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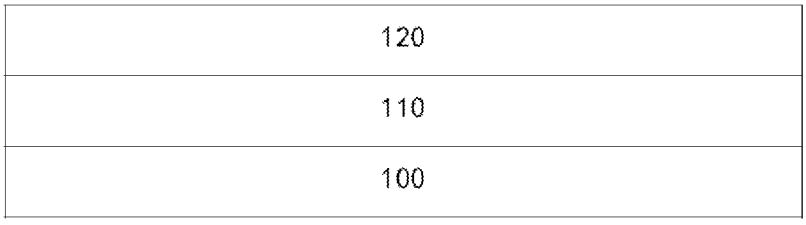

Figure 1

(57) Abstract: Provided is a laminate structure including: a glass substrate (100), a functional layer (110) disposed on a surface of the glass substrate (100), and a modified polyurethane coating (120) disposed on the surface of the functional layer (110).

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LAMINATE STRUCTURE

This application claims the benefit of priority to Chinese Patent Application No. CN201610634502.9 titled "LAMINATE STRUCTURE", filed with the Chinese State Intellectual Property Office on August 4, 2016, the entire disclosure of which is incorporated herein by reference.

FIELD

The present disclosure relates to the glass laminate field, and in particular to a glass laminate structure including a functional layer whose surface is covered with a modified polyurethane coating.

BACKGROUND

Silver-coated glass has good solar cutting performance, and meanwhile can maintain the transparency of glass. In existing technology, there are various types of silver-coated glass. However, the silver coating is susceptible to oxidization when being exposed to the ambient environment. Accordingly, at present, applications of the silver-coated glass are limited to laminate structured glass (e.g., windshield), where the silver coating can be protected by being isolated from the ambient environment via two pieces of glass and one polyvinyl butyral (PVB) layer.

To date, there is no successful development in the art to apply the silver coating to a single piece of glass (e.g., a piece of toughened glass applied on an automobile).

SUMMARY

An embodiment of the present disclosure provides a novel glass laminate structure, so as to solve the deficiencies and problems of the

existing technology.

Polyurethane (PU) is a generic term of polymer compounds whose main chain includes a carbamate (-NH-COO-) unit. The polyurethane is a multi-block polymer stepwise polymerized by polyols, small molecule chain extenders and polyisocyanates, where the polyols constitute soft segments, and the small molecule chain extenders and the polyisocyanates constitute hard segments. Thus, by adjusting compositions and proportions of the soft segments and the hard segments, a molecular structure, a physical property and a chemical property of the polyurethane can be effectively controlled. Moreover, the hard segments have a strong polarity and are easy to gather; and a micro-phase separation structure can be formed given a thermodynamic incompatibility between the hard segments and the soft segments. The micro-phase separation structure of the polyurethane makes the polyurethane have a better biocompatibility in comparison with other polymers, which makes the polyurethane capable of being used as a surface coating of medical devices or articles. In addition, the polyurethane has excellent and adjustable physical and mechanical properties, which makes the polyurethane capable of being widely used in surface coating. However, the polyurethane, especially the polyurethane coating, may inevitably be subjected to various external conditions in the course of use, which will directly effect the safety and service life of the polyurethane.

The inventors of the present disclosure found that, a modified polyurethane coating formed by modifying a polyurethane with specific materials can effectively prevent cracking and oxidization phenomenon. Thus, when the modified polyurethane is coated onto a functional layer, the functional layer can be effectively prevented from cracking and oxidating. Based on the above finding, the present disclosure has been

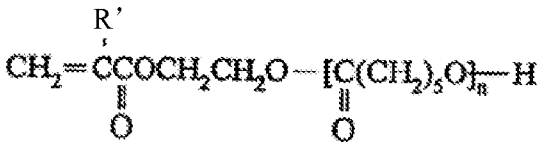
accomplished.

