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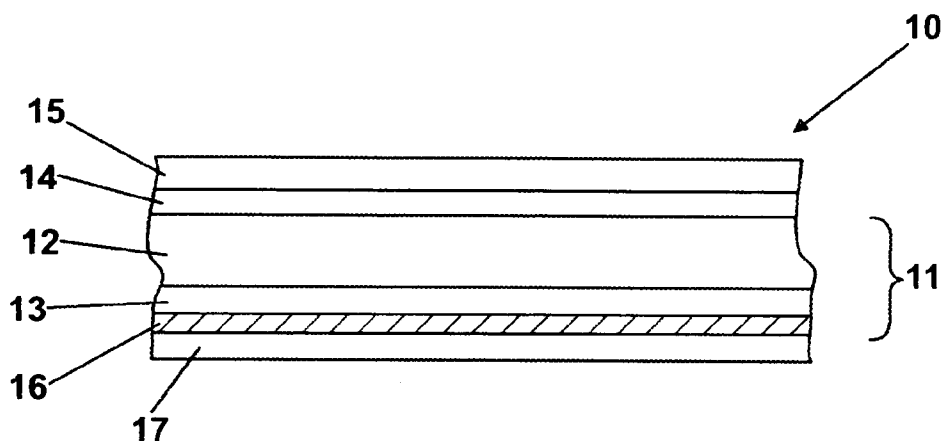


Fig. 1

(57) Abstract: A transparent exterior window film composite (10) comprising a base sheet material (11) having an outer PET (polyethylene terephthalate) film layer (12) which has been surface treated to elevate adhesion thereto and which is impregnated with an ultra violet light absorber (UVA), the treated surface being coated with a UV stabilized acrylic polymer or co-polymer undercoat (14) having a UV stabilized hard coat layer (15) thereon formed from at least one aliphatic urethane acrylate oligomer and at least one multifunctional acrylate monomer, the undercoat layer having a thickness which is at least as thick as the hard coat.

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Exterior Window Film

Field

This invention relates to exterior window film which is adhered to the outside of window
5 glazing typically but not exclusively to the window glazing of buildings.

Background of the Invention

Window film is adhered to the outside of windows for many different reasons for
example, the film may act as a safety film to prevent the shattering of glass when
10 impacted. Another use of window film is solar control film which may reflect light at
particular wavelengths or may absorb light of particular wavelengths. As is disclosed in
US 6773 778, exterior window film typically comprises a base film or film laminate
which is provided with a hard coat layer on its external surface.

15 The film surface to which the hard coat layer is applied may be subjected, if desired, to
surface treatment by an oxidation method or a roughening method for the purpose of
elevating the adhesive property with a layer provided on the surface thereof.

Exterior window film typically has a service life in the order of about 24 months and
20 typically fails due to the loss of hard coat adhesion to the base substrate. When cycled in
an Atlas Xenon Weatherometer according to ASTM G 155, a typical window film will
fail in 600-900 hours due to loss of adhesion between the hard coat and the base film
material when subjected to tape testing.

25 US 6787 236 describes a window film having a primer layer provided on one face of the
base material with the hard coat applied over the primer layer. The primer layer is

required to have a good adhesion with both the hard coat layer provided thereon and the base material. Conventionally known primers, are for example, primers of an acryl base, a polyester base, a polyurethane base, a silicone base and a rubber base.

- 5 The present invention provides for an exterior window film laminate with improved adhesion between the hard coat and the base material and which has improved resistance to the weather.

Statements of Invention

- 10 According to a first aspect of the present invention there is provided a transparent exterior window film composite comprising a base sheet material having an outer PET (polyethylene terephthalate) film layer which has been surface treated to elevate adhesion thereto and which is impregnated with a ultra violet light absorber (UVA), the treated surface being coated with an acrylic polymer or co-polymer undercoat having a
- 15 urethane-acrylate hard coat layer thereon formed from at least one urethane acrylate oligomer and at least one multifunctional acrylate monomer, the undercoat having a thickness which is at least as thick as the hard coat .

