

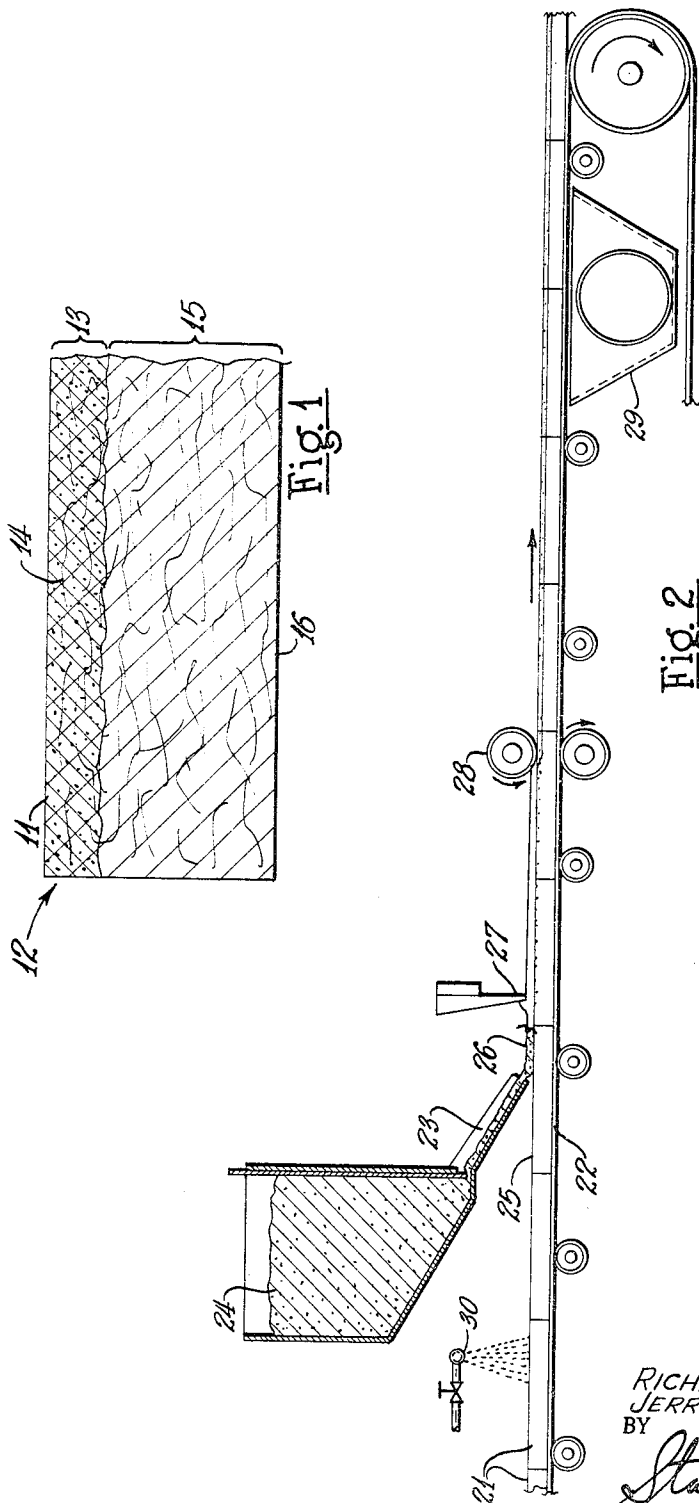
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HIGH TEMPERATURE RESISTANT ACOUSTICAL BOARD

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HIGH TEMPERATURE RESISTANT ACOUSTICAL BOARD

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ABSTRACT OF THE DISCLOSURE

An acoustical and thermal insulating element capable of maintaining structural integrity at temperatures in excess of 1000° F., composed of a plurality of siliceous fibers interbonded into an integral, void-containing mass having a density of between 1 and 30 pounds per cubic foot and shaped in the form of a panel having two substantially parallel major surfaces and a thickness of between 250 and 3000 mils, and kerfed side walls which extend between the major surfaces, and deposited within the voids present between the fibers and adjacent to one of the surfaces and to a depth of at least 125 mils, an impregnant containing between 75% and 98% by weight of aluminum silicate particles consisting essentially of between 10% and 90% by weight of hydrated aluminum silicate containing between 50% and 75% by weight of SiO₂ and between 15% and 35% by weight of Al₂O₃, and between 10% and 90% by weight of an alumino-silicate of a metal selected from the group consisting of sodium, potassium, calcium, magnesium, barium and combinations of the metals, in which the metal is present in a quantity in excess of 6% by weight, and admixed with and inter-adhering the aluminum silicate particles between 2% and 25% by weight of a binder phase consisting essentially of between 1% and 20% by weight of colloidal silica, and between 1% and 15% by weight bentonite, the remainder of the panel and the portion of the panel immediately adjacent to the second of the major surfaces to a depth of at least 50 mils being substantially devoid of the impregnant and retaining the density of 1 to 30 pounds per cubic foot.

This application is a continuation-in-part of our co-pending application Ser. No. 457,984, filed May 24, 1965, now Patent No. 3,286,785.

The present invention concerns structural, acoustical and thermal insulating boards which are capable of retaining their integrity and dimensional stability at temperatures in excess of 1000° F., and particularly low density siliceous fiber boards possessing an inorganic lamina at one surface and within the voids existing in the fibrous low density structure.

