Feb. 16, 1943. P. S. DENNING 2,311,588

METHOD OF MAKING REFRACTORY INSULATING BLOCKS

Filed June 14, 1940

Fig. 1

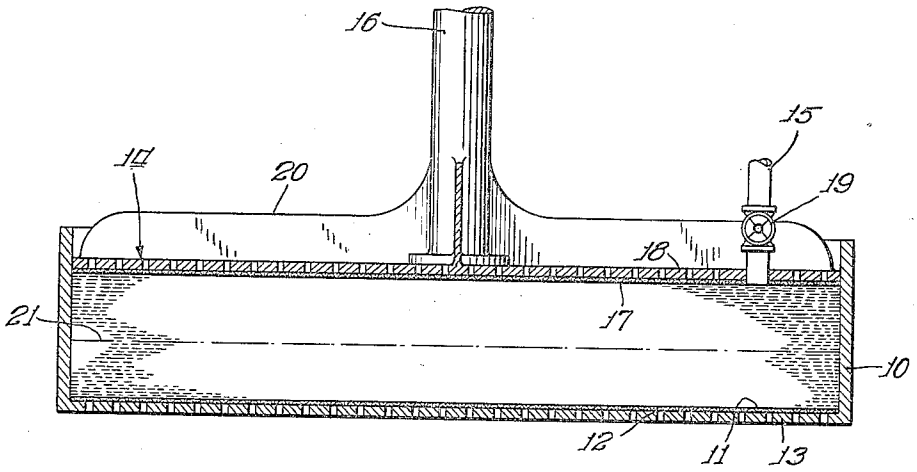


Fig. 2

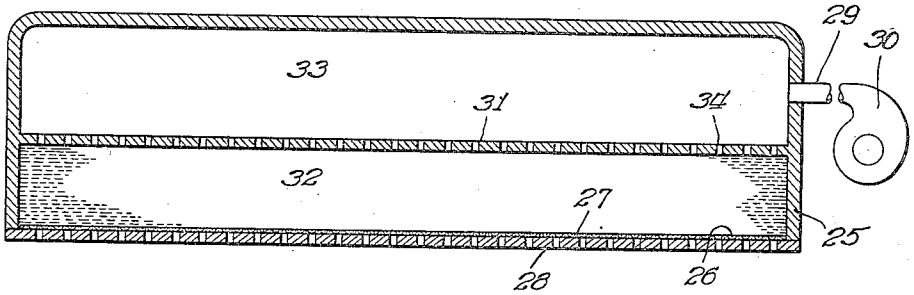
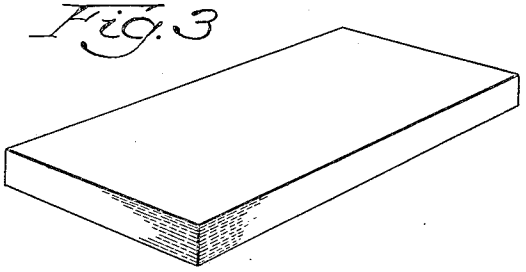


Fig. 3



Inventor:
Paul S. Denning
By: Roland C. Rehm
Atty.

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METHOD OF MAKING REFRACTORY INSULATING BLOCKS

Paul S. Denning, Joliet, Ill., assignor to F. E. Schundler & Co., Inc., Joliet, Ill., a corporation of Illinois

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6 Claims. (Cl. 18—48)

This invention relates to the manufacture of molded insulating material and, among other objects, aims to provide relatively inexpensive refractory insulation of improved insulating efficiency.

The nature of the invention may be readily understood by reference to an exemplary insulating material and its method of manufacture embodying the invention and illustrated in the accompanying drawing.

In said drawing:

Fig. 1 is a sectional elevation of molding apparatus;

Fig. 2 is a similar sectional elevation of a different form of molding apparatus; and

Fig. 3 is a perspective view of a typical molded insulation.

Most thermal insulating materials efficient at low temperatures cannot be used at temperatures substantially in excess of 600° F. either because they cannot physically withstand such temperatures or because they lose their insulating efficiency. Even such an efficient insulator as 85% magnesia is not satisfactory for temperatures above 600° F. Diatomaceaus earth bonded by clay materials or the like is one of the very few insulating materials having a reasonably satisfactory insulating efficiency at temperatures in excess of 600° F. This material, however, is relatively heavy and expensive, and for many purposes has inadequate strength. Its insulating efficiency, i. e. K factor, is about .72 at 500° F.; and .94 at 1200° F. The K factor is defined as the amount of heat in B. t. u. per hour passing through one square foot area of material when the temperature gradient is one degree Fahrenheit per inch of thickness. In this connection it should be borne in mind that the K factor varies somewhat with the particular apparatus used for determining it. At the present time there is considerable disagreement as to the most reliable apparatus for this purpose.

