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DESCRIPTION

Technical field

[0001] The present invention relates to a sash unit comprising a sash of a plastic material, a pane element comprising one or more sheet elements having a surface, and an organo silane layer, the organo silane layer being provided between the pane element surface and the plastic material, and methods for production of such sashs.

Background

[0002] Organo silanes have found various applications in attaching compounds together.

[0003] In the biochemical field, organo silanes have been used in the attachment of bio-molecules like polypeptides or nucleic acids to a surface. Illustrative examples are provided in WO 00/21967 and US2011/0143966.

[0004] WO 00/21967 discloses functionalised organo silanes, methods for their synthesis and their use. In one embodiment, functionalised organo silanes may be covalent bound to surfaces comprising silica, e.g. glass, to provide a functionalised glass surface, wherein molecules, including polypeptides or nucleic acids may be attached thereto. This is advantageous in conducting high volume nucleic acid hybridization assays.

[0005] US2011/0143966 discloses a method for forming an array of nucleic acids, wherein functionalized organo silanes are covalent attached to surfaces of substrates comprising silica, including microparticles. Thermal stability of particles conjugated with labelled oligonucleotides is demonstrated.

[0006] In the construction field, organo silanes have been used as a primer for attaching a window sheet to components of a window frame. Examples are provided below.

[0007] US 4951927, on which the preamble of claim 1 is based, discloses RIM moulded polymeric window frame wherein a glass surface is primed with Glass Primer 43518 comprising 3-aminopropyltriethoxysilane. However, the organo silanes suggested do not when wiped on the glass surface have the necessary durability against degradation when the final product is exposed to UV-light, and additional agents are needed to improve the adhesion of the RIM moulded polymeric window frame to the glass surface. Thus, it is therefore necessary to protect the primer layer with a second agent (blackout primer), such as Betaseal Glass Primer 43520. I.e. the RIM moulded polymeric window frame is masked with a second priming agent to improve the durability against degradation from UV-ligth.

[0008] Further, it is well-known that other masking agents can be a ceramic coating, UV hardening lacquer, a one- or two-component lacquer or any other suitable material. However, the use of an additional process step increases the total process costs.

[0009] US 7762028, which also relate to a RIM moulded polymeric window frames, discloses the use of organo silanes such as vinyl silanes, acryloxy compounds, epoxysilanes, aminosilanes, and esters of organosilanes, such as Glycidyloxypropyl-trimethoxysilane and 3-aminopropyl-triethoxysilane. US 7762028 specifically discloses the use of Glass Primer 43518 comprising 3-aminopropyl-triethoxysilane in combination with Betaseal 43520. I.e. the use of masking agents is still required in order to have sufficient durability against degradation from UV-light. US 8028478 relates to RIM mould skylight frames and suggest the use of the same silanes as US 7762028. According to US 8028478, two or more coupling agents are still required for preparing RIM mould skylight frames.

[0010] Additionally, priming of the pane element may be performed by use of e.g. Betaprimer 5500 or Betawipe VP 04206, which also are known to contain organo silanes. However it is still required to use masking agents in order to obtain a sufficient durability of the coating layer against degradation from UV-light, moisture and water.

[0011] WO2009039240 discloses two pane elements, such as glass, separated by a spacer member having a defined moisture vapour transmission rate. The surfaces of the pane elements directed towards the space member may be treated with an organo silane, such as glycidoxypropyl-trimethylsilane. The pane elements are initially cleaned with a 50% isopropyl and 50% water mixture, whereafter the primers are sprayed onto the surface and wiped off. The use of organo silanes as primers enhances the adhesion of the spacer member to the pane elements.

[0012] The lifespan of a sash unit and a pane element depend amongst other on the long term adhesion strength of the plastic material (e.g. polyurethane) to the primer or coating agent and the pane element surface. The adhesion strength of the plastic material (e.g. polyurethane) to the primer or coating agent and the pane element surface deteriorates when exposed to UV-light, water and moisture. Thus, sash units can ultimately, when exposed to UV-light, water and moisture for long time, break down.

[0013] Common failure modes for the sash unit may be cohesive failure in the bulk of the plastic material, adhesion failure between the plastic material and the primer/coating agent, adhesion failure between the primer/coating agent and the pane element surface, and stress of the plastic material at the time when maximal stress is measured. Unwanted breaking or failure of the plastic material may result in entry of moisture, which e.g. can condense in the space between two pane elements.

[0014] Thus, a requirement exist for providing sash units being more resistant against adhesion failure and more durable when exposed to UV-light, moisture, water, etc, e.g. during long term use.

Description of the invention

[0015] The present invention provides a sash unit comprising a sash of a plastic material, a pane element comprising one or more sheet elements having a surface, and an organo silane layer, the organo silane layer being provided between the pane element surface and the plastic material, wherein the organo silane layer comprises a dipodal organo silane, the organo silane layer adheres to the pane and to the plastic material of the sash and the sash at least partially surrounds the pane element encasing the edge of at least one sheet element.

[0016] The sash unit is found to be durable when exposed to UV-light, moisture and/or water.

[0017] Further, the present invention provides a method for production of a sash unit comprising a sash of a plastic material, a pane element comprising one or more sheet elements having a surface, and an organo silane layer, the organo silane layer being provided between the pane element surface and the plastic material, to obtain a sash at least partially surrounding the pane element encasing the edge of at least one sheet element, the method comprises the steps of cleaning the pane element surface, coating a dipodal organo silane on the pane element surface, and moulding the sash of a plastic material on the dipodal organo silane layer.

[0018] In one embodiment, the cleaning of the pane element surface is done by a plasma treatment.

[0019] It has surprisingly been found that if the pane element surface has a coating of a dipodal organo silane between the pane element surface and the plastic material then a sash unit comprising a sash made of plastic material and a pane element surface is obtained, which is durable and resistant against degradation when exposed to changing temperature, moisture, water and/or UV light.

[0020] In a certain aspect of the invention the plastic material is polyurethane or polyolefin. A particular high adhesion even after long term exposure to UV-radiation, water immersion and changing temperature may be obtained when polyurethane is contacted with the pane element using a reaction injection moulding (RIM),

[0021] In one embodiment of the invention, the layer of dipodal organo silanes applied on the pane element surface should be 100 nanometer or lower, preferably 50 nanometer or lower, and more preferably 20 nanometer or lower and even more preferably 5 nanometer or lower.