Structural and decorative elements formed from inter-bonded or intermeshed siliceous fibers, such as glass, mineral wool or asbestos fibers, have achieved extensive use in applications such as ceiling and wall tiles, panels or boards, in which they provide excellent acoustical and thermal insulating properties. Such elements normally comprise an integral relatively rigid mass of fibers grouped into a thin panel-like structure having two major

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opposed and substantially parallel surfaces, a thickness in the order of one-tenth or less of the major dimension of the structure, and possessing a density in the range of 1 to 30 pounds per cubic foot. Due to the non-combustibility of the siliceous fibers, it might be expected that such structures would be valued for their fire resistance, for example in the insulation and fireproofing of beams, steel girders and concrete decks of buildings and for the bulkheads, beams, and decks aboard ships.

However, it has been found that these non-combustible elements fail to prevent flame spread or building collapse as a result of their loss of integrity at temperatures experienced during a fire. In the first instance, these non-combustible elements can be employed to mask or seal off such readily combustible elements as wooden studs, joists, partitions, decking, and the like. At relatively low temperatures, e.g. 500° F., these low density materials perform the desired function in an adequate fashion by thermally insulating the combustible materials which they conceal and thereby preventing the transfer of temperatures adequate to initiate combustion. However, at higher temperatures, e.g. 1000° F., and despite their own incombustibility, these elements undergo structural failures which permit the transfer of heat or flame to the combustible elements. Such failures result from the sagging, slumping, warping, shrinking, delamination or the like of the incombustible boards and the ignition of the combustible elements occurs in one of several manners. For example, the sagging, slumping, warping or shrinking of the elements results in their separation from their fastening or suspension means and the formation of a gap or opening between abutting or adjacent boards. Such apertures permit the transfer of heat and/or flame capable of igniting combustible materials located behind the incombustible boards. Alternatively, delamination provides apertures for heat or flame transfer through the fibrous boards.

In addition, heat or flame transfer through these siliceous fiber boards may also weaken concealed non-combustible structural elements such as steel beams or girders and concrete decks, to cause a partial or total collapse of the building structure.

As a consequence of this discovery many localities have amended building codes to require prolonged resistance to flame and heat transfer at high temperatures, and particularly in public buildings. Insurance rates have also been adjusted in recognition of this hazard since a fire-damaged building may be saved if damage to the structural elements can be avoided.

Accordingly, methods and tests for measuring the resistance of a structure to flame and heat transfer, and the consequent spreading of the fire, have been developed. Typical of such tests and the resultant standards, are the Underwriters Laboratory fire rating tests in which the thermal-acoustical boards are erected in their usual manner of installation, within a furnace chamber approximating the dimensions of a room. According to one such test, thermocouples are positioned to measure temperatures of structural members and on the top side of a concrete slab below which is suspended the board to be tested. Flames are then ignited within the chamber and the temperature is progressively elevated, i.e. 1000° F., five minutes after the initiation of the test, 1700° F. at one hour, 1850° F. at 2 hours, etc. as specified by ASTM Test E 119, to simulate the conditions which would be experienced during an actual fire. The test is terminated whenever any

of the thermocouples senses a temperature which would be adequate to ignite combustible building elements or to weaken noncombustible elements such as steel beams or concrete decks. This test yields a fire rating based upon the length of endurance of the medium tested under the prescribed conditions. For example, an acoustical insulating board which endured for one hour before permitting the transfer of temperatures adequate to initiate combustion or cause the collapse of noncombustible elements would receive a one hour rating. A failure is experienced whenever any single thermocouple above the slab reaches 325° F., above ambient temperature, or when the average temperature of such thermocouples reaches 250° F., above ambient temperature. A failure also occurs if the concrete slab cracks and cotton waste thrown on a crack ignites, when any thermocouple indicating the temperature of a steel structural member reads 1200° F. above ambient, or when the average reading of such thermocouples is 1000° F. above ambient. This test has been described in detail in order to illustrate the improvements made possible by the inventive materials, in terms of the conditions which must be endured.

Consequently, the present invention is directed to the provision of low density decorative and structural elements formed from siliceous fibers, which exhibit structural integrity and dimensional stability at temperatures in excess of 1000° F., while retaining the acoustical and thermal insulating properties for which they are valued.

Another object is the provision of methods for the preparation of such low density decorative and structural elements.

A further object is the provision of impregnants capable of imparting the prescribed integrity to such decorative and structural elements.

Structural integrity, as used herein, is employed to define the ability to resist the transfer of heat and flames through apertures resulting from the delamination or disintegration of the structure, or through apertures resulting from the separation of the low density boards from their fastenings, suspension systems or from abutting, adjacent boards, upon exposure to temperatures in excess of 1000° F. Consequently, the term "structural integrity" contains an aspect of dimensional stability in respect to resistance to shrinkage, expansion and warping or sagging. It should also be noted that the acoustical properties of the structures include both sound absorption and resistance to sound transmission. The former is provided by the low density, void-filled region which is retained at one major surface of the board, while the latter is improved by virtue of the formation of a dense zone which is substantially impervious to sound at the opposite major surface. At the same time, the insulating values of the boards are enhanced by virtue of the opacifying effect of the dense layer.

The above objects and their achievement are described in detail in the following specification and the attached drawings in which:

FIGURE 1 is a fragmentary sectional view of a product prepared in accordance with the invention; and

FIGURE 2 is a schematic representation of an apparatus suitable for the practice of the methods of the invention.

The foregoing objects are achieved by means of the deposition of a continuous inorganic structural phase, or layer, within the voids present within a resin-bonded, low density siliceous fiber medium and immediately adjacent to one of the two major surfaces of the low density fibrous board. The extent of such deposition is limited and controlled in order to prevent the impregnation of the other or opposite major surface, and to thereby retain the desirable thermal and acoustical properties of the low density medium of which the second major surface is composed.