Another refractory insulating material is exfoliated vermiculite. This is produced in granular form and is an alteration product of certain micaceous minerals which exfoliate or expand to many times their original size upon the application of heat. The expression "exfoliated vermiculite" is here used to designate micaceous minerals, including that specifically named vermiculite, capable of exfoliating or expanding upon the application of heat. A granule of the material is characterized by a multitude of slightly separated polished laminae and has a very high insulating efficiency at high temperatures in a direction perpendicular to the laminae but not parallel thereto. To endow a piece of molded insulating material with equal insulating efficiency, it is important that a large majority of the laminae lie substantially parallel to the face of the insulation. In this connection it should be understood that a porous material which is an efficient insulator at low temperatures may be a poor one at high temperatures where most of the heat energy is in the form of radiant heat against which mere air spaces or voids are not effective. Radiant heat increases as the cube (or higher) of the temperature. Therefore, at 1200° F. the radiant heat is eight or more times that at 600° F. Exfoliated vermiculite has proved to be a very efficient insulator against radiant heat due to the multitude of slightly separated heat reflecting laminae lying transversely to the path of the heat rays.

It is also important that the vermiculite or other micaceous material be bonded together to provide adequate strength without substantially impairing either the refractory properties of the insulation or its insulating efficiency. The bonding cement must be properly distributed and strong to minimize the amount required and the resulting weight of the product. In the illustrative material the vermiculite is bonded together with calcium silicate in conjunction with the mechanical arrangement of the vermiculite flakes. Calcium silicate is a refractory cement capable of withstanding 2700° F.

While the present insulating material and its method of manufacture may technically be called improvements over those disclosed in Russ Patent 1,812,306 and in my copending application, Serial No. 187,038, of which this application is a continuation in part, the material utilizes commercially new principles in high temperature insulation. Insulation against radiant heat is provided by the multitude of substantially parallel polished laminae lying transversely to the path of the heat rays. These laminae are microscopically thin and each is more effective to retard heat rays than cell walls in other types of insulation. Moreover the number of laminae which must be traversed per inch of thickness greatly exceeds the number of cell walls per inch in other forms of insulation. Large pores or voids are eliminated both because they are not efficient insulators and because they are sources of weakness. Reduction in efficiency through shrinkage and cracking at high temperatures is minimized by elimination so far as possible of all appreciable voids. So far as possible the

spaces in the material are confined to the microscopically thin spaces between the individual laminae of the flakes of vermiculite.

In the Russ patent generally the same materials are employed as in the present material but the granules of micaceous material (there referred to as exfoliated jefferisite) are not arranged or oriented and so much diatomaceous earth and lime are used as to reduce the insulating efficiency of the material substantially below that of bonded diatomaceous earth. In other words, the insulating efficiency of the Russ material is not high enough to make the material competitive with other known refractory insulation such as bonded diatomaceous earth. Moreover, its weight is so high as to preclude its use where light weight is essential.

The illustrative method of manufacture produces a substantially higher orientation of the vermiculite laminae into positions parallel with those of the insulation than has heretofore been possible and also avoids formation of cracks in the material developed during the dewatering process.

In the present material the exfoliated vermiculite is prepared in flake form. These flakes though generally comprising more than a single laminae are nevertheless quite thin as compared to their face area. Preferably the vermiculite is first exfoliated to produce the conventional granules which are generally cubical in shape though, of course, many such granules break up along the lines of cleavage into smaller thinner granules. After exfoliation, the granules are treated to subdivide them along their lines of cleavage into a plurality of relatively thin flakes. Such subdivision may be effected either by skiving the granules so as to split them along their lines of cleavage or to grind them to smaller size during which grinding process the vermiculite is split up along the cleavage planes into relatively thin flakes. The vermiculite may advantageously be exfoliated at temperatures somewhat higher than normal to make the granules friable (a condition not ordinarily desired) to facilitate splitting of the granules during grinding. The grinding process, of course, produces flakes which are much smaller in area than flakes produced by a skiving process. Nevertheless the flakes, though very thin, generally comprise a plurality of miscroscopically thin laminae which have been slightly separated by exfoliation. The important characteristic of the material is the thinness of the flakes in relation to their area.