[0022] When a very thin coating layer of dipodal organo silane is between the pane element surface and the RIM moulded polyurethane, then this will result in a strong adhesion between to pane element surface and the layer of dipodal organo silane and the layer of dipodal organoe silane and the RIM moulded polyurethane. Thus, a sash unit having a layer of dipodal organo silane between the pane element surface and the RIM moulded polyurethane is more durable when exposed to UV-light, water and moisture.

[0023] According to the present invention, the sash unit may optionally be adapted to be installed in a window frame or alternatively, substituting a window frame as such.

[0024] According to the present invention, the pane element includes one or more sheet elements, such as sheets of glass.

[0025] The number of surfaces and edges of a sheet element vary, as these depend on the specific form and shape of the sheet element. Depending on the form and shape of the sheet element, these numbers may vary.

[0026] If the pane element comprises more than one sheet element, the sheet elements may optionally be parallel and/or separated by one or more spacer members provided at the edge.

[0027] If the pane element comprises more than one sheet element, the surfaces of a first sheet element, which are not directed towards a surface of a second sheet element, are considered as being outer surfaces. Surfaces of a first sheet element, which are directed towards a surface of a second sheet element, are considered as being inner surfaces.

[0028] An edge surface extends from the edge of a pane or sheet element towards the middle of one of the surfaces of the pane or sheet element.

[0029] In one embodiment, the edge surface may cover the whole surface of one of the surfaces of the sheet element. In a preferred embodiment, the edge surface extends from 1 to 40 mm from the edge of the pane or sheet element. In a more preferred embodiment, the edge surface extends from 1 to 20 mm from the edge of the pane or sheet element. In an even more preferred embodiment, the edge surface extends from 3 to 15 mm from the edge of the pane or sheet element.

[0030] In a specific embodiment, the edge surface is only partially coated. Hence, the first 1 to 10 mm from the edge of the pane or sheet element is not coated with an organo dipodal silane.

[0031] In a preferred embodiment, one or more of the edge surface(s) of the pane or sheet element(s) can optionally be cleaned and coated with a dipodal organo silane. Thus, an improved adhesion to the encasted plastic material is obtained.

[0032] In a preferred embodiment, the edge surface of the sheet element(s), which are not considered as being the outer sheet elements in a sash unit (e.g. if the pane element constitutes three of more sheet elements) is cleaned and coated with a dipodal organo silane.

[0033] According to the present invention, the sash surrounds the pane element, at least partially, encasing the edge of at least one sheet element. The sash gives a continuous support along the entire edge of the pane element and may be attached directly to a window frame.

[0034] According to the present invention, the term "encase" should not be understood as if the sash encloses or embraces the entire edge of the pane element; the mere contact between surfaces of the sash and pane element may give a sufficient durable adhesion.

[0035] The fact that the sash encases the edge of the pane element or at least a sheet element thereof means that the pane element is securely retained without the use of glue or the like, leading to a secure and resistant durable adhesion.

[0036] According to the present invention, the term "frame" covers both stationary and moveable frames including traditional sashes. Furthermore, the term "frame" includes such elements, which includes other elements as well, and the pane element may be used with any type of window regardless of the number of sash units or frames forming part of the window.

[0037] When using conventional thermo pane elements and the like, the sash may encase the edge of the pane element entirely. Other types of pane elements, however, have projecting edges that may be used for the attachment of the border element. One example is step unit pane elements, where the edge of one of the glass sheet elements projects over the edge of the other and over the spacer members. The border element may then be attached to the edge of the projecting sheet element.

[0038] The pane element will usually be composed of monolithic glass elements. In this context the term "monolithic glass" covers annealed glass, tempered glass, laminated glass, wired glass, figured or patterned glass as well as other types of glass that are used in conventional pane elements.

[0039] Even though the sheet elements of the pane element are referred to as being made from glass, it is to be understood that Plexiglas (also known as Perspex) or any other sheet element, transparent or not, which is suited for the particular use of the window, may also be employed, including luminescent materials.

[0040] The glass may have coatings on one or both sides. The cavity between the sheet elements may be filled with dry air, gas such as Ar, Kr or Xe, or with gas mixtures suitable for improving the insulating properties of the pane element by reducing its L) value. A vacuum pane element may also be used as may a pane element with a layer of aerogel filling the space between the sheet elements. If using a pane element type that can best be made in relatively small units, such as vacuum pane elements, a series of pane elements may be arranged side-by-side for the formation of a larger element of the desired size. This method may also be used for providing different areas of the pane element with different properties such as colour, opacity, insulation etc.

[0041] Regardless of the pane element type, the sheet elements may be parallel to each other, as is most commonly the case, or one may be inclined in relation to the other so that the distance between them vary. This latter kind of pane element has particularly good sound insulating properties and the principle may also be applied to three-sheet pane elements to thereby achieve an even better sound-proofing. Also, a combination of several pane elements arranged side-by-side or one above another in a single sash may be used, the former being advantageous when using vacuum pane elements that are difficult to make in larger sizes and the latter allowing the formation of multi-sheet pane elements.

[0042] Pane elements are usually rectangular, but other shapes such as square, circular, semi-circular or trapezoidal may also be used. The sash will usually be of the same shape as the pane element, but variations are possible. For example a trapezoidal pane element may be encased in a rectangular sash, the width of the sash varying to compensate for the difference in shape, or a rectangular pane element may be encased in a trapezoidal border element to thereby make it appear trapezoidal.

[0043] Dipodal organo silane comprises of at least two silyl groups ($-SiX_{(4-n)}$), wherein n = 1, 2, 3. Each silyl group has one, two or three X group(s) attached. The X group(s) may be identical or different and may be hydrolysable, typically alkoxy, acyloxy or chlorine, preferably methoxy, ethoxy, propoxy, which enables the silicon group to bond with inorganic substrates. The silyl groups are connected to each other with a linker group, R.

[0044] Presently, preferred dipodal organo silanes include Bis(triethoxysilylpropyl)amine, Bis(trimethoxysilylpropyl)amine, Bis(3-(trimethoxysillyl)propyl)etylenediamine, Bis(3-(triethoxysilyl)propyl)urea, Bis(trimethoxysilylpropyl)urea, Bis(methyldiethoxysilylpropyl)amine, and Bis(methyldiethoxysilylpropyl)-N-methylamine. These dipodal organo silanes make a strong bonding to the pane element surface and the RIM moulded polyurethane which may withstand exposure to UV-light, water and moisture. A particular preferred dipodal organo silane is Bis(3-(trimethoxysillyl)propyl)etylenediamine, SB1834 (Gelest).