- The polymerizable acrylic and acrylate matrices of the undercoat and hard coat contain
- 20 one or more crosslinking agents that are useful in the production of the crosslinked under coat and hardcoat of the present invention. The crosslinking agents are selected from di- or polyfunctional moieties which are capable of crosslinking oligomers. In general, the reactive functionalities which serve as active sites for such crosslinking are chemical groups containing double bonds as is well known to those skilled in the art, who similarly
- 25 recognize that it is necessary for such agents to have at least two reactive functionalities.

The surface treatment may include any suitable method, for example an oxidation method or a roughening method. The oxidation method may include, for example, corona discharge treatment, chromic acid treatment (wet type), flame treatment, hot air treatment, ozone and UV ray irradiation treatment. The surface treatment may further include a
5 chemical treatment such as an acrylic primer layer.

To prevent or inhibit photodegradation of a composite, at least one ultraviolet light stabilizer can be incorporated into or applied to one or more of the layers comprising the composite. UV stabilizers include materials that inhibit photoinitiation by absorbing UV
10 light (UVAs). Other stabilizers quench excited states of atoms capable of initiating undesired breakdown chemical reactions. One known agent is nickel phenolate found in Cyasorb UV 1084. Some stabilizer additives scavenge radicals formed during photodegradation of molecules upon weathering. Examples of these scavengers are hindered phenolics commercially found as Irganox 565 or hindered benzoates such as
15 Cyasorb UV 2908. The class of hindered amines are useful for scavenging radicals as well to protect the coating ingredients from break down such as Cyasorb 3346 or Tinuvin 123. Hydroperoxide decomposition is another method for preventing chemical breakdown during weathering of coatings. The peroxides formed can be arrested by thioesters such as Cyanox LTDP and nickel dibutyl dithiocarbamates found in the
20 antiozone compound agent NIBUD from Akrochem.

UVAs function by competitively absorbing the UV energy that causes photodegradation of the structure. A wide variety of ultraviolet light-absorbing compounds are available including, for example, benzophenones (e.g., materials sold under the trade names
25 CYASORB UV-531 (available from Cytec Industries Inc., located in West Paterson,

N.J.), and UVINUL 3008 (available from BASF, located in Mount Olive, N.J.)),
benzotriazoles (e.g., materials sold under the trade names CYASORB UV-5411
(available from Cytec Industries Inc.), and TINUVIN 329, TINUVIN 360, and TINUVIN
571 (available from Ciba Specialty Chemicals North America, located in Tarrytown,
5 N.Y.)), triazines (e.g., materials sold under the trade names CYASORB UV-1164
(available from Cytec Industries Inc.), and TINUVIN 400, 460, 477, and 479 (available
from Ciba Specialty Chemicals), oxanilides (e.g., materials sold under the trade names
TINUVIN 312 (available from Ciba Specialty Chemicals North America), and
SANDUVOR VSU (available from Clariant AG, located in Muttenz, Switzerland)),
10 benzoxazinones (e.g., CYASORB UV-3638 (available from Cytec Industries Inc.),
cyanoacrylates (e.g., UVINUL 3039 (available from BASF)), and benzilidine malonates
(e.g., HOSTAVIN PR-25 (available from Clariant AG). Inorganic UV absorbers include
Titanium dioxide, Zinc oxide, and cerium oxide which are added in the form of small
particles, preferably as nanoparticles.

15 Preferably the undercoat comprises a coating of a cured acrylic or urethane-acrylic based
resin, preferably formed from an aqueous dispersion and containing UV absorber,
preferably up to 3% by weight of organic UV absorber or up to 5% by weight of inorganic
absorber.

20 The undercoat preferably has a thickness of between 6 and 20 microns and, preferably
between 6-12 microns.

The urethane acrylate oligomer of the hard coat is preferably an aliphatic urethane
25 acrylate oligomer, including those having aliphatic cyclic structures, having a glass

transition temperature, or T_g , higher than 80°C with an elongation less than 10%, preferably less than 5%.

Preferred multifunctional acrylate monomers are dipentaerythritol pentaacrylate,
5 pentaerythritol tetraacrylate, pentaerythritol triacrylate, ditrimethylolpropane tetraacrylate, trimethylolpropane triacrylate, tricyclodecane dimethanol diacrylate, 1,6 hexanediol diacrylate, cyclohexane dimethanol diacrylate, and dioxane glycol diacrylate.

The hard coat contains at least one UV stabiliser and preferably has a thickness of
10 between 6-8 microns preferably with an abrasion resistance as measured by change in haze measurements after abrasion testing of less than 12%.

