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(54) **FLAME-RESISTANT INSULATION**

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(57) **ABSTRACT**

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Flame-resistant insulation may be prepared by coating a glass fiber substrate, such as a fiberglass board, with an aqueous dispersion of vermiculite and expandable graphite. The insulation may also be pre-coated with a vermiculite dispersion prior to coating with a vermiculite/expandable graphite coating, and the vermiculite/expandable graphite coating may be covered with an FSK facing, or with a gypsum or portland cement layer. The resulting coated insulation board has superior flame resistance, and may be used as a component of a building or in vehicles.

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FLAME-RESISTANT INSULATION**BACKGROUND OF THE INVENTION****[0001]** 1. Field of the Invention

[0002] The present invention relates to flame and heat-resistant insulation, a process for making the insulation flame-resistant, and a method of protecting insulation from the effects of fire. The flame-resistant insulation of the present invention comprises insulation coated with a mixture comprising vermiculite and expandable graphite.

[0003] 2. Discussion of the Background

[0004] Insulating materials are commonly used in the fabrication of components of buildings, such as doors, walls, ceilings and roofs, or in vehicles such as automobiles, trucks, aircraft, and ships. Insulating materials are commonly made of inorganic fibers, such as glass or mineral fibers, in order to provide a degree of fire-resistance. Preferably, the fibers comprise a glass.

[0005] The majority of glass fiber products used in insulating materials are composed of either sodium borosilicate glass having a softening point of approximately 1290° F. or E-type borosilicate glass having a softening point as low as about 1529° F. More refractory glass fibers are also known, such as aluminosilicate S-glass, but these higher melting point glass fibers are relatively expensive, and therefore are unsuitable for low cost applications, such as building insulation, and are used primarily in specialty applications where the higher melting point of these fibers is critical.

[0006] Although glass or mineral fiber insulation materials have superior flame resistance compared to insulation materials based on flammable fibers (e.g., lignocellulose derived fibers), glass insulation materials have only modest flame and heat resistance because of their relatively low melting point. When exposed to flames, glass fiber insulation materials melt, thereby allowing flames and heat to penetrate through the insulation material, and consequently allowing the fire to spread. It is therefore desirable to enhance the flame and heat resistance of glass fiber-based insulation materials.

[0007] Re. 34,020 describes a fibrous composite material that is coated with a lamellar material such as vermiculite to enhance the fire-resistance of the material. While the fire-resistance of the coated fiberglass material is enhanced, the flame-retardancy and thermal insulation properties of the resulting composite are still inadequate.

[0008] U.S. Pat. No. 4,888,233 describes fire-resistant composite materials prepared by coating a polymeric substrate with a mixture of chemically delaminated vermiculite and a copolymer of ethylene and a vinyl monomer. While this coating decreases the flammability of the polymeric substrate, improved flame and heat resistance is still desirable.

[0009] U.S. Pat. No. 5,968,669 describes a fire-retardant coating for lignocellulosic materials which comprises a mixture of expandable graphite particles, an absorbent material such as calcium carbonate to absorb toxic gases, a polymeric binder, a "carbonific" material such as pentaerythritol, which forms a network binding expanded units of expandable graphite together, a blowing agent to generate an intumescent char foam, and a wetting agent to improve

wetting of the coating onto the substrate. However, U.S. Pat. No. 5,968,669 only describes coated lignocellulose-based materials, and the resulting coated articles, while flame-resistant compared to uncoated lignocellulose-based materials, are still flammable.

[0010] U.S. Pat. No. 5,972,434 describes fire-resistant glass fiber products coated with a nitrogen-containing compound and a boron-containing compound, which upon exposure to fire or high temperatures react with each other to form a refractory compound on the surface of the fibers.

[0011] However, there is still a need to provide inexpensive insulating materials having improved flame and heat resistance.

SUMMARY OF THE INVENTION

[0012] Accordingly, it is an object of the present invention to provide an improved flame and heat resistant insulating material comprising glass fibers coated with a mixture comprising vermiculite and expandable graphite. A second object of the present invention is to provide a method of preparing such flame-resistant insulation. A third object of the present invention is to provide a method of protecting insulation with a coating comprising vermiculite and expandable graphite.