An embodiment of the present disclosure provides a laminate structure, including: a glass substrate, a functional layer disposed on a surface of the glass substrate, and a modified polyurethane coating disposed on a surface of the functional layer, wherein raw materials of the modified polyurethane coating include following main ingredients:

(A) isocyanate, wherein each molecule of the isocyanate has three or more isocyanate groups;

(B) long chain alkyl alcohol, wherein a number of carbon atoms included in the long chain alkyl alcohol ranges from 10 to 25; and

(C) polycaprolactone-modified (meth) hydroxy ethyl acrylate having a formula as follow:



where, R' denotes $-CH_3$ or –H; and n=1-25, and

wherein, by mole, hydroxyl in the long chain alkyl alcohol (B) : the isocyanate groups in the isocyanate (A) : hydroxy in the polycaprolactone-modified (meth) hydroxy ethyl acrylate (C) = 1 : (3-50) : (2-60).

In a preferred embodiment, the raw materials of the modified polyurethane coating further include: 0.01 to 5% by weight of an anti-scratch agent, 0.01 to 1% by weight of a catalyst, 0.01 to 1% by weight of an antistatic agent, 0.01 to 1% by weight of a polymerization inhibitor, and a solvent serving as a balance, wherein the main ingredients of the modified polyurethane coating are 100% by weight.

In another preferred embodiment, the anti-scratch agent includes organic beads and inorganic beads.

In another preferred embodiment, the organic beads include:

polymethyl methacrylate, polyurethane, nylon, and rubber.

In another preferred embodiment, the catalyst is 0.1 to 0.5 % by weight; and the catalyst includes dibutyltin dilaurate and dibutyl tin diethyl n-hexanoate.

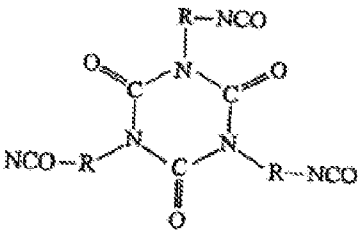
In another preferred embodiment, the antistatic agent includes: an antistatic agent containing lithium, sodium or potassium salt, a cationic antistatic agent containing amine salt or quaternary ammonium salt, and a nonpolar antistatic agent containing C8 to C12 alkyl or alkaryl.

In another preferred embodiment, the polymerization inhibitor includes hydroquinone monomethyl ether.

In another preferred embodiment, the solvent includes: xylene, ketone and ester.

In another preferred embodiment, the ketone includes acetone, methyl ethyl ketone, methyl isobutyl ketone and cyclohexanone; and the ester includes ethyl acetate, propyl acetate, isobutyl acetate and butyl acetate.

In another preferred embodiment, the isocyanate includes:



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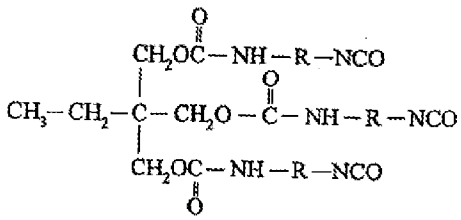
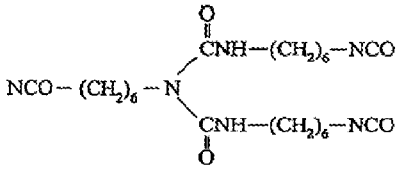
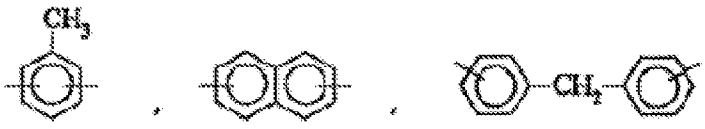
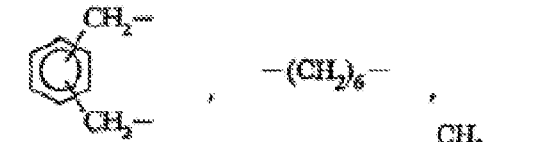


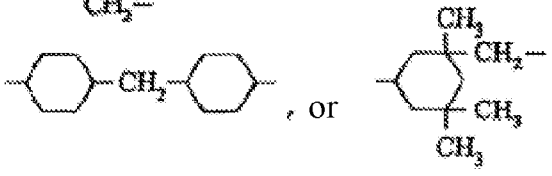


and

where, R denotes







, or

In another preferred embodiment, the long chain alkyl alcohol includes: tridecanol, tetradecanol, hexadecyl alcohol, stearyl alcohol, behenyl alcohol, polyoxyethylene monostearate, polyoxyethylene hexadecyl ether, polyoxyethylene stearyl ether and glycerol monostearate.

