

PATENT SPECIFICATION

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(72) Inventor JOHN HURST.

(54) ADHESIVE ROLLS

(71) We, W. R. GRACE & Co., a Corporation organized and existing under the laws of the State of Connecticut, United States of America, of 7, Hanover Square, New York 5, New York, United States of America, 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:—

This invention relates to a structure in the form of a roll and is an improvement in or modification of the invention described in Patent Application No. 1,230,753; the rolls are related to the structures and methods of Patent Applications Nos. 1,230,754—6. The rolls are particularly useful for application to concrete surfaces and will be described herein chiefly by reference to such use. It is to be understood, however, that the same description applies, *mutatis mutandis*, to the sealing of other surfaces, particularly such as are found in buildings and civil engineering structures.

It is known to seal surfaces, particularly of concrete by forming thereon a membrane of a bituminous composition or the like which is substantially impermeable to moisture and water vapour. [The term "bituminous composition" is used in this specification to include compositions containing asphalt, tar or pitch.] It is also known that such a composition may form an adhesive for a layer of cladding material, e.g. tiles and

panels of linoleum, polyvinyl chloride, thermoplastic material, natural or reconstituted stone or cork, and wood block and wood strip flooring. In the past such membranes have been formed by the application *in situ* of hot liquid bitumen, tar or pitch or a cold solution or emulsion of bitumen, tar or pitch. If desired, the membrane may be reinforced with latex, asbestos fibres or other types of filler. It is also known to provide a waterproofing membrane by the use of films of polyethylene or synthetic or natural rubber, or of bituminous roofing felt.

These known methods suffer from serious disadvantages. Thus in general the known procedures require formation of the layer of sealant *in situ* with the consequent difficulties in ensuring a uniform layer and the expense of labour at the site. Furthermore where the surface is a concrete floor which is subject to rising moisture, the adhesive must form a water-impermeable and alkali-resistant membrane; in practice this means that a bituminous composition must be applied hot to the floor, which is highly inconvenient, or a solvent-based bituminous composition must be used, which is expensive and involves fire risk. Generally the adhesive compositions currently in use require, in order to permit evaporation of solvent or aqueous content, to be placed in position some time before the cladding material is applied if a satisfactory bond is to be obtained, and this again is inconvenient.

[Price 25p]

It is also known to provide the rear surface of cladding materials, particularly floor tiles, with a layer of contact adhesive covered by a protective sheet which is removed before placing the cladding material in position. However, it is not possible to provide a satisfactory water- and alkali-resistant continuous coating under the cladding material by this means, particularly on a typical rough dusty concrete surface.

Our Patent Application No. 1,230,753 relates to the use of preformed membranes of water-proofing pressure-sensitive adhesives, which provide substantial advantages over the known procedures. Thus in its broadest aspect our Patent Application No. 1,230,753 discloses and claims a structure comprising a sheet-like substrate and contiguous thereto a membrane of a water-proofing pressure-sensitive adhesive, the structure being at least 15 cm. in width and the membrane being at least 0.025 cm. thick.

As disclosed in Patent Application No. 1,230,753, the membrane can be a layer of the adhesive alone or the adhesive can contain reinforcing material, particularly to give the membrane mechanical strength; if desired the membrane can contain a plurality of layers of water-proofing pressure-sensitive adhesive (the adhesive being the same or different in the different layers), and the layers can be separated by reinforcing material in the form of a sheet.

The structures of Patent Application No. 1,230,753 can be divided into two broad types. The structures of the first type are those designed to provide a continuous waterproof membrane adherent to a surface, the outer surface of the membrane having a non-adhesive surface. The structures of the second type are those designed to provide on a surface a layer of a pressure-sensitive contact adhesive, either over the whole of the surface, thus simultaneously providing a continuous waterproof membrane, or over selected parts of the surface only.

In the structure of the first type, the substrate is a sheet-like support (e.g. a film of an organic polymer such as polyethylene, as explained in detail below) to which the membrane is permanently adherent (that is to say the support and the membrane remain adherent to each other when the structure is used), and the surface of the support remote from the membrane is non-adhesive. In the structures of the second type, the substrate is a protective coating (e.g. siliconised paper or like materials as explained in detail below) which can be physically removed from the membrane without substantial damage to the membrane.