DETAILED DESCRIPTION OF THE INVENTION

[0013] The insulation of the present invention comprises a glass fiber substrate coated with a mixture comprising vermiculite and expandable carbon. The substrate may include products formed from at least one layer of fibers. The fiber layers may comprise loose fibers, or may be woven, knitted, needle punched, felted, or otherwise combined in various ways to provide a unified structure. The fibers may be continuous, filamentary fibers, or discontinuous, staple fibers or agglomerations of such fibers. The fibers may comprise any conventional glass fiber commonly used in insulation products, including an E-glass, a C-glass, or a high boron content C-glass.

[0014] The fibrous substrate may be flexible or rigid. For example, the fibrous substrate may be a flexible batt, mat, blanket, fabric, or sheet, or a rigid slab or board. The density of the fibrous substrate may range from 1.0 lb/ft³ to 10 lb/ft³, including 1, 1.5, 2, 2.5, 3, 3.5, 4, 4.5, 5, 5.5, 6, 6.5, 7, 7.5, 8, 8.5, 9, and 9.5 lb/ft³, inclusive of all values and subranges therebetween. If the substrate is a rigid board, the density is preferably 2.5 to 6 lb/ft³. The substrate may be as thin as ½ inch or as thick as two inches, preferably 1 to 2 inches thick.

[0015] A binder may be used to capture and hold the fibers of the fibrous substrate together. The binder can be organic or inorganic. The binder can be a thermosetting polymer, a thermoplastic polymer, or a combination of both thermoplastic and thermosetting-polymers. Preferably, the thermosetting polymer is a phenolic resin, such as a phenol-formaldehyde resin, which will cure or set upon heating. When binder is used in the insulation product, the amount of binder can be from 1 to 30 wt %, preferably from 3 to 25 wt %, more preferably from 6 to 18 wt %.

[0016] The fibrous substrate may be composed only of glass fibers, or may have at least one additional layer laminated thereto. The additional layer may include a woven

or non-woven fiberglass fabric layer, or may be a FSK (foil-scrim-kraft) sheet glued onto the surface, a gypsum cement layer, or a quick-set cement layer. For example, the quick-set cement layer may be a conventional Portland type cement product. The gypsum cement may be any cementitious material containing gypsum. These coatings can be applied to one or both surfaces of the fiberglass substrate.

[0017] The coating of the present invention, which is applied to the substrate, comprises a dispersion of vermiculite mixed with expandable graphite. The vermiculite may include the minerals known both scientifically and commercially as vermiculite, including the chloride-vermiculites. For example, the vermiculite may be any naturally occurring micaceous hydrated magnesium-aluminum-silicate which has been chemically delaminated, for example by dispersing the vermiculite in an aqueous solution containing cations such as alkylammonium cations, and lithium cations. Particularly effective vermiculite dispersions include MicroLite® dispersions (W.R. Grace & Company). The dispersion of vermiculite may consist of only the chemically delaminated vermiculite and water, but may also include vermiculite dispersions which include a small amount of an organic binder or other additives to stabilize the dispersion and facilitate coating and adhesion to the fibrous substrate (e.g., dispersing agents or organic adhesion promoters). The dispersion of vermiculite may have as low as 1% solids, or levels as high as 50% solids. Solids contents of approximately 5 to 25%, more preferably 10 to 20%, most preferably approximately 16.5% are desirable.

[0018] Any conventional type of expandable graphite may be used. Expandable graphite differs from other forms of graphite in that it is specially treated to expand in volume upon heating. In order to become expandable, the graphite may be treated, for example, with sulphuric acid in order to intercalate sulphuric acid between the layers of the graphite. The treated graphite is then washed and dried, providing a dry pourable material. Upon heating, the intercalated material, e.g., sulphuric acid, volatilizes between the graphite layers, thereby expanding the volume of the graphite flake. The high volume provided by this expansion provides an insulative layer which reduces heat transfer through the material, mass loss, and reduces the generation of smoke.