In another preferred embodiment, n = 2-5.

In another preferred embodiment, the modified polyurethane coating has a thickness ranging from 20 microns to 100 microns.

In another preferred embodiment, a glass transition temperature Tg of the modified polyurethane coating ranges from 30 ^{o}C to 50 ^{o}C.

In another preferred embodiment, at an ambient temperature, the modified polyurethane coating has a storage modulus ranging from 20 MPa to 100 Mpa.

In another preferred embodiment, the functional layer includes a metal layer or a non-metal layer.

In another preferred embodiment, the functional layer is a low radiation layer including one to three silver layers.

Accordingly, the present disclosure has following advantages:

In comparison with existing silver-coated glass laminate structures, the glass laminate structure of the present disclosure includes a functional layer whose surface is covered with a modified polyurethane coating, which makes the glass laminate structure of the present disclosure have better crack resistance and oxidization resistance performances.

BRIEF DESCRIPTION OF THE DRAWINGS

Figure 1 is a schematic diagram of a structure of a glass laminate structure according to an embodiment of the present disclosure.

DETAILED DESCRIPTION OF EMBODIMENTS

Laminate Structure

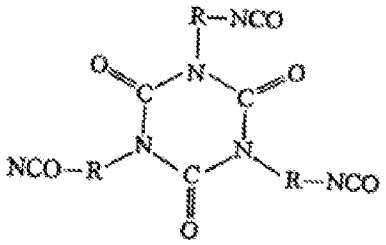
An embodiment of the present disclosure provides a laminate structure including: a glass substrate, a functional layer disposed on a surface of the glass substrate, and a modified polyurethane coating disposed on a surface of the functional layer.

Modified Polyurethane Coating

In the laminate structure according to an embodiment of the present disclosure, raw materials of the modified polyurethane coating include following main ingredients:

(A) Isocyanate

Each molecule of the isocyanate has three or more isocyanate groups, wherein the isocyanate groups include, but not limited to,



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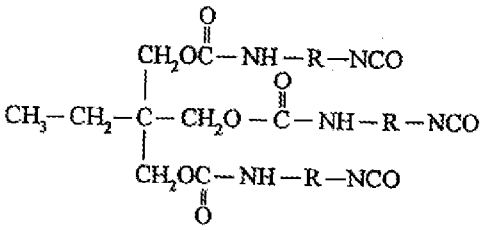

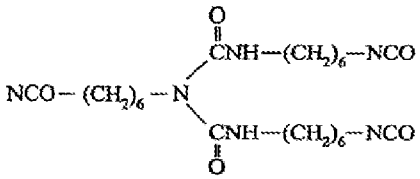

and

wherein, R denotes

,

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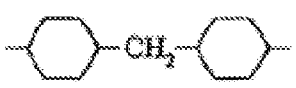

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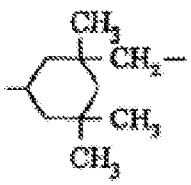

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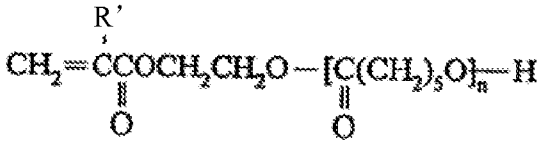
(B) Long Chain Alkyl Alcohol

A number of carbon atoms included in the long chain alkyl alcohol ranges from 10 to 25. The long chain alkyl alcohol includes, but not limited to, tridecanol, tetradecanol, hexadecyl alcohol, stearyl alcohol, behenyl alcohol, polyoxyethylene monostearate, polyoxyethylene hexadecyl ether, polyoxyethylene stearyl ether and glycerol monostearate.

