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A. A. FOWLER ET AL

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REFRACTORY MATERIAL AND METHOD OF MAKING THE SAME

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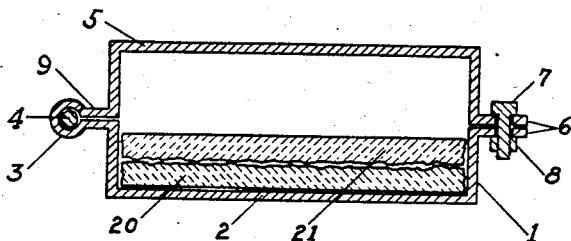


Fig. 1

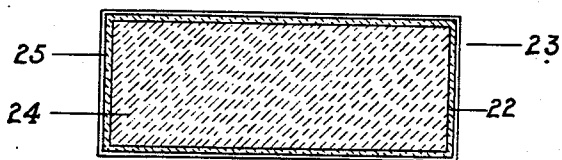


Fig. 2

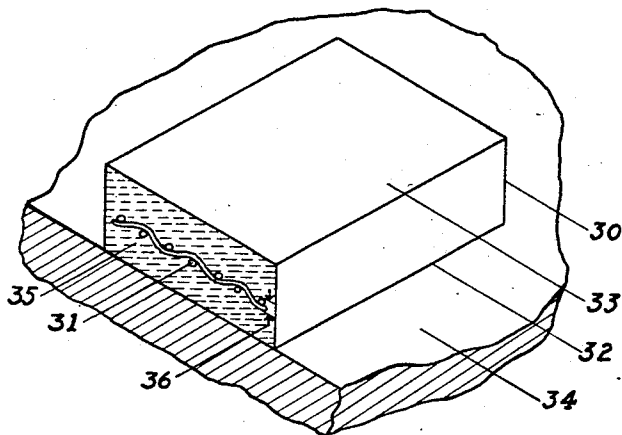


Fig. 3

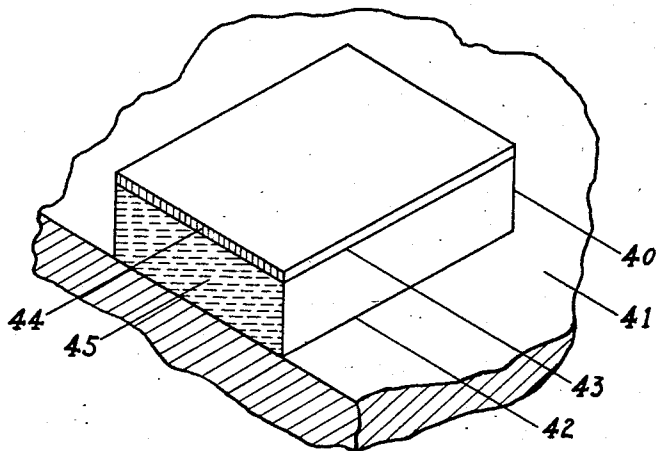


Fig. 4

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REFRACTORY MATERIAL AND METHOD
OF MAKING THE SAMEAlbert A. Fowler, North Hollywood, and Russell
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Application March 27, 1933, Serial No. 663,034

17 Claims. (Cl. 25—156)

In this specification and the accompanying drawing, we shall describe and show a preferred form of our invention and specifically mention certain of its more important objects. We do not
5 limit ourselves to the forms disclosed, since various changes and adaptations may be made therein without departing from the essence of our invention as hereinafter claimed; and objects and advantages other than those specifically mentioned are included within its scope.

10 An object of our invention is to produce a very light refractory material which shall be a good heat insulator.

15 Another object of our invention is to provide a method of making a heat insulator, which material shall be cheap, fireproof, strong, and indestructible by insects and vermin.

20 Another object is to make a material in the interstices of which there shall exist a partial vacuum.

Another object is to produce a light material which shall be stout enough to be employed in load bearing portions of building structures.

25 A further object is to provide a heat insulator that is water-proof.

Still another object of our invention is to make a material that is a good absorber of sound.