[0019] The volume expansion of the expandable graphite may be up to 100 times the original thickness of the flake, depending on the specific type and amount of intercalant used. The percent expansion is typically in the range of 50 to 250% by volume, depending upon the amount of intercalant added to the graphite. The onset temperature of the expansion typically occurs at temperatures between 230° C. and 280° C. Suitable particle sizes are in the range of 50 to 220 mesh.

[0020] The relative amounts of vermiculite and expandable graphite in the coatings of the present invention may range from 10:1 to 1:10 (dry weight of vermiculite: dry weight of expandable graphite). The preferred ratio of vermiculite to expandable graphite is 5:1 to 1:5, more preferably 3:1 to 1:3, most preferably approximately 2:1. The total solids content of the coating of the present invention, prior to application, may be approximately 15 to 60 wt. %, preferably from 15 to 50 wt. %, more preferably from 15 to 30 wt. %.

[0021] The coating may be prepared by mixing an aqueous dispersion of vermiculite, optionally containing dispersing

agents, binders, and adhesion promoters, with an aqueous dispersion of expandable graphite, or dry expandable graphite. Alternatively, the vermiculite and expandable graphite may be mixed as dry powders, and then dispersed in water using conventional dispersing equipment. The coating of the present invention may be coated onto the substrate by any conventional method such as brushing, spraying, roller coating, gravure coating, curtain coating, and slot-die coating.

[0022] The coating of the present invention may be applied as a single coating to the substrate, or applied in two or more coating steps. In addition, a coating of vermiculite or expandable graphite may be applied to the substrate, prior to coating with the mixture of vermiculite and expandable graphite. The coating may be air-dried, or oven-dried. The dry weight of the coating may be 5 to 30 g/100 in², preferably 10 to 30 g/100 in², more preferably 15 to 30 g/100 in², most preferably 15 to 25 g/100 in².

EXAMPLES

[0023] The flame and heat-resistance of insulation boards, both coated and uncoated with the coating composition of the present invention, were tested for flame resistance in the following manner. The coated board was mounted horizontally above a Bunsen burner flame so that the coated surface of the board was exposed directly to a flame having an approximate flame temperature of 1750° F. The temperature of the board was measured over a one hour period at the middle of the thickness of the board, directly above the flame, and the opposing surface of the board directly above the flame (i.e., the surface opposite the side directly exposed to the flame). The maximum temperature measured at the center of the thickness of the board (i.e., the maximum center of thickness temperature) and the maximum temperature at the top surface (i.e., the maximum top surface temperature) were measured for one hour after exposing the board to the flame. Lower temperatures are indicative of better flame and heat-resistance.

Example 1

[0024] A 4.8 lb/ft³ fiberglass insulation board (ULTRA-DUCT™, CertainTeed), having a thickness of 1 inch was coated with a mixture of MicroLite® HTS vermiculite dispersion (W. R. Grace) and MYZR802 expandable graphite (Hebei Maoyuan Chemical Industry Company of China) in a weight ratio of 2:1 vermiculite:expandable graphite, and a solids content of 17.2 wt. %. The mixture was painted onto one surface of the board using a paint brush, and after air drying for 48 hours, had a dry weight of 18.7 g/100 in². After exposure to a Bunsen burner flame, as described above, the maximum center thickness temperature was 666° F. and the maximum top surface temperature was 424° F. The surface of the board was somewhat discolored by exposure to the flame, but no obvious melting of the glass fibers was observed.

Example 2

[0025] A 4.6 lb/ft³ fiberglass insulation board was coated as in Example 1, above, except that the solids content of the coating mixture was 46.9 wt. %, and dry weight of the coating was 21.3 g/100 in². After exposure to a Bunsen burner flame, the maximum center thickness temperature was 476° F. and the maximum top surface temperature was 488° F.

