Nov. 30, 1965 H. B. THOMPSON 3,221,090

PROCESS FOR INCREASING THE STRENGTH OF NONWOVEN ARRAYS OF POLYETHYLENE TEREPHTHALATE FIBERS

Original Filed March 21, 1962

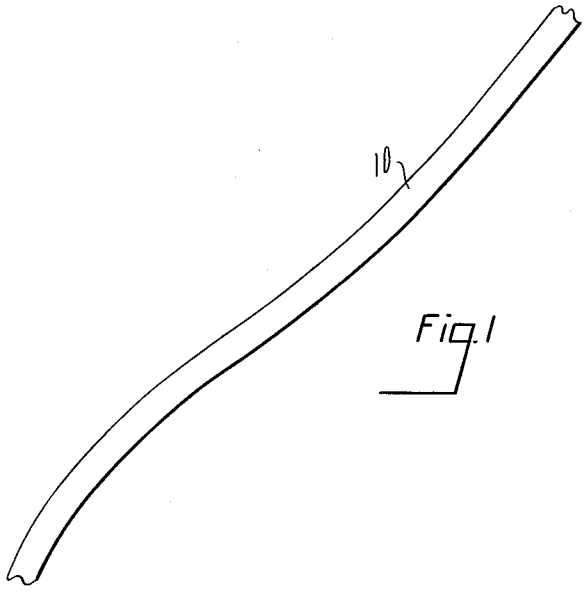


Fig. 1

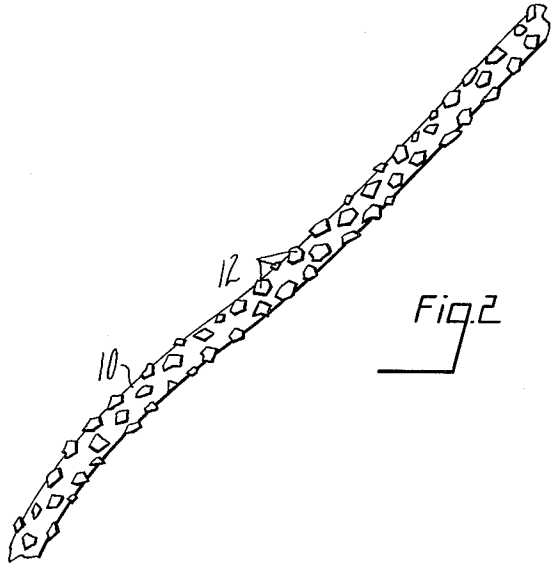


Fig. 2

United States Patent Office 3,221,090 Patented Nov. 30, 1965

3,221,090

PROCESS FOR INCREASING THE STRENGTH OF NONWOVEN ARRAYS OF POLYETHYLENE TEREPHTHALATE FIBERS

Hugh Brian Thompson, Milton, Mass., assignor to The Kendall Company, Boston, Mass., a corporation of Massachusetts

Original application Mar. 21, 1962, Ser. No. 181,354. Divided and this application Feb. 7, 1964, Ser. No. 348,319

3 Claims. (Cl. 264—345)

This application is a division of my copending application Serial No. 181,354, filed March 21, 1962.

This invention relates to a method for enhancing the strength of fibrous arrays comprising certain synthetic fibers.

More particularly it is concerned with a method for substantially increasing the strength of non-woven fabrics prepared from certain polymeric fibers, said fabrics being primarily dependent for their enhanced integrity upon frictional engagement of the fibers.

Non-woven fabrics produced from synthetic fibers, particularly of the polyester type, are increasingly important articles of commerce, especially in the form of lightly felted batts or arrays which may be compressed to a felt-like consistency, usually with the accompaniment of a certain degree of needling, punching, or other processing for entangling, bonding, or intermeshing the fibers into a unitary, coherent structure. Non-woven fabrics of this type are finding increasing usage in the interlining field, as well as in electrical and other industrial fields.

The degree to which such non-woven fabrics of synthetic fibers may be utilized, however, is definitely limited by the low strength which characterize prior art nonwovens of this type. In general, non-woven fabrics derived from a given weight of fiber are very much lower in tensile strength than woven fabrics derived from the same weight of staple fiber. The process of spinning and twisting the fibers into yarns brings inter-fiber frictional forces into play, which greatly enhances the strength of the fabric woven from such yarns. Additionally, in a conventional yarn structure, an applied load is distributed simultaneously over a large number of fibers, whereas in a non-woven fabric the cooperative contact between fibers is of a low order of magnitude, and the potential strength of the fibers is not realized since they react to stress almost singly and independently.

In an effort to enhance the strength of non-woven fabrics, various expedients have been resorted to, such as:

(1) Saturating fibrous batts with heavy concentrations of bonding agents. This not only adds foreign material to the fibrous array, but has an adverse effect on the desirable properties of softness and flexibility, which are often primary requisites as in the interlining field.

(2) Needle-punching the fibrous batts by repeated passes through a needle-loom. Each pass, however, makes the array more dense and compact, and is in addition uneconomical.

(3) Reinforcing the fibrous batts with yarns or fabrics. This is not only expensive, but it decreases the pliability, conformability, and extensibility of the batt.