The grinding treatment generally subdivides the material so that from 30 to 70 per cent of it will pass through a 65 mesh screen. Such material, though quite fine, is still in the form of flakes whose face areas are very much larger than their thickness. Much coarser material may advantageously be used provided the flakes are sufficiently thin. As will presently appear, it is the flake characteristic of the vermiculite which facilitates the orientation of the flakes essential to efficient insulation against radiant heat at high temperatures.

In preparing the molded material, the vermiculite flakes are mixed in slurry form with the binding material which in the present instance is formed by the reaction of hydrated lime and silica. The silica is preferably in such form as to provide a high surface area in relation to its mass. One form of such silica is diatomaceous earth whose particles present a large area in relation to their mass. Another form of silica having a still larger surface area in relation to mass is acid treated vermiculite, known on the market as lamisilite. To produce material of this character, vermiculite or similar micaceous materials are treated with acid to reduce the material to silica. One method of producing material of this character is disclosed in Guthrie et al. Patent No. 1,898,774.

The flaked exfoliated vermiculite, silica and hydrated lime are mixed with sufficient water to produce a slurry of such thinness that the flakes of vermiculite are free to orient themselves. If relatively large vermiculite flakes be used, the slurry should be thinner than for small flakes to provide ample freedom for orientation. A small amount of fiber, such as asbestos fiber, is also advantageously used both as a reinforcing and to facilitate the dewatering of the material after introduction to the mold by holding the flakes of vermiculite in slightly separated condition to make the material more free filtering. Fiber reinforcement of this character is desirable where blocks of insulation material need be partly cut through and slightly bent in applying the same. The fiber prevents complete breakage of the material where it is cut or mitered.

The aforesaid slurry is then molded or otherwise formed in such a way as to facilitate orientation of the vermiculite flakes so that they will lie transversely or perpendicularly to the direction of travel of the heat rays. For flat slab material the flakes would lie parallel to a face of the slab, and for curved insulation, such as pipe insulation, the flakes would lie perpendicular to the radius or tangential to the curved surface.

According to the illustrative process, the slurry is introduced into a mold or the like and during dewatering the vermiculite flakes are maintained in such condition that they are free progressively to orient themselves to build up a molded article of the desired thickness. During this progressive orientation care is taken to eliminate forces which might disturb the vermiculite flakes and prevent their proper orientation. Moreover during the progressive orientation and extraction of water, entrance of air to replace the water is prevented; indeed as the water leaves the material it is replaced by vermiculite flakes, with the result that relatively large voids and the weakness caused thereby are eliminated. Large pores or voids produce objectionable shrinkage and cracking. Moreover, they are not efficient in resisting transmission of radiant heat. This explains why many porous materials efficient at low temperatures where there is little radiant heat are inefficient at high temperatures where most of the heat energy is in the form of radiant heat.

In this connection it should be understood that the vermiculite flakes, presenting as they do large surfaces in relation to their mass and having a low gravity (7 to 9 pounds per cubic foot as against graphite flakes at 20 to 30 pounds per cubic foot), are particularly susceptible as regards orientation to disturbances such as currents in the slurry. Avoidance of disturbances of the vermiculite flakes during and after proper orientation may be attained by various methods of which those herein disclosed are illustrative. The method illustrated in Fig. 1 is characterized by dewatering a quantity of slurry of such volume as to contain enough solids to form a molded article of the desired thickness, while the slurry is in a quiescent state. To prevent entrance of air into the material to replace the water, the volume of the mold is progressively reduced. In fact light

pressure is maintained on the material to cause sufficient lateral shifting of the vermiculate flakes to fill voids left by the water.

The illustrative mold is represented by a rectangular box 10 having a filtering face 11, in this instance formed by a filter cloth or screen 12 supported on a perforated bottom 13. The depth of the mold is somewhat greater than that necessary for holding a volume of slurry containing enough solids to form a slab of insulation of the desired thickness. The opposite face of the mold is closed by a follower plate 14 which slidably fits inside the mold preferably with such closeness as to permit maintenance at a steady pressure and to avoid such leakage between the edges of the plate and the mold as to set up disturbing currents inside the mold.