Example 3

[0026] A 4.3 lb/ft³ fiberglass insulation board, as in Example 1, was first coated with MicroLite® HTS vermiculite dispersion (16.4 wt. % vermiculite), and dried for 24 days, thereby forming a vermiculite film having a dry weight of 6 g/100 in². A coating of a 2:1 mixture (by weight) of a MicroLite® HTS vermiculite dispersion and MYZR802 expandable graphite (solids content 47.5 wt. %), was then applied over the vermiculite film and dried for 12 days. The coating of the mixture of vermiculite and expandable graphite had a dry weight of 15.1 g/100 in². The coated board was then exposed to a flame, as in Example 1. The maximum center thickness temperature was 698° F. and the maximum top surface temperature was 348° F. The surface of the board was somewhat discolored by exposure to the flame, but no obvious melting of the glass fibers was observed.

Example 4

[0027] A 4.5 lb/ft³ fiberglass insulation board was first coated with a MicroLite® HTS vermiculite dispersion (16.1 wt. % vermiculite), then coated with a mixture of MicroLite® HTS vermiculite and MYZR802 expandable graphite (weight ratio of 2:1; solids content of 46.9 wt. %) as in Example 3, above. The dry weight of the vermiculite coating was 5.2 g/100 in² and the dry weight of the vermiculite/expandable graphite coating was 14.4 g/100 in². After exposure to a flame, the maximum center thickness temperature was 382° F. and the maximum top surface temperature was 426° F.

Example 5

[0028] A 4.7 lb/ft³ fiberglass insulation board, as in Example 1, was coated with a mixture of MicroLite® HTS vermiculite and MYZR802 expandable graphite (weight ratio 2:1; solids content of 45.3 wt. %). After drying for 10 days, the coating had a dry weight of 18.7 g/100 in². A 6.4 g FSK facing (COMPAC FB30) having a dry weight of 7.3 g/100 in² (0.9 grams of dry glue) was applied over the vermiculite/expandable graphite coating with a water-based polyvinyl acetate type adhesive manufactured by Henkel Corporation. The side of the board having the FSK facing was then exposed to a Bunsen burner flame, as described above. The maximum center of thickness temperature was 584° F., and the maximum top surface temperature was 449° F. The FSK facing was melted and distorted by the expansion of the expandable graphite, but no melting of the glass fibers was observed.

Example 6

[0029] A 4.5 lb/ft³ fiberglass insulation board was first coated with a mixture of MicroLite® HTS vermiculite and MYZR802 expandable graphite (weight ratio 2:1; solids content of 45.6 wt. %), then covered with a FSK facing as described in Example 5, above. The dry weight of the vermiculite/expandable graphite coating was 16.9 g/100 in² and the dry weight of the FSK facing and adhesive (6.1 g facing; 2.4 g adhesive) was 8.5 g/100 in². After exposure to a Bunsen burner flame, the maximum center thickness temperature was 756° F., and the maximum top surface temperature was 366° F.

Example 7

[0030] A 4.4 lb/ft³ fiberglass insulation board was first coated with mixture of MicroLite® HTS vermiculite and

MYZR802 expandable graphite (2:1 weight ratio; solids content of 48.5 wt. %), air dried for 10 days, then coated with a layer of gypsum. The gypsum was allowed to cure for 72 hours. The dry weight of the vermiculite/expandable graphite coating was 19.2 g/100 in² and the dry weight of the gypsum cement coating was 71 g/100 in². After exposure to a Bunsen burner flame, the maximum center of thickness temperature was 645° F., and the maximum top surface temperature was 368° F. The gypsum layer flaked off of the surface of the board, and the surface of the board was somewhat discolored by exposure to the flame, but no obvious melting of the glass fibers was observed.

Example 8

[0031] A 4.8 lb/ft³ fiberglass insulation board was first coated with a mixture of MicroLite® HTS vermiculite and MYZR802 expandable graphite (weight ratio 2:1; solids content of 52.5 wt. %), as in Example 7, dried for 10 days, and then coated with a layer of quick-set cement. The quick-set cement was allowed to cure for 72 hours. The dry weight of the vermiculite/expandable graphite layer was 23.2 g/100 in², and the dry weight of the quick-set cement was 56.4 g/100 in². After exposure to a Bunsen burner flame, the maximum center of thickness temperature was 636° F., and the maximum top surface temperature was 454° F. The quick-set cement layer flaked off of the surface of the board where it was exposed to the flame, and the board was somewhat discolored by exposure to the flame, but no obvious melting of the glass fibers was observed.

