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(54) PROCESS FOR PRODUCING A LAMINATE

(71) We, SUMITOMO BAKELITE COMPANY LIMITED, a corporation organized under the laws of Japan, of 2—2, Uchisaiwaicho-1-chome, Chiyoda-ku, Tokyo, Japan, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention relates to a process for producing a laminate, and more particularly to an improvement for producing a laminate comprising a metal support and a cured thermosetting resin layer, having a glossy or mat surface.

To make a laminate from a thermosetting resin-impregnated sheet and a metal plate or foil there have usually been used two procedures, that is, interposing an adhesive paper impregnated with a solution of a mixture of a thermoplastic resin and the thermosetting resin between the sheet and the plate or foil, or coating the solution on the plate or foil and then placing the sheet thereon, and thereafter heating the assembly in both cases under pressure between two caul plates or rollers of a continuous processing machine to cause integration of the components.

The thus obtained laminate has the disadvantage that it tends to become easily delaminated. The delaminating phenomenon is caused by the break of adhesion occurring at edges where the laminate is subjected to such processings as cutting, punching and holing because the resin is too rigid to tolerate the concentration of shear stress or caused by heat-stress due to the difference of heat-expansion between metal and resin.

The laminate formed usually has a surface as glossy as a mirror. However, if a laminate having a mat surface is needed, it is necessary according to conventional procedure either to use finely mat finished caul plates

or rollers, or to grind the surface of the laminate so as to give it a mat finishing which uniformly scatters the reflected light. In the former method, the caul plate or roller could not be reused once its surface is injured even by a slight scratch. Furthermore, a series of caul plates or rolls are necessary in the production of various mat laminates. Another drawback of the method resides in the careful handling of the caul plate or roller. These methods are rather complicated, troublesome, and inefficient in production and therefore the production cost is increased. In the latter method, there are also drawbacks necessitating the very troublesome procedure of processing one by one and highly delicate skill in mechanical processing for obtaining various, desired surfaces.

An object of the present invention is to provide a process which can produce a laminate having an excellent adhesion which can tolerate the concentration of shear stress.

Another object of the present invention is to provide a process for producing with ease a mat decorative laminate having an optional mat pattern on the surface.

The present invention provides a process for producing a laminate comprising a metal support and a cured thermosetting resin layer by heating a metal plate or foil and a thermosetting resin-impregnated sheet under pressure, which comprises interposing a film of a thermoplastic resin (referred to hereinafter as the "A-film") between the thermosetting resin-impregnated sheet and the metal plate or foil and heating the assembly under pressure to melting of the surface of the film of the thermoplastic resin with a peroxide or a mixture thereof with an organic acid or an anhydride of an organic acid present in the assembly, the peroxide decomposing during the step of heating under pressure, so as to integrate the laminate components.

[Price 25p]

Furthermore, the present invention relates to a process for producing a laminate having a mat surface comprising interposing a first film of thermoplastic resin between a thermosetting resin-impregnated sheet and a metal plate or foil, putting a second film of thermoplastic resin (referred to hereinafter as the "B-film") on the outside of the resin-impregnated sheet, and heating the assembly under pressure to melting of the surface of the said first film with a peroxide or a mixture thereof and an organic acid or an anhydride of an organic acid present between the outer surface of the second film and the metal plate or foil, the peroxide decomposing during the step of heating under pressure, so as to integrate the laminate components.

The term "to integrate" or "integration" used herein means that the thermosetting resin-impregnated sheet and the metal plate or foil are caused to adhere to each other firmly.

The "A-film" used in the present invention includes films of polyethylene, polypropylene, copolymers containing structural units of an olefin, for example, ethylene or propylene, polyvinylchloride and the blends of any of such resins and other thermoplastic resins or a laminated composite film of the films and those of other thermoplastic resins. For the integration, the "A-film" should be subjected to such a condition of heating under pressure that the surface of the film melts and flows. Thus, for instance, when using a condition of heating at a temperature of 120°C. and a pressure of 10 kg/cm² for laminating an unsaturated polyester resin-impregnated paper or cloth containing benzoyl peroxide as a curing agent and steel plate, the object of the present invention would be attained by the use of polyethylene film having a thickness of 40 microns but hardly by the use of polypropylene film.