(C) Polycaprolactone-modified (meth) Hydroxy Ethyl Acrylate

The polycaprolactone-modified (meth) hydroxy ethyl acrylate has a formula as follow:



where, R' denotes methyl or H; and n=1-25, optionally, n=2-5;

wherein, by mole, hydroxyl in the long chain alkyl alcohol (B) : the isocyanate groups in the isocyanate (A) : hydroxy in the polycaprolactone-modified (meth) hydroxy ethyl acrylate (C) = 1 : (3-50) : (2-60).

Additives

The additives include:

0.01 to 5% by weight of an anti-scratch agent, wherein the anti-scratch agent includes, but not limited to, organic beads and inorganic beads, and the organic beads include, but not limited to, polymethyl methacrylate, polyurethane, nylon, and rubber;

0.01 to 1% by weight of a catalyst, wherein the catalyst is optionally 0.1 to 0.5 % by weight, and the catalyst includes, but not limit to, dibutyltin dilaurate and dibutyl tin diethyl n-hexanoate;

0.01 to 1% by weight of an antistatic agent, wherein the antistatic agent includes, but not limited to, an antistatic agent containing lithium, sodium or potassium salt, a cationic antistatic agent containing amine salt or quaternary ammonium salt, and a nonpolar antistatic agent containing C8 to C12 alkyl or alkaryl;

0.01 to 1% by weight of a polymerization inhibitor, wherein the polymerization inhibitor includes, but not limited to, hydroquinone monomethyl ether; and

a solvent serving as a balance, wherein the solvent includes, but not limited to, xylene, ketone and ester, the ketone includes, but not limited to, acetone, methyl ethyl ketone, methyl isobutyl ketone and cyclohexanone, and the ester includes, but not limited to, ethyl acetate, propyl acetate, isobutyl acetate and butyl acetate;

wherein the main ingredients of the polyurethane coating layer are 100% by weight.

In the laminate structure provided by an embodiment of the present disclosure, the modified polyurethane coating has a thickness ranging from 20 microns to 100 microns.

In the laminate structure provided by an embodiment of the present disclosure, a glass transition temperature Tg of the modified polyurethane coating ranges from 30 ^{o}C to 50 ^{o}C.

In the laminate structure provided by an embodiment of the present disclosure, at an ambient temperature, the modified polyurethane coating has a storage modulus ranging from 20 MPa to 100 Mpa.

Functional Layer

In the laminate structure of the present disclosure, the functional layer includes, but not limited to, a metal layer and a non-metal layer.

In the laminate structure of the present disclosure, metal included in

the metal layer includes, but not limited to, silver and NiCr.

In the laminate structure of the present disclosure, non-metal included in the non-metal layer includes, but not limited to, $SiZrN_x$ (where $SiZrN_x$ refers to a doped silicon/zirconium nitride), ZnO, $SnZnO_x$ (where $SnZnO_x$ refers to a doped tin/zinc oxide) and Si_3N_4.

In the laminate structure of the present disclosure, the functional layer is a low radiation layer (i.e., a value of $g \leq 60\%$, where g denotes a total transmission sum), and the low radiation layer includes one to three silver layers.

The present disclosure will be illustrated as follow with reference to the drawing.

Figure 1 is a schematic diagram of a structure of a glass laminate structure according to an embodiment of the present disclosure. As shown in Figure 1, the glass laminate structure includes: a glass substrate 100, a functional layer 110 disposed on a surface of the glass substrate 100, and a modified polyurethane coating 120 disposed on a surface of the functional layer 110.

Embodiments

The invention will be further illustrated with reference to specific embodiments. The following embodiments are provided to illustrate and describe the present disclosure and but not to limit scope of the disclosure. In the following embodiments, test methods which are not given a specific condition are implemented according to conventional conditions or in accordance with conditions recommended by the manufacturer. All percentages and parts are measured by weight unless otherwise indicated.