Specifically, an aqueous slurry containing a particular

combination of inorganic ingredients formulated to provide the desired structural integrity and dimensional stability is applied to one major surface of the low density fibrous element and then deposited within the voids present in the fibrous medium and adjacent to the point of application to provide a structural layer. In determining the depth of the structural layer adequate to provide the prescribed structural integrity, and of the low density zone of the opposite surface adequate to yield the desired acoustical and thermal insulating properties, it has been found that there are several important considerations. These considerations will be more easily understood from a brief description of conventional ceiling board and tile, and particularly with reference to the manner of installation. There are two rather widely used installation methods. Ceiling boards are supported on narrow ledges provided by a suspension grid, while tiles have a kerf slot which receives a supporting spline. The grid or spline system is usually fastened to the deck or flooring thereabove, for example by wires. The primary difference lies in the fact that ceiling boards rest on a narrow ledge which frames the entire circumference of the board on one of its major surfaces, while tiles are supported by splines received in the slot or groove in the narrow sides thereof. The properties described for ceiling board and ceiling tiles apply equally to wall board and wall tile but for purposes of simplicity only the ceiling board and tile will be discussed. It has been found that a structural layer having a thickness of 125 mils is adequate for the ceiling board, but that in the ceiling tile, the structural layer must extend at least below the kerf, which is normally located about 1/4 inch from the impregnated surface. Preferably, the structural layer extends to the acoustical-thermal low density zone or layer which in both cases must have a thickness of at least 50 mils and preferably 125 mils. The low density boards which are treated in accordance with the invention normally have a thickness of at least 375 mils, and the structural layer or impregnated zone normally has a thickness of at least 125 mils for the ceiling board and at least 250 mils for the ceiling tile.

After depositing and positioning the impregnant to the desired depth, the aqueous component is dispelled by drying to leave a strong rigid structural phase or layer.

I.—Impregnants

The inventive impregnants contain both inorganic particulate matter, and a binder phase which is capable of interadhering the inorganic particles both at ambient conditions and during exposure to temperatures in excess of 1000° F. Specifically, the binder phase contains between 5 to 95% by weight of colloidal silica and between 5 to 95% by weight of bentonite, and comprises between 2 to 25% by weight of the total solids present in the impregnant. The impregnant contains between 1 to 20% by weight of colloidal silica and between 1 to 15% by weight of bentonite.¹ It should be noted that references to quantities of colloidal silica relate to the quantities of silica solids, although these compositions are normally employed as dispersions in a liquid carrier medium such as water.

The inorganic particles which are interbonded by the foregoing binder phase include a combination of both materials possessing a melting point below 2000° F., and materials which melt above that temperature. The

¹ Bentonite, usually predominantly sodium montmorillonite, beidellite or a combination of the two, serves both as a binder for the impregnant and, by increasing the viscosity of a slurry thereof, as a process aid facilitating the required degree of penetration by the slurry without filtration of solids therefrom. Calcium montmorillonite is somewhat less effective as a binder and does not function as a process aid unless subjected to high shear or connected to the sodium cycle. Alginates, methylcellulose and other thixotropic materials can be used in place of bentonite where only the process aid function is required.

lower melting materials are the aluminosilicates of the Group I and II metals, sodium, potassium, calcium, magnesium, and barium, and aluminosilicates containing two or more of these metals. The high melting materials comprise certain hydrous aluminum silicates which are subsequently described. This combination is based upon the discovery that the formation of a molten or ceramic phase is highly desirable in the attainment of structural integrity at temperatures experienced during a fire or a fire test.

It is believed that the lower melting materials provide a new or auxiliary adhesive phase upon exposure to temperatures in the range of 1000–2500° F., which serves to interadhere the higher melting particles and to maintain structural integrity. While structural integrity resulting from a "liquid" or molten phase may at first appear contradictory, the nature of this "liquid" must be considered. Specifically, the Group I and/or Group II metals function as fluxes which facilitate the melting of a portion of the ingredients of the impregnant and form an extremely viscous adhesive. For example, molten glass compositions commonly possess viscosities in the range of 10^4 to 10^7 poises and consequently are not highly fluid liquids in the ordinary sense of the word. The value of thermally inert particles interbonded by the described "liquid" adhesive, over thermally inert but structurally weak impregnants is aptly demonstrated by the fire ratings achieved with the inventive impregnants.

The previously described binder phase, i.e. bentonite and colloidal silica, is employed to interbond between 75 to 98% by weight of an admixture of low and high melting materials. In turn, this admixture comprises between 10 to 90% by weight of the higher melting hydrated aluminum silicate particles and between 10 to 90% by weight of the lower melting sodium, potassium, calcium, magnesium and barium aluminosilicates. Consequently, the impregnant composition contains from 7.5 to 88.2% by weight of the lower melting material and from 7.5 to 88.2% of the higher melting material.

Thus, the impregnants generally comprise:

	Percent by weight
Colloidal silica (solids) -----	1-20
Bentonite -----	1-15
Hydrated aluminum silicate -----	7.5-88.2
Na, K, Ca, Mg and Ba aluminosilicates -----	7.5-88.2

Preferably the impregnants contain:

	Percent
Colloidal silica (solids) -----	2-6
Bentonite -----	1-5
Ball clay -----	15-25
Feldspar -----	65-80

Colloidal silica and silica sols are commercially available compositions and materials of this nature, as well as methods for their preparation, are disclosed by U.S. 2,244,325, 3,083,167, 3,041,140 and 3,128,251.

Bentonite is a commonly known clay mineral com-

posed essentially of montmorillonite and beidellite; it is colloidal hydrated aluminum silicate or sodium montmorillonite.

