavity, the crucible mold being inverted during the jolting so that the portion of the mold cavity forming the lip of the crucible is lowermost during the jolting operation. 10 15 20

12. The process of molding a refractory crucible, comprising filling a crucible mold with a plastic mix of crucible material, applying consolidating pressure to the top of the mix, and jolting the filled mold in a direction parallel to the axis of the crucible to compact the mix, the portion of the mold cavity forming the lip of the crucible being lowermost during the jolting operation. 25

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