The hard coat and undercoat are cured by any suitable process such as electron beam curing, or light curing using one or more photoinitiators. In general, the photoinitiator(s)
15 are at least partially soluble (e.g. at the processing temperature of the resin) and substantially colorless after being polymerized. The photoinitiator may be (e.g. yellow) colored, provided that the photoinitiator is rendered substantially colorless after exposure to the UV light source. Preferably the hard coat and undercoat are cured by UV light and suitable UV photoinitiators, include monoacylphosphine oxide and bisacylphosphine
20 oxide. Commercially available mono or bisacylphosphine oxide photoinitiators include 2,4,6-trimethylbenzoyldiphenylphosphine oxide, commercially available from BASF (Charlotte, NC) under the trade designation "Lucirin TPO"; ethyl-2,4,6-trimethylbenzoylphenyl phosphinate, also commercially available from BASF under the trade designation "Lucirin TPO-L"; and bis(2,4,6-trimethylbenzoyl)-phenylphosphine
25 oxide commercially available from Ciba Specialty Chemicals under the

trade designation "Irgacure 819". Other suitable photoinitiators include 2-hydroxy-2-methyl-1-phenyl-propan-1-one, commercially available from Ciba Specialty Chemicals under the trade designation Darocur 1173. Other photoinitiators are commercially available under the tradenames "Darocur 4265", "Irgacure 651", "Irgacure 1800",
5 "Irgacure 369", "Irgacure 1700", and "Irgacure 907". The photoinitiator is generally added at about .5 to 4 phr (part per hundred resin) to the resin composition. More or less can be employed depending on the requirements for color and cure speed, and other design criteria.

10 The hard coat has preferably been cured under an inert atmosphere such as a nitrogen atmosphere.

The hard coat may be made hydrophobic by the addition of a hydrophobic additive which helps the run-off of water by reducing the surface energy. This improves dirt shedding
15 properties of the film composite. Such additives include silicone acrylates, preferably oligomers of silicone acrylates and fluorinated urethane acrylates, preferably up to 4% by weight.

The hard coat may contain a nanoparticle dispersion therein. Nanoparticles should have a
20 size of less than 0.1 microns in diameter which are utilised as inorganic UVA's as discussed above, or to increase surface hardness. Inorganic nanoparticles such as zinc oxide, and cerium oxide may be used as UVAs as discussed above and silicone dioxide and aluminium oxide may be used to increase surface hardness and abrasion resistance as is well known.

The PET (polyethylene terephthalate) outer film layer of the base sheet material is about 1/2 - 7 mil (0.0125 – 0.18mm) thick which has been surface treated by chemical treatments to improve adhesion. The film may be further treated with a UV absorbing material as is disclosed in US 6221 112. The film composite may further include as a base material any solar control film or safety film. Examples of solar control film are disclosed in US 6416872 and US 6007901.

The base sheet material on its other surface of the polymeric film may be coated with a pressure sensitive adhesive, preferably containing UVA, which in use adheres to the film layer and in use may adhere the composite to glazing. Prior to use of the composite, the adhesive may be covered with a release liner.

Glazing includes any suitable transparent sheet material which may be used for motor cycle helmet visors, vehicle windscreens, aircraft canopies and windscreen and windows, PC Monitor Screens etc. and which include glass, acrylic sheet, polyester sheet, polycarbonate sheet.

The composite film has a visible light transmission in the range of 5- 85% VLT. VLT is visible light transmission calculated using CIE Standard Observer (CIE 1924 1931) and D65 Daylight.

Composite film when tested for adhesion after being subjected to weather testing in accordance with ASTM G 155 exceeded 1200 hours before adhesion failure of the hard coat and with the preferred hard coat formed from a mixture of acrylate monomers the minimum life expectancy before adhesion failure was between 1500-2400 hours.

Preferably the hard coat when cured under inert conditions provides for a composite having a life expectancy of at least 3000 hours before failure of the adhesion of the hard coat to the PET base material.

