PATENT SPECIFICATION

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(54) MANUFACTURE OF FIRE PROTECTION MATERIALS

(71) We, BASF AKTIENGESELLSCHAFT, a German Joint Stock Company of 6700 Ludwigshafen, Federal Republic of Germany, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention relates to a process for the manufacture of a laminate useful for fire protection and to laminates made by the process.

It has been disclosed that sheets or strips of hydrated alkali metal silicates, when exposed to high temperatures in the event of a fire, form a fine-celled, firm, non-combustible and heat-insulating foam which, as a result of developing a foaming pressure, is able to protect gaps, slits and other openings and passages in buildings against the penetration of fire and smoke.

German Published Applications DAS 1,169,832 and 1,471,005, disclose a process for the manufacture of such fire protection sheets. In this process, reinforcing fibers or fabrics are embedded in a cast layer of an alkali metal silicate solution, normally containing more than 50% by weight of water; thereafter, the water is partially removed by heating and the layer is solidified to form a sheet. The drying of relatively thick sheet-like materials is, however, expensive and time-consuming, above all if the sheets are manufactured individually and in large numbers. The drying time of commercial fire protection sheets prepared from hydrated alkali metal silicate is several hours even if efficient installations are used.

It has also been disclosed that combustible materials, e.g., wood, paper or textiles, can be fireproofed by impregnating them with a waterglass solution. It is true that the materials treated in this way are difficult to ignite, but it is not possible to prevent their combustion on sustained exposure to a flame. Furthermore, the insulating action and mechanical stability of the materials is poor.

The present invention seeks to provide a process for the manufacture of fire protection materials which can be carried out simply and with relatively little expenditure of energy.

According to the present invention, there is provided a process for the manufacture of a laminate useful for fire protection, which comprises the following steps:

A) applying a paste of an alkali metal silicate, containing from 25 to 55% by weight of water, to a sheet-like carrier

B) heat-treating the resulting carrier to effect gelling of the alkali metal silicate without foaming, and

C) bringing together two or more layers of the resulting carrier and preferably bonding the layers to one another.

The sheet-like carriers preferably consist of inter-connected fibers or wires which below 150°C do not soften and do not stretch substantially. Examples of suitable materials are textile fabrics, above all cellulose-based fabrics, glass fibermats, metal gauzes and metal fabrics, and, above all, cellulose-based papers. The paper used should preferably have received a wet-strength finish, for example with a urea-aldehyde resin or a melamine-aldehyde resin or with a polyamido-imide, and should preferably weigh from 40 to 300 g/m². The thickness of the paper may vary within wide limits, e.g. from 40 to 400 μm. Further details of the types of paper, their manufacture and their properties are to be found in Ullmanns Encyklopädie der technischen Chemie, volume 13, pages 112—159. In the

preferred, continuous embodiment of the process, endless webs or strips of the carrier, which may be from 0.5 to 100 cm in width, are coated, the webs preferably having a width of from 50 to 150 cm and the strips preferably having a width of from 1 to 10 cm.

The carriers are coated with a paste of an alkali metal silicate which contains from 25 to 55, preferably from 30 to 50, % by weight of water. The alkali metal silicate may be a sodium silicate, potassium silicate or lithium silicate having a molecular ratio $Me_2O:SIO_2$ of from 1:1 to 1:6, especially from 1:3 to 1:4. The use of sodium waterglass is preferred. To obtain the prescribed water content, it is possible either to start with waterglass powder of low water content and add the requisite amount of water, or to start with a waterglass solution having the conventional water content of from about 60 to 70% by weight and add waterglass powder until the water content is from 5 to 25% by weight. The alkali metal silicate is in the form of a paste which in general has a viscosity, at 5°C, of from 1,000 to 10,000 mPas. In addition to the alkali metal silicate, the paste may contain conventional additives, e.g., dyes, pigments, foaming assistants and wetting agents, as well as carbohydrates or cellulose powder, which in the event of a fire form at high temperatures, a carbon skeleton.