The peroxides or organic acids or anhydrides used therewith, used in the present invention to activate the A-film, may be, for example, benzoyl peroxide, *p*-chlorobenzoyl peroxide, methyl ethyl ketone peroxide, cyclohexanone peroxide, cumene hydroperoxide, tert.-butyl perbenzoate, di-tert.-butyl perphthalate, and tert.-butyl peracetate as the peroxide, and acrylic acid, methacrylic acid, crotonic acid, isocrotonic acid, maleic acid, fumaric acid, cinnamic acid, benzoic acid, phthalic acid, isophthalic acid, pyromellitic acid, oxalic acid, succinic acid, formic acid, acetic acid, propionic acid, maleic anhydride, itaconic anhydride, cinnamic anhydride, phthalic anhydride, benzoic anhydride, tetrahydrophthalic anhydride, hexahydrophthalic anhydride, succinic anhydride and acetic anhydride and the like as the organic acid and the anhydride. The activator may be used by impregnating in the thermosetting resin-impregnated sheet or coating on the surface of the sheet, the surface of the "A-film" or

that of the metal plate or foil which contacts with the "A-film". When using as the thermosetting resin for the sheet an unsaturated polyester resin or allyl resin such as diallyl phthalate resin, diallyl isophthalate resin, diallyl maleate resin and diallyl chloroendate resin, which already contains the activator, it is not necessary to add further peroxide activator or the mixture of a peroxide and an organic acid or the mixture of a peroxide and an anhydride.

It is surprising in the present invention that despite the generally recognised difficulty of bonding of a polyolefin resin to a metal plate the resin adheres firmly thereto according to the present invention, resulting in a laminate tolerable to the concentration of the shear-stress caused by processing such as cutting, punching and bending. Though the inventors do not exclude the possibility of other theories, it is believed that such a good adhesion is given by the activation of "A-film" caused by the peroxide activator which decomposes during the step of heating under pressure. It is also surprising that the activation is effected even when the activator is originally contained in the resin-impregnated sheet probably due to the permeation of activator through the A-film.

When made according to the method mentioned above, the laminate may have a surface as glossy as a mirror with the use of a conventional caul plate or roller, and an excellent adhesion property tolerable of the concentration of shear stress caused by processing as mentioned probably due in part to the toughness of the thermoplastic resin film used.

According to the present invention, a mat laminate can be made with ease by following the same procedures as mentioned above with exception that a film of thermoplastic resin is put on the outside of the thermosetting resin-impregnated sheet and removed after the procedures.

The "B-film" used in the present invention may be any thermoplastic resin film commercially available, and includes films made of a polyester, polyacetal, polytetrafluoroethylene, polyethylene, polypropylene, polybutadiene, polycarbonate, polyamide, polyvinylchloride, polyvinylidene chloride, polyvinylalcohol, polyvinylacetal, polyacrylate, polystyrene, polyvinylacetate and the like, as well as copolymers consisting of monomers thereof capable of copolymerization.

When the softening point of the "B-film" is much higher than the heating temperature, the glossiness of the laminate is higher than when a "B-film" of a resin having a softening point substantially equal to the heating temperature is used. The softening point of the "B-film" may be slightly below the heating temperature. Thus, a laminate having a desired glossy surface can be obtained by

selecting the "B-film". It goes without saying that the "B-film" may be mat for the purpose.

There are many embodiments in the present invention for producing a mat laminate. A laminate having a desirable pattern on its surface may be produced by selecting either the sort of sheet to be impregnated with resin or the "B-films" to which various uneven patterns have been applied. In the use of such "B-film", the higher the softening point and the less the elongation under heating of the "B-film" are, the better the pattern on the surface of the laminate obtained.

The application of an uneven pattern to the "B-film" may be achieved by a conventional method such as passing the film through a roller for embossing under pressure and, if necessary, heating, or moulding the original resin in a mould having a corresponding pattern.

In another embodiment of the present invention, another article for applying the pattern may be used together with the "B-film". For instance, a laminate having a net-like pattern may be produced by putting a wire net on the outside of the "B-film" and following the laminate formation, removing the wire net. In place of the wire net, cloths of natural or synthetic fibre, asbestos and glass fibre may be used for such purpose.

