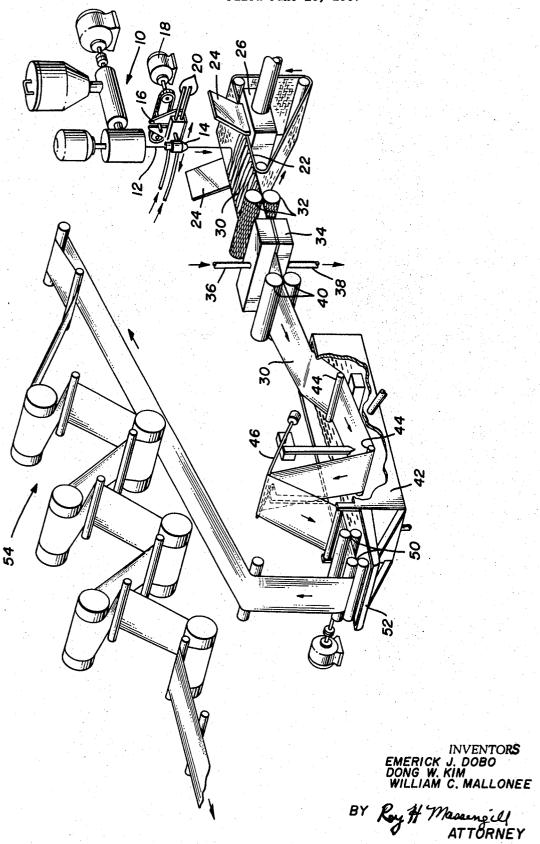
PROCESS FOR PRODUCING A NYLON NON-WOVEN FABRIC

Filed June 16, 1967



United States Patent Office

Patented Nov. 24, 1970

1

3,542,615 PROCESS FOR PRODUCING A NYLON NON-WOVEN FABRIC

Emerick J. Dobo, Cary, and Dong W. Kim and William C. Mallonee, Chapel Hill, N.C., assignors to Monsanto Company, St. Louis, Mo., a corporation of Delaware

Filed June 16, 1967, Ser. No. 646,720 Int. Cl. D04h 3/12

U.S. Cl. 156-181

ABSTRACT OF THE DISCLOSURE

A process for producing self-bonded non-woven fabrics from polyamides in a continuous operation employing an 15 activating gaseous medium such as hydrogen chloride gas to promote the bonding and apparatus for carrying the process to completion.

BACKGROUND OF THE INVENTION

The production of non-woven fabrics in a continuous process from a melt is known. However, the bonding of the known non-woven fabrics has been accomplished by the addition of external binders or by softening of the fiber with heat, solvent or plasticizer. External binders may be applied as powder, solution, emulsions, or even in the form of fibers. These methods suffer from several disadvantages. The use of an outside binder presents 30 problems in uniform application and may limit the properties of the entire web to those of the binder. Thus, for example, if a fiber with a relatively low melting point is used as a bonding material, the temperature conditions to which the web or resulting fabric may be subjected are 35 limited by the melting point of the binder fibers.

Autogenous bonding by the previous methods is not easily controlled, and frequently tends to alter the aesthetic properties of the web. For example, in solvent bonding, achieving adequate adhesiveness in the fibers 40 without dissolving the entire web or at least impairing the physical properties thereof is difficult. Furthermore, the intersections at which the fibers are bonded frequently have a swollen appearance or other evidence of solution and redeposition of polymer which is generally referred 45 to as polymer migration. In most instances these swollen areas around the bonds do not possess the same dye acceptance level because of changes in the crystalline structure which is localized at the bond site thereby causing nonuniform dyeing. With the foregoing problems in mind, 50 it is a primary object of the present invention to provide bonded polyamide structures and blends thereof which are free from external bonding agents and visible polymer migration at the bond points.