Embodiment 1: preparing a glass laminate structure

The preparing process includes following steps:

(1) Preparing a modified polyurethane (where, by mole, hydroxyl in the long chain alkyl alcohol (B) : the isocyanate groups in the isocyanate (A) : hydroxy in the polycaprolactone-modified (meth) hydroxy ethyl acrylate (C) = 1: 3: 2).

Specifically, 60 parts of toluene and 10 parts of stearyl alcohol are added to a reaction kettle and stirred at 40°C until the stearyl alcohol is completely dissolved. Thereafter, 37 parts of 1,6 hexamethylene diisocyanate (molecular weight is 168) are added, heated to 70°C, and stirred for 30 minutes. Thereafter, 0.02 parts of dibutyltin laurate are added and continuously stirred for 3 hours at an unchanged temperature. Thereafter, 25 parts of polycaprolactone modified hydroxyethyl acrylate (molecular weight is 344), 0.02 parts of dibutyltin laurate and 0.02 parts of hydroquinone monomethyl ether are added and stirred for 3 hours until the reaction is completed. Then, 60 parts of toluene and 5 parts of PMMA organic beads are added and dispersed uniformly to obtain the modified polyurethane coating.

(2) Preparing a glass laminate structure

1) Depositing a low radiation functional coating (e.g., Saint-Gobain glass Kappa coating) on a plate glass by means of magnetron sputtering;

2) Cutting, edging, printing and bending the plate glass having the functional coating, so as to form desired automotive glass size and type;

3) Uniformly and densely spraying the above-obtained polyurethane coating on a surface of the functional coating and

4) Curing at 80 °C for 40 minutes.

Comparative Example 1

The preparing process includes following steps:

(1) Preparing a glass laminate structure

1) Depositing a low radiation functional coating (e.g., Saint-Gobain glass Kappa coating) on a plate glass by means of magnetron sputtering; and

2) Cutting, edging, printing and bending the plate glass having the functional coating, so as to form desired automotive glass size and type.

Performance Test

1) The prepared sample is placed in a laboratory condition at 23 ^{o}C and 50% relative humidity for 168 hours.

2) The prepared sample is wrapped with wet cotton (relative humidity is approximately 100%), placed in an aging box, stays at 70 ^{o}C for 168 hours, and stays at minus 20 ^{o}C for 2 hours.

Test Result

1) Appearance of the sample prepared according to embodiments of the present disclosure did not change, and spotted rust was observed on the sample prepared according to comparative example 1.

2) Appearance of the sample prepared according to embodiments of the present disclosure did not change, and large area of rust was observed on the sample prepared according to comparative example 1.

It should be noted that the above described embodiments are given for describing rather than limiting the present disclosure, and it is to be understood by those skilled in the art that modifications and variations may be made to the disclosure without departing from the spirit and scope of the present disclosure. Such modifications and variations are considered to be within the scope of the present disclosure and the appended claims.

All documents mentioned in the present disclosure are hereby incorporated by reference as if each individual document was individually incorporated by reference. Furthermore, it is to be understood by those skilled in the art that, various changes and modifications can be made upon a reading of the foregoing teachings of the present disclosure, which also fall into the scope of the claims appended hereto.

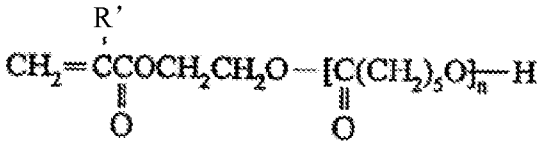
CLAIMS

1. A laminate structure, comprising: a glass substrate (100), a functional layer (110) disposed on a surface of the glass substrate (100), and a modified polyurethane coating (120) disposed on a surface of the functional layer (110), wherein raw materials of the modified polyurethane coating comprise following main ingredients:

(A) isocyanate, wherein each molecule of the isocyanate has three or more isocyanate groups;

(B) long chain alkyl alcohol, wherein a number of carbon atoms comprised in the long chain alkyl alcohol ranges from 10 to 25; and

(C) polycaprolactone-modified (meth) hydroxy ethyl acrylate having a formula as follow:



where, R' denotes $-CH_3$ or $-H$; and n=1-25, and

wherein, by mole, hydroxyl in the long chain alkyl alcohol (B) : the isocyanate groups in the isocyanate (A) : hydroxy in the polycaprolactone-modified (meth) hydroxy ethyl acrylate (C) = 1 : (3-50) : (2-60).

