AUSTRALIA 651496 P/00/001
Section 29

Patents Act 1990

PATENT REQUEST: CONVENTION PATENT

We, being the person identified below as the Applicant, request the grant of a patent to the person identified below as the Nominated Person, for an invention described in the accompanying standard complete specification

Full application details follow:-

Applicant:	FLACHGLAS AKTIENGESELLSCHAFT
Address:	Otto-Seeling-Promenade 10-14, D-8510 Furth 1, Germany
Nominated Person:	FLACHGLAS AKTIENGESELLSCHAFT
Address:	Otto-Seeling-Promenade 10-14, D-8510 Furth 1, Germany
Invention Title:	PROCESS FOR THE MANUFACTURE OF A LAMINATED GLASS PANE WITH SINGLE OR DOUBLE CURVATURE, IN PARTICULAR FOR MOTOR VEHICLES
Name(s) of actual Inventor(s):	Gerhard Tunker
Address for service in Australia:	CALLINAN LAWRIE, 278 High Street, Kew 3101, Victoria, Australia
Attorney Code:	CL

Convention Details

Basic Applicant	Application Number	Country	Country Code	Date of Application
FLACHGLAS AKTIENGESELLSCHAFT	P4132 652.0-45	Germany	DE	1 October 1991

D A T E D this 14th day of April, 1994.

FLACHGLAS AKTIENGESELLSCHAFT
By their Patent Attorneys:
CALLINAN LAWRIE

CALLINAN LAWRIE
278 High Street, Kew
Victoria 3101, Australia
Facsimile (613) 853.0062

This is a Multiple Use Form Covering:
Convention
Non-Convention
PCT National Phase

NOTICE OF ENTITLEMENT

Insert name of applicant

We, FLACHGLAS AKTIENGESELLSCHAFT

of, Otto-Seeling-Promenade 10-14, D-8510 Furth 1, Germany

being the applicant in respect of the Application No. 25391/92 state the following:-

Complete only for non-PCT applications

The person(s) nominated for the grant of the patent:

~~(i) is/are the actual inventor(s)~~

(i) has entitlement from the actual inventor(s) by virtue of being a person who would, if a patent was to be granted on an application made by the actual inventor, be entitled to have the patent assigned to it; and

(ii) is/~~are~~ the applicant(s) of the basic application(s)

~~(ii) has entitlement from the applicant(s) of the basic application(s) listed on the patent request form by virtue of~~

Complete only for PCT applications

~~The person(s) nominated for the grant of the patent:~~

~~(i) is/are the applicant(s) of the application(s) listed in the declaration under Article 8 of the PCT~~

~~(i) has entitlement from the inventor(s) and/or the applicant(s) of the application(s) listed in the declaration under Article 8 of the PCT by virtue of~~

Complete for all Convention cases

The basic application(s) listed:

(i) on the request form

~~(i) in the declaration made under Article 8 of the PCT~~

is/~~are~~ the first application(s) made in a Convention country in respect of the invention.

.....
Jeffrey P. Winch
Registered Patent Attorney

14 April 1994
.....
Date

AU9225391

(12) PATENT ABRIDGMENT (11) Document No. AU-B-25391/92
(19) AUSTRALIAN PATENT OFFICE (10) Acceptance No. 651496

(54) Title
PROCESS FOR THE MANUFACTURE OF A LAMINATED GLASS PANE WITH SINGLE OR DOUBLE CURVATURE, IN PARTICULAR FOR MOTOR VEHICLES

International Patent Classification(s)
(51)[5] **B32B 031/12** **B32B 017/10**

(21) Application No. : **25391/92** (22) Application Date : **28.09.92**

(30) Priority Data

(31) Number	(32) Date	(33) Country
4132652	**01.10.91**	**DE GERMANY**

(43) Publication Date : **08.04.93**

(44) Publication Date of Accepted Application : **21.07.94**

(71) Applicant(s)
FLACHGLAS AKTIENGESELLSCHAFT

(72) Inventor(s)
GERHARD TUNKER

(74) Attorney or Agent
CALLINAN LAWRIE , Private Bag 7, KEW VIC 3101

(56) Prior Art Documents
AU 511247 19294/76
AU 495954 18706/76

(57) Claim

1. A process for the manufacture of a laminated glass pane with single or double curvature, which consists of a glass inner pane, a glass outer pane and a plastics material sheet interlayer and incorporates on said plastics material sheet side of at least one of said panes, a coating applied by a screen-printing process, wherein a screen-printable enamel ink is used which consists of glass paste, at least a ceramic pigment and an inorganic vehicle and is prepared with an organic screen-printing oil; said coating is applied with said enamel ink to the appropriate surface of said pane with the aid of a silk-screen printing process; said coating is dried at a temperature at which said screen-printing oil is volatile, said screen-printing oil evaporating in the process; the two panes with the dried coating are placed on top of one another, heated to deformation temperature and are bent as a pair according to the single or double curvature required, the coating being baked on simultaneously in the process; and the curved panes of the pair are removed from one another, the plastics material sheet placed in position and the panes of the pair are again placed on top of one another with the plastics material sheet interposed, heated and combined to form the laminated glass pane.