In a further embodiment, the "B-film" may be integrated with such other things as cloths or paper for producing the laminate having patterns according to the present invention. Such a "B-film" may be produced, for example, by impregnating or applying a varnish of the thermoplastic resin to the paper or cloth, evaporating the solvent of the varnish, if necessary, and applying pressure and heat, if necessary, to the impregnated paper or cloth between two embossed caul plates to form the desired pattern thereon. The "B-film" integrated with the paper or cloth can be used in the present invention.

As seen from the disclosures, it will be desirable that the relation of the temperatures of softening point of the "A-film" (represented by T_1), decomposition temperature of the peroxide (T_2), applied temperature during the heating step (T_3) and softening point of the "B-film" (T_4), satisfy the following formula

$$T_1 \leq T_2 < T_3 \leq T_4$$

wherein the symbols are referred to above.

The method according to the present invention provides both glossy laminates and mat laminates which are suitable for the materials of upholstery, for electric instruments

and as laminate plates for the interior of vehicles and houses, especially for decorative plate.

The present invention will be more clearly illustrated by the following Examples. The parts mentioned therein are by weight.

The cross-cut test referred to therein is explained as follows:

Figure 1 shows a schematic side view of the test apparatus. A test specimen (1) is set on the table of the machine (2) with the layer of the resin-impregnated sheet upward, and given by means of a razor blade (11) straight scratches at 2 mm intervals, each crossing each other at right angles, which reach the metal plate, so as to form 100 squares like a go board. A piece of pressure-sensitive tape (3) is pressed to adhere thereon over a length of 20 mm and the remainder of the tape is also allowed to adhere to a suitable fixing plate (4). The plate is pulled upward at an angle of 45° with the specimen by a load of 500 g connected therewith through a fixed pulley (5) and hanging at a height of 20 cm above the specimen. The number of the squares removed from the metal plate of the specimen is enumerated. The less the number is, the stronger is the adhesive force.

The test of hardness against scratches is explained as follows:

Figure 2 shows a hardness tester resembling that of Martin but having a diamond needle whose vertical angle is 45°. The tester is used under variable loads to scratch the surface of the specimen over a length of 5 cm. The load is counted when the width and length of the scratch conforms to a series of standards as measured on the surface of a slide glass for a microscope passing the test of Japanese Industrial Standard R-3707 in the hardness test by means of the same hardness tester under the load of 420 g.

Other tests for heat resistance, hot water resistance, discoloration resistance and flammability are carried out by Japanese Industrial Standards K-6902, K-6902, K-6902 and K-6744, respectively.

EXAMPLE 1

(A) 93 Parts of diallyl phthalate prepolymer, 7 parts of diallyl phthalate and 2 parts of benzoyl peroxide are dissolved in 150 parts of acetone. A sheet of diallyl phthalate resin-impregnated paper was prepared by impregnating with the solution a sheet of printed decorative paper having 80 g/m² as basis weight and drying. A polyethylene film having a thickness of 50 microns was placed on a corrosion-resistively alloyed aluminium plate having a thickness of 0.8 mm, and the resin-impregnated paper was put on the polyethylene film. The assembly was heated at a temperature of 125°C under a pressure of 12 kg/cm² between two caul plates for 10 minutes and allowed to cool.

A diallyl phthalate resin-decorative laminate supported by the corrosion-resistively alloyed aluminium plate was thus produced. The properties of the decorative plate obtained are shown in the following Table I.

From the Table, the heat-resistance, hot water-resistance, and decoloration-resistance as well as adhesion after punching of the decorative laminate are excellent.

TABLE I

Properties	Test method	Result
Heat-resistance	JIS K—6902	No change
Hot water-resistance	JIS K—6902	No change
Discoloration resistance	JIS K—6902	No change
Flammability	JIS K—6744	Extinguished promptly
Punching	By press for punching	Good
Adherence	Cross-cutting test	No peeling

(B) The procedure as described in the above Example (A) were repeated with the exception that a polyethylene film was put on the outside of the resin-impregnated paper and removed after the completion of the procedures. A mat, decorative laminate of diallyl phthalate resin supported by corrosion-resistively alloyed aluminium plate, was thus produced.