In process state A, the carrier is coated with the paste, preferably on both sides. The thickness of one layer may be from 0.1 to 1.5 mm; preferably it is from 0.3 to 0.8 mm. Coating is preferably carried out continuously by drawing an endless web of the carrier, eg. in the form of a strip from 1 to 10 cm wide, through a vessel filled with the alkali metal silicate paste, from which vessel the web issues via a slot die, by means of which the thickness of the coating can be adjusted. However, the alkali metal silicate paste can also be applied to the carrier by knife-coating or calendering. Loose carriers, e.g. glass fiber mats, may be impregnated with the alkali metal silicate.

In stage B, the coated carrier is subjected to a heat-treatment, whereupon the alkali metal silicate gels, i.e. solidifies. Heating may be effected by means of hot gas, e.g. hot air, or by infrared radiation. The temperature is selected so that gelling takes place very rapidly, but as yet unaccompanied by foaming of the alkali metal silicate. Preferably, the surface temperature of the alkali metal silicate layer is above 40°, especially above 80°C. As a result of the fact that in general some water evaporates during gelling, the temperature of the alkali metal silicate layer does not rise above 100°C even if the temperature of the environment is somewhat higher. The duration of the heat treatment depends on the water content and on the thickness of the layer, and may be from a few seconds to several minutes, preferably from 1 to 10 minutes.

Finally, in stage C, two or more, especially three, layers of the coated material are brought together and are preferably bonded to one another. Bonding may be effected by the use of pressure or by welding the material whilst it is thermoplastic, or by a combination of both measures. In a preferred embodiment, the coated webs are first heated at from 50 to 100°C. This softens the alkali metal silicate. On then pressing the individual layers together, the alkali metal silicate coatings bond to one another. Pressing may be effected by means of conventional rollers or belt presses, which are preferably cooled to prevent the coating from sticking to the machine. Pressing somewhat reduces the overall thickness of the individual layers, so that the resulting finished laminates generally have a thickness of from 0.5 to 8 mm, preferably of from 1.0 to 5 mm, and a density of, preferably, from 1.2 to 1.8 g/cm^3. The pressing may be accompanied by molding the laminates to form profiles or half-shells. It is not absolutely essential to bond the layers of the coated material firmly to one another during manufacture of the laminates; such bonding may alternatively take place on storage or even only in the event of a fire.

After manufacture, the laminates may be trimmed to the desired length and width and may or may not be dusted with release agents, e.g. talc, to facilitate handling. Preferably, they are provided with a coating, or wrapped in a film, in a further process stage D. Suitable coatings are finishes based on polyvinyl chloride, epoxy resins or polyurethanes, which may or may not be cured subsequently. The laminates, which may or may not have been coated, can also be wrapped in plastic films or metal foils, preferably aluminum foils, to protect them against attack by water and carbon dioxide. It is particularly advantageous to employ sealable aluminum foils, e.g. an aluminum/polyethylene composite foil, and to seal the laminates therein.

The laminates manufactured in accordance with the invention can be used as fire protection materials in connection with openings and passages in buildings,

above all in connection with doors. In the event of a fire, the water still present in the alkali metal silicate in an amount of, preferably, from 25 to 45%, evaporates and the layer foams up. This generates a substantially unidirectionally acting foaming pressure, whereby the gaps and openings become filled with foam and, as a result, insulating. If fire protection materials which consist of only one layer of a coated carrier are used, foaming takes place in all directions. The foam exudes from the gap and the foaming pressure generated in many cases is insufficient for complete filling of the gap.

In the Examples, parts and percentages are by weight.

EXAMPLE 1

A paper which had received a wet-strength finish, weighed 90 g/m² and had a thickness of 150 μm, was used in the form of a 2.5 cm wide, rolled-up strip. The coating paste was brought to a solids content of 54% by mixing a 38% strength sodium waterglass solution (SiO_2:Na_2O=3.3:1) with an 81% strength sodium waterglass powder. The viscosity of the paste at 5°C was 10,000 mPa.s. The paper strip was passed at a speed of 3.0 m/min through a vessel filled with the paste and drawn off through a slot die, the latter being adjusted to give various thicknesses of coating. The strip, coated on both sides, was heated by means of infrared radiation over a 2 m long heating zone, during which treatment the alkali metal silicate gelled, and some water evaporated, at a surface temperature of only just 100°C. 3 strips were then brought together via rollers, heated by means of hot air to about 80°C and pressed together by means of water-cooled nip rollers. The water content of the finished laminates was determined in terms of the loss of weight on calcination at 600°C. To determine the foaming pressure, strips were placed next to one another, over an area of 400 cm², between aluminum foils, and introduced in this form into a gap of a platen press, the gap width being somewhat greater than the thickness of the strip. The press was heated to 300°C, causing the strips to foam up. After about 3 minutes, the resulting pressure was measured.

