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(54) **Composite polymeric film**

(57) A composite film consists of a thin film layer and a backing layer. The backing layer is soluble in a solvent in which the thin film layer (<10  $\mu\text{m}$ ) is not soluble. The composite film may be installed in a device in the same position in which it is sought to finally

emplace the thin film. The backing layer is then selectively dissolved in the solvent to leave the insoluble thin film layer as an unbacked film. The method permits a very thin film to be successfully installed in devices where the fragility of the film would preclude handling and installation by conventional methods.

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## SPECIFICATION

**Composite polymeric film and method for its use in installing a very thin polymeric film in a device**

## 5 Background of the Invention

The invention disclosed herein relates generally to plastic polymeric films and methods of making the same. More particularly, this invention relates to methods of forming and installing a very thin polymeric film in a device, under conditions where the fragility of the film would ordinarily make its handling and installation difficult or impossible.

10 Very thin (<10  $\mu\text{m}$ ) polymeric films are useful in many well known applications. Representative applications in connection with which the present invention was developed include the use of polymeric films as barriers to moisture, dust and gases; and as frequency-specific radiation filters. In such applications, the film is ordinarily  
20 emplaced in a device where it is subject only to small static stresses under controlled conditions. Once installed, the film, even though very fragile, is sufficiently strong and flexible to perform its intended function without rupturing. It is often  
25 difficult, however, to initially assemble such devices. This difficulty lies not so much in the formation of the film itself, as there are various well known methods of making thin films, but rather in the handling and installation of the film  
30 without tearing it. In some cases, this difficulty effectively imposes a lower limit on the thickness of film which may be routinely installed in a device.

35 Accordingly, it is the object and purpose of the present invention to provide a method of forming and installing very thin polymeric films.

It is also an object of the present invention to provide a method of forming and installing very thin polymeric films *in situ* in a device in which the  
40 film is to be utilized.

Additionally, for reasons that will be apparent from the description of the invention set forth below, it is an object of the invention to provide a composite film for use in installing a thin film in a  
45 device.

Additional objects, advantages and novel features of the invention will be set forth in part in the description which follows, and in part will become apparent to those skilled in the art upon  
50 examination of the following or may be learned by practice of the invention. The objects and advantages of the invention may be realized and attained by means of the instrumentalities, combinations and methods particularly pointed  
55 out in the appended claims.

## Summary of the Invention

To achieve the foregoing objects and other objects, and in accordance with the purposes of the present invention as embodied and broadly  
60 described herein, there is provided a composite polymeric film and a method for its use for the purpose of installing a very thin polymeric film in a device. The composite film comprises two layers;

65 a thin film layer, which is the film to be installed in the device, and a relatively thick backing layer. The backing layer supports the thin film layer and enables it to be handled in a routine manner without tearing. In accordance with the method of application, the composite film is installed in the  
70 device where it is sought to emplace the thin film. The device, together with the composite film, is then immersed in a suitable solvent to selectively dissolve the backing layer and leave the thin film layer as an unbacked film formed *in situ* in the  
75 device.

In accordance with a preferred aspect of applicant's invention, the composite film comprises a backing layer consisting essentially of polyvinyl alcohol and a thin film layer consisting  
80 essentially of polyvinyl formal. The polyvinyl alcohol backing layer is readily soluble in water at room temperature, whereas the polyvinyl formal film is insoluble in water. Further, the surface chemistries of these materials are particularly  
85 compatible so as to permit a very thin, uniform film of polyvinyl formal to be cast onto a backing layer of polyvinyl alcohol. Such a composite film can be prepared well in advance of its installation and can be handled, stored, shipped, and installed  
90 without undergoing damage to the thin film layer. Other combinations of polymeric materials that may be used to form the composite film are set forth below.

## Detailed Description of the Preferred Embodiment

95 In accordance with the preferred embodiment of the invention, a composite film is formed which consists of a polyvinyl alcohol backing layer and a thin layer of polyvinyl formal. This may preferably be done by solution casting the polyvinyl alcohol  
100 onto a smooth surface of glass or stainless steel. The polyvinyl formal layer may be subsequently deposited, also by solution casting, onto the polyvinyl alcohol layer while it is still on the substrate surface. The resulting composite film  
105 may then be peeled from the substrate surface and subsequently handled, shipped, or stored with little danger of tearing or puncturing.