[0045] Dipodal organo silanes are commercially available today and can amongst other be bought at Gelest Inc. Morrisville, PA 19057 (US) or UCT Spcialities, LLC, Bristol, PA 19007 (US).

[0046] Methods for producing dipodal organo silanes are amongst other disclosed in US 2005 027138, which hereby is incorporated by reference. However, dipodal organo silanes can also be produced by other known methods.

[0047] According to the present invention, cleaning of the pane element surface of unwanted surface contaminants such as oil, grease, other organic or inorganic materials, may be made by any conventional cleaning methods, such as wet chemical solvent or aqueous cleaning, e.g. rinsing with alcohol, wiping and drying, or plasma surface cleaning. Other suitable and/or conventional methods form also part of the invention. A preferred cleaning method is plasma surface cleaning.

[0048] According to the present invention, the coating of the pane element may be made by any conventional coating method such as chemical vapour deposition, physical vapour deposition, spraying, roll-to-roll coating, roll-on coating, and any other suitable method.

[0049] According to the present invention, plasma treatments are used to clean pane and glass element surfaces.

[0050] Plasmas, when used in plasma treatment such as plasma surface cleaning, are generated by the application of electric and/or magnetic fields. Different types of power source may be applied, such as direct current (DC), radio frequency (RF), and microwaves.

[0051] According to the present invention, different dischargers and/or electrode configurations to generate plasma can be used.

[0052] Thus, plasma can e.g. be generated when an electrical current is applied on electrodes wherein a dielectric gas can pass.

[0053] Plasma can be generated at vacuum pressure, i.e. lower than 1 Pa, moderate pressure; i.e. between 1 Pa and 100 kPa, preferably between 50 Pa and 50 kPa, more preferably between 90 Pa and 1 kPA, even more preferably between 90 Pa and 200 Pa, and atmospheric pressure.

[0054] An example of a vacuum or moderate pressure discharges is a glow discharge which generate non-thermal plasmas by the application of DC or low frequency RF (<100 kHz) to the gap between two metal electrodes. Other examples of vacuum or moderate pressure discharges are capacitively coupled plasma and inductively coupled clasma.

[0055] An example of atmospheric pressure discharge is an arc discharge which is a high power discharge of very high temperature (~10,000 K). Another atmospheric pressure discharges can be a corona discharge which is a non-thermal discharge generated by the application of high voltage to sharp electrode tips. Dielectric barrier discharge is another example of non-thermal discharge generated by the application of high voltages across small gaps wherein a nonconducting coating prevents the transition of the plasma discharge into an arc.

[0056] According to the present invention, the sash may be of any type of plastic material preferably thermoplastic material such as polyurethane or polyolefin. Other conceivable materials include thermoplastic materials such as PVC, PE or PP, a thermoplastic elastomeric (TPE) and thermoset elastomer materials such as ethylene propylene diene monomer (EDPM).

[0057] Reaction injection moulding (RIM) or low pressure moulding may be used for the manufacture of the sash.

Figures

[0058]

Fig. 1 shows a sash (31) encasing a pane element comprising two sheet elements (1), a space member (22) and a pane element sealing (23), wherein a layer of dipodal organo silane (32) is coated on the glass element surface.

Fig. 2 shows a H-piece RIM module, comprising a sash (31) made of polyurethane encasing two sheet elements (1), wherein a layer of organo silane (32) is coated of the sheet (glass) element surface. The dimensions of the H-piece RIM module is as follows X = 15 mm, Y = 20 mm, Z = 15 mm.

Fig. 3 shows the temperature cycle and the UV-A cycle when testing the H-piece RIM modules in a Mathilde test.

Fig. 4A, Fig. 4B and Fig. 4C show results from the water immersion test at 40°C.

Fig. 5A and Fig. 5B shows results from the Xenon chamber test when exposed to an UV intensity of 460 MJ.

Fig. 6 shows results from test in the Mathilde box after 15 weeks exposure.

Detailed description

[0059] In one embodiment of the invention, the pane element or any part thereof is cleaned by plasma surface cleaning. Plasma surface cleaning with oxygen plasma eliminates natural and technical oils and grease at the nano-scale level and reduces contamination significantly when compared with traditional wet cleaning methods. One theory is that the ultra-violet light generated in the plasma is very effective in the breaking of most organic bonds of surface contaminants. This helps to break apart oils and grease. The cleaning action is carried out by the energetic oxygen species created in the plasma. These species react with organic contaminants to form mainly water and carbon dioxide, which are continuously removed (pumped away) from the chamber during processing. These contaminants are not harmful.

[0060] The advantage of plasma surface cleaning is a precisely controllable process through variation of the power supply, pressure, gas type, processing time etc, and no organic residues remain on the cleaned surface. The contaminants are harmless and always in gaseous form that can be liberated directly to atmosphere. UV radiation and active oxygen species from the plasma break up separating agents, silicones and oils from the surface. These are pumped away by the vacuum system. Active oxygen species (radicals) from the plasma bind to active surface sites all over the material, creating a surface that is highly 'active' to bonding agents. Technical oxygen is usually used as the process gas, however, many plasma activations can also be carried out with just ambient air.

[0061] Plasma cleaning produces a pristine surface, ready for bonding or further processing, without any harmful waste material. The cleaned surface remains active for a few minutes up to several months, depending on the particular material that has been plasma treated. Free SiOH groups of the pane elements or glass elements are during plasma cleaning activated and can react with any suitable chemical group which these groups are exposed to.

[0062] In one embodiment of the invention, plasma generators using atmospheric plasma generators are preferable. The advantage of using these types of generators is that they are versatile for stand-alone surface treatments and can operate as a fully integrated plasma equipment in automated production lines.

[0063] According to the invention, preferred dipodal organo silanes of formula (II) are part of the invention

(SiX_3)-R-(SiX_3) Formula(II)

R: R_1-NR_3-R_2,

R_1-NR_3-R_4-NR_5-R_2

wherein the X groups independently are hydrogen, alkyl, alkoxy, acyloxy, or halogen, preferably methyl, ethyl, propyl, methoxy, ethoxy and propoxy, R_1 and R_2 are independently branched or unbranched alkylene, preferably methylene, ethylene, propylene, and butylene, R_3 and R_5 is hydrogen, alkyl, alkoxy, acyloxy, or halogen, preferably hydrogen, methyl, ethyl, propyl, methoxy, ethoxy, propoxy, , and R_4 is alkylene, carbonyl, preferably ethylene, and carbonyl (urea group).