Our Patent Application No. 1,230,753 then goes on to disclose that for storage purposes the surface of the membrane remote from the substrate must have a protective coating

thereon, and that the structures as produced in the factory, stored and brought to the site are in the form of laminates of the protective coating, the membrane and either a sheet-like support (the first type) or a second protective coating (the second type). The structures are conveniently available in the form of rolls.

It has now been realised, in accordance with the present invention, that in rolls of the structures as produced in the factory, stored and brought to the site, it is possible for the protective coating on the face of the membrane, which face is to be applied to the surface to be sealed and/or provided with a layer of contact adhesive, to be provided by the substrate itself, if the substrate is such that when the roll of the structure is unrolled, the substrate is physically separated from the said face of the membrane without substantial damage to the membrane. Thus the invention provides a structure in the form of a roll and comprising (a) a water-proof membrane comprising a waterproofing pressure-sensitive adhesive, and, contiguous to the membrane, (b) a sheet-like substrate, one face of the substrate being substantially more readily physically separable from the membrane than the other face, the membrane being at least 0.025 cm. thick and the substrate being at least 15 cm. wide.

Such rolls can be prepared by forming a substrate one face of which is substantially more readily separable from the pressure-sensitive adhesive than the other face, preparing a laminate of the substrate and a membrane of the pressure-sensitive adhesive with the said other face contiguous thereto, and rolling up the laminate. The said more readily separable face can be rendered more readily separable before, during or after the lamination with the membrane.

In structures of the first type described above, the differential separability of the two faces of the sheet-like support can readily be obtained by forming a release coating on one face only of the support; preferably the release coating is on the outer surface of the rolled-up support. The release coating can be formed in known manner, for example by deposition of a silicone, such as by the application to the membrane of a dispersion of a silicone compound which is cured with the aid of a catalyst and/or heat and forced ventilation. The outer and inner faces (or surfaces) of the substrate are those facing away from, and forcing towards, respectively, the centre of the roll.

In structures of the second type, where the substrate is a protective coating which is ultimately removed altogether from the membrane, a release coating is formed on each face of the substrate, e.g. as described above, but coatings of different levels of release properties are of course required in order to

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obtain the desired differential separability. Preferably the outer face of the rolled-up protective coating is more readily separable from the layer of adhesive than is the inner face of the protective coating.

When the rolls of the invention are used, they are simply unrolled, thus exposing one surface of the membrane, which surface is applied to the surface to be sealed and/or provided with a layer of contact adhesive; in the latter case the protective coating is removed at an appropriate stage to expose the other surface of the membrane ready for the application thereto of cladding material. If necessary the formation of a continuous waterproof seal over the whole surface is achieved by further applications so that each membrane overlaps its neighbour.

The widths of the novel rolls can be up to 120 cm. and even more, depending on the use to which they are to be put; generally they will be up to 91 cm. wide, with widths in the upper part of this range, e.g. 60 to 91 cm., being preferred where the whole of a surface is to be rendered waterproof. The thickness of the membrane and substrate can also vary widely depending on the intended end use, but will generally each be 0.025 to 0.6 or 0.9 cm.

A variety of adhesives can be used successfully for the membrane, but the adhesive must be such that the membrane will stick to the required surface without the use of heat or additional bonding agents; thus for application to surfaces of concrete, which are comparatively rough and dusty, the layer of adhesive must be at least 0.063 to 0.5 cm. thick; the thicker the layer of adhesive, the better the waterproofing effect, but in general a layer 0.063 to 0.4 cm. thick is satisfactory, with thicknesses at the upper end of this range being preferred. Thus for rolls of the first type a support having on it a single layer of adhesive 0.23 to 0.35 cm. thick is preferred, and for rolls of the second type a similar thickness is preferred; this may be achieved for example by two layers of adhesive, each 0.1 to 0.18 cm. thick, either side of a central reinforcing material.