EXAMPLE 2

(A) 75 Parts of an unsaturated polyester resin having an acid number of 20 prepared from phthalic anhydride, maleic anhydride and diethylene glycol by a conventional method, 25 parts of diallyl phthalate and 3 parts of a solution containing methyl ethyl ketone peroxide in an amount of 50 per cent by weight are mixed in the presence of 50 parts of acetone with stirring. The thus obtained mixture was applied to a non-woven cloth weigh-

ing 30 g/m² as basis weight of acrylic synthetic fibre which had been printed, and the sheet so impregnated with unsaturated polyester resin had a 60 per cent resin content.

An unsaturated polyester resin-laminated glossy laminate supported by a steel plate was produced by putting as the "A-film" a film having a thickness of 50 microns of ethylene-vinyl acetate copolymer containing the latter monomer in an amount of 4 mole per cent by weight, upon a steel plate having a thickness of 0.3 mm which has been subjected to degreasing, further placing on the "A-film" the resin-impregnated sheet of non-woven cloth, and heating the thus prepared assembly between two caul plates at a temperature of 140°C. under pressure of 6 kg/cm² for ten minutes and removing after cooling.

The results of the tests for the laminate showed excellent properties, as shown in the following Table II.

TABLE II

Property	Test method	Result
Punching	By press for punching	Good
Bending	Winding around metal pipe	Possible to radius of 2 mm
Heat-resistance	140°C — 5 hours	No change
Adhesion	Cross-cut test	No peeling

(B) The procedures as described in the above Example (A) were repeated with exception that a polyvinyl alcohol film was placed on the outside of the unsaturated polyester-impregnated sheet as the "B-film" and removed after the completion of the procedures. A mat laminate whose properties were the same as those of the laminate obtained in the above Example (A) was produced.

EXAMPLE 3

(A) 90 Parts of diallyl phthalate prepolymer, 10 parts of diallyl phthalate and 3 parts of tert.-butyl perbenzoate were dissolved in 100 parts of methyl ethyl ketone. A paper

containing a pigment was impregnated with the diallyl phthalate resin solution and dried.

A diallyl phthalate resin-decorative laminate was produced by inserting a polypropylene film having a thickness of 25 microns between the resin-impregnated paper and corrosion-resistively alloyed aluminium plate having a thickness of 1.2 mm, integrating the assembly by heating at 160°C under pressure of 20 kg/cm² between two caul plates and cooling. The thus obtained decorative laminate is of excellent heat-resistance and processability as shown in Table III.

TABLE III

Property	Test method	Result
Heat resistance	170°C. — 4 hours	No change
Cutting	By shearing machine	No change
Punching	By press for punching	Good
Drilling	By drills	Good
Scratch hardness	By diamond needle	Load 100 g

(B) The procedures described in the above Example (A) were repeated with the exception that a polyethylene terephthalate film having an uneven pattern thereon was placed on the outside of the resin-impregnated paper and removed after the completion of the procedures. A decorative plate having an uneven pattern on its surface was produced.

EXAMPLE 4

An unsaturated polyester resin having a softening point of 65°C was prepared according to a conventional method by condensing 1,000 g of isophthalic acid, 465 g of maleic anhydride and 800 g of ethylene glycol in an atmosphere of nitrogen.

An unsaturated polyester resin-impregnated paper containing benzoyl peroxide was produced by dissolving 95 parts of the unsaturated

polyester resin, 25 parts of diallyl phthalate and 4 parts of benzoyl peroxide in 120 parts of acetone, and impregnating a pigment-containing paper having 80 g/m² as basis weight with the solution and drying.

An unsaturated resin-laminated steel decorative plate was produced by placing as "A-film" a polyethylene film having a thickness of 20 microns on a steel panel having a thickness of 0.8 mm and having been degreased, further placing on the polyethylene film the resin-impregnated paper, and integrating the thus prepared assembly by heating at a temperature of 125°C under pressure of 10 kg/cm² for 25 minutes between two caul plates and cooling.

As shown in Table IV, the obtained decorative plate has excellent properties.