[0064] In one embodiment of the invention, preferred dipodal organo silanes are Bis(triethoxysilylpropyl)amine, Bis(trimethoxysilylpropyl)amine, Bis(3-(trimethoxysillyl)propyl)etylenediamine, Bis(3-(triethoxysilyl)propyl)urea, Bis(trimethoxysilylpropyl)urea, Bis(methyldiethoxysilylpropyl)amine, and Bis(methyldiethoxysilylpropyl)-N-methylamine.

[0065] Polyurethanes are produced by the polyaddition reaction of a polyisocyanate with a polyalcohol (polyol) in the presence of a catalyst and other additives polyisocyanates contain two or more isocynate groups. Isocyanates can be classed as aromatic, such as diphenylmethane diisocyanate or toluene diisocyanate; or aliphatic, such as hexamethylene diisocyanate or isophorone diisocyanate. Aliphatic polyols are preferred because they are less prone to degeneration in sunlight. An example of a polymeric isocyanate is polymeric diphenylmethane diisocyanate, which is a blend of molecules with two-, three-, and four- or more isocyanate groups. Polyols contain two or more hydroxyl groups. The polymerization reaction may be catalyzed by tertiary amines, such as dimethylcyclohexylamine, and organometallic compounds, such as dibutyltin dilaurate or bismuth octanoate. Furthermore, catalysts can be chosen based on whether they favor the urethane (gel) reaction, such as 1,4-diazabicyclo[2.2.2]octane, or the urea (blow) reaction, such as bis-(2-dimethylaminoethyl)ether, or specifically drive the isocyanate trimerization reaction, such as potassium octoate.

[0066] The adhesion of the plastic material to the coated organo silane layer is established during the moulding process. Thus, a strong adhesion between the plastic layer and the coated organo silane layer or coated organo silane layer on the activated glass surface is achieved.

[0067] In one embodiment of the invention, the sash is preferably made from polyurethane by moulding around the pane element so that edges of one or both pane elements are encased in the moulding material. The sash may surrounds the entire border of the pane element, but it is to be understood that it may also be U-shaped surrounding the pane element or glass element on three of its four sides or that separate elements may be used on each side leaving the corners of the pane element free. Similarly it is to be understood that pane elements with other geometrical configurations, i.e. semi-circular or triangular, are also conceivable.

[0068] In one embodiment of the invention, the sash unit may be produced by using any suitable moulding technique, but injection moulding, e.g. reaction injection moulding (RIM), is preferred. When using the RIM process, current-carrying components, plastic or metal components contributing to strength and stiffness, screws etc. may be embedded in the moulding material. Furthermore, the RIM process allows the integration of details such as pane element sealings and space members, if needed.

[0069] Reaction injection moulding (RIM) is a process that is well known per se. During moulding, a two-component curing polyurethane is mixed in the mould containing the pane element to be encased. In the mould a pressure of approximately 6 to 10 bar is obtained during the curing process. The cured module is ready to be handled within approximately 45 to 60 seconds. During the RIM process itself the temperature of the material and the mould lies between 80 and 110 °C depending on the configuration of the mould and whether the polyurethane used is of the aromatic or the aliphatic kind. Depending on the kind of polyurethane used, different Shore A hardnesses may be obtained. In the example polyurethane having a cured hardness of 60-90 Shore A may be used.

[0070] Wherever polyurethane is mentioned in the description it is to be understood that other materials may also be used, possibly with slight adaptations, which will be obvious to the person skilled in the art. Further, any suitable polyurethane coating may be used, including the catalyst components, cross-linkers and surfactants necessary to form the polymer coating.

[0071] It should be noted that embodiments and features described in the context of one of the aspects of the present invention also apply to the other aspects of the invention.

Example 1

[0072] A H-piece RIM module was prepared as follows:

[0073] Two glass elements were initially cleaned by surface plasma cleaning using a Tantec atmospheric plasma generator apparatus.

[0074] The glass element surfaces were then coated by wiping a solution of 2% w/w organo silane or dipodal organo silane, 94% w/w ethanol and 6% w/w water with a filtpath. After 1 minute, the surface of the glass element was cleaned with ethanol. The glass element was hardened in an oven at 60°C for 1 hour (< 6% relative humidity).

[0075] Finally, polyurethan was moulded by using the RIM method on the two glass element surfaces such that the glass element surfaces were connected to each other.

[0076] The following H-piece RIM modules (Fig. 2) with dipodal organo silanes as primer system were prepared:

Table 1: Prepared H-piece RIM modules

H-piece RIM module Sample	Primer
A	Bis-3-trimethoxysilylpropyl-ethylenediamine, SIB 1834 Gelest Incorporation
I	Bis[(3-trimethoxysilyl)propyl)ethyleneamine, Gelest Incorporation

[0077] As reference samples, a H-piece RIM module was prepared as follows:

[0078] Two glass elements were wiped with a 2% dipodal organo silane solution and dried.

[0079] The glass element surfaces were then coated by wiping a solution of 2% w/w organo silane or dipodal organo silane, 94% w/w ethanol and 6% w/w water with a filtpath. After 1 minute, the surface of the glass element was cleaned with ethanol. The glass element was hardened in an oven at 60°C for 1 hour (< 6% relative humidity).

[0080] Finally, polyurethan was moulded by using the RIM method on the two glass element surfaces such that the glass element surfaces were connected to each other.

[0081] The following H-pieces with conventional primers were prepared:

Table 2: Prepared H-piece RIM module

H-piece RIM module Sample	Primer system
B	Aminopropyltriethoxysilane, SIA 0610 Gelest Incorporation
C	N-Methylaminopropyltrimethoxysilane, SIM 6500 Gelest Incorporation
D	Aminopropylmethyldiethoxysilane, SIA 0605 Gelest Incorporation

H-piece RIM module Sample	Primer system
E	Glycidyloxypropyltrimethoxysilane, obtained from ABCR
F	Isocyanatopropyltriethoxysilane, SIA 6455 Gelest Incorporation
G	Betawipe VP 04604, Dow Chemical
H	Betaprimer 5500, Dow Chemical

Example 2

Water immersion

[0082] H-piece RIM modules were placed in a water bath at constant temperature, 40°C, for 3, 6, 9, 12 and 15 weeks, respectively. Each test consists of a dataset of 5 samples.

