

Cross-linkable base layer for interlinings applied in a double-dot method

The invention relates to a crosslinkable hotmelt
5 adhesive coating based on a powder mixture comprising a commercially customary, OH-group-terminated copolyester based on terephthalic acid, isophthalic acid and butanediol or butanediol in combination with small amounts of up to 12 mol%, preferably from 6 to 10 mol%,
10 of other diols such as hexanediol or polyethylene glycol or PTHF, having melting points of from 100 to 150°C, and a pulverized free or blocked isocyanate, an aqueous epichlorohydrin or a liquid or solid epoxide, for producing a base dot as a barrier to strikeback in
15 double dot coating. The upper dot consists of an amine-regulated copolyamide, in order to ensure good attachment to the lower dot. The invention relates in particular to a hotmelt adhesive composition for the halftone coating of bondable interlinings for the
20 clothing industry, especially outerwear.

In order to solve the problems regarding the reduced stability to laundering and dry cleaning and regarding the relatively weak adhesion, improved hotmelt adhesive
25 compositions, but also improved coating technologies, have been developed. Duodot or double dot coatings are described, for example, in the patents DE-B 22 14 236, DE-B 22 31 723, DE-B 25 36 911 and DE-B 32 30 579.

30 The coating supports have been improved by using finer yarns with low-denier individual fibers down to the microfiber range and also synthetic yarns, examples being high-bulk acrylic yarns or polyester yarns. The wovens originally used have largely been replaced by
35 stitch-bonded fabrics, which are a combination of nonwovens and knitted fabrics. These new combinations lead to very soft but also very open constructions which place even greater requirements on the coating methods

and hotmelt adhesive compositions, especially with regard to strikeback and strikethrough of the hotmelt adhesive composition.

5 Costs and quality reasons have led to a marked reduction in the amount of coating applied per m^2 of interlining material. Whereas formerly the amounts applied were commonly 10 - 20 g/m^2 , they are nowadays 7 - 12 g/m^2 .
10 Despite these small amounts, it is necessary to ensure sufficient adhesion and resistance; in other words, the hotmelt adhesive must not strike away into the interlining, since then it would no longer be available for the actual bonding.

15 The object on which the invention was based, therefore, was to find an effective strikeback barrier which with a reduced amount of coating features high bond strength, effective attachment of the upper dot to the base layer and high stability to laundering and dry cleaning.

20 A range of strikeback barriers are known: crosslinking acrylate dispersions or polyurethane dispersions, or powder-filled pastes based on high-melting acid-regulated copolyamides and polyethylene or on high-
25 viscosity thermoplastic polyurethane powders.

All systems have more or less severe disadvantages in terms of application, attachment to the upper dot or stability to laundering. In addition, the dispersions
30 cannot be employed on coarse, hairy interlinings.

When self-crosslinking acrylate or polyurethane dispersions are coated, there is partial crosslinking even in the course of coating, which leads to a deposit on the
35 stencils and thus to clogging of the stencil holes. Laborious cleaning of the unit is required. Great difficulties are experienced when there is production-induced standstill. A further problem is the attachment of the

upper dot to the base layer. The high-viscosity powder-filled systems based on acid-regulated polyamide, polyethylene and polyurethane do not achieve the required strikeback security.

5

In DE 198 08 809 a description is given of how a free isocyanate is stabilized to water: the free isocyanate is incorporated by extrusion into an inert polyethylene matrix and subsequently finely ground again. By this means success was achieved in creating a stable crosslinkable system for the base dot. The disadvantage of this system is the complicated and hence expensive preparation of the water-stable isocyanate; additionally, the polyethylene matrix hinders the diffusion rate, which signifies a reduction in the reaction rate.

In a preferred embodiment a commercially customary copolyester with OH end groups is mixed with a passivated trimerized diisocyanate (as described in DE 35 17 333 A1) and the mixture is processed as an aqueous paste by rotary screen printing.

It has now surprisingly become possible to prepare a very reactive system and its preparation in accordance with the claims, having an activation temperature in the range from about 100 to 130°C and having water resistance. The crosslinkable hotmelt adhesive composition according to the invention for the coating and/or lamination of sheetlike structures has the feature that the reactive components present in the hotmelt adhesive composition do not react, with crosslinking, until in the melt.

Polyisocyanates, particularly solid polyisocyanates, are dispersed with isocyanate-reactive media, such as diamines (hexamethylenediamine), and by that means are surface-stabilized with respect to the surrounding

medium. This deactivation is brought about by treating the isocyanate particles on the surface with a deactivating agent in amounts which are stoichiometric with respect to the total isocyanate content. The
5 passivated fraction is in the range from 0.01 to 10%, preferably from 0.1 to 5%.

It is also possible, however, to use other crosslinkers, such as aqueous epichlorohydrin or epoxides. In the
10 course of subsequent drying in the oven at about 100 to 130°C, crosslinking is initiated within a few seconds to give a crosslinked strikeback barrier for the double dot. In this way it is possible to avoid the customary problems of isocyanate-containing systems, which are for
15 example that capped isocyanates (with caprolactam or oximes as capping agent or obtained by dimerization) require excessive activation temperatures; furthermore, no extraneous substances should be released in the course of bonding. The other problem was that non-
20 blocked isocyanates react with water while still in the paste.