P/00/011
Regulation 3.2

AUSTRALIA

651496

PATENTS ACT 1990

COMPLETE SPECIFICATION

FOR A STANDARD PATENT

ORIGINAL

TO BE COMPLETED BY APPLICANT

Name of Applicant: FLACHGLAS AKTIENGESELLSCHAFT

Actual Inventor(s): Gerhard Tünker

Address for Service: CALLINAN LAWRIE, 278 High Street, Kew, 3101, Victoria, Australia

Invention Title: "PROCESS FOR THE MANUFACTURE OF A LAMINATED GLASS PANE WITH SINGLE OR DOUBLE CURVATURE, IN PARTICULAR FOR MOTOR VEHICLES"

The following statement is a full description of this invention, including the best method of performing it known to me:-

Specification

The invention concerns a process for the manufacture of a laminated glass pane with single or double curvature, in particular for motor vehicles, which consists of a glass inner pane, a glass outer pane and a plastic sheet interlayer and incorporates on the plastic sheet side of at least one of the panes (in the case of motor vehicles on the plastic sheet side of the outer pane) a coating applied by the screen-printing process, in particular edge coating. The coating can in the case of such laminated glass panes fulfill a wide variety of functions. In the case of motor vehicles, it usually takes the form of edge coating. As edge coating of this nature, it is used for the obscuration of other components of the motor vehicle. It can also take the form of a printed antenna and/or printed heating element conductors. Exacting requirements are imposed on the coating. It must satisfactorily outlast the statistical service life of a printed laminated glass pane. It may neither discolour, nor may it become detached from the glass pane involved. The coating itself must be clearly defined, provide uniform cover, be free from blisters and present an overall favourable impression. A printed coating consisting of an enamel ink would meet the requirements, but requires an elaborate process for the manufacture of the laminated glass panes with single or double curvature as a whole.

In the manufacture of laminated glass panes with single or double curvature, the following basic procedure is known: the two panes which should be to form the laminated glass pane are placed on top of one another, heated to deformation temperature and are bent as a pair to form the single or double curvature. The curved panes of the pair are removed from one another, the plastic sheet placed in position and the panes of the pair are again placed on top of one another with the plastic sheet interposed, heated and combined to form the laminated glass pane. This has proved satisfactory and, on account of the formation of the pair and their curvature, leads to the two panes possessing the same curvature, apart from negligible tolerances, which is important to prevent optical distortion. This tried and tested procedure cannot be readily carried out, if the coating is applied by the silk-screen printing process using enamel ink and should meet the requirements. The procedure is different:

In the case of the process known in practice, on which the invention is based, a normal enamel ink is used for applying the coating to a glass pane. This enamel ink which incorporates an organic vehicle, e.g. ethyl cellulose, is applied to the flat, uncurved glass pane to which the coating is to be applied and the coating is baked onto the flat glass pane, that is to say at baking temperatures of around 555°C, as is usual. Therefore, pre-baking takes place. Only afterwards are the two panes with the baked-on coating placed on top of one another, heated to deformation temperature and bent as a pair to the required single or double curvature. Then the process continues as described above. The interposed stage of the process described above, where the flat glass pane has a coating of enamel ink applied to it and the coating is baked onto the flat glass pane, is costly and not readily integratable in existing production lines for laminated glass panes with single or double curvature. In addition, it is not possible when baking the coating on to prevent the planeness tolerance of the glass pane involved suffering deterioration, which can lead to optical faults. If an attempt is made to dispense with the pre-baking described above and if, in conjunction with this, the normal enamel ink is applied so as to heat the glass panes to deformation temperature after mating and to bend them as a pair according to the single of double curvature required, baking of the enamel ink onto the glass pane taking place simultaneously, the adhesion of the enamel ink to the glass pane will not be adequate and undesirable blistering will be observed. This is also true if special, costly screen-printing oils are used.

The purpose of the invention is to specify a process for manufacture of a laminated glass pane of the construction described with single or double curvature at the beginning, in particular for motor vehicles, which makes it possible to produce a durable coating of enamel ink while dispensing with the pre-baking described.