[0083] The adhesion property between the polyurethane material for RIM and the glass element surface of the different H-piece RIM modules after the water immersion test was measured in a tension test by pulling the H-piece RIM modules. The H-piece RIM modules were pulled at a speed of 5 mm/min and the adhesion strength / breakage failure mode of the H-piece RIM modules was measured.

Score

[0084] If the measured average adhesion strength is over 3 MPa then the measured adhesion strength is strong.

[0085] If the measured average adhesion strength is over 1 MPa and lower than 3 MPa then the measured adhesion strength is poor.

[0086] If the measured average adhesion strength is lower than 1 MPa then the adhesion strength is lost.

Table 4: H-piece RIM module tested in water bath at 40°C, Figure 4A, Figure 4B and Figure 4C.

Sample	Test Exposure / weeks	Average adhesion strength Score
Reference A	No water immersion	Strong
A	Water immersion: 3, 6, 9, 12, and 15 weeks	Strong
B, C, D, E, F	Water immersion: 3 weeks	Lost
G	Water immersion: 3 weeks	Poor
H	Water immersion: 3 weeks	Strong
I	Water immersion: 3, 6, 9, 12, and 15 weeks	Strong

[0087] Reference A was kept dry and was therefore not exposed to water immersion.

[0088] The observed failure mode was the adhesion failure between polyurethane and the primer and adhesion failure between primer and glass surface. Thus, the polyurethane material itself was not the reason for failure (cohesive failure and stress of the polyurethane material).

[0089] The reference sample has a higher adhesion strength than the samples that have been exposed to water immersion. This is due to the fact that the polyurethane material of the reference sample has not taken up any water, which deteriorates the adhesion strengths of the polyurethane material.

[0090] Xenon chamber: H-pieces were exposed to UV light (300-400 nm) in a Xenon Ci4000 weather-O-meter according to the Japanese standard JS 1900.

[0091] The adhesion strength between the polyurethane material for RIM and the glass element surface of the different H-piece RIM modules after the Xenon chamber test was measured in a tension test by pulling the H-piece RIM modules. The H-piece RIM modules were pulled at a speed of 5 mm/min and the adhesion strength / breakage failure mode of the H-piece RIM modules was measured.

Score

[0092] If the measured average adhesion strength is over 3 MPa then the measured adhesion strength is strong.

[0093] If the measured average adhesion strength is over 1 MPa and lower than 3 MPa then the measured adhesion strength is poor.

[0094] If the measured average adhesion strength is lower than 1 MPa then the adhesion strength is lost.

Table 3: H-piece RIM module tested in Xenon chamber, Figure 5A and Figure 5B.

Sample	Average adhesion strength, Score
A - Figure 5A	Strong
G - Figure 5A	Strong
H - Figure 5A	Poor
I - Figure 5B	Strong

[0095] The observed failure mode was the adhesion failure between polyurethane and the primer and adhesion failure between primer and glass surface. Thus, the polyurethane material itself was not the reason for failure (cohesive failure and stress of the polyurethane material).

Example 3

[0096] The H-piece RIM modules were placed in a closed chamber, "Mathilde box". The aging cycles related to temperature, exposure to water and UV light follows the profile disclosed in figure 2. The bottom edge of the H-piece RIM modules, was covered by water, when the temperature in the climate chamber is higher than 4°C. At the same time the bottom edge of the pane elements was illuminated with UV-A light with an effect of 80 W/m^2. The aging test consists of a dataset of 5 samples.

[0097] The adhesion property between the polyurethane material for RIM and the glass element surface of the different H-piece RIM modules after the Mathilde box test was measured in a tension test by pulling the H-piece RIM modules. The H-piece RIM modules were pulled at a speed of 5 mm/min and the adhesion strength / breakage failure mode of the H-piece RIM modules was measured.

Score

[0098] If the measured average adhesion strength is over 3 MPa then the measured adhesion strength is strong.

[0099] If the measured average adhesion strength is over 1 MPa and lower than 3 MPa then the measured adhesion strength is poor.

[0100] If the measured average adhesion strength is lower than 1 MPa then the adhesion strength is lost.

Table 4: H-piece RIM module tested in Mathilde box in 18 weeks

Sample	Average adhesion strength Score
A	Satisfactory
G	Poor
H	Lost

[0101] The observed failure mode was the adhesion failure between polyurethane and the primer and adhesion failure between primer and glass surface. Thus, the polyurethane material itself was not the reason for failure (cohesive failure and stress of the polyurethane material).

Summary Example 1-3

[0102] Coated bis(trimethoxysilylpropyl)ethylenediamine maintained a good adhesive strengths after exposure to water and UV-light. Conventional primers comprising a organo silane such as Betaprimer 5500 and Betawipe VP 04604 lost their adhesive strength when exposed to UV-light or water immersion, respectively. Further, conventional primers comprising a organo silane such as glycidyloxypropyltrimethoxysilane and aminopropyltriethoxysilane, lost their adhesive strength when exposed to UV-light or water immersion

Example 4

[0103] Sample A was exposed to water immersion for 20 weeks at 25°C, 40°C and 70°C, and a Mathilde test for 30 weeks. Subsequently, sample A were evaluated in three different tests; a tensile test, peel test and shear test. Subsequently, sample A was evaluated in the tensile test as described above.

Table 5: Test of H-piece RIM module, sample A

Test	Adhesion strength
Water immersion 20 weeks at 25°C	No long term deterioration of the adhesive strength
Water immersion 20 weeks at 40°C	No long term deterioration of the adhesive strength
Water immersion 20 weeks at 70°C	No long term deterioration of the adhesive strength
Temperature interval -20°C-75°C + water and UV-light, 30 weeks in total	No long term deterioration of the adhesive strength
Xenon test 1000 MJ according to method JS 1900 (standard within industry),	No long term deterioration of the adhesive strength

Example 5: Insulating sash units

[0104] Sash units comprising 2 or 3 sheets elements of glass were prepared according to the procedure in Example 1. All the borders and edges of the sheet elements were encasted such that a closed space between the individual sheet elements in the sash unit could comprise an inert gas.Characteristics of the prepared polyurethane sash units:

Example 5.1: Dipodalsilane: Bis(3-trimethoxysilyl)propyl)amine; 2 layer of glass - 432 mm x 615 mm (4 mm, 16 mm Argon, 4 m).