30 The exact nature of the invention will be better understood from a description thereof in connection with the accompanying drawing.

Fig. 1 is a sectional drawing of the form used to mold one type of our material, showing in cross-section the contents of the form as arranged in one stage of a process for manufacturing this material.

35 Fig. 2 is a cross-sectional view of the finished insulation material.

40 Fig. 3 is a drawing in perspective of one type of insulating material applied to a surface being insulated, one end of the insulation being shown in section.

45 Fig. 4 is a drawing in perspective of another type of insulation material applied to a surface being insulated, one end of the insulation being shown in section.

50 The material resulting from the practice of our invention is essentially a silicate which has been given a spumiform character and while in this state has been solidified by desiccation. This material may be made in several different types and the method of making each type is necessarily varied somewhat to account for the product being formed. It is a common quality of
55 all these types of material, however, that they are

of spumiform character, that is, they have the appearance and the structure of foam; but they are nevertheless solidified. Whether the material is in the form of powder or of thick slabs, close examination will reveal this foam-like structure, and the word spumiform is intended to describe this internal structure regardless of the external size or shape of the body possessing it.

Another characteristic is that they are substantially completely desiccated, by which we mean that they are substantially devoid of all moisture. It is the process of desiccation performed after the manner of our invention that results in the spumiform state of the silicate and in its solidification while in that state.

One type of our material we make by boiling a solution of a water soluble silicate for about six hours at a temperature gradually increasing from room temperature up to about 650 deg. F. to 850 deg. F., and then cooling it slowly to room temperature again. Ordinarily commercial sodium silicate or water glass of 42 deg. Bé. specific gravity is used. If it is desired to form the material to any particular shape, the liquid silicate is placed in a form which is provided with some means of escape for the steam generated during the process. A loosely fitting but confined cover will permit the escape of steam without allowing the silicate to escape. In order to avoid sticking of the molded article to the form, the form is preferably made of glass, or is of metal and coated with graphite, or it may be lined with waxed paper in which case the paper is burned during the firing operation. One quart of the liquid silicate is used for every 250 cubic inches of space required to be filled by the molded article.

In making this type of material it is necessary to desiccate the silicate very slowly or large holes will be found in the finished article. When the material is to be ground up for use as powder, this defect is not important but in a slab of heat insulating material it cannot be tolerated.

A method of making our spumiform material which has marked advantages over that above-mentioned consists of two steps and is as follows:

The first step consists in a preliminary cooking operation. The purpose of this cooking operation is to produce a partially desiccated solid silicate which may be more advantageously handled than the liquid silicate, and which will not tend to stick to a molding form, but which when further desiccated, as specified, will result in the spumiform material desired. Sodium silicate so-

Formed Silicate Bond Form

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lution is poured into shallow open pans and is cooked over an open flame, not in an oven, for from 1 to 1½ hours at from 300 deg. F. to 200 deg. F.

- 5 It is desirable not to cook it in an oven because when this is done the top skin of silicate in the pan becomes hard, tends to prevent the escape of steam from below and the resultant pressure causes the whole mass to puff and rise.
- 10 If, however, the silicate is cooked in an open pan over a fire, the bottom will be hotter than the top and consequently water will be evaporated from the bottom first while the top is still liquid. When the cooking is done as specified and the
- 15 pan with its silicate content is allowed to cool, a solid cake of material results. The top layer is generally of glassy appearance while the part next to the pan is white and more spumiform in character than the glass-like upper layer due to its more nearly complete desiccation. A metal
- 20 pan is used, and is coated with graphite or plated with tin to prevent the silicate cake from sticking. In this preliminary cooking operation, the process of desiccation is stopped when the silicate be-
- 25 comes sufficiently gummy that, on cooling to ordinary atmospheric temperature, it becomes a solid as distinct from a liquid. In this state it may be described as a hydrous solid silicate, one containing water, as distinct from the completely
- 30 desiccated or anhydrous silicate.