TABLE

Experiment	Thickness in mm Single strip	Laminates	Water content in %	Foaming pressure in N/mm²
a	0.8	2.3	39.5	0.58
b	1.0	2.5	37.4	0.58
c	1.2	2.8	39.2	0.61
d	1.4	3.2	40.9	0.56

EXAMPLE 2

Using the method described in Example 1, an endless paper strip was coated with a paste of 71% solids content, which was prepared by mixing 84% strength waterglass powder with 38% strength waterglass solution. The viscosity of the paste was 2,500 mPa.s.

The water content of the finished laminate was 32% and the thickness 2.5 mm.

The 2.5 cm wide strips were laid on a 7.8 cm wide composite foil of aluminum (15 μm) and polyethylene (20 μm). The foil was folded over the strip and sealed by heating to just above 100°C under light pressure.

EXAMPLE 3

An 8 cm wide, commercial glass mat weighing 50 g/m², and bonded with a urea resin, was impregnated with a sodium waterglass paste (having a water content of 45% and a viscosity of 4,000 mPa.s at 5°C). The impregnated strip was 0.9 mm thick. Gelling of the waterglass was carried out as described in Example 1. Two strips were then brought together, and pressed together, as described in Example 1 The water content of the finished laminate was 34% and the total thickness 1.6 mm. The foaming pressure was found to be 0.65 N/mm².

WHAT WE CLAIM IS:—

1. A process for the manufacture of a laminate useful for fire protection, comprising the following process steps:

A) applying a paste of an alkali metal silicate, containing from 25 to 55% by weight of water, to a sheet-like carrier,

B) heat-treating the resulting carrier to effect gelling of the alkali metal silicate without foaming, and

C) bringing together two or more layers of the resulting carrier.

2. A process as claimed in claim 1, wherein the carrier is cellulose-based paper.

3. A process as claimed in claim 1, wherein the carrier is a glass fiber mat.

4. A process as claimed in any of claims 1 to 3, wherein the sheet-like carrier is an endless web in the form of a strip having a width of from 1 to 10 cm.

5. A process as claimed in any of claims 1 to 4, wherein the alkali metal silicate is sodium waterglass.

6. A process as claimed in any of claims 1 to 5, wherein the sheet-like carrier is coated on both sides in stage A.

7. A process as claimed in any of claims 1 to 6, wherein the surface temperature of the alkali metal silicate layer in stage B is from 40 to 100°C.

8. A process as claimed in any of claims 1 to 7, wherein the layers are bonded to one another in stage C.

9. A process as claimed in claim 8, wherein three layers are bonded to one another in stage C.

10. A process as claimed in any of claims 1 to 7, wherein, in stage C, the layers are heated at from 40 to 100°C and the individual layers are then pressed together, causing welding of the softened alkali metal silicate coating.

11. A process for the manufacture of a laminate useful for fire protection carried out substantially as described in any of the foregoing Examples.

12. A process for the manufacture of a fire protection material wherein a laminate is manufactured by a process as claimed in any of claims 1 to 10 and the laminate is provided with a coating and/or wrapped in a foil in a subsequent process stage D.

13. A laminate useful for fire protection when manufactured by a process as claimed in any of claims 1 to 12.

14. A laminate as claimed in claim 13 which is in the form of a strip from 1 to 10 cm wide and from 1 to 5 mm thick, which laminate comprises at least 2 superposed paper strips which are coated with alkali metal silicate coatings, which contain from 25 to 45% of water, and are bonded to one another.

15. A laminate as claimed in claim 13 which is in the form of a web from 50 to 150 cm wide and from 1 to 5 mm thick, which laminate comprises at least 2 superposed paper webs which are coated with alkali metal silicate coatings, which contain from 25 to 45% of water, and are bonded to one another.

16. A laminate as claimed in any of claims 13 to 15, which is provided with a surface coating and/or is wrapped in a metal foil or plastics film.

17. A laminate as claimed in any of claims 14 to 16, wherein there are 3 of the superposed coated paper webs bonded to one another.

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