Example 5.2: Conventional silane: betaprimer 5500; 2 layer of glass - 432 mm x 615 mm (4 mm, 16 mm Argon, 4 mm).

Example 5.3: Dipodalsilane: Bis(3-trimethoxysilyl)propyl)amine; 3 layer of glass - 432 mm x 615 mm (6 mm, 10 mm Argon, 3 mm,10 mm Argon, 4 mm).

Example 5.4: Conventional silane: betaprimer 5500; 3 layer of glass - 432 mm x 615 mm (6 mm, 10 mm Argon, 3 mm,10 mm Argon, 4 mm).

[0105] The 4 sash units were placed in a closed chamber, "Mathilde box". The aging cycles related to temperature, exposure to water and UV light follows the profile disclosed in figure 2. The bottom edge of the H-piece RIM modules, was covered by water, when the temperature in the climate chamber is higher than 4°C. At the same time the bottom edge of the pane elements was illuminated with UV-A light with an effect of 80 W/m^2. The aging test consists of a dataset of 5 samples.

[0106] The adhesion property between the polyurethane material for RIM and the glass element surface of the different sash units after the Mathilde box test was measured in a tension test by pulling the sash units. The sash units were pulled at a speed of 5 mm/min and the adhesion strength / breakage failure mode of the sash units was measured.

Score

[0107] If the measured average adhesion strength is over 3 MPa then the measured adhesion strength is strong.

[0108] If the measured average adhesion strength is over 1 MPa and lower than 3 MPa then the measured adhesion strength is poor.

[0109] If the measured average adhesion strength is lower than 1 MPa then the adhesion strength is lost.

Table 6: Sash units tested in Mathilde box in 10/20 weeks

Sample	Average adhesion strength, Score
Example 5.1	Strong, after 20 weeks
Example 5.2	Poor, after 10 weeks
Example 5.3	Strong, after 20 weeks
Example 5.4	Poor, after 10 weeks

[0110] The observed failure mode was the adhesion failure between polyurethane and the primer and adhesion failure between primer and glass surface. Thus, the polyurethane material itself was not the reason for failure (cohesive failure and stress of the polyurethane material).

[0111] The sash units, Example 5.1 and Example 5.3, wherein Bis(3-trimethoxysilyl)propyl)amine was in between the surface of the glass element and the encasted polyurethane, maintained their adhesive strengths after exposure to the Mathilde Boxfor 20 weeks whereas sash units prepared with the the conventional silane compounds, Example 5.2 and Example 5.4, lost most of the adhesive strength after 10 weeks of exposure.

REFERENCES CITED IN THE DESCRIPTION

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Patent documents cited in the description

- WO0021967A **[0003] [0004]**
- US20110143966A **[0003] [0005]**
- US4951927A **[0007]**
- US7762028B **[0009] [0009] [0009]**
- US8028478B **[0009] [0009]**
- WO2009039240A **[0011]**
- US2005027138A **[0046]**

PATENTKRAV

1. Rammeenhed, der omfatter en ramme af et plastmateriale, et rudelement, der omfatter et eller flere pladeelementer, der har en overflade, hvilken ramme mindst delvist omgiver rudeelementet, der omslutter kanten af mindst ét pladeelement, og et organisk silanlag, hvilket organisk silanlag er tilvejebragt mellem rudeelementoverfladen og plastmaterialet, **kendetegnet ved**, at det organiske silanlag omfatter en dipodal organisk silan og ved, at det organiske silanlag klæber til rudeelementet og til rammens plastmateriale.

2. Rammeenhed ifølge krav 1, hvor rudeelementet omfatter to parallelle pladeelementer adskilt af et afstandsstykke.

3. Rammeenhed ifølge kravene 1 eller 2, hvor det organiske silanlag er tilvejebragt ved en ydre kantoverflade(r).

4. Rammeenhed ifølge kravene 1 til 3, hvor den ydre kantoverflade strækker sig 1 til 50 mm fra kanten af rudeelementet.

5. Rammeenhed ifølge kravene 1 til 4, hvor plastmaterialet er polyurethan eller polyolefin.

6. Rammeenhed ifølge kravene 1 til 5, hvor den dipodale, organiske silan er udvalgt fra gruppen bestående af
Bis(triethoxysilylpropyl)amin,
Bis(trimethoxysilylpropyl)amin,
Bis(3-(trimethoxysillyl)propyl)ethylendiamin,
Bis(3-(triethoxysilyl)propyl)urea,
Bis(trimethoxysilylpropyl)urea,
Bis(methyldiethoxysilylpropyl)amin og

Bis(methyldiethoxysilylpropyl)-N-methylamin.

7. Rammeenhed ifølge kravene 1 til 6, hvor tykkelsen af det dipodale organiske silanlag er 100 nm eller mindre.

8. Rammeenhed ifølge kravene 1 til 7, hvor tykkelsen af den dipodale organiske silan er mindre end 20 nm.

9. Fremgangsmåde til fremstilling af en rammeenhed, der omfatter en ramme af et plastmateriale, et rudelement der omfatter et eller flere pladeelementer, der har en overflade, og et organisk silanlag, hvilket organiske silanlag er tilvejebragt mellem rudeelementoverfladen og plastmaterialet, omfattende trinnene med

   a. rengøring af rudeelementoverfladen,
   b. coating af den dipodale organiske silan på rudeelementoverfladen, og
   c. støbning af rammen med et plastmateriale på det dipodale organiske silanlag,

   for at opnå en rammeenhed, der har en ramme, der mindst delvist omgiver rudeelementet, der indkapsler kanten af mindst ét pladeelement.

10. Fremgangsmåde ifølge krav 9, hvor den dipodale organiske silan er udvalgt fra gruppen bestående af
Bis(triethoxysilylpropyl)amin,
Bis(trimethoxysilylpropyl)amin,
Bis(3-trimethoxysillyl)propyl)ethylendiamin,
Bis(3-(triethoxysilyl)propyl)urea,
Bis(trimethoxysilylpropyl)urea,
Bis(methyldiethoxysilylpropyl)amin og

Bis(methyldiethoxysilylpropyl)-N-methylamin,

11. Fremgangsmåde ifølge kravene 9 eller 10, hvor lagtykkelsen af den dipodale organiske silan er 100 nm eller mindre.

12. Fremgangsmåde ifølge et hvilket som helst af kravene 9 til 11, hvor plastmaterialet er polyurethan eller polyolefin.

13. Fremgangsmåde ifølge et hvilket som helst af kravene 9 til 12, hvor coatingen af en dipodal organisk silan er påført en eller flere af de ydre kantoverflade(r) af et rudelement, hvilke ydre kantoverflade(r) af rudeelementet strækker sig 1 til 50 mm fra kanten af rudeelementet.