The second step, completing the process under consideration, consists in cooking the material resulting from step one described above, until complete desiccation is achieved. As desiccation

35 proceeds, the silicate puffs into a spumiform material presumably because of the passage of evaporated water through the remaining viscous substance. As the process continues the spumiform material gets larger and more viscous until

40 when all the water has been evaporated, puffing has ceased and the viscous material has solidified into an anhydrous spumiform material of five times the volume, that is composed of millions of

45 tiny air cells enclosed by exceedingly thin silicate walls—a material that is amazingly light and strong and an excellent insulator of heat. The cooking is preferably conducted at 650 deg. F. to 850 deg. F. in a closed oven so that uniform temperature exists at all surfaces of the material being cooked. The required time of cooking depends upon the shape and mass of material being treated. The important consideration determining how long cooking shall continue is that

50 substantially all the water must be removed from the partially desiccated or hydrous silicate and yet the remaining spumiform material must not be melted down. If the water is not substantially all removed, further heating at any later time will cause unwanted puffing of the material, and

60 also a partially desiccated material is not as good an insulator as a completely desiccated one. If heating at a high temperature is prolonged beyond the point of complete desiccation, the silicate may melt and destroy the fine spumiform structure because while water was still present the material was limited in temperature by the evaporation process, but after complete desiccation the increased temperature may be excessive.

- 70 This second step may be carried out in a variety of ways to conveniently secure the spumiform material in various forms and shapes:

For certain applications it is desirable to have it in the form of a powder. To produce this

75 form, the material resulting from step 1, de-

scribed previously, is pulverized to desired size and step 2 is carried out by cooking this partially desiccated pulverized silicate in a revolving hot drum until complete desiccation has been achieved. Usually 15 minutes per batch at 650 deg. F. to 850 deg. F. is sufficient. The result is an exceedingly light white powder composed of individual particles each of which appears upon detailed examination as a small round spumiform mass, itself composed of a multitude of minute air cells enclosed by thin silicate walls. The size of the individual particles is determined by the size of the partially desiccated particles from which they are produced, and very small spumiform particles may be made by simply pulverizing the partially desiccated material to extreme fineness. The finished powder of spumiform material should not be confused with powder made by pulverizing the completely desiccated silicate, for the particles of the former material are stronger, more symmetrical and uniform, and contain more air cells. The powder resulting from the process here specified is used as heat insulation by pouring it into a space surrounding the body to be insulated, or it is packed in boxes of asphalted paper for refrigerator use. It is also packed in sealed cellophane packages which are placed in the walls of refrigerators. This packing of the powder into boxes is more desirable than pouring it into a space surrounding the body to be insulated because in packing, the individual thin-walled particles of spumiform silicate are compressed. Being under compression, any tendency of the particles to settle and occupy a smaller volume is thwarted, since the particles merely expand sufficiently to maintain the total volume the same. In practice, it is found that in a box into which the powder has been packed, the particles remain in compression throughout the life of the insulation, and hence the box is always full. Asphalted paper and cellophane are used as box materials because they are moisture-proof and prevent the accumulation of moisture in the interior of the insulation.

The powder may also be mixed with a dilute liquid solution of sodium silicate or with the pulverized partially desiccated silicate and the whole mass fired in a form at 650 deg. F. to 850 deg. F., preferably 45 minutes at 750 deg. F., to produce a tile. When the binder consists of dilute liquid solution of sodium silicate, the action of the heat is to solidify this binding substance to form a hard, firm bond joining the particles of powder. If the partially desiccated silicate is employed as a binder, it is softened by the heat causing it to hold tenaciously to the powder, and continued heating then solidifies the binder. When the particles of the completely desiccated powder employed in this operation are made large enough, the resulting tile is an excellent absorber of sound. This is because the individual particles touch and are bonded together at only a few points, leaving rather large interstitial intercommunicating voids through which the impinging sound can pass to contact the large internal absorbing surface of the tile.