To use the composite film in accordance with the invention, it is installed in the same manner  
110 and position in which the thin film layer is desired to be ultimately positioned. The assembly is then immersed in a water bath to dissolve the backing layer and leave the thin film layer as an unbacked film properly positioned in the device.

115 Polyvinyl alcohol is ordinarily prepared by hydrolysis of polyvinyl acetate. In this regard, it is found that optimum solubility in water at room temperature is obtained with polyvinyl acetate that is not completely hydrolyzed. Adequate  
120 solubility in water at room temperature is obtained with polyvinyl acetate that is between 75 and 95% hydrolyzed. Optimum solubility is obtained with polyvinyl acetate that is approximately 88% hydrolyzed, i.e., a polyvinyl polymer consisting of  
125 88% polyvinyl alcohol and 12% polyvinyl acetate. A suitable polyvinyl alcohol polymer of this type is commercially available, for example, from Aldrich

Chemical Company of Milwaukee, WI.

The polyvinyl alcohol backing layer is preferably formed by solution casting onto a mold or other solid substrate, which may be coated with a mold release agent. The mold is briefly dipped in an aqueous solution of polyvinyl alcohol and subsequently air dried to form a backing layer of polyvinyl alcohol approximately 25  $\mu\text{m}$  thick. To prepare such a film requires an aqueous solution of polyvinyl alcohol having a viscosity between approximately 200—10,000 centipoises, with the preferred range being from approximately 1000—3000 centipoises. These ranges correspond to aqueous solutions having from 6 to 15% and from 8 to 12% by weight polyvinyl alcohol, respectively.

Other suitable water soluble film materials which may be used to prepare the backing layer include cellulose ethers, sodium carboxymethyl cellulose, polyoxyethylene, and the like.

Polyvinyl formal is the preferred material for the formation of the thin film layer because it is insoluble in water and, also, because solutions of polyvinyl formal in organic solvents are found to thoroughly wet the surface of a dry polyvinyl alcohol film, such that a uniform thin film layer can be cast onto a backing layer of polyvinyl alcohol. In this regard, it is important that the solution used to form the thin film layer have the proper surface tension characteristics such that it uniformly wets the first film surface, or backing layer.

Another polymeric material which is suitable for forming the thin film layer is di-p-xylylene, which is sold under the tradename Parylene by Union Carbide Corporation. Di-p-xylylene is relatively inert to chemical attack and is not soluble in water. A thin film of di-p-xylylene may be formed on a suitable backing layer, for example a water-soluble film of polyvinyl alcohol formed as described above, by a technique known as vapor phase polymerization. This technique has been previously known for forming thin films of di-p-xylylene, and may be utilized to form a thin film layer approximately 1  $\mu\text{m}$  thick on a backing layer of polyvinyl alcohol.

Another polymeric material which may be used to form a water-insoluble thin film layer is cellulose acetate. A thin film layer of cellulose acetate may be formed on a polyvinyl alcohol backing layer by solution casting from a solution of cellulose acetate dissolved in acetone.

In an inverse application of the immediately foregoing example, a thin film layer of polyvinyl alcohol may be cast from a water solution onto a backing layer of cellulose acetate. After mounting of the composite film, the cellulose acetate layer may be selectively dissolved by immersion in acetone, leaving an unbacked thin film of polyvinyl alcohol.

In actual tests of the method, thin polymeric films ranging in thickness from 0.5 to 25  $\mu\text{m}$  have been produced. The backing layer is typically 10 to 250  $\mu\text{m}$  thick. The following description of a demonstrative test more clearly illustrates the practice of the method.

#### EXAMPLE 1

In an exemplary demonstration of the method described above, a stainless steel ring having a highly polished, cylindrical inner surface was selected as a casting surface. The ring was dipped in an aqueous solution containing approximately 9.8% by weight polyvinyl alcohol (88% hydrolyzed), and 2.4% by weight glycerin as a plasticizer. The ring was subsequently air dried. This produced a backing layer approximately 10  $\mu\text{m}$  thick. The coated ring was then dipped in a solution consisting of polyvinyl formal (10% by weight) dissolved in a mixture consisting of 20% (by volume) methyl benzoate, 48% by volume toluene, and 32% by volume ethanol. The wetted ring was then air dried. This produced a thin polymer film layer approximately 1  $\mu\text{m}$  thick on the underlying polyvinyl alcohol backing layer. The resulting composite film was manually peeled from the ring and was found to be sufficiently rugged to withstand ordinary handling.