5 Description of Drawings

The invention will be described by way of Example and with reference to the accompanying drawings in which

Fig. 1 is a schematic drawing a film composite according to the present invention, and

10 Fig. 2 is a schematic of the film composite of Fig. 1 in situ on a glazing panel

Detailed Description of the Invention

With reference to Fig. 1 of the accompanying drawings there is shown an exterior window
15 film composite 10 comprising a base sheet material 11 which in the present example is a solar control film laminate but could be safety film or anti-graffiti film.

In the present example the base laminate 11 comprises a first layer 12 of a transparent polyester film laminated to a metallized film layer 13. Both the first layer and the
20 metallized film layer 13 comprise polyethylene terephthalate (PET) film. The base material 11 is coated in an undercoat layer 14 which is covered with a protective hard coat 15. The other side of the base sheet material 11 may be coated in an adhesive layer 16 for adhering the window film 10 to glazing 19 (see Fig. 2). The adhesive in turn is covered by a release liner 17.

The adhesive layer is a pressure sensitive adhesive, preferably an acrylic based adhesive for example, Gelva 263 available from Cytec Industries, preferably containing upto 8 wt% UVA, typically a hydroxy benzophenone type such as Maxgard 800 . The release liner 16 may comprise a polyethylene coated paper, or a silicone release liner, that can be peeled from the adhesive leaving the adhesive on the base material 11. Other suitable adhesives types and adhesives that are commonly used in adhering films such as solar control films, anti-graffiti film or safety film to glazing may also be used.

The PET first film layer 12 of the base material 11 comprises a surface treated PET film about .0125- 0.18 mm [1/2 to 7 mil] in thickness and which has preferably been chemically treated with an acrylic surface treatment to improve adhesion. A suitable PET film is DuPont Teijin Films' Melinex 454. The film 12 is treated with a UV absorber as is described in US patent 6221 112B so as to absorb in the order of 99% of UV Radiation.

The undercoat 14 may be formed from a solvent based or water based composition comprising acrylic or acrylic-urethane based polymer cured by UV, isocyanates or aziridines. Some acrylic content is necessary to promote adhesion to the PET base material 12. The preferred undercoat layer 14 is formed from a water based UV cured acrylic-urethane hybrid dispersion having the following composition given in table 1:

Table 1

Photoinitiator	0.4- 2.0 parts
UV absorber	3.0-6.0 parts
Acrylic Urethane Dispersion	100 parts

The parts are parts by weight.

The preferred photoinitiator is an alpha-hydroxyketone type which is suitable for use in water based systems such as Esacure KIP EM (Lamberti), Irgacure 500 (Ciba Specialty Chemicals), or Irgacure 2959 as a solution in a water miscible solvent (Ciba Specialty Chemicals).

5

The UV absorber can be an organic UV absorber suitable for use in water based resins such as Tinuvin 477-DW which is an aqueous dispersion of hydroxyphenyl-triazine, available from Ciba Specialty Chemicals or an inorganic UV absorber such as Rhodigard W 200 available from Rhodia and which is an aqueous dispersion of cerium oxide. The preferred UV absorber is the organic UVA which provides a composite having an increased resistance to weathering as compared with the inorganic UVA.

10

The undercoat 14 is coated onto the base material by any suitable method for example bar coating, slot die, or reverse gravure process to provide a dry film thickness of between 6-20 microns and preferably in the order of 6-12 microns. The undercoat layer 14 is dried in a 100°C-105°C oven for 60-90 seconds. The applied coating 14 can be cross linked using an appropriate method.

15

The preferred method of curing is using UV radiation, preferably using UV light having a wavelength between about 360-440 nm is preferred, with light having a wavelength of about 395-440 nm being most preferred. A variety of UV light sources can be employed. Representative sources include but are not limited to a FUSION(TM) H-bulb high-intensity mercury lamp (which emits three bands centered at 254, 313, 365 nm and is commercially available from Fusion UV Systems, Inc.), a FUSION D-bulb iron-doped mercury lamp (which adds emission at 380-400 nm but which may emit less at lower

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wavelengths, and is commercially available from Fusion UV Systems, Inc.) and a FUSION V-bulb gallium-doped mercury lamp (which adds emission at 404-415 nm but which may emit less at lower wavelengths, and is commercially available from Fusion UV Systems, Inc.). In general, lower wavelengths promote surface cure and higher wavelengths promote bulk cure. A FUSION D-bulb generally represents a desirable overall compromise.