14. Fremgangsmåde ifølge et hvilket som helst af kravene 9 til 13, hvor rengøringen er plasmaoverfladerengøring.

15. Anvendelse af dipodal organisk silan som et coatingmateriale mellem en rudeelementoverflade og et rammeplastmateriale, hvorved det dipodale organiske silanlag klæber til rudeelementoverfladen og til rammens plastmateriale.

DRAWINGS

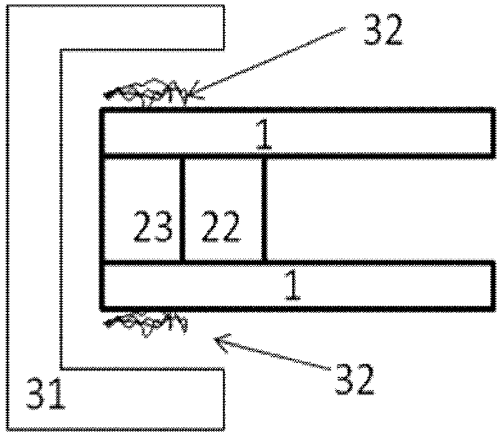


Figure 1

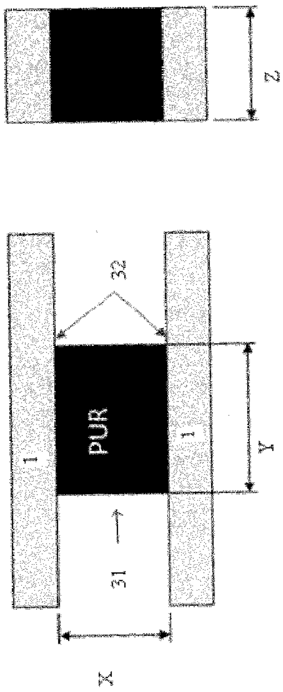
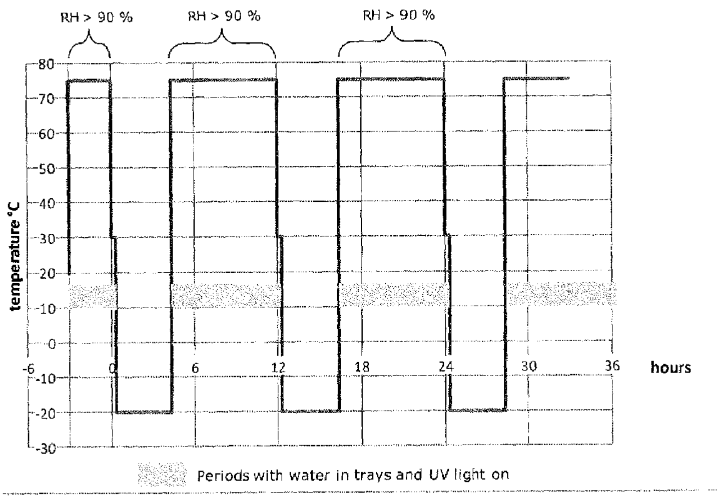


Figure 2



RH > 90 %
RH > 90 %
RH > 90 %
temperature °C
80
70
60
50
40
30
20
10
0
-10
-20
-30
-6
0
6
12
18
24
30
36
hours
Periods with water in trays and UV light on

Figure 3

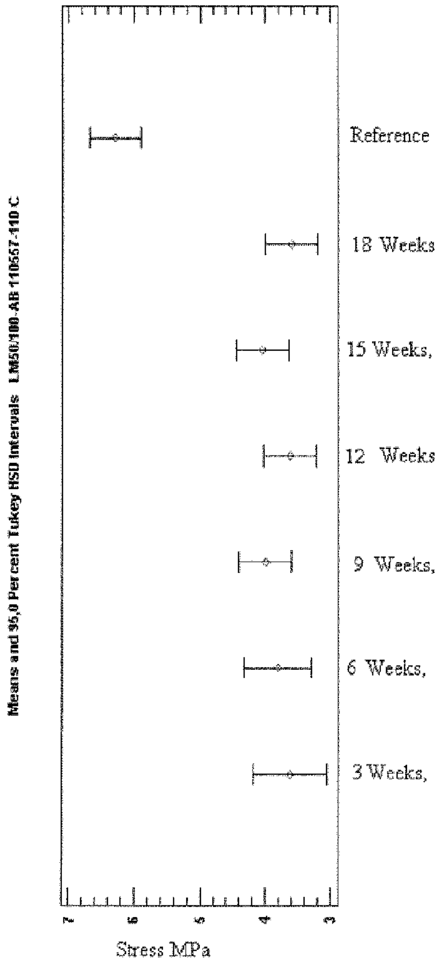

Means and 95,0 Percent Tukey HSD Intervals LM50/100-AB 110557-110 C
Reference
18 Weeks
15 Weeks,
12 Weeks
9 Weeks,
6 Weeks,
3 Weeks,
7
6
5
4
3
Stress MPa

Figure 4A

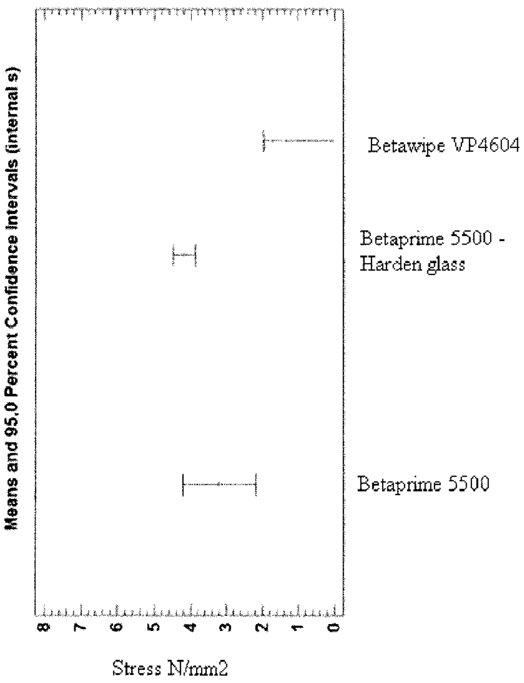

Means and 95,0 Percent Confidence Intervals (internal s)
Betawipe VP4604
Betaprime 5500 - Harden glass
Betaprime 5500
8 7 6 5 4 3 2 1 0
Stress N/mm2