The hydrated aluminum silicates employed by the invention are ball clays consisting primarily of kaolinite, illite and montmorillonite, with minor quantities of ferric oxide, finely divided silica and titanium dioxide, and trace amounts of lime, magnesia, soda, and potash. More precisely, they contain between 50–75% by weight of SiO_2 , between 15–35% by weight of Al_2O_3 and between 0.5–10% by weight of Fe_2O_3 with conventional silica, alumina and iron contents of approximately 57:27:1. Ball clays are distinguished from the more common clays such as kaolin clays, by their lower alumina contents, e.g. 15–35% as opposed to 37–50% for kaolin clays. In addition, between 50–90% of the particles which make up the ball clays employed by the invention have a diameter of 10 microns or less. Furthermore, and as contrasted with the subsequently discussed alkali metal aluminosilicates, the ball clays have a total alkali and alkaline earth metal content of between 0.5–4% by weight. It should be noted that in references made to the quantity of ingredients present in clays or minerals, the total weight of the composition from which the percentages are derived may contain minor quantities of organic materials. Typical of these hydrated aluminum silicates are the Maryland and Tennessee ball clays generally, and such specific materials as Saxon clay, Yankee ball clay, Rex Clay, XB ball clay, pyrophyllite, and the like.

The lower melting material is preferably feldspar but one may generally employ the Group I and II metal aluminosilicates of sodium, potassium, calcium, magnesium or barium aluminosilicates, combinations thereof, or aluminosilicates which contain two or more of the specified metal ions, i.e. Na, K, Ca, Ba, or Mg. In addition, these metals are present in a quantity of between 6–20% by weight, and preferably 8–15% by weight. Examples of such aluminosilicates include the feldspars such as orthoclase, albite, hydropheane, microcline, anorthite, anorthoclase, etc.; and such compounds as nepheline, cancrinite, thomsonite, eucryptite, grossularite, aluminite, phillipsite, etc. In addition, the majority of the particles making up these compositions also have diameters of no more than 20 microns.

The examples in the following table provide a number of formulations suitable for use in the present invention in which the quantities of ingredients are expressed as percentages by weight:

TABLE 1

Ingredients	Examples							
	1	2	3	4	5	6	7	8
Feldspar I. -----	47	73.3	13	-----	-----	36.75	-----	76
Feldspar II. -----	-----	-----	-----	-----	-----	-----	70	-----
Feldspar III. -----	-----	-----	-----	-----	-----	-----	-----	70
Nepheline. -----	-----	-----	73.5	-----	-----	-----	-----	-----
Ball clay I. -----	47	18.3	79	-----	46.0	18.5	17	17
Ball clay II. -----	-----	-----	18.5	-----	-----	-----	19	-----
Ball clay III. -----	-----	-----	-----	46.0	36.75	-----	-----	-----
Colloidal silica (solids) -----	2.7	5.1	3	3	3	3	10	1.7
Bentonite. -----	3.3	3.3	5	5	5	5	3	3.3

Feldspar I in the above table comprised the following quantities of ingredients expressed in percentages by weight:

SiO_2 -----	67.53
Al_2O_3 -----	19.40
Fe_2O_3 -----	.06
TiO_2 -----	Trace
CaO -----	1.36
MgO -----	Trace
Na_2O -----	6.83
K_2O -----	4.58
Organic -----	.22

Its sieve analysis was a residue of 0.3% on a 200 mesh, and 3.2% on a 325 mesh (68.2% of the particles possessed a diameter of 20 microns or less). Feldspars II and III were similar in respect to ingredients and total alkali and alkaline earth metal content, i.e. approximately 12-16% by weight, but feldspar II possessed a higher K_2O and lower Na_2O content, while feldspar III contained a higher CaO and MgO content.

Ball clays I and II in the above table contained the following quantities of ingredients and possessed the specified properties:

	Ball Clay I	Ball Clay II
SiO_2	60.19	58.66
Al_2O_3	26.47	25.40
Fe_2O_3	0.89	2.29
TiO_2	1.72	1.21
CaO	0.31	0.13
MgO	0.23	0.75
Na_2O	0.33	0.32
K_2O	0.44	2.55
Organic.....	9.42	8.69
pH.....	4.4	3.9
Oil absorption percent.....	34.5	-----
Residue on 200 mesh percent.....	0.8	-----
Particles having diameter of 10 microns or less percent.....	81.0	93.0
Shrinkage at 2,000° F. at.....	2.4	3.6

Ball clay III was similar to ball clay I with the exception that a portion of the aluminum silicate was present as pyrophyllite.

The colloidal silicas of the foregoing examples were employed as aqueous dispersions containing between 10-60% by weight of colloidal silica and preferably 40% by weight. However, the quantities recited refer to the amount of silica solids employed.

The impregnants of the above examples were prepared by admixing the ingredients with water to form a slurry. While a slurry containing 20-70% and preferably 50% by weight of water is preferred in most cases, the ratio must be gauged to yield an impregnant of the viscosity desired for the specific applicator system and substrate employed. Sodium hexameta-phosphate may also be added to reduce viscosity and such addition is optional. In the case of low density fibrous substrates, the viscosity of the impregnant is normally increased by increasing the percentage of solids. This may be employed to control the rate and depth of impregnation. However, the impregnation depth and rate may also be controlled by means independent of the impregnant. For example, vacuum or mechanically pressurized impregnation, e.g. a doctor blade, may be employed to force a highly viscous impregnant to the desired depth within the fibrous substrate. Alternately, the fibrous substrate may be pre-treated with a wetting agent, e.g. sprayed with a solution of an organo-silicone fluid, to facilitate and control the rate and depth of impregnation by viscous slurries. Similarly, the wetting agent may be incorporated in the impregnant. The siliceous fibers which make up the low density boards treated in accordance with the invention, frequently contain a water repellent which causes the board to resist wetting and penetration by the aqueous slurries which are employed. In such cases, either the board should be pre-treated with a wetting agent, or an agent capable of modifying the surface tension properties of the slurry should be incorporated in the slurry. A wetting agent which has proved highly satisfactory for the above purpose is sodium dioctyl sulfosuccinate having a molecular weight of 444 and available from American Cyanamid under the trade-name Aerosol OT.