The present undercoat is cured under the UV lights on a conveyor running at 30 feet per minute. The undercoat can be either UV cured in air or under inert conditions, e.g., under a nitrogen atmosphere to provide inert curing.

The cured undercoat layer 14 should be sufficiently hard to prevent blocking and surface deformation when the film is wound into a roll prior to the application of the hard coat.

The preferred cured undercoat has a König hardness of 100 according to ASTM D4366-65. The undercoated film may be held in storage before the addition of the hard coat 15.

The preferred aqueous dispersion is Lux 285 which is available from Alberdingk-Boley and the composition of the preferred undercoat is given in table 2 below:

Table 2

Irgacure 2959	2 parts *
Tinuvin 477-DW	6 parts
Lux 285	100 parts

* The photoinitiator Irgacure 2959 is used for curing in air and is added in the form of a 40% solution by weight in a water miscible solvent

The cured undercoated film is then passed onto a further coating process for the application of the hard coat 15. The hard coat 15 is a cured acrylate based resin which is formed from a liquid composition which is applied to the undercoat layer 14 by any suitable process, for example bar coating, slot die coating, and reverse gravure process.

5 The hard coat composition is given in table 3 below.

Table 3

	Photoinitiator	0.5-5.0 parts
	UV stabilizers	1.0- 3.0 parts
	Urethane Oligomer	36-70 parts
10	Acrylate monomer	20-50 parts
	Solvent	30- 80 parts

The parts are parts by weight

Acetate and ketone solvents are preferred in these formulations. Suitable solvents are
15 acetone, methyl ethyl ketone, methyl isobutyl ketone, n-butyl acetate, and ethyl acetate.

The hard coat composition can contain one or more of said solvents. Suitable UV photoinitiator and UV stabilizers have been discussed previously.

The oligomer should be an acrylate oligomer, preferably an aliphatic urethane acrylate
20 oligomer having a glass transition temperature, or Tg, higher than 80°C with an elongation of less than 10%, preferably less than 5%. Aliphatic acrylate oligomers include cycloaliphatic and heterocyclic aliphatics. The harder non- yellowing urethane oligomers are preferred. Suitable urethane acrylate oligomers are CN983, CN963B80, CN985B88, and CN2920 from Sartomer (Total).

Suitable acrylate monomers are diacrylates or a mixture of polyacrylates. A suitable polyacrylate is dipentaerythritol pentaacrylate (available as Sartomer SR399), and a suitable mixture of diacrylates comprises 10-30 parts of Sartomer SR-399 with 10-20 parts of at least one of tricyclodecane dimethanol diacrylate (available as Sartomer SR833 S), and dioxane glycol diacrylate (available as Sartomer CD536).

Samples of the base material having the preferred undercoat given in table 2, were coated with the hard coat compositions according to table 3 to a cured hard coat thickness of 6.0-8.0 microns. The hardcoat is then dried in an 80°C-85°C oven for 30-45 seconds. The coating is cured by a suitable method, in this case using UV curing, preferably under inert conditions with an oxygen concentration of between 600-900 parts per million. The running speed is 80-100 feet per minute. UV cure details are similar to the conditions for the undercoat 14 given above.

Samples were then tested for adhesion of the hardcoat 15 to the base material 11 by weathering the composite 10 using an Atlas Xenon Weatherometer according to ASTM G 155 and then testing for adhesion of the hard coat every 300 hours. Samples were failed due to loss of adhesion between the hard coat and the base film material 11. All samples were evaluated on a pass/fail determination based on testing with 3M 810 and 600 tape.

The tape was pressed onto the sample surface and left for approximately 10 seconds and then pulled off at a 180° angle. The adhesion test was in accordance with ASTM D 3359 excepting that the samples were not cross-hatched. The samples were not cleaned or marked prior to testing.

It was found that samples had a minimum weathering life of 1200 hours before adhesion failure. This life expectancy could be increased to 1500-2400 hours before adhesion failure by using a mixture of acrylate monomers in the hard coat.

- 5 It has been noted that the life expectancy of the samples can be further increased by curing the hard coat in a nitrogen atmosphere. This may be due to the increase in the efficiency of the curing process due to the absence of oxygen which may react with free radicals in the coating and inhibit the curing process. When curing in air it may be necessary to increase the photoinitiator content which may be as high as 5%.