SUMMARY OF THE INVENTION

The invention contemplates the production of a selfbonded, non-woven fabric from a polymer melt in a single continuous operation. Continuous filaments are spun from molten polyamides and pneumatically attenuated prior to deposition in a random pattern onto the surface of a conveyor belt to form a coherent, uniform web. Preferably, the web is sprayed with a dope solution on the surface to facilitate better handling of the unbonded web by improving web integrity until bonding is ac-

complished. The web is advanced through a chamber filled with an activating gas wherein residence time is sufficient to permit surface absorption of gas into the filament. Contact between the filaments at their intersections is improved by calendering between opposed rolls or platens either before or after the web is exposed to the activating gas. Thereafter the gas is desorbed from the filaments in a wash bath or in a heated environment prior to take-up. The removal of the activating gas leaves a 14 Claims 10 strong bond between intersecting filaments that were under tension at the intersection.

The activating gas may include the hydrogen halides, boron trifluoride, sulfur dioxide and sulfur trioxide. Because of its faster absorption and desorption rate in addition to comparative ease of handling, hydrogen chloride gas is highly preferred. Therefore, for the sake of brevity and convenience, hydrogen chloride will be referred to herein as the activating gas in the description of the in-

Preferably the activating gas should be maintained at about 20 to 25° C. for uniform treating conditions and handling convenience although bonding can be accomplished at substantially higher and lower temperatures. The absorption rate is a function of the temperature with faster absorption occurring at elevated temperatures. It has been found however, that the amount of activating gas absorbed decreases with a corresponding increase in temperature until no absorption at all takes place when the temperature exceeds approximately 110° C.

While aqueous solutions of hydrogen chloride (hydrochloric acid) are known solvents for many polyamides, substantially non-ionized pure hydrogen chloride is not a solvent, and the process of this invention can be carried out under totally anhydrous conditions. The bonding process is not dependent upon solution and reprecipitation of polymer. Further indication that this is not a solvent effect lies in the fact that polymers other than polyamides which are also soluble in hydrochloric acid do not undergo this bonding reaction when subjected to gaseous hydrogen chloride in accordance with the techniques of this invention. It has been discovered however that better bonding is accomplished when the process is carried out in atmospheric conditions wherein the humidity level is maintained above about 25 percent and preferably between 40 to 60 percent.

To obtain this bonding reaction the structures must be in very close contact. In the case of two intersecting fibers, this condition may be achieved by holding the fibers crossed under tension. In a mat of fibers, it may be accomplished by shrinking entagled filaments after the mat has been formed, or by subjecting the mat to pressure. Pressing of the mat can precede or follow exposure to hydrogen chloride. In practice, the "pre-pressing" has the advantage that the pressing equipment need not be exposed to the activating gas. Post-pressing however, has the advantage of imparting greater strength to the mat because of an increase in pressure imposed on the fiber intersections.

The discovery has been made that polymers which can be self-bonded under the influence of hydrogen chloride gas (HCl) have in common in their structure the -NHCO- group. In order to exhibit this bonding property, the polymer needs an adequate concentration of these groups which are accessible and attached to groups which do not alter basicity unfavorably. It has been -3

found that polyamides containing some aromatic groups will undergo this bonding reaction, but certain wholly aromatic polyamides do not undergo the reaction despite concentrations of —NHCO— groups comparable to that in polyhexamethylene adipamide (nylon 66) which bonds very easily. This may result from the rigidity of the structure or from the effect of the aromatic rings on the basicity of the amide group of from a combination of these effects.

While the mechanism of the bonding is not completely 10 understood, it is believed that it is based on disruption of hydrogen bonds between the polymer chains by formation of an HCl complex with the amide group. In the polymer art it is well known that many of the physical properties of polyamides depend to a great extent on the 15 intermolecular hydrogen bonds between the —CO— and -NH- groups in adjacent polymer chains. The bonds form cross links between the molecular chains, increasing such properties as melting points and tensile strengths. When these bonds are disrupted by the action of the 20 hydrogen chloride, the polymer chains within the structure become more flexible and tend to shift to relieve the stress caused by tension or pressure on the structure. The complex formation is reversible, and when the hydrogen chloride is desorbed, the hydrogen bonds reform. In the 25 shifted position of the polymer chains, many of the new bonds are between chains in two different structures. Photomicrographs of cross-sections of filaments bonded by this process show a homogeneous structure at the site of the bond with no indication of a boundary between 30 the two filaments. Further support for this theory lies in the fact that self-bonding polymers cannot be bonded to polymers which are not self-bonding under the conditions of this process. However, two different polyamides which are self-bonding can be bonded to one another. 35