2. The laminate structure according to claim 1, wherein the raw materials of the modified polyurethane coating further comprise: 0.01 to 5% by weight of an anti-scratch agent, 0.01 to 1% by weight of a catalyst, 0.01 to 1% by weight of an antistatic agent, 0.01 to 1% by weight of a polymerization inhibitor, and a solvent serving as a balance, where the main ingredients of the modified polyurethane coating are 100% by weight.

3. The laminate structure according to claim 2, wherein the anti-scratch

agent comprises: organic beads and inorganic beads.

4. The laminate structure according to claim 3, wherein the organic beads comprise: polymethyl methacrylate, polyurethane, nylon and rubber.

5. The laminate structure according to claim 2, wherein the catalyst is 0.1 to 0.5 % by weight; and the catalyst comprises: dibutyltin dilaurate and dibutyl tin diethyl n-hexanoate.

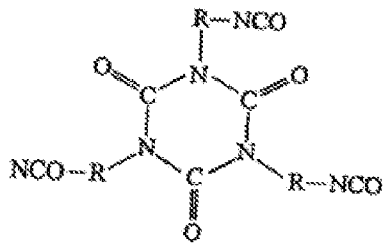
6. The laminate structure according to claim 2, wherein the antistatic agent comprises: an antistatic agent containing lithium, sodium or potassium salt, a cationic antistatic agent containing amine salt or quaternary ammonium salt, and a nonpolar antistatic agent containing C8 to C12 alkyl or alkaryl.

7. The laminate structure according to claim 2, wherein the polymerization inhibitor comprises: hydroquinone monomethyl ether.

8. The laminate structure according to claim 2, wherein the solvent comprises: xylene, ketone and ester.

9. The laminate structure according to claim 8, wherein the ketone comprises: acetone, methyl ethyl ketone, methyl isobutyl ketone and cyclohexanone; and the ester comprises: ethyl acetate, propyl acetate, isobutyl acetate and butyl acetate.

10. The laminate structure according to claim 1, wherein the isocyanate comprises:



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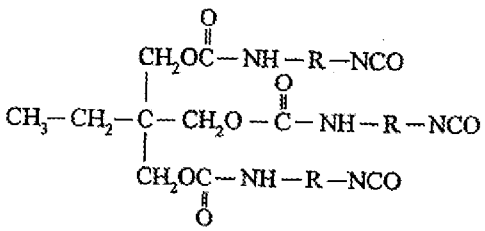

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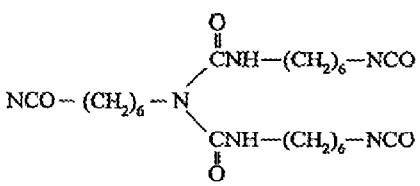
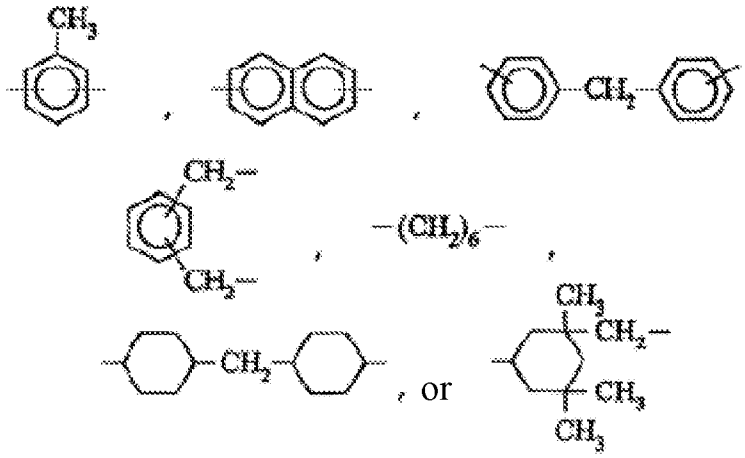


where, R denotes



11. The laminate structure according to claim 1, wherein the long chain alkyl alcohol comprises: tridecanol, tetradecanol, hexadecyl alcohol, stearyl alcohol, behenyl alcohol, polyoxyethylene monostearate, polyoxyethylene hexadecyl ether, polyoxyethylene stearyl ether and glycerol monostearate.