All of the impregnants of the above examples were applied to siliceous fiber acoustical boards and subjected to a simulated fire test to yield fire ratings of at least 2 hours. These ratings represent a substantial improvement

over untreated boards which fail after approximately 15-45 minutes, or boards treated or impregnated with clays, silica, alumina, and the like, which normally provide a one hour rating.

Various other ingredients such as reinforcements, opacifiers, fillers, wetting agents, antifoaming agents, dispersing agents, and the like, may be added to the inventive impregnants. Typical of such additives are asbestos, asbestine, wollastonite, titania, zircon, zirconia, alumina, carbon black, crystalline silica, calcium carbonate, barium sulfate, magnesium carbonate, ferric sulfate, sodium hexametaphosphate, and the like.

It is apparent that the inventive combinations of higher and lower melting materials may be simulated by adding fluxing agents to hydrated aluminum silicates having a high melting point, e.g. 2000-3500° F. For example, a fluxing agent such as sodium oxide may be added to a ball clay in a quantity adequate to exert a fluxing effect upon only a portion of the clay, e.g. 8% by weight. Such an expedient also results in a molten or ceramic adhesive phase but is more expensive and difficult to prepare, since minerals which naturally contain a fluxing agent content, e.g. feldspar, are readily available.

II.—Impregnated products

As previously mentioned, the products of the invention comprise an acoustical panel, board, or tile formed from siliceous fibers bonded or entangled into a mass having a density in the range of 1 to 30 p.c.f., and possessing two substantially parallel major surfaces, one of which is impregnated with the inventive compositions. The resultant product retains its desirable acoustical and thermal insulating values, while yielding highly improved structural integrity and resistance to heat or flame transfer when exposed to high temperatures. In order to preserve the acoustical absorption properties of the board, an unimpregnated zone of at least 50 mils and preferably 125 mils must be preserved at the surface opposite the impregnated region. This unimpregnated zone also contributes to the thermal insulating properties of the boards. In turn, an impregnated region having a thickness of at least 125 mils is adequate for the ceiling board and an impregnated region extending at least below the kerf slot of the ceiling tile provides structural integrity permitting the boards to endure temperatures as high as 2500° F. for short periods of time without failing. It should be realized that in the case of thicker boards, the temperatures which are experienced by the impregnant are lower as the result of the layer of low density thermal insulation which is present at the outset between the impregnant and the heat source.

The glass fiber boards which are impregnated according to the invention have an apparent density of from 1 to 30 pounds per cubic foot and are from 250 to 3,000 mils thick. Preferred embodiments of the invention comprise fibrous glass boards having a density in the range of 8 to 15 pounds per cubic foot, a thickness of from 350 to 1500 mils, and an impregnated region having a thickness of about 125 to 750 mils and generally about 125 to 200 mils for the ceiling boards; and an impregnated region for the ceiling tiles of about 250 to 750 mils and generally about 300 to 350 mils. Normally, the unimpregnated zone will comprise about 125 mils. Examples of such structures comprise one inch thick ceiling board with an impregnated zone or structural layer having a thickness of 125 mils, and a ½ inch thick ceiling tile having an impregnated zone having a thickness of 250 to 300 mils. In the case of boards having a density in the range of 9 to 13 pounds per cubic foot and a thickness of no more than one inch, impregnants constituting between 0.3 to 0.8 pound (solids) per square foot of the impregnated board have proved satisfactory. It is preferred, however, that the impregnants constitute about 0.4 pound (solids) per square foot of the impregnated

board in the case of the ceiling boards and about 0.65 pound per square foot of the impregnated board in the case of the ceiling tiles. For example, a preferred product comprises a ceiling board having a density in the range of 9 to 13 pounds per cubic foot, a thickness of between 350 to 1500 mils, and containing 0.4 pound of the impregnant for each square foot of the impregnated board. Similarly, a preferred ceiling tile comprises a board having a density in the range of 9 to 13 pounds per cubic foot, a thickness of between 350 to 1500 mils and containing 0.65 pound of the impregnant for each square foot of the impregnated board. As previously noted, however, boards having densities of between 1 to 30 pounds per cubic foot and a thickness as great as 3 inches have been prepared and have had the desired properties. It has also been found that ceiling boards and tiles having an overall weight of at least 1 pound per square foot are particularly valuable for their acoustical properties; in common with unimpregnated boards and tiles they do not reflect sound to a substantial extent, but, in addition, such impregnated products have a Sound Transmission Coefficient of about 40, which means that they do not transmit sound well. For example, a $\frac{3}{4}$ " x 2' x 5' board having an overall density of 16 pounds per cubic foot would meet these requirements, and could comprise a glass fiber board having an apparent density of 12 pounds per cubic foot and an applied impregnant according to the invention amounting to 0.25 pound of solids per square foot. Such a board is doubly useful: as a fire-rated acoustical material, as discussed above, and for sound attenuation, e.g., when used in a dropped ceiling structure with a plenum above the dropped ceiling, and partitions extending only as high as the dropped ceiling.