Suitable isocyanates are solid isocyanates containing more than 2 free NCO groups and having a melting range
25 of from 100 to 130°C (e.g. Vestanat T 1890 from Degussa AG). The fraction of the polyisocyanate relative to the polyester used is in the range from 3 to 20% by weight, preferably from 5 to 10% by weight.

30 Also suitable as crosslinking component are epoxides having a melting range of from 90 to 130°C, preferably from 100 to 120°C, a molecular weight range of from 2 000 to 6 000, preferably 2 500 to 3 000, and more than 2 epoxide groups per molecule; bisphenol A may be
35 mentioned as an example.

A surprising finding was particularly effective attachment of the lower dot to the upper dot material

employed. The amine-regulated copolyamide, as upper dot material, reacted with the reactive components of the lower dot paste and forms an optimum attachment at the boundary of the two dots, which is not possible with normal, acid-regulated polyamides. Suitable products for the base dot and upper dot are low-melting grades of low viscosity. The melting point should be between 90 and 150°C, preferably between 115 and 130°C, with a solution viscosity η_{rel} in the range from 1.2 to 1.7, preferably from 1.25 to 1.5. By this means, the boundary layer reacts with the crosslinker-containing paste and creates a highly stable bonding of the two dots. The coating amounts for the base dot should be from 1.5 to 5 g/m², preferably from 2 to 4 g/m², and for the upper dot, depending on application, should be from 4 to 8 g/m², especially from 5 to 7 g/m². The base dot can be applied as a paste in a halftone formation. The copolyamides employed are based on dicarboxylic acids with chain lengths of C₆ - C₁₅, Cl, LL and diamines (piperazine, hexamethylenediamine, MPD, IPD C₉ + C₁₀).

The fraction of the polyester (based on solids content) in the base paste is in the range from 1 to 20% by weight, preferably from 5 to 15% by weight.

As acrylate and/or polyurethane dispersions it is possible to employ all common types. Particularly suitable are self-crosslinking butyl acrylates such as PLEXTOL BV 411 from Degussa AG, for example. Polyester:acrylate (PU) ratio based on solids is 1:5 - 1:20, preferably 1:7 - 1:15.

Examples:

A powder mixture comprising an OH-terminated copolyester (VESTAMELT 4680-P1) and a trimerized polyisocyanate from Degussa (VESTAGON T 1890) was dispersed with a diamine (e.g. hexamethylenediamine) and a common dispersant in water and the dispersion was passivated (diamine in an

equimolar deficit relative to NCO groups, 1:50). A common acrylate dispersion, e.g. PLEXTOL BV 441, and a thickener, e.g. Mirox TX, from Stockhausen, were processed to a printable paste, as described in
5 DE-B 20 07 971, DE-B 22 29 308, DE-B 24 07 505 and DE-B 25 07 504, and the paste was printed with a rotary screen printing unit having a CP 66 stencil onto a 25 g polyester knit comprising high-bulk yarn. The amount applied was 3 g/m². VESTAMELT X 1027-P816 was
10 scattered onto the paste dot while the dot was still wet, the excess was removed by suction, and the material was dried and partially sintered in a drying oven at 130°C. The upper dot (VESTAMELT X 1027-P816) had an addition of 5 g/m², so that the overall weight was 8 g/m².

15

Paste formulation of the base dot:

500 g of water

10 g of Mirox TX (polyacrylic acid derivative)

20 g of Intrazol 12/18/5 (ethoxylated fatty alcohol)

20 400 g of PLEXTOL BV 441

20 g of VESTAMELT 4680-P1

3 g of VESTANAT T 1890

0.15 g of hexamethylenediamine

25 **Result:**

A 5 cm wide strip of this interlining was bonded to a siliconized shirt material comprising a polyester/cotton blend at a joining temperature of 127°C and a linear pressure of 4 N for 10 s, and then the assembly was
30 subjected to laundering at 60°C.

Primary adhesion: 13 N/ 5 cm

60°C wash: 10 N/ 5 cm

Back-riveting: 0.1 N/10 cm

35

Comparative example 1: (state of the art)

A paste system based on an acid-regulated polyamide and on a polyethylene was applied to the same interlining

and the material was scattered with the same upper dot material (VESTAMELT X 1027-P518), dried and sintered. The same amounts of base dot and upper dot were applied.

5 **Paste formulation:**

1500 g of water

35 g of Mirox TX

40 g of Intracol 12/18/5

400 g of Schättifix 1820 (LP polyethylene)

10 200 g of VESTAMELT 250-P1

Schättifix 1820 is a low-pressure polyethylene having a melting point of 128 - 130°C and an MFR of 20 g/10 min.