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TABLE IV

Property	Test method	Result
Heat-resistance	JIS K—6902	No change
Hot water-resistance	JIS K—6902	No change
Boiling water-resistance	4 hours in boiling water	No change
Cutting	Cutting by shearing machine	Good
Punching	By press for punching	Good
Adherence	Cross-cut test	No peeling

COMPARATIVE EXAMPLE

(B) The procedures described in the above Example (A) were repeated except that no polyethylene film was used as the "A-film". The decorative plate obtained shows inferior adhesion in the punching test, showing delamination at the edge of the surface subjected to punching.

EXAMPLE 5

A diallyl phthalate resin-impregnated sheet was prepared by impregnating a printed acrylic type non-woven cloth of 30 g/m² with a solution of 93 parts of diallyl phthalate prepolymer, 7 parts of diallyl phthalate and 2 parts of benzoyl peroxide in 150 parts of acetone, and had a resin content of 70 per cent by weight of the impregnated sheet.

A steel panel having a thickness of 0.3 mm and having been degreased was treated with

a solution of 1 part of benzoyl peroxide, 10 parts of maleic anhydride in a mixture of 15 parts of toluene and 65 parts of acetone and dried to obtain the panel coated with a mixture of benzoyl peroxide and maleic anhydride.

A diallyl phthalate resin-laminated steel decorative plate was produced by placing a rigid polyvinyl chloride film having a thickness of 50 microns as the "A-film" on the treated surface of the panel, further placing thereon the diallyl phthalate resin-impregnated sheet, integrating the assembly by heating at a temperature of 135°C under pressure of 20 kg/cm² for 20 minutes between two caul plates and cooling.

The thus obtained decorative laminate has excellent properties, as shown in the following Table V.

TABLE V

Property	Test method	Result
Punching	By press for punching	Good
Bending	Winding on pipe	Possible to 5 mm diameter
Adherence	Cross-cut test	No peeling

(B) The procedures described in the above Example (A) were repeated with the exception that a polypropylene film was put on the outside of the diallyl phthalate resin-impregnated sheet and removed after completion of the procedures. A mat diallyl phthalate-laminated steel decorative plate was produced.

EXAMPLE 6

A 10 per cent solution of tert.-butyl perbenzoate in acetone was sprayed on the unprinted side of a melamine-impregnated sheet for use as decorative plate to coat the sheet with the peroxide.

A melamine-laminated decorative laminate

was produced by placing a polypropylene film having a thickness of 25 microns on a corrosion-resistively alloyed aluminium panel having a thickness of 1.2 mm and having been applied with a 3% solution of oxalic acid in methanol, further placing the melamine-impregnated sheet on the film, integrating the assembly by heating at a temperature of

165°C under pressure of 120 kg/cm² for 30 minutes and cooling.

The produced decorative plate has excellent properties in scratch hardness, heat-resistance, discolouring resistance, peeling resistance against punching and cutting, as shown in the following Table VI.

TABLE VI

Property	Test method	Result
Scratch hardness	By diamond needle	200g
Heat-resistance	JIS K—6902	No change
Discolouring resistance	JIS K—6902	No change
Punching	By press for punching	Good
Cutting	By shearing machine	Good

COMPARATIVE EXAMPLE

(B) The procedures described in the above Example (A) were repeated with the exception that spraying the tert.-butyl perbenzoate solution was omitted.

The decorative laminate obtained is poor in adhesion between the metal and polypropylene film, as shown by easy peeling off in the cross-cut test.

(C) The procedures described in the above Example (A) were repeated with the exception that a polyethylene terephthalate film having a thickness of 20 microns was placed on the outside of the melamine resin-impregnated sheet and removed after the completion of the procedures.

A unique semi-glossy decorative plate was thus produced.

WHAT WE CLAIM IS:—

1. A process of producing a laminate comprising a metal support and a cured thermosetting resin layer by heating a metal plate or foil and a thermosetting resin-impregnated sheet under pressure, which comprises interposing a film of thermoplastic resin between the thermosetting resin-impregnated sheet and the metal plate or foil, and heating the assembly under pressure to melting of the surface of the film of the thermoplastic resin with a peroxide or a mixture thereof with an organic acid or an anhydride of an organic acid present in the assembly, the peroxide decomposing during the step of heating under pressure, so as to integrate the laminate components.