10

The preferred hard coat 16 is formed from a composition given in table 4 below for UV cure in inert conditions

Table 4

	Photoinitiator (Irgacure 819)	0.65%
15	UV Stabilizers (Tinuvin 477 and 123 in a 4:1 ratio by weight)	2.5%
	Urethane Oligomer (Sartomer CN985B88)	36.0%
	Tricyclodecane Dimethanol Diacrylate (Sartomer SR833 S)	10.0%
	Dipentaerythritol pentaacrylate (Sartomer SR399)	20.0%
	Solvent	Remainder

- 20 The percentages are approximate percentages by weight.

Samples having the preferred hard coat have a life of 3000 hours or longer when cured under nitrogen.

The abrasion resistance of the preferred hard coat was measured as a change in haze in accordance with ASTM D1003 after abrasion using a Taber Abrader in accordance with ASTM D 1044 using CS-10 Type 3 wheels each loaded to 500g. The results are quoted in a change in haze value after 50 cycles.

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The surface of the hard coat may be made hydrophobic by the addition of up to 4% by weight of a hydrophobic material additive to the composition. Potential additives include silicone and fluorinated acrylate oligomers that render the hardcoat hydrophobic. Other beneficial attributes may include easy clean, dirt release, anti-stain, anti-soil, and

10 antigrffiti properties. Suitable additives include CN4000, NTX7980 (Sartomer), Ebecryl 1360 (Cytec Surface Specialties), Fluorolink 5113X, MD 700 (Solvay Solexis), BYK 371, BYK-UV 3570, BYK-SILCLEAN 3710 (BYK Chemie), CoatOSil 3503, CoatOSil 3509 (Momentive Performance Materials), TEGO Rad 2250, TEGO Rad 2500, TEGO Rad 2600 (Evonik Industries).

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The preferred additive is a fluorinated urethane acrylate, e.g. Fluorolink 5113X available from Solvay Solexis, Italy, which is added prior to curing under nitrogen.

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Claims

1. A transparent exterior window film composite (10) comprising a base sheet material (11) having an outer PET (polyethylene terephthalate) film layer (12) which has been surface treated to elevate adhesion thereto and which is impregnated with an ultra violet light absorber (UVA), the treated surface being coated with an acrylic polymer or co-polymer undercoat (14) having a urethane-acrylate hard coat layer (15) thereon formed from at least one urethane acrylate oligomer and at least one multifunctional acrylate monomer, the undercoat (14) having a thickness which is at least as thick as the hard coat.
10
2. A film composite as claimed in Claim 1 wherein the outer PET film layer (12) of the base sheet material is between 1/2 - 7 mil (0.0125 – 0.18mm) thick and contains UVA to absorb at least 99% of ultra violet light.
- 15 3. A film composite as claimed in Claim 1 or Claim 2 wherein the undercoat (14) comprises a coating of at least one cured acrylic based resin or a urethane-acrylic based resin containing UV stabilizer.
4. A film composite as claimed in any one of Claims 1 to 3, wherein the undercoat (14)
20 contains up to 5% by weight of UV absorber.
5. A film composite as claimed in Claim 3, wherein the undercoat (14) contains up to 3 % by weight of organic UV absorber or up to 5% by weight of inorganic UV absorber.
- 25 6. A film composite as claimed in any one of Claims 1 to 5, wherein the undercoat (14) has a thickness of between 6 and 20 microns.

7. A film composite as claimed in Claim 6, where the undercoat (14) has a thickness of between 6 and 12 microns.

8. A film composite as claimed in any one of Claims 1 to 7, wherein the urethane acrylate oligomer of the hard coat (15) comprises an aliphatic acrylate urethane oligomer having a glass transition temperature (T_g) higher than 80°C with an elongation less than 10%, preferably less than 5%.

9. A film composition as claimed in any one of Claims 1 to 8, wherein the multifunctional acrylate monomer forming the hard coat (15) comprises dipentaerythritol pentaacrylate.

10. A film composite as claimed in Claim 9, in which the hard coat comprises at least two multifunctional acrylate monomers and further includes tricyclodecane dimethanol diacrylate.

11. A film composite as claimed in Claim 6 and any one of Claims 7 to 10 when depending from Claim 6, wherein the hard coat (15) has a thickness of between 6-8 microns.