A product prepared in accordance with the invention is depicted by FIGURE 1 which provides a fragmentary, sectional view through a fibrous ceiling board prepared in accordance with the invention. As may be seen, one surface 11 of the low density, fibrous board 12 has been impregnated to yield a dense layer 13 which serves as a structural member. As also may be observed, the dense layer 13 also contains the fibers 14 of which the board 12 is composed, and which provide an additional benefit in reinforcing the dense layer 13. In turn, a low density unimpregnated phase 15 is left intact at the opposite surface 16 of the board 12 to provide the desired thermal and acoustical properties.

It will be appreciated from the foregoing discussion that a ceiling tile is similar in appearance except that an edge kerf is provided, and the structural member extends at least as far as the kerf. It should be noted that in the illustrated products the structural value of the dense layer 13 functions primarily after the fibers of the low density unimpregnated phase 15 have been weakened, softened or melted and following pyrolysis of the resin binder. At such time, the dense layer 13 becomes the primary or sole supporting, spanning, or suspending means and may provide the sole source of structural integrity for the board 12.

As previously mentioned, in the use of the inventive impregnants, i.e. containing a Group I or II metal aluminosilicate, it is believed that the dense layer 13 does not remain inert upon exposure to temperatures in the range of 1500° F. or higher. Specifically, when inorganic impregnants resistant to temperatures as high as 3000° F. were employed as the dense layer 13, the structures still failed at temperatures far below the melting points of the impregnants. It is believed that such failures were the result of the absence of an adequate binder phase within the impregnants. In effect, the inert impregnants suffered from thermal erosion or the like, lost integrity, and failed. It was found that the use of the inventive binder phase, i.e. bentonite and colloidal silica, overcame this inadequacy. It was further found that the presence of an ingredient which became molten upon exposure to temperatures in the range of 1500–2500° F., actually improved

the integrity of the structure when it was exposed to those temperatures.

The low density fibrous boards employed as the substrate utilized in the above examples and impregnated in the practice of the invention are typified by those disclosed by U.S. 2,790,741, 2,791,289, 2,882,764, 2,984,312, 3,082,143, 3,111,188, 3,118,516, 3,159,235, and similar structures employing glass, mineral wool or asbestos fibers.

Usually, the ceiling board is impregnated with an aqueous slurry of an impregnant according to the invention which contains 50% solids by weight and has a viscosity of approximately 100 to 200 centipoises. Impregnation to the desired depth i.e., approximately 125 to 180 mils, is conveniently achieved by knife coating which takes only a matter of seconds. In producing ceiling tiles, basic boards previously impregnated to the required depth and dried are cut to the desired dimension, kerfed, and painted. Since a greater depth of impregnation is required in the case of the ceiling tiles, impregnation can be effected by using a somewhat more dilute aqueous slurry of the impregnant according to the invention or by applying vacuum to a board carrying the same slurry to achieve the required depth of impregnation. Naturally, the viscosity should not be such that complete penetration or even partial penetration of the zone which is necessarily left unimpregnated for preservation of acoustical properties results. Alternately, the fibrous board may be first washed, but not thoroughly impregnated, i.e. without filling the voids throughout the thickness of the board and destroying insulating and acoustical value, with an inorganic material such as a clay slurry. The above described measures are advantageous for improving the total fire resistance of the structure but alone fail to provide adequate structural integrity at high temperatures.

III.—Preparation of the impregnated structures

As previously mentioned, the impregnated structures are prepared by depositing a slurry of the inventive impregnant upon one surface of the fibrous board, introducing the impregnant within the voids present upon and below one surface of the porous board without filling the voids present upon and adjacent the opposite surface of the board, and drying the impregnant.

A method for fabricating these products is depicted by FIGURE 2 which shows a schematic representation of apparatus suitable for the impregnation of low density siliceous fiber substrates. As shown, fibrous boards 21 are carried by means of a foraminous conveyor belt 22 beneath a trough 23 which dispenses an impregnating slurry 24 upon the upper surface 25 of the boards 21. The deposited impregnant 26 is then passed beneath a metering knife blade 27 which controls the thickness and consequently the quantity of the deposited impregnant 26. The boards 21 are then passed beneath and in contact with a pressure roll 28 which forces the deposited impregnant within the upper surface of the boards 21. To facilitate and control the depth of penetration of the boards 21 by the impregnant, the boards 21 and foraminous conveyor belt 22 may then be passed over a suction box 29 which serves to draw the impregnant within the boards 21 and may implement or replace the effect of the pressure roll 28. Alternatively, or in conjunction with the foregoing process, a sprinkler 30 may deposit a wetting agent such as a silicone fluid upon the upper surface 25 of the boards 21 prior to the deposition of the impregnant. The use of such a wetting agent facilitates the rapidity and extent of the penetration of the boards 21 by the impregnant. The drying of the impregnant may be accomplished by conventional oven treatments. For example, boards having a density of approximately 11.5 p.c.f., a thickness of between 350 to 1000 mils, and containing an impregnant layer weighing approximately 0.8 pound (aqueous slurry, 50% solids) per square foot of the board, may be oven dried at a temperature of 400° F., in approximately one hour.

Obviously, the primary sources of control for both the rapidity and the degree of impregnation reside in the matching of the viscosity and/or surface tension of the impregnant in relation to the density of the board the slurry may be merely metered and evenly deposited upon the upper surface of the board, and will immediately flow to a desired depth within the upper surface of the board, without penetrating to the opposite surface.

The following example is presented for the purpose of illustration and is intended in no way to limit the invention.