Bituminous adhesives are generally suitable except where their colour is a disadvantage. If a bituminous adhesive is used, it is preferably formed of natural or synthetic rubber, virgin or reclaimed, blended into bitumen to provide a smooth mix. The ratio by weight of bitumen to rubber is suitably greater than 80:20, preferably up to 95:5, especially about 90:10. Other types of contact adhesive composition may include polychloroprene, butyl rubber, oil-based mastics or compositions containing rosins or rosin derivatives. Generally, suitable compositions have softening points (measured by the Ring and Ball method) of 60° to 140°, preferably 60°C to 110°C.

and penetration values of 50 to 400, preferably 150—300 at 25°C. (100 g. 5 secs—Institute of Petroleum method).

A wide variety of materials can be used to provide protective coatings in the rolls. Paper having a release coating thereon, e.g. siliconised paper or suitable material formulated from or with polytetrafluoroethylene, is satisfactory. Other materials include treated or modified films of organic polymers.

A wide variety of materials can be used as the sheet-like support, which forms part of the novel rolls of the first type noted above, and it is a particular advantage of such rolls that they can be used to provide a waterproof seal over a surface which combines the properties of the membrane and the support. It is generally desirable that the support should be substantially impermeable to water. For many purposes it is desirable that the support should be such that after application to the surface it is capable of stretching with movement of the concrete or other material, e.g. as a result of shrinkage, with maintenance of a moisture and moisture-vapour proof seal. To this end it is desirable that the combined structure of the support and the membrane should have, at 20°C., an elongation at break of at least 300%, a tensile strength of at least 1.5 kg. per cm. width and an Elmendorf tear strength of at least 750 gm. [The term "Elmendorf tear strength" as used herein refers to the test described in Elmendorf, Paper, 1920, 26, April 21st, page 302]. However, where stretchability and flexibility are less important than other properties, supports not fulfilling these requirements can be utilised. For example it is desirable for some purposes to use a metallic foil, particularly of copper or aluminium, as the support.

The preferred supports are films of natural rubber or of a synthetic organic polymer such as polyethylene, which is preferred, e.g. that commercially available under the trade name Polythene ("Polythene" is a Registered Trade Mark), polypropylene or other polyolefin, a polyamide, a polyester, e.g. polyethylene terephthalate, a polyurethane, polyvinyl chloride, a copolymer of vinyl chloride and vinylidene chloride, a synthetic rubber such as polychloroprene or butyl rubber, regenerated cellulose, cellulose ethers or cellulose esters. Inorganic or metallic supports can also be used.

Particularly for use in road-building it is desirable that the support should be such that hot asphalt (base course) can be poured directly onto it without deleterious effect on the waterproof seal. For this purpose the support should be able to resist a temperature of at least 150°C., preferably at least 175°C., for asphalt mixes such as sand course asphalt, and even higher temperatures for

hot asphalt (mastic), which is applied at temperatures up to 250°C. A particularly suitable material for this purpose is one consisting of a woven fabric of organic polymer fibres having on one face a waterproof layer of a pressure-sensitive bitumen-rubber adhesive as described above and on the other face a waterproof layer of a bitumen adhesive which is not a pressure-sensitive adhesive when cold but becomes tacky at elevated temperature, as when a hot asphalt mix is poured thereon.

Although cellular films can be used as supports, the supports are preferably substantially incompressible and have a thickness of at most 0.6 cm.

Other sheet-like supports include woven and non-woven fabrics of inorganic or organic natural or synthetic fibres (i.e. staple fibres or continuous filaments), e.g. a woven fabric of fibres of a synthetic organic polymer, glass tissue, hessian, cotton or other fibre scrim or bituminous roofing felt.

In Patent Application No. 47390/71 (Serial No. 1,265,953), there is disclosed and claimed a structure in the form of a roll and comprising (a) a waterproof membrane comprising a waterproofing pressure-sensitive adhesive which is a bituminous-rubber composition, and, adhered to the membrane, (b) a sheet-like support of polyethylene or polypropylene, one face of the support being substantially more readily physically separable from the membrane than the other face, the membrane being at least 0.025 cm. thick and the support being at least 15 cm. wide, and a method of forming a seal on a surface by unrolling the structure and applying the membrane so exposed to the surface. In this Specification, no claim is made to the structure or method of Patent Application No. 47390/71 (Serial No. 1,265,953).

