

PATENT SPECIFICATION

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(54) SEALANTS AND ADHESIVES

(71) We, W. R. GRACE & Co., a Corporation organised 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 sealants and adhesives in the form of pre-formed sheet-like structures. The structures of the invention 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

[Price 25p]

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- and alkali-resistant

SPECIFICATION AMENDED - SEE ATTACHED SLIP

Price 33p

membrane, which means that water-based materials are not particularly desirable 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.

It is also known to provide the rear surface of cladding materials, particularly floor tiles, with a layer of a 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.

It has now been realised that considerable advantages are obtained from the use of pre-formed membranes of water-proofing pressure-sensitive adhesives. Such membranes can be produced on a large scale in a factory and then used on site. In one aspect, therefore, the invention provides 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.

The term "membrane of a pressure-sensitive adhesive" as used herein refers to those membranes of adhesive which when forming part of the structures enables the structures, when pressed by normal hand pressure against a concrete surface without any prior treatment of the membrane or the concrete, to remain stuck thereto.

The membrane can be a layer of the adhesive alone or the adhesive can contain reinforcing material, for example a scrim of glass fibres or an organic polymer, 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 the invention 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 have 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 water-proof membrane, or over selected parts of the surface only.

In the structures 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 hereinafter) which can be physically removed from the membrane without substantial damage to the membrane: It will of course be clear that for storage purposes the surface of the membrane remote from the substrate must have a said protective coating thereon. Thus the structures of the invention 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.

The structures can be used to form a waterproof seal on a surface by applying an exposed face of the membrane to the surface to be sealed. When a structure of the second type is used in order to provide a layer of pressure-sensitive adhesive, the other surface of the membrane is exposed at an appropriate stage after application of the membrane ready to the surface to be sealed 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. This method is discussed further in Patent Application No. 22789/70 (Serial No. 1,230,755).

The widths of the structures 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 90 cm., being preferred where the whole of a surface is to be rendered waterproof. The thickness of the structures can also vary widely depending on the intended end use, but will generally be up to 0.63 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 is suitably 0.063 to 130

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5 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 structures of the first type a support having on it a single layer of adhesive 0.23 to 0.33 cm. thick is preferred, and for structures 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.

15 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 preferably from 80:20 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 temperatures (measured by the Ring and Ball method) of 60 to 110°C. and penetration values of 150—300 at 25°C. (100 g. 5 secs. — Institute of Petroleum method).

30 A wide variety of materials can be used to provide protective coatings in the structures of the invention. Paper having a release coating thereon, e.g. siliconised paper or paper having a coating thereon of a vinyl chloride polymer, is satisfactory. Other materials include films of organic polymers, particularly vinyl chloride polymers.

35 A wide variety of materials can be used as the sheet-like support, which forms parts of the structures of the first type noted above, and it is a particular advantage of such structures 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).

65 However, where stretchability and flexi-

bility is less important than other properties, supports not fulfilling these requirements can be utilised. For example, it is desirable for some purposes to use a metal foil, particularly of copper, 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, polypropylene or other polyolefin, a polyamide, a polyester, e.g. polyethylene terephthalate, a polyurethane, polyvinyl, chloride, a copolymer of vinyl chloride and vinylidene chloride, or a synthetic rubber such as polychloroprene or butyl rubber.

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.

While cellular films can be used as supports, the supports are generally substantially incompressible and have a thickness of at most 0.63 cm.

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

If desired, the surface of the support remote from the membrane can be treated to protect it from damage and/or so that it offers an improved keying surface for other materials, e.g. concrete, subsequently applied thereto. Thus this surface of the support can be textured and/or can carry a layer of adhesive, which is preferably water-resistant and may be the same as or different from the adhesive in the membrane, to which layer is adherent a deposit of granular or powdered material, e.g. finally divided aggregate or other organic or inorganic material, e.g. sand or cork granules.

The structures of the invention can be prepared without difficulty by techniques known *per se*. It is preferred to use adhesives which are not solvent-based and which are heated. Thus the adhesive may be bonded to the substrate to form a uniform layer, to the substrate. If it is desired to incorporate a sheet-like reinforcing layer in the membrane, this can be achieved, for example by pressing a scrim into the layer of adhesive. Such a reinforcing layer is not generally required when a support is used, but is often helpful when there is no support, in order to prevent excessive distortion of the membrane when the protective coatings are removed.