EXAMPLE

A binder composition was prepared, as described below, from the following ingredients:

	Parts by weight dry solids basis
Water	-----
Phenolic resin A ²	80
Pinewood pitch extract ²	20
Asbestine ²	56.6
TiO ₂	14.2

² Subsequently identified.

The water was added to a mixing tank provided with a propeller-type agitator, and agitation was begun. The other ingredients were then added in the order in which they are listed. Agitation was allowed to continue for about five minutes after the completion of the additions to assure substantial uniformity of the completed binder composition.

The above binder composition was sprayed into a forming hood for association with glass fibers which were simultaneously being projected onto a foraminous conveyor passing through the forming hood and collected in a wool-like open, non-woven, intermeshed mass. The wool-like mass of glass fibers and associated binder was advanced on the conveyor into a curing oven under a compression member to provide an apparent density of 12 pounds per cubic foot and a thickness of 3/4". In the curing oven at a temperature of about 400° F., the compressed mass of fibers and associated binder was converted to a hardened condition binding the fibers relative to one another at points of contact producing a hard board material. The time in the curing oven was about 240 seconds. The continuous board produced was cut into 1' x 1', 2' x 4', and 4' x 8' lengths.

The final boards had an ignition loss of about 12 percent, which means that approximately 12 percent of the total weight of the board was the organic binder composition. When subjected to the Underwriter's Laboratories Test as previously described, these board products received a fire rating of 1 hour.

In the above described binder composition, phenolic Resin A was produced by charging a reaction vessel with 80 parts of phenol, 123 parts of a 50 percent water solution of formaldehyde, 19.3 parts of water and 12 parts of barium hydrate (Ba(OH)₂·8H₂O), and heating the resulting charge for a total of seven hours during which time it was stirred by a propeller-type agitator. The charge first was heated to 110° F., and maintained at about such temperature for approximately two hours, and was then heated to and held at about 140° F. for the remaining five hours, at which time the refractive index of the reaction mixture was 1.4620 and the infra-red absorption analysis indicated that it was substantially free of unreacted phenol and also of methylene groups. The reaction products were then cooled to approximately 100° F., and neutralized with sulfuric acid to a pH of about 7.5. A 28 part charge of dicyandiamide was then added to the neutralized reaction products, and the resulting mixture was heated to and maintained at approximately 140° F. for an additional one hour period. The reaction products were then cooled to approximately

room temperature of 75° F., and neutralized with further sulfuric acid to a pH of approximately 7.4.

The pinewood pitch extract used in the binder composition was resinous in nature. It can be isolated as described in U.S. Patent 2,391,368 (page 2, col. 1, lines 34 and following). It had the following analysis:

	Percent
High melting furfural condensate (methanol insoluble)	6
Neutral oils (hydrocarbons, esters and ethers)	4
Rosin	9
Belro-phenol-lactone (probably C ₁₈ H ₁₄ O ₃ ·2(OCH ₃ OH))	5
Flavone type polyphenol (possibly about C ₁₅ H ₇ O ₂ ·3OH)	5
Fumic acid type compound	2
Pectic acid type compound	0.2
Air oxidized resin acid (unfused)	6
Strongly acidic compound	3
Weakly acidic, high melting phenolic compound	38
Relatively neutral phenol ethers and esters	19
Water soluble carbohydrates, etc.	1

The asbestine used in the binder composition set forth above had a specific gravity at 20° C. of 2.78, and the following chemical analysis:

	Percent
SiO ₂	58.61
MgO	28.88
CaO	4.97
Fe ₂ O ₃	0.30
Al ₂ O ₃	0.85
CO ₂	1.95
Ignition loss	4.41
pH	9.4

It had a particle size distribution as indicated by the following table:

Percent fines, about:	Relative diameter in microns
98	30
70	10
46	5
29	3
18	1
7	.5

The TiO₂ actually used in the binder composition identified above was anatase, and had a Refractive Index of 2.55, a specific gravity of 3.9, and comprised about 98 percent TiO₂, 1 percent Al₂O₃, the balance being impurities, and had an average particle mean diameter of about 0.3 micron.

The analysis of the specific fibers with which the foregoing tests were performed was as follows:

Ingredient:	Percent
SiO ₂	55.83
CaO	20.17
MgO	6.98
Al ₂ O ₃	5.53
Na ₂ O	10.55
K ₂ O	0.27
MnO	0.30
Fe ₂ O ₃	0.20
TiO ₂	0.17

Impregnant (57% solids)

An impregnating composition was prepared, as described below, from the following ingredients.

	Composition percent by weight based upon solids
Water, 220 gals. -----	
Hydrous Colloidal Silica (30% by weight solids), 550 lbs. -----	5.4
Sodium hexametaphosphate, 12 lbs. -----	10
Feldspar, 2200 lbs. -----	72.9
Bentonite, 100 lbs. -----	3.3
Ball clay, 550 lbs. -----	18.2
Sodium dioctyl sulfosuccinate 25%, 5 lbs. -----	15
Silicone antifoam agent (10 parts Stoddard solvent — 1 part silicone), 10 lbs. -----	
	99.8

The water and colloidal silica were charged to a mixing tank equipped with a shear-type mixing blade. Mixing was begun at low speed (about 800 revolutions per minute) and continued throughout the charging of the sodium hexametaphosphate, the feldspar and the bentonite. The Ball clay was then charged and mixed at high speed (about 1200 revolutions per minute) for fifteen minutes before reducing mixing speed (about 800 revolutions per minute) and charging of the sodium dioctyl sulfosuccinate and of the silicone antifoam agent. Low speed mixing was continued for an additional 5 minutes to assure uniformity of the dispersion and to break up any clay agglomerates. The silicone foam agent or Stoddard solvent alone is used as needed to control foaming.