12. The laminate structure according to claim 1, wherein n = 2-5.

13. The laminate structure according to any one of claims 1 to 12, wherein the modified polyurethane coating has a thickness ranging from 20 microns to 100 microns.

14. The laminate structure according to any one of claims 1 to 12, wherein a glass transition temperature Tg of the modified polyurethane coating ranges from 30 °C to 50 °C.

15. The laminate structure according to any one of claims 1 to 12, wherein at an ambient temperature, the modified polyurethane coating has a storage modulus ranging from 20 MPa to 100 Mpa.

16. The laminate structure according to claim 1, wherein the functional layer comprises a metal layer or a non-metal layer.

17. The laminate structure according to claim 16, wherein the functional layer is a low radiation layer comprising one to three silver layers.

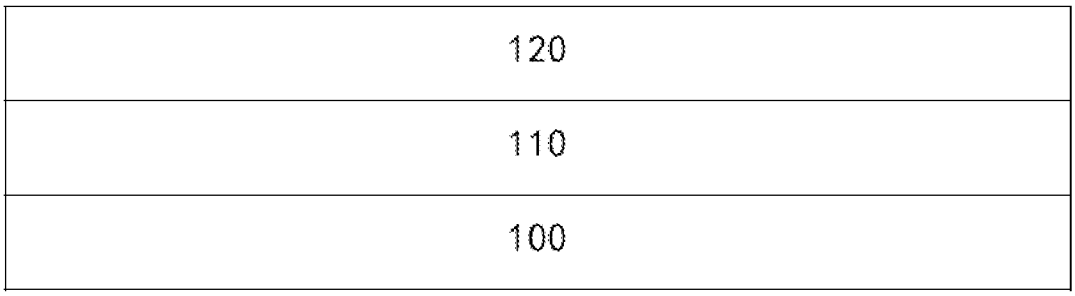


Figure 1

INTERNATIONAL SEARCH REPORT

International application No.
PCT/CN2017/074406

A. CLASSIFICATION OF SUBJECT MATTER

B32B 17/10(2006.01)i; C09D 175/16(2006.01)i

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

B32B; C09D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

CNPAT,CNKI,WPI:laminate,glass,substrate,layer,functional,polyurethane,isocyanate,alcohol,alkyl,caprolactone,acrylate

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	CN 1406259 A (NATOCO CO LTD) 26 March 2003 (2003-03-26) description, page 2 line 6 to page 11 line 28	1-17
X	CN 101445585 A (BAYER MATERIALSCIENCE LLC) 03 June 2009 (2009-06-03) description, page 2 line 16 to page 11 line 23	1-17
A	CN 105431391 A (SAINT-GOBAIN GLASS FRANCE) 23 March 2016 (2016-03-23) the whole document	1-17
A	CN 1576886 A (MITSUI CHEM INC) 09 February 2005 (2005-02-09) the whole document	1-17
A	US 2011261443 A1 (MITSUBISHI CHEM CORP) 27 October 2011 (2011-10-27) the whole document	1-17
A	TW 201542602 A (NIPPON SYNTHETIC CHEM IND CO LTD) 16 November 2015 (2015-11-16) the whole document	1-17

☐ Further documents are listed in the continuation of Box C.

☑ See patent family annex.

* Special categories of cited documents:

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier application or patent but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
03 May 2017	**19 May 2017**

Name and mailing address of the ISA/CN	Authorized officer
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INTERNATIONAL SEARCH REPORT
Information on patent family members

International application No.
PCT/CN2017/074406

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