When it is desired to form a slab of spumiform material having the advantage of structural strength, the partially desiccated material resulting from step 1, as described previously, is cooked to complete desiccation in a molding form. In Fig. 1, the lower half 2 of form 1 has at one end several circularly shaped portions 3 which are adapted to carry a rod 4. The upper

portion 5 of the form 1 also has circularly shaped portions 9 which surround rod 4 and which co-operate to form a hinge about rod 4. Both the top 5 and the bottom 2 have horizontally extending edges 6 which are adapted to be clamped together at intervals around the periphery of the form by bolts 7 and nuts 8. The form 1 is generally coated with graphite both inside and out to prevent oxidation and lessen the tendency of the silicate to stick to the walls. In preparation for the cooking operation, a cake 20 of the partially desiccated silicate which is shaped approximately to cover the bottom of the form but may be only one-fourth the height of the form, is placed in the form so that the side of the cake 20 which has been more nearly completely desiccated is toward the bottom of the form. The cake 20 can have been made in the required shape by cutting it from a larger piece or by partially desiccating it in a pan having the same area as the bottom of the form 1. Atop the cake 20 is placed another cake 21 identical to it, but with its more nearly completely desiccated side toward the top 5. The cakes 20 and 21 as prepared to completely fill the form 1 in this operation should have resulted from the partial desiccation of one quart of 42 deg. Bé. sodium silicate for each 250 cubic inches of the form to be filled. After the cakes 20 and 21 are in place, the top 5 is shut and bolted down by means of bolts 7 and nuts 8. It is not desirable to bolt the form so tightly closed that a dangerous pressure builds up in the form before the steam incident to desiccation can escape. The form is closed tightly enough to prevent the silicate from being forced out, however. For a form two inches high, cooking is carried out at 650 deg. F. to 850 deg. F., preferably for about 50 minutes at 750 deg. F. The cooking is determined in time and intensity by the condition that desiccation shall be complete, yet the material shall not be melted down. As desiccation proceeds, the inner portions of cakes 20 and 21 puff more than the outer more nearly completely desiccated portions and the cake 21 is pushed tightly against the top 5 of the form. As the process continues, the pressure in the form compresses the hot and still pliable layer next to the form into what develops, when cold, into a hard strong outer shell 22 on the finished slab 23. The interior 24 of the finished slab 23 is solidified and is of a light spumiform character.

After the cooking has been completed the form and contents are cooled slowly. If cooling takes place too rapidly the slab 23 may crack. Also, since at the time of solidification the air entrapped in the slab is at a high temperature, and therefore at a low density, when the temperature has decreased a partial vacuum exists in the slab of spumiform material and if the temperature decreases too rapidly this differential pressure will collapse the slab. The greater heat insulating value of an evacuated substance is taken advantage of by sealing the whole exterior of the slab 23 of spumiform material, immediately after removal from the form, with a sealing substance 25 such as sealing wax or a ceramic glaze. The slab can be removed from the form 1 soon enough after the cooking is complete to still retain most of the vacuum. After the sealing substance 25 is cold, the slab will consist of a multitude of partially evacuated air cells enclosed by thin silicate walls—an extremely good heat insulator.

As alternative to the air-tight sealing surface,

the slab is made water-proof by dipping in an asphalt paint. It is undesirable for any surface openings to exist which might permit the intrusion of water with its consequent reduction in heat insulation. Another effective water-proof exterior is often provided by applying a water-proof paper or fabric to the surface of the slab. Asphalted paper has been found well adapted to this use. The paper on one side of the slab is made to extend beyond it. The extending paper carries asphalt, and when the slab is put into place in a refrigerator the extending paper is pressed and sealed by the asphalt against the refrigerator frame, making a water-seal for the frame.

The advantages of making the material by a double cooking process as above specified rather than by a single cooking operation as previously described are that it is possible by the double cooking process to make a more uniform material; the outside shell is harder and stronger; and the silicate does not tend to stick to the form so much.