The mold is first filled with slurry, in this case through the supply pipe 15, to the necessary depth and as the water passes through the filter screen 11 the follower plate is advanced to prevent entrance of air and with sufficient pressure to cause the solid materials to shift laterally to fill the voids left by the water. Air should preferably be eliminated from the mold before pressure is applied to prevent the disrupting action which would otherwise occur if entrapped air were compressed in the mold. Such entrapped air would either subsequently disrupt the molded article after release of pressure or would find its way out at the point of least resistance, leaving a weak spot in the material. The pressure is carefully limited to avoid such pressure as would substantially disrupt the internal structure of the material. This may readily be accomplished by advancing the plate through its plunger 16 by pneumatic or hydraulic pressures limited in intensity. In the present instance a pressure of 20 to 30 pounds per square inch on the material has proved sufficient to accomplish the desired result without damaging the material. I have found that the pressure materially reduces drying shrinkage and cracking by elimination of substantial voids and reduction of water content prior to drying. Of course the pressure also hastens the dewatering process. For a block 5½ inches in thickness the dewatering time is from 60 to 80 minutes, depending on the fineness of the vermiculite flakes.

The follower plate 14 may advantageously be provided with a filter face comprising a screen 17 lying against a perforated back plate 18 similar to that on the opposite face of the mold, thereby permitting dewatering through both faces of the mold. This type of follower plate also makes elimination of air much easier than with an imperforate follower since the air will eventually rise to the top and pass through the plate. During the dewatering process pipe 15 is closed by a check valve 19 or the like to prevent escape of material through the pipe with the consequent creation of disturbing currents. The follower plate 14 is preferably reinforced by ribs 20 to prevent flexure under pressure.

As the water leaves the mold the vermiculite flakes first assemble in oriented position along the filtering face of the mold and progressively build up in oriented and overlapping felted condition until a slab of the desired thickness is formed. Where the follower plate is also perforated the building up of an oriented layer of flakes also occurs on this face of the mold. The orientation thus continues until all the flakes have been oriented and the mass has been reduced to reasonably solid form, the follower plate having in the meanwhile advanced to the dotted line position 21 which represents the final thickness of the slab. The greatest danger of disturbance of the vermiculite flakes occurs toward the end of the process where only a small amount of the material remains in a mobile condition. As the material loses its fluidity (and therefore orientation becomes more difficult), it is important that there be no disturbing influence interfering with the deposition of the vermiculite flakes in oriented position. Avoidance of such disturbance in this instance is attained through elimination of air and the advance of the follower plate 14 at such a rate that the water leaves the mold over the entire face of the mold, thus preventing any localized or concentrated currents. At the same time, the follower action causes a sufficient lateral shifting of the material to fill all voids without, however, disturbing the orientation of the flakes.

The cementing materials, in this case lime and silica (such as diatomaceous earth), are not carried out with the water but collect between the flakes. The fiber tends to prevent segregation of the cementing materials as well as to hold the flakes slightly separated to facilitate dewatering.

It is not necessary to employ suction to hasten dewatering of the material in the mold. Dewatering without suction is preferable to avoid interference with proper orientation of the flakes and to prevent entrance of air and disruption of the material. If suction be used, provision should be made to replace abstracted water with solid material and not air and it must be controlled to prevent concentration of the suction at the point of least resistance in the material. At such points there is likely to be a rush of water out of the mold with the consequent disturbance in orientation of the flakes and production of the weak spots in the material. As the flakes progressively orient themselves they overlie each other in a felting action which very materially contributes to the strength of the material and minimizes the use of bonding cement.

The present process contrasts with the method of my copending application in that in the latter air was permitted to enter the mold to replace the water, thereby causing weak spots, requiring the use of a larger amount of cement to provide adequate strength. Moreover, the exfoliated vermiculite was used in granule form. These were relatively bulky (many being generally cubical in shape) and tended to float in the liquid. These characteristics facilitated dewatering but resulted in poor orientation with the result that the material had low insulating efficiency and low strength. While I have not been able to determine exactly the percentage of orientation of the vermiculite flakes into parallel relationship, I have estimated that about 90% of the flakes are properly oriented by the present process even when the flakes are so fine that 30 to 70% will pass through a 65 mesh screen. Flakes larger in area and not substantially thicker would, of course, orient more readily and to a greater extent even than the finer material. The strength produced by the felting action of the flakes would also be correspondingly greater.