Figure 4B

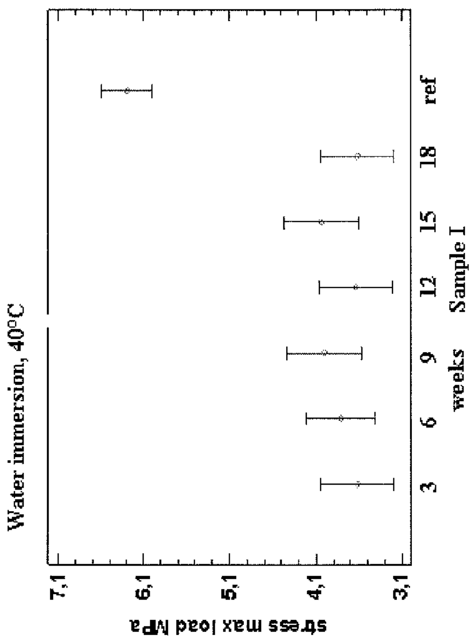

Water immersion, 40°C
stress max load MPa
7,1
6,1
5,1
4,1
3,1
3
6
9
12
15
18
ref
weeks Sample I

Figure 4C

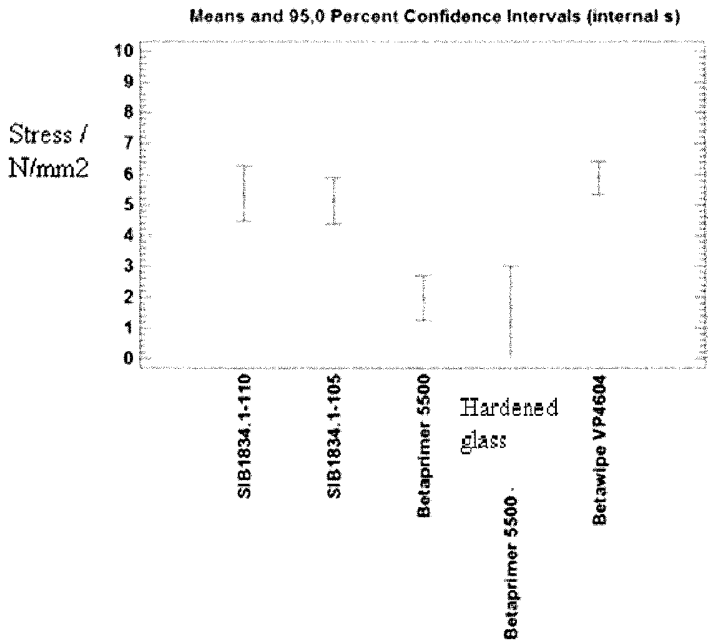

Means and 95,0 Percent Confidence Intervals (internal s)
Stress / N/mm2
10
9
8
7
6
5
4
3
2
1
0
SIB1834.1-110
SIB1834.1-105
Betaprimer 5500
Hardened glass
Betaprimer 5500
Betawipe VP4604

Figure 5A

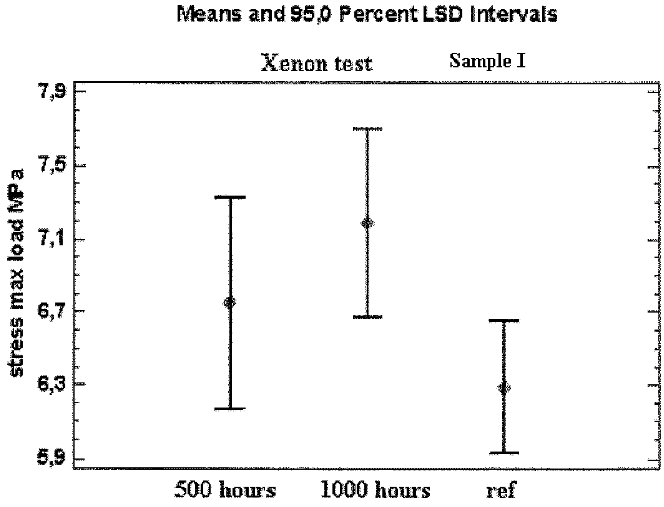
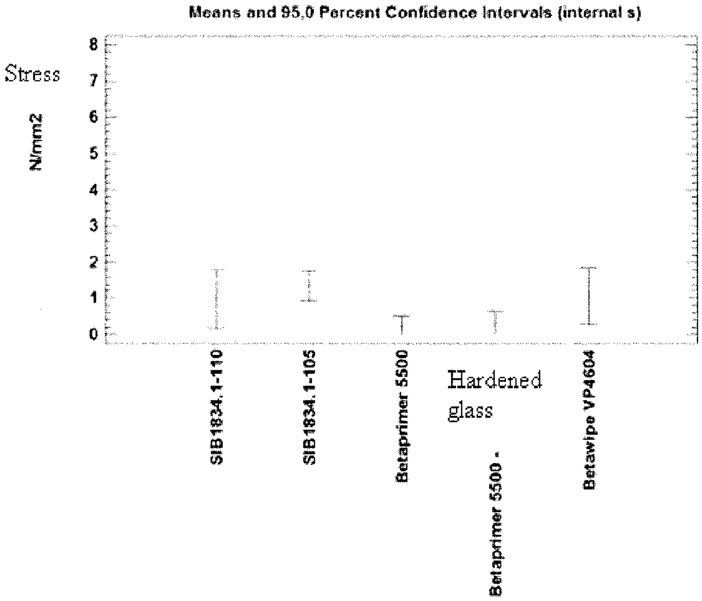

Means and 95,0 Percent LSD Intervals
Xenon test
Sample I
stress max load MPa
7,9
7,5
7,1
6,7
6,3
5,9
500 hours
1000 hours
ref

Figure 5B



Means and 95,0 Percent Confidence Intervals (internal s)
Stress
N/mm2
8
7
6
5
4
3
2
1
0
SIB1834.1-110
SIB1834.1-105
Betaprimer 5500
Hardened glass
Betaprimer 5500 -
Betawipe VP4604

Figure 6