SUBJECT TO THE FOREGOING DISCLAIMER,

WHAT WE CLAIM IS:—

1. A structure in the form of a roll and comprising (a) a waterproof membrane comprising a waterproofing pressure-sensitive adhesive, and, contiguous to the membrane, (b) a sheet-like substrate, one face of the substrate being substantially more readily physically separable from the membrane than the other face, the membrane being at least 0.025 cm. thick and the substrate being at least 15 cm. wide.

2. A structure according to claim 1 wherein the substrate is a sheet-like support having on one face a release coating.

3. A structure according to claim 2 wherein the outer face (as hereinbefore defined) of the support has the release coating.

4. A structure according to claim 2 or 3 wherein the release coating is provided by silicone deposition.

5. A structure according to claim 1 wherein the substrate is in the form of a protective coating which can be physically removed from the membrane without substantially damaging the membrane, the outer face (as hereinbefore defined) of the protective coating being more readily separable from the membrane than the inner face (as hereinbefore defined) of the protective coating.

6. A structure according to any one of claims 2 to 4 wherein the support is a water-impermeable film of a synthetic organic polymer.

7. A structure according to claim 6 wherein the polymer is a polyolefin.

8. A structure according to claim 7 wherein the polymer is polyethylene.

9. A structure according to claim 6 wherein the film has a softening temperature of at least 150°C.

10. A structure according to any one of claims 2 to 4 and 7 and 8 wherein the combined support and membrane have, at 20°C., an elongation at break of at least 300%, a tensile strength of at least 1.5 kg. per cm. width and an Elmendorf tear strength of at least 750 gm.

11. A structure according to claim 5 wherein the membrane comprises also a reinforcing sheet-like material which is a scrim of fibres of glass or of an organic polymer.

12. A structure according to claim 5 or 11 wherein the protective coating is paper having a release coating on one surface thereof.

13. A structure according to any one of the preceding claims wherein the membrane is 0.063 to 0.4 cm. thick.

14. A structure according to claim 13 wherein the membrane is 0.23 to 0.33 cm. thick.

15. A structure according to any one of the preceding claims wherein the adhesive is a bituminous composition.

16. A structure according to claim 15 wherein the bituminous composition is a blend of natural or synthetic rubber and bitumen, the ratio by weight of bitumen to rubber being greater than 80:20.

17. A structure according to claim 16 wherein the ratio is up to 95:5.

18. A structure according to any one of the preceding claims which is 15 to 120 cm. wide.

19. A structure according to claim 18 which is 15 to 91 cm. wide.

20. A structure according to claim 19 which is greater than 60 cm. wide.

21. A structure according to any one of claims 2 to 4 and 13 to 20 wherein the support is a woven fabric of polypropylene fibres.

22. A structure according to claim 21 wherein the fabric has on one face a water-

hot asphalt (mastic), which is applied at temperatures up to 250°C. A particularly suitable material for this purpose is one consisting of a woven fabric of organic polymer fibres having on one face a waterproof layer of a pressure-sensitive bitumen-rubber adhesive as described above and on the other face a waterproof layer of a bitumen adhesive which is not a pressure-sensitive adhesive when cold but becomes tacky at elevated temperature, as when a hot asphalt mix is poured thereon.

Although cellular films can be used as supports, the supports are preferably substantially incompressible and have a thickness of at most 0.6 cm.

Other sheet-like supports include woven and non-woven fabrics of inorganic or organic natural or synthetic fibres (i.e. staple fibres or continuous filaments), e.g. a woven fabric of fibres of a synthetic organic polymer, glass tissue, hessian, cotton or other fibre scrim or bituminous roofing felt.

In Patent Application No. 47390/71 (Serial No. 1,265,953), there is disclosed and claimed a structure in the form of a roll and comprising (a) a waterproof membrane comprising a waterproofing pressure-sensitive adhesive which is a bituminous-rubber composition, and, adhered to the membrane, (b) a sheet-like support of polyethylene or polypropylene, one face of the support being substantially more readily physically separable from the membrane than the other face, the membrane being at least 0.025 cm. thick and the support being at least 15 cm. wide, and a method of forming a seal on a surface by unrolling the structure and applying the membrane so exposed to the surface. In this Specification, no claim is made to the structure or method of Patent Application No. 47390/71 (Serial No. 1,265,953).