After the molding operation is completed, the slab is removed from the mold and subjected to a heating and drying action. The heat promotes reaction between the hydrated lime (probably the lime must first go into solution) and the silica of the diatomaceous earth (or other silica supplying medium) to form calcium silicate which

is the cementing or bonding agent. The relatively large surface area provided by the diatomaceous earth brings about a much more extensive reaction than would occur with other forms of silica having a small surface area in relation to mass, thereby permitting a substantial reduction in the amount of lime and diatomaceous earth necessary to produce a given amount of bonding agent. The formation of calcium silicate can also be increased by heating the slabs under pressure, e. g. 150 pounds steam pressure, in order to develop higher temperatures inside the slab with moisture present. Mere increase in drier temperatures (at atmospheric pressure) does not have the same result inasmuch as the internal temperatures of the material do not exceed the boiling point of water until the latter has been evaporated and after evaporation the reaction between the lime and silica would be very slow. If heating under steam pressure be resorted to, the slabs are preferably subsequently dried in an ordinary drier to eliminate surplus moisture.

After the molded material has been dried, it is preferably superficially trimmed or faced to provide true surfaces and square corners. During the drying process, the shrinkage is not absolutely uniform, thereby resulting in slightly uneven surfaces which if untrimmed would prevent the formation of tight joints between adjacent slabs. This material has a K factor of .64 at 500° F. and .83 at 1200° F.

One satisfactory form of insulating material comprises the following proportions of materials by weight:

	Per cent
Exfoliated vermiculite flakes	65.4
Silica, such as diatomaceous earth	15.7
Hydrated lime	12.8
Long fiber asbestos	6.1

I have found that the lime content need not be equal to that of the diatomaceous earth and may advantageously be somewhat lower than the latter without reduction in strength.

One satisfactory form of asbestos has a Canadian grading of 0–2–8–6. If the insulating material is likely to be subjected to temperatures in excess of 1400° F., it is preferable in order to minimize shrinkage of the insulation at such high temperatures to calcine the diatomaceous earth at approximately 2000° F. and to exfoliate the vermiculite under heat sufficient to raise the granules to about 1800°–1850° F.

Water is used in an amount to form a relatively thin slurry. For each 100 pounds of exfoliated vermiculite from 150 to 300 gallons of water are generally employed.

A somewhat different method, designed to accomplish the same result, is illustrated in Fig. 2. Therein disturbance of the vermiculite flakes during orientation is avoided by feeding the slurry into the mold slowly (i. e. commensurate with the rate of dewatering) and over such a wide area as to eliminate localized disturbance.

As here shown, the apparatus for performing this process is represented by mold casing **25** having a removable filter face **26**. This filter face may comprise, as in the mold shown in Fig. 1, a filter screen or cloth **27** supported by perforated plate **28**. The slurry is introduced into the mold through a pipe **29** by means of a pump **30** or other supply means. Distributed introduction of the slurry into the mold is effected by perforated distribution plate **31** which avoids concentration of a current of slurry at any one point and consequent interference with proper orientation of the vermiculite flakes. The distribution plate **31** is preferably spaced from the filter face **26** by a distance determined by the final thickness of the molded article.

Simultaneous supply of slurry and dewatering through the filter face occurs until the mold space **32** is filled with solid material. Pressure to effect complete displacement of the water and replacement by solid material is developed in this instance by the pressure supplied by the pump **30** or other supply means. The rate of supply should, therefore, be sufficient to maintain the desired pressure on the material. This rate gradually decreases as the block builds up due to gradual increase in resistance to expulsion of water. Air inside the mold is displaced into the upper part of the distributing space **33**. After the mold space has been filled and the distributing space **33** partly filled with slurry, continued introduction of slurry creates the necessary pressure to complete the operation. The air is, of course, slightly compressed in the upper part of the distributing space **33** but this pressure is not sufficient to blow through the material after molding has been completed. It can be utilized to eject the molded block from the mold. The holes **34** in the distribution plate are preferably made large enough and numerous enough to prevent substantial reduction in the pressure transmitted through the distribution plate. As water leaves the filter face of the mold, solid material continues to accumulate inside the mold space **32** until the latter is completely filled with properly oriented vermiculite flakes and other solid material. When this occurs, the operation is interrupted, the filter plate **28** and its screen removed, and the molded block withdrawn from the mold. It does no harm to continue the process for a longer time than necessary, the only result being the collection of solid material above or on the opposite side of the distribution plate **31**. This material can be flushed out and used again after the molded slab has been withdrawn.