To solve this problem, the subject of the invention is a process for manufacture of laminated glass pane with single or double curvature, in particular for motor vehicles, which consists of an inner pane of glass, an outer pane of glass and a plastic sheet interlayer, and on the plastic sheet side of at least one of the panes, in the case of motor vehicle windows on the plastic sheet

side of the outer pane, incorporates a coating applied by the silk-screen printing process, in particular edge coating, - with the following measures and process stages:

(a) A screen-printable enamel ink is used which consists of glass paste, at least a ceramic pigment and an inorganic vehicle and is prepared with an organic screen-printing oil,

(b) the coating is applied with the enamel ink in accordance with (a) to the appropriate surface of the pane with the aid of the silk-screen printing process,

(c) the coating is dried at a temperature at which the screen-printing oil is volatile, the screen-printing oil evaporating in the process,

(d) the two panes with the dried coating are placed on top of one another, heated to deformation temperature and are bent as a pair according to the single or double curvature required, the coating being baked on simultaneously in the process,

(e) the curved panes of the pair are removed from one another, the plastic sheet placed in position and the panes of the pair are again placed on top of one another with the plastic sheet interposed, heated and combined to form the laminated glass pane. The process according to the invention can be integrated without difficulty in existing production lines for laminated glass panes. The drying of the coating can be carried out continuously along a path of the production line which the printed glass pane has to pass anyway. The drying can be carried out with infrared radiation. Surprisingly, the coating adheres adequately after drying, so that it is possible to proceed in accordance with step (d) as outlined above. Although the only dried coating is encapsulated in a practically airtight manner during the process of deformation and the associated process of baking on, no fault occurs in the coating due

to nascent gases, running, or the like. The quality of the baked-on coating meets all requirements, that is to say even in long-term behaviour. In detail, there are within the scope of the invention several alternatives for development of the process. Thus, according to a preferred embodiment of the invention, at least a silica gel can be employed as an inorganic vehicle of the enamel ink used. The expression silica gel is used here in its most general sense. It also includes modifications . Preferably the gel will be selected from the group consisting of tetramethylorthosilicate, tetraethylorthsilicate, tetra-n-propylsilicate, tetramethylglycolsilicate, tetraethylglycolsilicate, ethylpolysilicate, silica ester vehicle, ethyl-glycol-free silica vehicle, modified silica vehicle, modified silica ester, butyglycolsilicate or mixtures thereof. An especially suitable inorganic vehicle, in particular one of the group specified above, can easily be established by trial and error and matched to the remaining composition of the enamel ink, and also to the composition of the glass of the pane.

It is advisable at all times according to the preferred embodiment of the invention to add at least an alcoholate to the vehicle. In this respect, it is preferred to work with an alcoholate from the group consisting of aluminium, titanium, zirconium, boron or mixtures thereof. If, contrary to expectations, gases still occur during baking on, their undesirable effect can be prevented by additionally adding an inorganic source of oxygen to the inorganic vehicle. Preferably, the inorganic source of oxygen will be selected from the group consisting of lead oxide, minium, manganese oxide or mixtures thereof. The appropriate quantity to be added can be established by trial and error.

Within the scope of the invention, it is possible to use all usual screen-printing oils. It has been found advisable to prepare the enamel ink with terpineol as screen-printing oil. It is also possible however to employ glycol or mixtures of terpineol or glycol as screen-printing oil.

The enamel ink which is used according to the invention has, apart from the special features stated, a composition usual for such enamel inks, e.g. with 40 to 80 weight %, preferably approximately 50 weight %, of glass paste and approx. 20 to 60 weight %, preferably 30 weight %, of a ceramic pigment. The deformation temperature for carrying out stage (d) of the process can be adjusted without difficulty so that baking on also takes place, or vice versa. According to a preferred embodiment of the invention, it is ensured that the drying according to stage (c) of the process is carried out at a temperature of below 300 °C, which can easily be achieved with the aid of infrared drying.

The enamel ink used within the scope of the invention is also in itself the subject of the invention.

Example

For manufacture of a laminated glass pane with single curvature in the form of a windscreen of a motor vehicle with printed edge coating, it was proceeded as follows for test purposes:

1) Using a commercially available screen powder pigment for laminated glass, three screen-printable enamel inks were prepared in paste form with different screen-printing oils, that is to say in accordance with the following Table:

Proportions in weight %	A	B	C
Glass paste (lead borosilicate)	50	50	50
Pigment (copper chrome spinel)	28	28	28
Terpineol	18	22	18
Ethyl cellulose	4	-	-
Silicate vehicle	-	-	4

2) A 5 cm wide all-round ornamental strip with a layer thickness of 10 - 15 µm was applied to three flat windscreens with the individual enamel inks with the aid of a 77 threads/cm polyester screen.