SUBJECT TO THE FOREGOING DISCLAIMER,

WHAT WE CLAIM IS:—

1. A structure in the form of a roll and comprising (a) a waterproof membrane comprising a waterproofing pressure-sensitive adhesive, and, contiguous to the membrane, (b) a sheet-like substrate, one face of the substrate being substantially more readily physically separable from the membrane than the other face, the membrane being at least 0.025 cm. thick and the substrate being at least 15 cm. wide.

2. A structure according to claim 1 wherein the substrate is a sheet-like support having on one face a release coating.

3. A structure according to claim 2 wherein the outer face (as hereinbefore defined) of the support has the release coating.

4. A structure according to claim 2 or 3 wherein the release coating is provided by silicone deposition.

5. A structure according to claim 1 wherein the substrate is in the form of a protective coating which can be physically removed from the membrane without substantially damaging the membrane, the outer face (as hereinbefore defined) of the protective coating being more readily separable from the membrane than the inner face (as hereinbefore defined) of the protective coating.

6. A structure according to any one of claims 2 to 4 wherein the support is a water-impermeable film of a synthetic organic polymer.

7. A structure according to claim 6 wherein the polymer is a polyolefin.

8. A structure according to claim 7 wherein the polymer is polyethylene.

9. A structure according to claim 6 wherein the film has a softening temperature of at least 150°C.

10. A structure according to any one of claims 2 to 4 and 7 and 8 wherein the combined support and membrane have, at 20°C., an elongation at break of at least 300%, a tensile strength of at least 1.5 kg. per. cm. width and an Elmendorf tear strength of at least 750 gm.

11. A structure according to claim 5 wherein the membrane comprises also a reinforcing sheet-like material which is a scrim of fibres of glass or of an organic polymer.

12. A structure according to claim 5 or 11 wherein the protective coating is paper having a release coating on one surface thereof.

13. A structure according to any one of the preceding claims wherein the membrane is 0.063 to 0.4 cm. thick.

14. A structure according to claim 13 wherein the membrane is 0.23 to 0.33 cm. thick.

15. A structure according to any one of the preceding claims wherein the adhesive is a bituminous composition.

16. A structure according to claim 15 wherein the bituminous composition is a blend of natural or synthetic rubber and bitumen, the ratio by weight of bitumen to rubber being greater than 80:20.

17. A structure according to claim 16 wherein the ratio is up to 95:5.

18. A structure according to any one of the preceding claims which is 15 to 120 cm. wide.

19. A structure according to claim 18 which is 15 to 91 cm. wide.

20. A structure according to claim 19 which is greater than 60 cm. wide.

21. A structure according to any one of claims 2 to 4 and 13 to 20 wherein the support is a woven fabric of polypropylene fibres.

22. A structure according to claim 21 wherein the fabric has on one face a water-



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proof layer of a pressure-sensitive adhesive bituminous composition as defined in any one of claims 15 to 17, and on the other face a waterproof layer of a bitumen adhesive which is not a pressure-sensitive adhesive when cold but which becomes tacky at an elevated temperature.

23. A structure according to claim 9 wherein the film has a softening temperature of at least 250°C.

24. A structure according to any one of claims 1 to 14, 18 to 21 and 23 wherein the pressure-sensitive adhesive is a non-saponifiable adhesive.

25. A structure according to claim 1 substantially as hereinbefore described.

26. A method of forming a waterproof seal on a surface, the method comprising unrolling a structure according to any one of claims 1 to 20 and applying the membrane so exposed to the surface, and then if necessary removing the protective coating, if present.

27. A method according to claim 26 wherein the surface is concrete.

28. A method according to claim 26 or

27 wherein a continuous waterproof seal is formed over the whole surface by the use of overlapping structures, and another surface is formed or placed in contact therewith. 30

29. A method of forming a waterproof seal on a surface, the method comprising unrolling a structure according to any one of claims 21 to 25 and applying the membrane so exposed to the surface, and then if necessary removing the protective coating, if present. 35

30. A method according to claim 26 substantially as hereinbefore described. 40

31. A surface whenever treated by the method according to any one of claims 26 to 28.

32. A surface whenever treated by the method according to claim 29 or 30. 45

J. A. KEMP & CO.,
Chartered Patent Agents,
14, South Square,
Gray's Inn,
London, WC1R 5EU.

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