Bonding is accomplished with exposure times which may vary from 1 sec. to several minutes, depending on composition and structure of the materials to be bonded. Effective bonding has been achieved with concentrations of HCl ranging from 100 percent to 25 percent mixtures with air. By regulation of exposure time, depth of penetration of the gas into the individual filaments can easily be controlled and limited to the depth necessary to obtain desired bonding. In practice, good bonding has been achieved with penetrations as low as 5 45 percent of the cross-sectional area of the filaments but penetration of between 20 and 50 percent is preferred. Optimum time of exposure varies with the polymer composition, the concentration of the activating gas, the filament diameter, and previous physical treatment of 50 the filaments. In general, finer filaments, because of greater surface area per unit weight, will require a shorter time of exposure than will heavier denier filaments from the same polymer composition. Also, it has been observed that freshly spun filaments which have not been drawn 55 generally require shorter exposure time than drawn filaments. Prolonged exposure times tend to lower tensile strength of filaments probably because of a depth of penetration which permits a substantial decrease in orientation.

Desorption of the activating gas may be achieved at room temperature by washing with water or a very dilute aqueous solution of a base, or it may be achieved under completely anhydrous conditions by application of heat. The latter method offers the advantage of simpler 65 recovery of the activating gas. Temperatures necessary for desorption are far below the softening or melting temperatures of the polymers and therefore do not alter the physical properties of the polymeric structures.

BRIEF DESCRIPTION OF THE DRAWING

The single figure is a perspective view showing the preferred arrangement of the apparatus employed for carrying out the process of the present invention.

In referring to the drawing, there is shown an arrange- 75 otherwise designated.

4

ment for converting a polymer to a fabric in a single, continuous process. More specifically, reference numeral 10 denotes a conventional melt extruder and associated equipment which is employed to form the continuous filaments 12 from a molten thermoplastic material. The extruded filaments are attenuated and forwarded by a pneumatically operated aspirator 14 which is mechanically traversed by the traversing mechanism 16. A motor 18 of a well known type reciprocates the traversing mechanism 16 on a pair of fixed guide bars 20. The aspirator 14 is traversed at a predetermined rate to deposit the filaments 12 in a random pattern on the conveyor belt 22. As the filaments are deposited upon the lay-down belt they are intermingled to the extent that a coherent web structure is formed. If desired, a polymeric solution containing formic acid or the like may be sprayed upon the web at the point of lay down to improve the maintenance of web integrity during its travel to the point where bonding occurs.

To overcome the problem of having a non-uniform web edge from being created by the circular lay down pattern and the resulting reduction in web uniformity, edge-forming deflector plates 24 are provided in close relationship to the collection belt 22. These plates are preferably inclined at about 15° from the vertical and are spaced apart at the desired web width. The traverse stroke imparted to the filaments by traverse mechanism 16 is somewhat greater than the ultimate width of the web which results in straight edges having a uniform density. A suction box 26 is provided in close proximity underneath the lay-down belt 22 for exhausting air emanating from the aspirator 14 and from the spray solution dispensing jet when employed. The vacuum created in the suction box 26 is advantageously employed to assist in the deposition of the filaments on the collection belt.

The web leaves the forwarding belt 22 and passes through a pair of pressure rolls 32 for calendering to improve web density and coherency prior to entering chamber 34 which is filled with an activating gas introduced at inlet 36 and the excess thereof exhausted at outlet 38. If a solution is sprayed onto the web, as mentioned above, the rolls 32 are radiantly heated by conventional means, not shown. The web should be calendered at a roll temperature hot enough to evaporate the solvent employed and pressure sufficient to establish the desired number of filament intersections with the filaments in contact for bonding. Patterned rolls may be employed to obtain a variety of patterns in the finished product.

The residence time in the chamber 34 is dependent upon the type of activating gas used and the concentration thereof. After sufficient exposure time of the filaments in the gas filled chamber to permit surface absorption of said gas, the web 30 is then forwarded by rolls 40 to a wash bath 42 filled with water or a mild alkaline bath.