15 **Result:**

Primary adhesion: 9 N/ 5 cm

60°C wash: 5 N/ 5 cm

Back-riveting: 0.9 N/10 cm

20 **Comparative example 2:**

A paste system based on an acid-regulated polyamide and on an acrylate dispersion was applied to the same interlining and the material was scattered with the same upper dot material (VESTAMELT X 1027-P816), dried and
25 sintered. The same amounts of base dot and upper dot were applied.

Paste formulation:

500 g of water

30 10 g of Mirox TX

20 g of Intracol 12/18/5

400 g of PLEXTOL BF 440

Primary adhesion: 6 N/ 5 cm

35 60°C wash: 3 N/ 5 cm

Back-riveting: 1.9 N/10 cm

The advantage of the new technology is that the lower

dot is crosslinked even under the drying conditions and, owing to its amine termination, the upper dot is cross-linked with the lower dot during melting, so giving an optimum attachment. Since there is a sharp increase in the molecular weight of the lower dot following coating, it is no longer able to sink into the knit. In the course of subsequent bonding, the low-viscosity polyamide of the upper dot is compelled to flow against the upper material that is to be bonded, since it is unable to flow off downward, hence giving very high adhesions even with very small amounts of hotmelt adhesive. The parting layer between upper dot and base dot, which hitherto has been the weak point of the system, especially in the course of laundering, is more resistant to hydrolytic attack than prior systems and therefore exhibits substantially higher stabilities.

Products employed:

VESTAMELT 4680-P1 is a copolyester from Degussa AG based on terephthalic acid, isophthalic acid, butanediol and polyglycol, having a melting point of 120°C.

VESTAMELT X 1027-P816 is a ternary copolyamide from Degussa AG with amine end groups 100 - 400 meq/kg, preferably 250 - 350 meq/kg, melting point 120°C.

VESTANAT T 1890/100 is a polyisocyanate having a functionality of 3 - 4, its melting point being at 100 - 115°C. It is a product of Degussa AG.

30

PLEXTOL BV 411

PLEXTOL BV 411 is an aqueous dispersion of a self-crosslinking acrylic polymer.

What is claimed is:

1. A hotmelt adhesive composition for the coating and/or lamination of sheetlike structures in accordance
5 with the double dot technique, characterized in that upper dot is based on an amine-terminated crosslinkable copolyamide and the lower dot is composed of an OH terminated polyester and further comprises a crosslinker and an acrylic and/or PU dispersion.
- 10 2. A hotmelt adhesive composition as claimed in claim 1, characterized in that the upper dot is an amine-regulated copolyamide.
- 15 3. A hotmelt adhesive composition as claimed in either of the preceding claims, characterized in that the upper dot is an amine-regulated copolyamide powder having a melting range of from 90 to 150°C and a solution viscosity η_{rel} in the range from 1.2 to 1.7.
- 20 4. A hotmelt adhesive composition as claimed in any of the preceding claims, characterized in that the lower dot is an OH terminated copolyester.
- 25 5. A hotmelt adhesive composition as claimed in claim 1, characterized in that the lower dot comprises an acrylate dispersion and/or polyurethane dispersion.
- 30 6. A hotmelt adhesive composition as claimed in any of the preceding claims, characterized in that the crosslinking component comes from the group of the isocyanates and has more than two reactive groups per molecule.
- 35 7. A hotmelt adhesive composition as claimed in any of the preceding claims, characterized in that the isocyanate has a melting range of from 100 to 130°C.

8. A hotmelt adhesive composition as claimed in any of the preceding claims, characterized in that an epoxide having a melting range of from 90 to 130°C, a molecular weight range of from 2 000 to 6 000 and more than two
5 epoxide groups per molecule is employed as crosslinking component.

9. A hotmelt adhesive composition as claimed in any of the preceding claims, characterized in that a
10 pulverulent free or blocked isocyanate is as crosslinking component.

10. A hotmelt adhesive composition as claimed in any of the preceding claims, characterized in that the
15 crosslinking component is an epichlorohydrin.

11. A hotmelt adhesive composition as claimed in any of the preceding claims, characterized in that the reactive, OH terminated copolyester is employed as base
20 dot for the double dot technology, as a strikethrough barrier.

12. A hotmelt adhesive composition as claimed in any of the preceding claims, characterized in that the base dot
25 consists of a passivated isocyanate and an OH terminated copolyester and is applied in halftone formation as a paste.

13. A hotmelt adhesive composition as claimed in any of the preceding claims, characterized in that the
30 crosslinking reaction is accelerated by catalysts.

14. A hotmelt adhesive composition as claimed in any of the preceding claims, characterized in that the
35 copolyester is based on terephthalic acid and/or isophthalic acid and/or adipic acid and/or dodecanedioic acid and on butanediol and/or hexanediol and/or polyglycol and/or PTHF.

15. The use of a hotmelt adhesive composition as claimed in any of the preceding claims for the coating and/or lamination of sheetlike structures.

5

16. An interlining material for clothing, characterized in that it has been provided with a hotmelt adhesive composition as claimed in any of the preceding claims.