Another method of performing step 2 of the double cooking process is as follows: The partially desiccated silicate resulting from step 1 is broken into chunks varying in size from about 1/8 inch to 1 inch in diameter. These chunks are placed in the form 1 and the form is closed. The amount of the partially desiccated silicate used for every 250 cubic inches of space in the form is that which results from cooking 1 quart of 42 deg. Bé. sodium silicate. In place of some of the partially desiccated silicate, an equivalent amount of the completely desiccated pulverulent spumiform material previously described may be employed. The form and contents are heated at 650 deg. F. to 850 deg. F., preferably 50 minutes at 750 deg. F. As heating and consequent further desiccation proceeds, the partially desiccated chunks of silicate puff and bond to one another, completely filling the available space, even exerting a pressure on the form that results in an exterior shell of considerable compactness. After cooking, the form and contents are slowly cooled. A sealing substance is applied, as described above, to retain the partial vacuum existing within the material, or a water-proof surfacing material such as asphalt is applied to the surface of the formed insulation. This method has the advantage over that in which cakes 20 and 21 are employed that it is not necessary to shape the partially desiccated silicate to the form being used, and it has the disadvantage that the molded spumiform material resulting has not quite so strong an outside shell.

When it is desired to make a molded article of spumiform material of superior strength, reinforcement is provided for the article. The spumiform material with reinforcement is very strong and may be employed as load bearing members in building structures, having the advantage of great lightness. The reinforcing members may be of any material to which the silicate will bond tightly. The variety of suitable reinforcing materials is very great; iron and aluminum are most satisfactory commercially but other materials that will withstand the temperature of cooking are also used, such as thin asbestos board. When the reinforcement is applied to the interior of the article, the reinforcing member, for example, an iron wire screen, is dipped in sodium silicate solution and placed during the filling of the form, in the desired

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position. In the case in which cakes 20 and 21 are employed in filling the form, the reinforcing screen is placed between the two cakes, and the cooking proceeds as previously described. In case the form is filled with chunks of partially desiccated silicate, the screen is placed in the form, after half the chunks have been put in, and the other half of the chunks are placed on top of the screen. The screen is placed in the form with its shortest dimension in a direction substantially perpendicular to the surface it is intended to heat insulate in order that the reinforcing material shall affect to a minimum degree the insulating value of the whole article. Thus in a slab 30 of material such as is made in form 1, the reinforcing screen 31 is shown enclosed by spumiform material 35 and parallel to and midway between surfaces 32 and 33. The slab 30 is applied to a surface 34 which it is desired to heat insulate. The surface 32 of slab 30 is higher in temperature than the surface 33 and heat flows from surface 32 to surface 33 through the slab 30. The reinforcing member 31 is positioned with its shortest dimension 36 substantially perpendicular to the surface 34 to be heat insulated.

Instead of a screen, a plate or sheet of reinforcing material is often used on one or on both sides of the molded article. This method of reinforcement has the advantage that a metal or other durable exterior may be provided to a heat insulator without the necessity of any fasteners such as nails, screws, etc. The sheet of reinforcing material is coated with a light coat of liquid sodium silicate on the side which is to be next to the insulator. It is then placed either on the bottom or on the top of the material in the form and cooked in the same manner as specified for making the material without reinforcement. Upon completion of cooking it will be found that the sheet of reinforcing material is bonded to the insulation with as strong a bond as the insulation has to itself. The sheet of reinforcement may also be applied to the completely desiccated material by coating the reinforcing sheet on one side with liquid sodium silicate, sticking it to the insulation and holding it in firm contact while the metal sheet is heated to about 750 deg. F. as by a blow-torch. In Fig. 4, the finished slab 40 is shown applied to a surface 41 which is being heat insulated. Surface 42 of slab 40 rests on surface 41. Surface 43 of the spumiform slab is covered with and joined to iron sheet 44. This iron sheet is nickel plated or polished to reduce the heat transfer by radiation from its upper surface. Heat conduction is through the spumiform material 45 in a direction perpendicular to surfaces 42 and 43 and it will be noted that this direction is also that of the shortest dimension of the reinforcing sheet 44. Thus, the reinforcing member 44 is positioned with its shortest dimension perpendicular to the surface 41 to be heat insulated.