Example 9

[0032] A 4.5 lb/ft³ fiberglass insulation board was first coated with a mixture of MicroLite® HTS vermiculite and MYZR802 expandable graphite (weight ratio 1:1; solids content of 42.7 wt. %), and dried for 10 days. The dry weight of the vermiculite/expandable graphite coating was 16.6 g/100 in². After exposure to a Bunsen burner flame, the maximum center of thickness temperature was 900° F., and the maximum top surface temperature was 434° F. The coating was notably expanded where the flame contacted the board, but no melting of the glass fibers was observed.

Example 10

[0033] A 4.6 lb/ft³ fiberglass insulation board was first coated with a mixture of MicroLite® HTS vermiculite and MYZR802 expandable graphite (weight ratio 3:1; solids content of 36.6 wt. %), and dried for 10 days. The dry weight of the vermiculite/expandable graphite coating was 14.1 g/100 in². After exposure to a Bunsen burner flame, the maximum center of thickness temperature was 1144° F., and the maximum top surface temperature was 442° F. The coating was notably expanded where the flame contacted the board, but no melting of the glass fibers was observed.

Example 11

[0034] A 4.6 lb/ft³ fiberglass insulation board was first coated with a mixture of MicroLite® HTS vermiculite and

MYZR802 expandable graphite (weight ratio 5:1; solids content of 29.9 wt. %), and dried for 10 days. The dry weight of the vermiculite/expandable graphite coating was 10.5 g/100 in². After exposure to a Bunsen burner flame, the maximum center of thickness temperature was 682° F., and the maximum top surface temperature was 367° F. The coating was notably expanded where the flame contacted the board, but no melting of the glass fibers was observed.

Example 12

[0035] A 4.6 lb/ft³ fiberglass insulation board was first coated with a mixture of MicroLite® HTS vermiculite and MYZR802 expandable graphite (weight ratio 10:1; solids content of 22.9 wt. %), and dried for 10 days. The dry weight of the vermiculite/expandable graphite coating was 8.2 g/100 in². After exposure to a Bunsen burner flame, the maximum center of thickness temperature was 796° F., and the maximum top surface temperature was 385° F. The coating was notably expanded where the flame contacted the board, but no melting of the glass fibers was observed.

Comparative Example 1

[0036] An uncoated 4.6 lb/ft³ fiberglass insulation board, which was otherwise identical to the insulation boards which were coated in Examples 1-8, above, was exposed to a Bunsen burner flame. The maximum center of thickness temperature was 972° F., and the maximum top surface temperature was 794° F. The board was discolored, and the glass fibers were melted nearly through the thickness of the board, where exposed to the flame.

Comparative Example 2

[0037] A 4.96 lb/ft³ fiberglass insulation board was coated with a dispersion of MicroLite® HTS vermiculite (solids content of 17.2 wt. %). The dry weight of the vermiculite coating was 5.6 g/100 in². After exposure to a Bunsen burner flame, the maximum center of thickness temperature was 800° F., and the maximum top surface temperature was 673° F. The surface of the board was somewhat discolored by exposure to the flame, and the glass fibers were melted to a depth of approximately half of the thickness of the board where exposed to the flame.

Comparative Example 3

[0038] A 4.5 lb/ft³ fiberglass insulation board was coated with a dispersion of MicroLite® HTS vermiculite (solids content of 17.0 wt. %). The dry weight of the vermiculite coating was 5.9 g/100 in². After exposure to a Bunsen burner flame, the maximum center of thickness temperature was 982° F., and the maximum top surface temperature was 631° F.