The fibrous bonded boards produced as previously described were impregnated with the above composition in order to provide structural integrity, especially at temperatures in excess of 1000° F. The manner of impregnation will be understood by reference to FIGURE 2. A ceiling board 21 was first charged onto a foraminous conveyor belt 22 beneath a trough 23 which dispensed the impregnating slurry 24 upon the upper surface 25 of the board 21. The board 21 and deposited impregnant 26 were then passed beneath a metering knife blade 27 which controlled the thickness and the quantity of the deposited impregnant 26 so that the thickness was 125 mils and the amount deposited was 0.4 pound per square foot of the finished board. The board 21 was then passed beneath and in contact with the pressure roll 28 which further smoothed and helped to control the depth of the impregnant deposited within the upper surface of the board 21. The board 21 was then advanced into an oven (not shown) at a temperature of 400° F. where it was dried for about an hour. When subjected to the Underwriter's Laboratory Test as previously described, the ceiling board received a fire rating of at least two hours.

In a similar manner, kerfed ceiling tiles were produced from ceiling boards 21 impregnated to a depth of about 300 mils, using vacuum, by cutting to size and kerfing. When subjected to the Underwriter's Laboratory Test as previously described, the tile received a fire rating of at least two hours.

The final weight of the ceiling board was 1 pound per square foot and the final weight of the ceiling tile was 1.2 pounds per square foot. The STC³ of both products ranged from 26 to 42.

In the impregnation of a fibrous glass board having a density of 9–13 p.c.f., it is preferred to employ a slurry having a viscosity of approximately 100–200 centipoises. In such case, the slurry flows entirely within the board to a depth of approximately 125–180 mils within a matter of 1 to 3 seconds.

³ Sound Transmission Coefficient.

While the majority of the specification has concerned glass fibers, it must be realized that boards of a comparable density prepared from other siliceous fibers, e.g. mineral wool, asbestos, etc., are equally benefited by the practices of the invention, and yield products which simultaneously provide thermal and acoustical insulating values and prolonged structural integrity at temperatures in excess of 1000° F.

The treating materials of this invention can also be used for bonding aggregates such as vermiculite, perlite, glass foam pellets, glass beads and the like to produce products such as high temperature resistant pipe insulation. Likewise these treating compositions can be used for near total impregnation coupled with one or more back-coatings to provide novel effects. The coatings can be used for steel and wood decks to provide improved fire ratings.

It is further apparent that various alterations and substitutions may be made in the compositions, products and methods without departing from the spirit of the invention.

We claim:

1. An acoustical and thermal insulating element capable of maintaining structural integrity at temperatures in excess of 1000° F., comprising a plurality of siliceous fibers interbonded into an integral, void-containing mass having a density of between 1 and 30 pounds per cubic foot and shaped in the form of a panel having two substantially parallel major surfaces, a thickness of between 250 and 3000 mils, and kerfed side walls which extend between the major surfaces, and deposited within said voids present between said fibers and adjacent to one of said surfaces and to a depth of at least 125 mils, and to a depth extending at least below the kerfs, an impregnant comprising between 75% and 98% by weight of aluminum silicate particles consisting essentially of between 10% and 90% by weight of hydrated aluminum silicate containing between 50% and 75% by weight of SiO₂ and between 15% and 35% by weight of Al₂O₃, and between 10% and 90% by weight of an aluminosilicate of a metal selected from the group consisting of sodium, potassium, calcium, magnesium, barium and combinations of said metals, in which said metal is present in a quantity in excess of 6% by weight, and admixed with and interadhering said aluminum silicate particles between 2% and 25% by weight of a binder phase consisting essentially of between 1% and 20% by weight of colloidal silica, and between 1% and 15% by weight of bentonite, the remainder of said panel and the portion of said panel immediately adjacent to the second of said major surfaces to a depth of at least 50 mils being substantially devoid of said impregnant and retaining said density of 1 to 30 pounds per cubic foot.

2. A method for the preparation of acoustical and thermal insulating elements capable of maintaining structural integrity at temperatures in excess of 1000° F., comprising applying to one major surface of a panel comprising a plurality of siliceous fibers interbonded into an integral void-containing mass having a density of between 1 and 30 pounds per cubic foot, having two substantially parallel major surfaces, a thickness of between 250 and 3000 mils, and kerfed side walls extending between the major surfaces, an aqueous dispersion of an impregnant comprising between 75% and 98% by weight of aluminum silicate particles and consisting essentially of between 10% and 90% by weight of hydrated aluminum silicate containing between 50% and 75% by weight of SiO₂, and between 15% and 35% by weight of Al₂O₃, and between 10% and 90% by weight of an aluminosilicate of a metal selected from the group consisting of sodium, potassium, calcium, magnesium, barium and combinations of said metals, in which said metal is present in a quantity in excess of 6% by weight, and between 2% and 25% by weight of a binder

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phase consisting essentially of between 1% and 20% by weight of colloidal silica and between 1% and 15% by weight of bentonite, depositing said dispersion within the voids present between said fibers adjacent to said one major surface, to a depth of at least 125 mils from said one major surface, and to a depth at least extending below the kerfs in said side walls by applying suction to said second major surface subsequent to the application of said impregnant, maintaining the remainder of said panel and the portion of said panel adjacent to the second of said major surfaces to a depth of at least 50 mils from said second major surface substantially devoid of said aqueous slurry, and drying said panel to remove the aqueous phase of said dispersion.

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3. A method as claimed in claim 2 in which said impregnant also contains a wetting agent.

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