It is obvious, of course, that our material can be molded in many different shapes such as in circular cylindrical sections for pipe coverings, etc. Water soluble silicates other than sodium silicate can be used—for example, potassium silicate, and we regard the application of these as coming within the scope of our invention. To those skilled in the art, other detail modifications and variations in our materials and methods of their manufacture will be apparent, and we wish, therefore, to be limited only by the scope of the appended claims.

What we claim as new and desire to secure by Letters Patent is:

1. A molded article containing solidified silicate having a spumiform structure, said article being provided with an air-tight outer shell within which exists a partial vacuum.

2. A method of making a pulverulent refractory material which consists in partially desiccating a silicate solution to form a solid silicate containing water, reducing said solid silicate to a powder and cooking said powder until desiccation is substantially complete.

3. A method of making a molded article which consists in partially desiccating a silicate solution to form a solid silicate containing water, reducing said silicate to chunks, confining said chunks of silicate in a molding form sufficiently closed to permit escape of water vapor, but not of silicate, cooking said silicate in said form at a temperature sufficiently high to create in it a spumiform structure, and continuing the process until desiccation is substantially complete.

4. A method of making a molded article which consists in forming a plurality of cakes of partially desiccated solid silicate in such manner that each cake has one side more nearly completely desiccated than the other side, confining said cakes in a molding form with the more nearly completely desiccated sides toward the walls of the molding form, cooking said silicate in said form at a temperature sufficiently high to create in it a spumiform structure, and continuing the process until desiccation is substantially complete.

5. A molded article comprising spumiform particles and a binder bonding said particles at their points of contact.

6. A molded article comprising particles containing spumiform silicate and a binder bonding said particles at their points of contact.

7. The method of making heat insulation which includes providing an intimate mixture of particles containing spumiform silicate with a non-solid binder, forming said mixture to desired shape, and solidifying said binder.

8. A heat insulator comprising a confined body of spumiform particles under compression containing substantially anhydrous silicate.

9. The method of producing a non-settling granular heat insulator from a body of spumiform particles containing substantially anhydrous silicate which includes compressing said body of particles and maintaining said body under compression.

10. A molded article comprising spumiform particles containing substantially anhydrous silicate and a binder bonding said particles at their points of contact, said particles being of such sizes that interstitial intercommunicating voids exist between the particles.

11. The method of making heat insulation which includes providing an intimate mixture of particles containing spumiform silicate with a solid binder, forming said mixture to desired shape, heating said mixture to soften said binder, and solidifying said binder while retaining said mixture in said shape.

12. The method of making a molded article which includes confining a multiplicity of particles containing hydrous solid silicate in a molding form sufficiently closed to permit the escape of water vapor, but not of silicate, cooking the contents at a temperature sufficiently high to create in them a united spumiform structure, and continuing the process until desiccation is substantially complete.

13. A heat insulator comprising a molded body of spumiform material having bonded to the exterior surface thereof a sheet of metal with a heat-reflecting surface.

5 14. A heat insulator comprising a molded spumiform body containing anhydrous silicate and having a sheet of metal bonded to the exterior surface thereof by a bond containing anhydrous silicate.

10 15. A non-settling granular heat insulator comprising a confined body of resilient spumiform particles in a state of compression.

15 16. The method of making non-settling granular heat insulation which includes heating hydrous particles of sodium silicate at a temperature sufficiently high to create in them a spumiform structure, continuing said heating until

desiccation of said particles is substantially complete, compressing said spumiform particles, and maintaining said spumiform particles under compression.

17. The method of providing a body with a metal exterior through a common bonding layer having a spumiform structure which includes the steps of forming on said metal a coating of hydrous soluble silicate, providing contact between said coating and said body, heating said coating to create therein a spumiform structure and bond said metal to said body, and continuing the heating until desiccation of said bonding layer is substantially complete.

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