A pair of separately space apart guide members 44 control the horizontal path of the web 30 through the bath to permit the desorption of the gas from the filaments whereby bonding occurs from the recrystallization of the hydrogen bonds. Thereafter, the self-bonded web passes over a tensioning bar 46 before being drawn through two sets of squeeze rolls 50 which are set at clearances to squeeze as much of the residual water from the web as is feasible prior to entering the drying step. A drain pan 52 is provided for returning the excess water to the wash bath. The web is then advanced over a series of steam heated drying rolls 54 operated at a steam temperature of about 135° C. before being taken up.

PREFERRED EMBODIMENTS

In the following examples, which are merely illustrative of the present invention, all parts are by weight unless otherwise designated.

Nylon 66 predried to a moisture level of 0.01 percent and having an RV of 29 is melt spun at about 290° C. through a spinnerette having 14 orifices of .009 inch diameter. The spinning rate is 0.78 pound per hour. The freshly spun filaments of approximately 1.5 den. are passed through an aspirator located 38 inches below the spinnerette operated at 40 p.s.i.g. with an air throughput of 6 s.c.f.m. These attenuated filaments are randomly dispersed and deposited upon a continuously moving fo- 10 raminous conveyor by the action of the aspirator jet, the suction box beneath the belt. The distance between the aspirator jet and the foraminous belt is 18 inches. The web is prepared in continuous sheet form by means of traversing the aspirator in a reciprocating manner at right angles to the direction of travel of the conveyor belt. The traverse rate was 20 cycles per minute while traveling approximately 9 inches to produce a web of 0.96 oz. per sq. yd. The web is sprayed with wet ligaments comprising 70 percent of formic acid and 30 percent of nylon 20 solids at a rate of 2.5 cc. per minute to improve web integrity until further processing. The wet ligament treated web is embossed with a patterned roll exerting a force of 16 lbs. per linear inch at a roll temperature of 100° C. which removes the formic acid. From the calendering rolls, the web is forwarded in ambient atmosphere to the hydrogen chloride gas application chamber. In this gas chamber, the web is subjected to a hydrogen chloride atmosphere maintained under a small vacuum to prevent leakage to the ambient surroundings. The web has a residence time within the chamber of approximately 10 seconds. The flow rate of HCl gas is 12 g. per minute. The residual HCl gas is removed from the fibrous web by washing in a mild caustic bath. Excess water is removed from the web by means of a pair of wringer rolls followed by drying on a plurality of heated rolls prior to take-up. The resulting fabric is firm drapable and clothlike having a basis weight of 0.96 oz. per. sq. yd. with a bending length of 1.4, a tenacity of 7.8 lbs. per in. per sq. yd. and a Stohl abrasion resistance of approximate 2000 cycles.

Example II

Nylon 66, having an RV of 29, was melt spun at about 290° C. through a spinnerette having 14 prifices of .009 inch diameter. The spinning rate was 1 pound per hour. The freshly spun filaments were passed through an aspirator located 38 inches below the spinnerette. The aspirator jet was operated at 40 p.s.i.g. with an air throughput of 6 s.c.f.m. The attenuated filaments were randomly dis- 50 persed and deposited upon the continuously moving foraminous conveyor belt by the action of the aspirator jet. The distance between the aspirator jet and the foraminous belt was 18 inches. The web was prepared in continuous sheet form by means of traversing the aspirator in a 55 reciprocating manner at right angles to the direction of travel of the foraminous conveyor belt. The traverse rate was 30 cycles per minute while the traverse stroke was 10 inches. To provide a web having a uniform selvage density, inclined plates were provided having a separation of 9 inches vicinal to the conveyor belt. Air control jets were provided to direct a flow of air down over the surface of the edge-forming plates which forced the filaments deposited thereon down onto the belt. These air jets were operated at 30 p.s.i.g.