3) The coating in accordance with 2) was dried with the aid of infrared radiation, a temperature of approx. 250°C being measured on the glass pane in the drying area. Escape of vapour of the screen-printing oils was observed. Drying was carried out until no further evaporation of screen-printing oil was perceptible. After cooling of the panes, the adhesion of the enamel inks was checked by sticking on adhesive strips and pulling the adhesive strips off. Enamel inks A and C adhered well. They could not be removed with the adhesive strips. Enamel ink B evidenced no adhesion and remained almost completely on the adhesive strip.

4) The glass panes prepared in accordance with 3) were each placed together with a second unprinted glass pane, heated in 8 minutes to

a temperature of 620°C, held at this temperature for a period of 1 minute and then bent at this temperature with the usual equipment. After the curved pairs of panes had cooled down, the second pane was lifted off. The quality of the baked-on enamel inks was assessed by visual inspection and by examination for adhesion:

Ink A evidenced inadmissibly pronounced variations of hue, pores and bonding to the mating pane.

Ink B evidenced uniform hue, but inadmissible pores and bonding to the mating pane. The outlines were indistinct.

Ink C evidenced an enamel layer of faultless density, no undesirable pores and no bonding to the mating pane.

5) The treated glass panes bent in accordance with 4) were combined as usual to form a laminated glass pane and their long-term behaviour was examined by simulation.

The examination results have been recorded in the following Table:

Ink	A	B	C
Storage in water bath 6 weeks 20°C	negative	negative	positive
Storage at 100°C/1 h in air	negative	negative	positive
Storage at 100°C/1 h in water bath	negative	negative	positive

In the case of Inks A and B, inadmissible defects were found in the plastic sheet interlayer after the tests for long-term behaviour, e.g. dampening and blisters. The panes to which a coating had been applied with Ink C were free from defects after the tests.

The claims defining the invention are as follows:

1. A process for the manufacture of a laminated glass pane with single or double curvature, which consists of a glass inner pane, a glass outer pane and a plastics material sheet interlayer and incorporates on said plastics material sheet side of at least one of said panes, a coating applied by a screen-printing process, wherein a screen-printable enamel ink is used which consists of glass paste, at least a ceramic pigment and an inorganic vehicle and is prepared with an organic screen-printing oil; said coating is applied with said enamel ink to the appropriate surface of said pane with the aid of a silk-screen printing process; said coating is dried at a temperature at which said screen-printing oil is volatile, said screen-printing oil evaporating in the process; the two panes with the dried coating are placed on top of one another, heated to deformation temperature and are bent as a pair according to the single or double curvature required, the coating being baked on simultaneously in the process; and the curved panes of the pair are removed from one another, the plastics material sheet placed in position and the panes of the pair are again placed on top of one another with the plastics material sheet interposed, heated and combined to form the laminated glass pane.

2. The process in accordance with claim 1, wherein at least a silica gel is employed as inorganic vehicle for said enamel ink used.

3. The process in accordance with claim 1 or claim 2, wherein a commercially available silica gel vehicle selected from the group consisting of tetramethylorthosilicate, tetraethylorthosilicate, tetra-n-propylsilicate, tetramethylglycolsilicate, tetraethylglycolsilicate, ethylpolysilicate, silica ester vehicle, ethylglycol-free silica vehicle, modified silica vehicle, modified silica ester, ~~butyglycolsilicate~~ butylglycolsilicate or mixtures thereof are used.

4. The process in accordance with claim 2 or claim 3, wherein at least an alcoholate is added to said inorganic vehicle.

5. The process in accordance with claim 4, wherein an alcoholate selected from the group consisting of aluminium, titanium, zirconium, boron, or mixtures

thereof, is added.

6. The process in accordance with any one of claims 2 to 6, wherein an inorganic source of oxygen is added to the inorganic vehicle.

7. The process in accordance with claim 6, where an inorganic source of oxygen selected from the group lead dioxide, minium, manganese oxide, or mixtures thereof, is added.

8. The process in accordance with any one of claims 1 to 7, wherein said enamel ink is prepared with terpineol as screen-printing oil.

9. The process in accordance with any one of claims 1 to 7, wherein said enamel ink is prepared with glycol as screen-printing oil.

10. The process in accordance with any one of claims 1 to 9, wherein drying is carried out at a temperature of below 300°C.

11. The process in accordance with any one of the preceding claims, for the manufacture of glass panes for motor vehicles.

12. The process as claimed in any one of the preceding claims, wherein said coating is on the plastics material sheet side of said outer pane.

13. A process for the manufacture of a laminated glass pane with single or double curvature, as claimed in claim 1 and using a screen-printable enamel ink, substantially as described herein with reference to the Example.

14. A laminated glass pane, when prepared by the process as claimed in any one of the preceding claims.

DATED this 25th day of May 1994.

FLACHGLAS AKTIENGESELLSCHAFT
By their Patent Attorneys:
CALLINAN LAWRIE