As is the case with the slab molded according to the process disclosed in Fig. 1, the greatest danger of interference with proper orientation of the vermiculite flakes occurs near the completion of the molding process. At the beginning of each of the processes, the initial layer of vermiculite flakes against the filter face of the mold is readily oriented and not easily distributed. However, as the slurry thickens toward the end of the molding process, orientation becomes more difficult and slight disturbances may, therefore, interfere with proper orientation. It is particularly important at this time that disturbances be avoided. The depth of the molding space **32** may be slightly greater than the final thickness of the block to allow for trimming off the top layer of material in which there may have been disturbances which prevented adequate orientation.

The block molded according to the process illustrated in Fig. 2 is also heated and dried as above described and then finished to true size.

The completed insulation is lighter in weight and has an insulating efficiency superior to any molded refractory insulation capable of resisting temperatures in excess of 1800° F. It weighs about 19 pounds per cubic foot as compared to 24 pound per cubic foot for bonded diatomaceous earth. Moreover, it is made of less expensive materials at no greater cost of manufacture than other molded refractory insulation.

Obviously the invention is not limited to the details of the illustrative process or methods herein disclosed. Moreover it is not indispensable that all features of the invention be used conjointly since various features may be used to advantage in different combinations and subcombinations.

Having described my invention, I claim:

1. The method of making a refractory insulating block or the like which comprises preparing a relatively thin slurry containing exfoliated vermiculite in relatively thin flakes, diatomaceous earth and hydrated lime, introducing the slurry into a mold having a filter face and withdrawing the water in the slurry through the filter face while the slurry is in a relatively quiescent state to cause the vermiculite flakes to collect progressively at the filter face of the mold in oriented and felted relationship with the flakes lying substantially parallel to said filter face and while withdrawing the water excluding the entrance of air to replace the water and instead filling the voids left by the water with solid material, and then subjecting the molded material to heat to produce a substantial reaction between the lime and diatomaceous earth to form calcium silicate to bond the vermiculite together.

2. The method of making a refractory insulating block or the like which comprises introducing into a mold having a filter face a slurry containing exfoliated vermiculite in relatively thin flakes and minor amounts of hydrated lime and silica in the form of diatomaceous earth, causing the water in the slurry to filter from the mold so as progressively to collect the flaked vermiculite at the filter face of the mold in oriented and felted condition with the flakes lying parallel to the filter face and while abstracting the water excluding the entrance of air into the voids left by the water by filling such voids with solid material, and then subjecting the molded material to heat to produce a substantial reaction between the lime and diatomaceous earth to form calcium silicate to bond the vermiculite together.

3. The method of making a refractory insulating block or the like which comprises introducing into a mold having a filter face a slurry containing exfoliated vermiculite in the form of relatively thin flakes, a refractory bonding material, causing the water in the slurry to filter from the mold so as progressively to collect the flaked vermiculite at the filter face of the mold in oriented and felted condition with the flakes lying parallel to the filter face, and while abstracting the water excluding the entrance of air into the voids left by the water by filling such voids with solid material.

4. The method of making a refractory insulating block or the like which comprises introducing into a mold having a filter face a slurry containing exfoliated vermiculite in relatively thin flakes and refractory bonding material, causing the water in the slurry to filter from the mold so as progressively to collect the flaked vermiculite at the filter face of the mold in oriented and felted condition with the flakes lying parallel to the filter face, and reducing the volume of the mold in a direction perpendicular to said filter face simultaneously with and at the same rate as the flow of water therefrom so that solid material will occupy the spaces left by the escaping water.

5. The method of making a refractory insulating block or the like which comprises introducing into a mold having a filter face a slurry containing exfoliated vermiculite in relatively thin flakes and refractory bonding material, causing the water in the slurry to filter from the mold through said filter face so as progressively to collect the flaked vermiculite at the filter face of the mold in oriented and felted condition with the flakes lying parallel to a filter face, and regulating the rate of outflow of water from said mold so that at no point in the mold will there be currents sufficient to disturb the orientation of said vermiculite flakes.

6. The method of making a refractory insulating block or the like which comprises introducing into a mold having a filter face a slurry containing exfoliated vermiculite in relatively thin flakes and refractory bonding material, causing the water in the slurry to filter from the mold through said filter face so as progressively to collect the flaked vermiculite at the filter face of the mold in oriented and felted condition with the flakes lying parallel to a filter face, causing solid material to move toward the filter face of the mold substantially at the rate of outflow of water therefrom to replace the spaces occupied by the water with the solid material, and regulating the rate of outflow from said mold to prevent creation of currents in the mold which might disturb the orientation of said vermiculite flakes.

PAUL S. DENNING.