2. A process according to claim 1, wherein the film is of polyethylene.

3. A process according to claim 1, wherein the film is of polypropylene.

4. A process according to claim 1, wherein the film is of a copolymer containing structural units of an olefin.

5. A process according to claim 1, wherein the film is of polyvinyl chloride.

6. A process according to any of claims 1 to 5, wherein the film is of a mixture of at least two thermoplastic polymers.

7. A process according to any one of claims 1 to 6, which comprises putting a second film of thermoplastic resin on the outside of the resin-impregnated sheet, the softening point of the said thermoplastic resin of the second film being substantially equal to or higher than the heating temperature but higher than that of the thermoplastic resin of the first film and heating the assembly so obtained under pressure to form the laminate.

8. A process as claimed in claim 7, wherein the second film of thermoplastic resin is removed from the resin-impregnated sheet, after obtaining the assembly.

9. A process according to claim 7, wherein the second film is provided with uneven pattern thereon.

10. A process according to claim 1, whenever carried out substantially as described in any one of the Examples herein.

11. A laminate produced by a process according to any one of claims 1 to 10.

(15)

5 was produced by placing a polypropylene film having a thickness of 25 microns on a corrosion-resistively alloyed aluminium panel having a thickness of 1.2 mm and having been applied with a 3% solution of oxalic acid in methanol, further placing the melamine-impregnated sheet on the film, integrating the assembly by heating at a temperature of

165°C under pressure of 120 kg/cm² for 30 minutes and cooling.

The produced decorative plate has excellent properties in scratch hardness, heat-resistance, discolouring resistance, peeling resistance against punching and cutting, as shown in the following Table VI.

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TABLE VI

Property	Test method	Result
Scratch hardness	By diamond needle	200g
Heat-resistance	JIS K—6902	No change
Discolouring resistance	JIS K—6902	No change
Punching	By press for punching	Good
Cutting	By shearing machine	Good

COMPARATIVE EXAMPLE

20 (B) The procedures described in the above Example (A) were repeated with the exception that spraying the tert.-butyl perbenzoate solution was omitted.

25 The decorative laminate obtained is poor in adhesion between the metal and polypropylene film, as shown by easy peeling off in the cross-cut test.

30 (C) The procedures described in the above Example (A) were repeated with the exception that a polyethylene terephthalate film having a thickness of 20 microns was placed on the outside of the melamine resin-impregnated sheet and removed after the completion of the procedures.

A unique semi-glossy decorative plate was thus produced.

35 WHAT WE CLAIM IS:—

40 1. A process of producing a laminate comprising a metal support and a cured thermosetting resin layer by heating a metal plate or foil and a thermosetting resin-impregnated sheet under pressure, which comprises interposing a film of thermoplastic resin between the thermosetting resin-impregnated sheet and the metal plate or foil, and heating the assembly under pressure to melting of the surface of the film of the thermoplastic resin with a peroxide or a mixture thereof with an organic acid or an anhydride of an organic acid present in the assembly, the peroxide decomposing during the step of heating under pressure, so as to integrate the laminate components.

2. A process according to claim 1, wherein the film is of polyethylene.

3. A process according to claim 1, wherein the film is of polypropylene.

4. A process according to claim 1, wherein the film is of a copolymer containing structural units of an olefin.

5. A process according to claim 1, wherein the film is of polyvinyl chloride.

6. A process according to any of claims 1 to 5, wherein the film is of a mixture of at least two thermoplastic polymers.

7. A process according to any one of claims 1 to 6, which comprises putting a second film of thermoplastic resin on the outside of the resin-impregnated sheet, the softening point of the said thermoplastic resin of the second film being substantially equal to or higher than the heating temperature but higher than that of the thermoplastic resin of the first film and heating the assembly so obtained under pressure to form the laminate.

8. A process as claimed in claim 7, wherein the second film of thermoplastic resin is removed from the resin-impregnated sheet, after obtaining the assembly.

9. A process according to claim 7, wherein the second film is provided with uneven pattern thereon.

10. A process according to claim 1, whenever carried out substantially as described in any one of the Examples herein.

11. A laminate produced by a process according to any one of claims 1 to 10.

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