[0039] As shown in the above Examples (Summarized in Table 1, below), a glass fiber insulation board coated with a mixture of vermiculite and expandable carbon provides significantly better flame and heat resistance compared to uncoated insulation, or insulation coated with vermiculite alone

TABLE 1

Horizontal Insulation board exposed to a Bunsen burner flame - approximately 1750° F.			
Description	Dry weight of Treatment for 100 square inch board	Maximum Center of Thickness Temperature within 1 hour	Maximum Top Surface Temperature within 1 hour
<u>Examples</u>			
1 2:1 by Weight HTS Vermiculite: Expandable Graphite	18.7	666	424
2 2:1 by Weight HTS Vermiculite: Expandable Graphite	21.3	476	488
3 1) HTS Vermiculite	6	698	348
2) 2:1 by Weight HTS Vermiculite: Expandable Graphite	15.1		
4 1) HTS Vermiculite	5.2	382	426
2) 2:1 by Weight HTS Vermiculite: Expandable Graphite	14.4		
5 1) 2:1 by Weight HTS Vermiculite: Expandable Graphite	18.7	584	449
2) 6.4 g FSK facing applied with Henkel glue	7.3		
6 1) 2:1 by Weight HTS Vermiculite: Expandable Graphite	16.9	756	366
2) 6.1 g FSK facing applied with Henkel glue	8.5		
7 1) 2:1 by Weight HTS Vermiculite: Expandable Graphite	19.2	645	368
2) Gypsum (43 g cement 21 g H ₂ O)	71		
8 1) 2:1 by Weight HTS Vermiculite: Expandable Graphite	23.2	636	454
2) Quick Set Cement (90 g cement 17.5 g H ₂ O)	56.4		
9 1:1 by Weight HTS Vermiculite: Expandable Graphite	16.6	900	434
10 3:1 by Weight HTS Vermiculite: Expandable Graphite	14.1	1144	442
12 5:1 by Weight HTS Vermiculite: Expandable Graphite	10.5	682	367
13 10:1 by Weight HTS Vermiculite: Expandable Graphite	8.2	796	385
<u>Comparative Examples:</u>			
1 Untreated Board		972	794
2 HTS Vermiculite	5.6	800	673
3 HTS Vermiculite	5.9	982	631

[0040] Obviously, numerous modifications and variations on the present invention are possible in light of the above teachings. It is therefore to be understood that within the scope of the appended claims, the invention may be practiced otherwise and as specifically described herein.

What is claimed as new and is intended to be secured by Letters Patent is:

1. Flame and heat-resistant insulation comprising a glass fiber substrate coated with a mixture comprising vermiculite and expandable graphite.

2. The flame and heat-resistant insulation of claim 1, wherein the weight ratio of vermiculite to expandable graphite in the coating is in the range of 10:1 to 1:10.

3. The flame and heat-resistant insulation of claim 1, wherein the mixture further comprises an organic binder.

4. The flame and heat-resistant insulation of claim 1, wherein the mixture further comprises an inorganic binder.

5. The flame and heat-resistant insulation of claim 1, wherein the coating has a dry weight of 5 to 30 g/100 in².

6. The flame and heat-resistant insulation of claim 1, wherein the coating has a dry weight of 10 to 30 g/100 in².

7. The flame and heat-resistant insulation of claim 1, wherein an FSK facing is adhered to the coating.

8. The flame and heat-resistant insulation of claim 1, wherein a cement layer is deposited on the coating.

9. The flame and heat-resistant insulation of claim 1, wherein a gypsum layer is deposited on the coating.

10. The flame and heat-resistant insulation of claim 1, wherein said glass fiber substrate is selected from the group consisting of a glass fiber flexible mat, a glass fiber fabric, a glass fiber sheet, and a glass fiber board.

11. The flame and heat-resistant insulation of claim 10, wherein the glass fiber substrate is a glass fiber board.

12. The flame and heat-resistant insulation of claim 11, wherein the board has a density of 2.5 to 6 lb/ft³.

13. A method of preparing the flame and heat-resistant insulation of claim 1, wherein a mixture comprising vermiculite and expandable graphite are dispersed in water, said dispersion is coated onto a glass fiber substrate, and dried.

14. The method of claim 13, wherein said depositing is selected from the group consisting of spraying, roller coating, gravure coating, curtain coating, slot-die coating and brushing.

15. A door panel comprising the flame and heat-resistant insulation of claim 1.

16. A wall comprising the flame and heat-resistant insulation of claim 1.

17. A building insulation comprising the flame and heat-resistant insulation of claim 1.

18. A vehicle insulation comprising the flame and heat-resistant insulation of claim 1.

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