In Patent Application No. 22788/70 (Serial No. 1230754) there is described and

claimed a structure comprising a sheet-like support of polyethylene or polypropylene, the support having adherent thereto a membrane of a water-proofing pressure-sensitive adhesive which is a bituminous rubber composition, the structure being at least 15 cm. wide and the membrane being at least 0.025 cm. thick, while in Patent Application No. 22790/70 (Serial No. 1230756) there is described and claimed a structure comprising a sheet-like support of aluminium, the support having adherent 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. In this specification, no claim is made to the structures of Patent Applications Nos. 22788/70 and 22790/70 (Serial Nos. 1230754 and 1230756).

SUBJECT TO THE FOREGOING DISCLAIMERS, WHAT WE CLAIM IS:—

1. 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.

2. A structure according to claim 1 wherein the substrate is a sheet-like support having the membrane permanently adherent thereto.

3. A structure according to claim 2 wherein the support is a water-impermeable film of natural rubber.

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

5. A structure according to claim 4 wherein the polymer is a polyester.

6. A structure according to claim 4 wherein the film is of a polyolefin.

7. A structure according to claim 6 wherein the film is of polyethylene.

8. A structure according to any one of claims 4 to 6 wherein the film has a softening temperature of at least 150°C.

9. A structure according to claim 8 wherein the film has a softening temperature of at least 175°C.

10. A structure according to claim 2 wherein the support is a woven or non-woven fabric.

11. A structure according to any one of claims 2 to 4, 6, 7 and 10 wherein the support and membrane have, at 20°C, an elongation at break of at least 300%, tensile strength of at least 1.5 kg. per cm. width and an Elmendorf tear strength (as hereinbefore defined) of at least 750 g.

12. A structure according to any one of claims 2 to 4, 6, 7, 10 and 11 wherein the support is substantially incompressible and has a thickness of at most 0.63 cm.

13. A structure according to claim 2 wherein the support is a film of a metal.

14. A structure according to claim 13 wherein the metal is copper.

15. A structure according to any one of claims 3, 4, 6, 7, and 10 to 12 wherein the surface of the film remote from the layer of granular or powdered material.

16. A structure according to any one of claims 2 to 4, 7 and 10 which comprises also a layer of adhesive one face of which is adherent to the surface of the support remote from the membrane and the other face of which has adherent thereto a deposit of granular or powdered material.

17. A structure according to claim 16 wherein the adhesive of said second layer is the same as the adhesive of the membrane.

18. A structure according to any one of the preceding claims which also comprises a protective coating over the surface of the membrane remote from the support, the said protective coating being such that it can be physically removed from the membrane without substantially damaging the membrane.

19. A structure according to claim 1 wherein the substrate is in the form of a protective coating which can be physically removed from the layer of adhesive without substantially damaging the membrane.

20. A structure according to claim 19 wherein the membrane is sandwiched between two said protective coatings.

21. A structure according to claim 19 or 20 wherein the membrane of pressure-sensitive adhesive has incorporated therein a reinforcing sheet-like material.

22. A structure according to claim 21 wherein the reinforcing material is a scrim of fibres of glass or an organic polymer.

23. A structure according to claim 19 or 20 wherein the protective coating is paper having a release coating on the surface adjacent to the membrane.

24. A structure according to claim 23 wherein the protective coating is siliconised paper.

25. A structure according to claim 23 wherein the protective coating is a film of an organic polymer.

26. A structure according to claim 25 wherein the protective coating is a film of a vinyl chloride polymer.

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

28. A structure according to claim 27 wherein the membrane is 0.23 to 0.33 cm. thick.

29. A structure according to any one of claims 2 to 4, 10, 16 and 17 wherein the pressure-sensitive adhesive is a bituminous composition.

30. A structure according to claim 29

claimed a structure comprising a sheet-like support of polyethylene or polypropylene, the support having adherent thereto a membrane of a water-proofing pressure-sensitive adhesive which is a bituminous rubber composition, the structure being at least 15 cm. wide and the membrane being at least 0.025 cm. thick, while in Patent Application No. 22790/70 (Serial No. 1230756) there is described and claimed a structure comprising a sheet-like support of aluminium, the support having adherent 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. In this specification, no claim is made to the structures of Patent Applications Nos. 22788/70 and 22790/70 (Serial Nos. 1230754 and 1230756).