The tubular composite film was then cut along one side to form a planar film. This composite film was stretched over a rectangular frame having an open area of dimensions of 5 x 20". The film was clamped at the edges and the assembly was immersed in water, taking care to avoid disturbance in the water which would rupture the thin film. Dissolution of the polyvinyl alcohol backing layer was complete in a few seconds, leaving the thin polyvinyl formal film spanning an unbacked area of approximately 100 square inches.

The foregoing description of a preferred embodiment of the invention has been presented for purposes of illustration and description. It is not intended to be exhaustive or to limit the invention to the precise form disclosed, and obviously many modifications and variations are possible in light of the above teaching. In particular, there may be various combinations of film materials which may be apparent to a chemist of ordinary skill as being suitable for the practice of the invention. The embodiments described above were chosen in order to best explain the principles of the invention and its practical application to thereby enable others skilled in the art to best utilize the invention in various embodiments and with various modifications as are suited to the particular use contemplated. It is intended that the scope of the invention be defined by the claims appended hereto.

#### CLAIMS

1. A composite polymeric film for use in installing a very thin polymeric film in a device, comprising a backing layer and a thin film layer, said backing layer being relatively thicker than said thin film layer and consisting essentially of a polymeric material susceptible to dissolution in at least one solvent in which said thin film layer is substantially insoluble.

2. The composite polymeric film defined in claim 1 wherein said backing layer consists essentially of polyvinyl alcohol.

3. The composite polymeric film defined in claim 2 wherein said thin film layer consists essentially of polyvinyl formal.
4. The composite polymeric film defined in claim 3 wherein said polyvinyl alcohol is between 75 and 95 percent hydrolyzed.
5. The composite polymeric film defined in claim 4 wherein said backing layer further includes a plasticizer.
6. The composite polymeric film defined in claim 5 wherein said plasticizer consists of glycerin.
7. The composite polymeric film defined in claim 1 wherein said thin film layer consists essentially of polyvinyl formal and said backing layer consists essentially of a cellulose ether.
8. The composite polymeric film defined in claim 1 wherein said thin film layer consists essentially of polyvinyl formal and said backing layer consists essentially of sodium carboxymethyl cellulose.
9. The composite polymeric film defined in claim 1 wherein said thin film layer consists essentially of polyvinyl formal and said backing layer consists essentially of polyoxyethylene.
10. The composite polymeric film of claim 2 wherein said thin film layer consists essentially of di-p-xylylene.
11. The composite polymeric film of claim 2 wherein said thin film layer consists essentially of cellulose acetate.
12. A method of installing a thin polymeric film in a device, comprising the steps of:  
forming a composite film consisting essentially of a backing layer and a thin film layer, said backing layer being relative thicker than said thin film layer, said backing layer being soluble in at least one solvent in which said thin film layer is insoluble;
- installing said composite film in a device in a position in which it is desired to install said thin film; and  
immersing said device together with said composite film in a suitable solvent to dissolve said backing layer and leave said thin film layer as an unbacked thin polymeric film installed in the device.
13. The method of claim 12 wherein said thin film layer consists essentially of polyvinyl formal.
14. The method of claim 13 wherein said backing layer consists essentially of polyvinyl alcohol.
15. The method of claim 14 wherein said backing layer consists essentially of polyvinyl alcohol which is between 75 and 95 percent hydrolyzed.
16. The method of claim 15 wherein said backing layer further includes a plasticizer.
17. The method of claim 16 wherein said plasticizer is glycerin.
18. The method of claim 12 wherein said thin film layer consists essentially of di-p-xylylene.
19. The method of claim 12 wherein said thin film layer consists essentially of cellulose acetate.
20. The method of claim 18 or 19 wherein said backing layer consists essentially of polyvinyl alcohol.
21. The methods of claim 20 wherein said polyvinyl alcohol is between 75 and 95 percent hydrolyzed.