12. A film composite as claimed in Claim 8, wherein the hard coat is made hydrophobic by the addition of up to 4% by weight of a hydrophobic material additive.

13. A film composite as claimed in any one of Claims 1 to 12, wherein the hard coat further contain a nanoparticle dispersion therein.

14. A film composite as claimed in any one of Claims 1 to 13, wherein the base sheet material (11) on its other surface of the polymeric film is coated with a pressure sensitive adhesive (16) which adheres to the PET film layer and in use may adhere the composite to glazing.

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15. A film composite as claimed in Claim 14, wherein the adhesive (16) is covered with a release liner (17).

16. A film composite as claimed in any one of Claims 1 to 15, wherein the film composite has a visible light transmission in the range of 5- 85% VLT.

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17. A film composite as claimed in claim 1 and which when tested for adhesion after exposure to weather testing in accordance with ASTM G 155 exceeds 1200 hours before adhesion failure between the hard coat and the base material.

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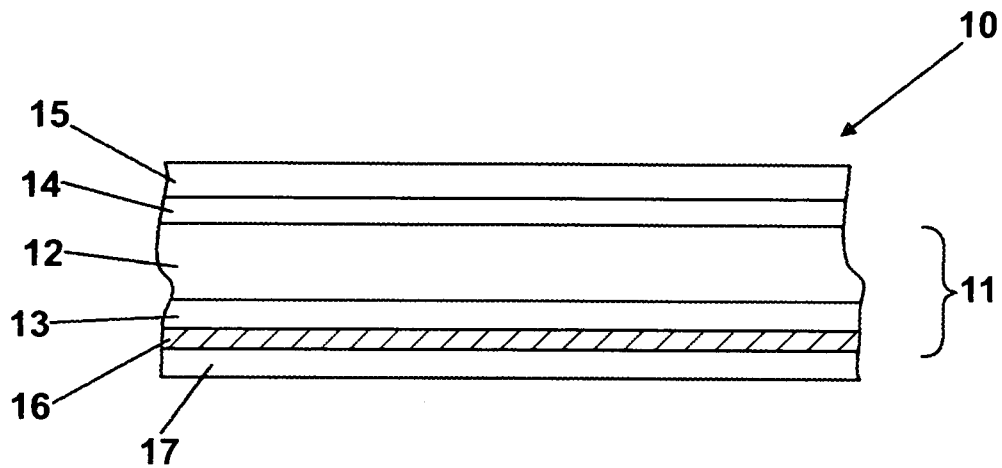
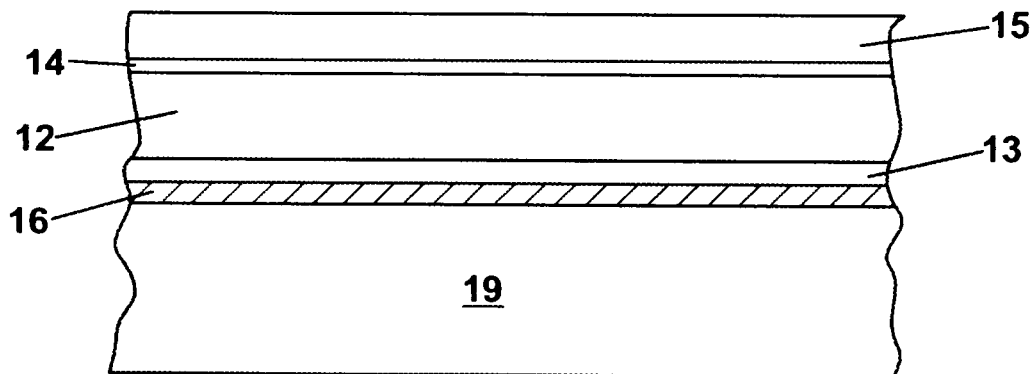
18. A film composite as claimed in Claim 11, when tested for adhesion after exposure to weather testing in accordance with ASTM G 155 has a life between 1500-2400 hours before failure between the hard coat and the base material.

19. A film composite as claimed in claim 11, cured in inert conditions and which when tested for adhesion after weather testing in accordance with ASTM G 155 exceeds 3000 hours before failure between the hard coat and the base material.

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*Fig. 1**Fig. 2*