SUBJECT TO THE FOREGOING DISCLAIMERS, WHAT WE CLAIM IS:—

1. 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.
2. A structure according to claim 1 wherein the substrate is a sheet-like support having the membrane permanently adherent thereto.
3. A structure according to claim 2 wherein the support is a water-impermeable film of natural rubber.
4. A structure according to claim 2 wherein the support is a water-impermeable film of a synthetic organic polymer.
5. A structure according to claim 4 wherein the polymer is a polyester.
6. A structure according to claim 4 wherein the film is of a polyolefin.
7. A structure according to claim 6 wherein the film is of polyethylene.
8. A structure according to any one of claims 4 to 6 wherein the film has a softening temperature of at least 150°C.
9. A structure according to claim 8 wherein the film has a softening temperature of at least 175°C.
10. A structure according to claim 2 wherein the support is a woven or non-woven fabric.
11. A structure according to any one of claims 2 to 4, 6, 7 and 10 wherein the support and membrane have, at 20°C, an elongation at break of at least 300%, tensile strength of at least 1.5 kg. per cm. width and an Elmendorf tear strength (as hereinbefore defined) of at least 750 g.
12. A structure according to any one of claims 2 to 4, 6, 7, 10 and 11 wherein the support is substantially incompressible and has a thickness of at most 0.63 cm.

13. A structure according to claim 2 wherein the support is a film of a metal.

14. A structure according to claim 13 wherein the metal is copper.

15. A structure according to any one of claims 3, 4, 6, 7, and 10 to 12 wherein the surface of the film remote from the layer of granular or powdered material.

16. A structure according to any one of claims 2 to 4, 7 and 10 which comprises also a layer of adhesive one face of which is adherent to the surface of the support remote from the membrane and the other face of which has adherent thereto a deposit of granular or powdered material.

17. A structure according to claim 16 wherein the adhesive of said second layer is the same as the adhesive of the membrane.

18. A structure according to any one of the preceding claims which also comprises a protective coating over the surface of the membrane remote from the support, the said protective coating being such that it can be physically removed from the membrane without substantially damaging the membrane.

19. A structure according to claim 1 wherein the substrate is in the form of a protective coating which can be physically removed from the layer of adhesive without substantially damaging the membrane.

20. A structure according to claim 19 wherein the membrane is sandwiched between two said protective coatings.

21. A structure according to claim 19 or 20 wherein the membrane of pressure-sensitive adhesive has incorporated therein a reinforcing sheet-like material.

22. A structure according to claim 21 wherein the reinforcing material is a scrim of fibres of glass or an organic polymer.

23. A structure according to claim 19 or 20 wherein the protective coating is paper having a release coating on the surface adjacent to the membrane.

24. A structure according to claim 23 wherein the protective coating is siliconised paper.

25. A structure according to claim 23 wherein the protective coating is a film of an organic polymer.

26. A structure according to claim 25 wherein the protective coating is a film of a vinyl chloride polymer.

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

28. A structure according to claim 27 wherein the membrane is 0.23 to 0.33 cm. thick.

29. A structure according to any one of claims 2 to 4, 10, 16 and 17 wherein the pressure-sensitive adhesive is a bituminous composition.

30. A structure according to claim 29



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wherein the bituminous composition is a bituminous rubber composition.

31. A structure according to claim 30 wherein the bituminous rubber composition is a blend of natural or synthetic rubber and bitumen, the ratio by weight of bitumen to rubber being from 80:20 to 95:5.

32. A structure according to any one of claims 2 to 4, 7, 10, 16, 17, 29 and 30 which is up to 120 cm. wide.

33. A structure according to claim 32 which is up to 0.63 cm. thick.

34. A structure according to any one of claims 6, 11, 12 and 15 which is up to 91 cm. wide.

35. A structure according to any one of the preceding claims which is 60 to 91 cm. wide.

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

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PATENTS ACT 1949

SPECIFICATION NO 1230753

In accordance with the Decision of the Principal Examiner, acting for the Comptroller-General, dated 30 August 1973, this Specification has been amended under Section 14 in the following manner:-

Page 3, line 27, *delete* temperatures *insert* points

Page 3, line 41, *delete* parts *insert* part

Page 3, line 92, *delete* incompressible *insert* incompressible

Page 4, line 9, *after* 1230756) *insert* as published,

Page 4, line 16, *after* thick *delete* full stop *insert* ; the subject matter claimed in Patent Application No 22790/70 (Serial No 1230756 was subsequently linked by amendment.

Page 4, *delete* lines 17, 18 and 19 *insert* to the structures of Patent Application No 22790/70 (Serial No 1230756) as published or the structures of Patent Application No 22788/70 (Serial No 1230754).

THE PATENT OFFICE
7 March 1974

R 74607/16

ERRATA

SPECIFICATION NO 1230753

Page 1, line 27, *for* trem *read* term *SLIP NO. 2*

Page 2, line 2, *after* desirable *insert* ;

Page 3, line 64, *after* dorf *insert* ,

Page 3, line 78, *after* polyvinyl *delete* ,

Page 4, line 72, *delete* whole line *insert* of adhesive is textured.

THE PATENT OFFICE
8 April 1974

R 74645/1