As the conveyor belt moved forward at a predetermined speed to control web weight, a 9-inch wide uniformly dense, continuous web was formed from edge to edge. The as-formed web was sprayed with wet ligaments comprising 70 percent formic acid and 30 percent nylon solids at the rate of 0.41 cc. per minute to provide ease in further processing. The wet ligament treated web was embossed with a patterned calendering roll exerting a force of 130 pounds at a temperature of 130° C. From the calendering rolls the web was forwarded in the ambi-

ß

ent atmosphere, maintained at a relative humidity of 60 percent at 70° F., to the HCl gas application chamber. In the HCl gas application chamber the web was subjected to HCl gas maintained at a vacuum of 16 inches of mercury. The web had a residence time within the chamber of approximately 7 seconds. The flow rate of the HCl gas was 8 grams per minute. The residual HCl gas was desorbed from the fibrous web and washed in a bath containing NaOH maintained at a Ph value in the range of 11–13 at 30° C.

Excess water was removed from the web by means of a pair of wringer rolls followed by drying over a plurality of heated rolls prior to take-up.

The resulting fabric was firm, drapable and clothlike having a base weight of 3.0 ounces per square yard, a thickness of 25 mils, a tensile strength 9.3 pounds/inch/ounce/square yard, an elongation of 50 percent, a bending length of 2.1 inches, and a Stohl flex abrasion resistance of 1775 cycles.

Example III

Nylon 66 dried to a moisture level of 0.01 percent having an RV of 30 is melt spun at 290° C. through a spinneret having 14 holes and passed through an air aspirator located below the spinneret. The filaments are attenuated and randomly dispensed on a moving foraminous conveyor. The web is formed continuously and is composed of filaments having 1.9 denier with 3.8 grams per denier tenacity and 134 percent elongation to break. The web is sprayed with wet ligaments to control its formation and ease of handling until the filaments are bonded. The web weight is 2.6 oz./yd.2. It is pressed flat to consolidate the filaments at 223 p.s.i.g. at 150° C. by a gas application chamber maintained at one inch of mercury vacuum, the web is exposed to pure HCl gas for 10 seconds. The gas is passed through the web at a rate of 20 grams per minute. By an ambient atmosphere, the gas is removed with dry heat from an infra-red source at 150° C. for 20 seconds. The resulting product is a strong, flexible fabric having a strength of 11.2 lbs./in./oz./yd.2 with a bending length of 1.9 inches.

What is claimed is:

- 1. A method for preparing non-woven fabrics comprising the steps of spinning continuous nylon filaments from a nylon polymer melt, pneumatically attenuating the filaments, depositing the filaments onto a moving belt in a random pattern to form a uniform web, advancing the web through a chamber filled with a chemically activating gas wherein the filaments absorb said gas, said gas being at a temperature of less than 100° C., removing the absorbed gas from said nylon filaments to bond said filaments together at a substantial number of the filament cross-over points.
- 2. The method of claim 1 in which the web is calendered prior to entering the gas-filled chamber.
- 3. The method of claim 2 in which the activating gas is a hydrogen halide.
- 4. The method of claim 3 in which the hydrogen halide is hydrogen chloride.
- 5. The method of claim 1 in which the activating gas is 60 boron trifluoride.
 - 6. The method of claim 1 in which the activating gas is sulfur dioxide.
 - 7. The method of claim 1 in which the activating gas is sulfur trioxide.
 - 8. The method of claim 1 in which the activating gas is removed in a wash bath.
 - 9. The method of claim 1 in which the activating gas is desorbed in a substantially dry environment heated to between about 90° C. and 200° C.
 - 10. The method of claim 1 in which the web is further consolidated and stabilized by spraying a solution upon said web near the point of deposition thereof.
 - 11. The method of claim 10 in which the solution is comprised of a mixture of nylon ligaments and formic acid

3,542,615

9,9	-14	OIO		
7				8
12. The method of claim 11 in which the web is calendered at a temperature sufficient to vaporize the formic		3,184,358 3,276,944		Utz 156—306 X Levy 156—306 X
acid.		3,314,840		Lloyd et al 156—181 X
 13. The method of claim 1 in which the web is calendered after leaving the gas-filled chamber. 14. The method of claim 1 in which the continuous filaments are traversed across the width of a foraminous 		CARL D. QUARFORTH, Primary Examiner G. SOLYST, Assistant Examiner		
by a traverse mechanism.			. 1	U.S. Cl. X.R.
References Cited	10	156305, 30	06	
UNITED STATES PATENTS				
2,398,831 4/1946 Hoffman 156—181 X			s	