(4) Strengthening the batt by using thermoplastic binder fibers, or by mixing the synthetic fibers with naturally feltable fibers, or alternatively by employing certain retractible synthetic fibers in a retracting shrinkage process, as set forth in the patent to Lauterbach, U.S. 2,910,763. Various other proposals have been made wherein the fiber shrinkage is accompanied by a certain amount of fusion and sticking together of thermosensitive fibers.

Such processes employing fusion and retraction, however, are difficult to control insofar as they depend on the use of fibers which retract when heated. The retraction of the fibers inevitably results in shrinkage of the whole assembly, and in a certain degree of densification whereby the soft appealing hand of the original fibrous array is to some extent lost or destroyed.

It is an object of this invention to provide a strong non-woven felt-like array of synthetic fibers which is not characterized by having undergone shrinkage or fiber fusion. It is a further object of this invention to provide a process for substantially increasing the strength of such a felt-like array without substantial retraction or fusion of the synthetic fibers. It is also an object of this invention to provide a process for making a high tensile strength array of fibers which retains essentially all of the softness, drape, and conformability of the unprocessed array.

It is a further object of this invention to provide an array of synthetic fibers characterized by a substantially increased degree of inter-fiber friction, whereby the tensile strength of the array is similarly increased.

These and other objects will be apparent from the following description of the invention.

In general, the elements of this invention are based on considerations of and means for enhancing the fiber-to-fiber friction of fibrous arrays, without adding foreign substances or shrinking the fibers to the extent that the density, compactness or conformability of the array become objectionable. In this respect, my process is applicable to a fibrous array, such as a needled batt of polyethylene terephthalate fibers, and operates so as to impart to such a batt a 10 to 20 fold increase in strength without substantial shrinkage, and without surrendering to any significant degree the desirable properties of softness, flexibility, porosity, etc., associated with what I term "fiber freedom."

It is known to enhance fiber-to-fiber friction by the use of colloidal silica or similar materials. However, the presence of added foreign materials is often undesirable, and their efficiency in promoting fiber friction is limited, since they are merely in contact with the fiber surface.

I have found that unexpectedly large-scale increases in fiber-to-fiber friction may be brought about by a heat treatment of certain synthetic polymeric fibers, as typified by fibers made from polyethylene terephthalate. Commercially available fibers of polyethylene terephthalate, such as offered by Du Pont as Dacron 54, or by Celanese as Fortrelle, contain a small amount of lower molecular weight polymeric material which is principally a trimer of the ethylene glycol-terephthalic acid condensation product, in cyclic form. This trimer may be associated with smaller amounts of other oligomers, the presence of said trimer and other oligomers in commercial polyethylene terephthalate having been established by Ross et al. (Journal of Polymer Science, vol. 13, page 406, 1954).

Normally, the presence of these lower molecular weight materials in commercial polyester fibers is not obvious or apparent even under microscopic examination at very high magnifications. The lower polymer apparently exists within the body of the fiber, which shows a smooth surface. By exposing the fiber to a heating process, however, I have found that I can cause to appear on the fiber surface an irregular crystalline growth which clings tenaciously to the fiber surface, converting the fiber profile from an originally relatively smooth, low-friction contour to one which is marked by rough, sharp-edged protuberances. This effect is illustrated in the drawing, wherein FIGURE 1 is a representation of a portion of the length of a commercial synthetic fiber. FIGURE 2 is a representation of the same portion of fiber after treatment according to this invention, showing crystals **12**, derived from the body of the fiber **10**, the crystals being adherent to and lying on the surface of the fiber.

As set forth more fully below, this can increase the in-

ter-fiber friction to such an extent that a heat-treated needled felt made from suitable fibers may have a tensile strength 20 times the strength of an identical needled felt before heat treatment.

It is not known to me whether the lower molecular weight material, which I will call trimer for convenience, exists in the form of crystals within the body of the unheated polyester fiber, or whether it is disposed in crystallites, or in an amorphous, non-crystalline state of aggregation.

In any case, it appears that the process of this invention yields products which are chemically and physically unique with respect to surface roughness and fiber-to-fiber friction, so far as man-made fibers are concerned. The art of adding foreign crystalline material to fibers, to enhance friction, is known. It is also possible to bond friction-promoting agents to fibers, either by adhesives, or by heating thermosensitive fibers in the presence of such agents, or by swelling certain sensitive fibers and adding foreign material while the fibers are surface-softened or surface-swollen. Several considerations make such treatment undesirable, however. No matter what sort of adhesive is used, there is always difficulty in promoting a maximum degree of adhesion of friction-enhancing agent to fiber, since the ideal conditions for specific adhesion between two different chemical species are hard to establish. Second, a foreign material, no matter how finely divided, can rarely be so dispersed that it is distributed over the fiber surface with maximum efficiency. The addition of foreign friction-enhancing agents from an external phase, onto the fiber surface, will always remain a different process from the promotion, from within the fiber substance, of crystalline growths which grow outwardly from the fiber surface in the form of rough protuberances. In the latter case, the protuberances are of the same basic chemical composition as the main body of the fiber, so that questions of specific adhesion do not arise, nor is a foreign chemical identity added to the fiber system.

It should also be appreciated that the fiber-to-fiber frictional enhancement of this invention differs from the results obtained by etching fiber surfaces. In an etching or partial-dissolution process, highly irregular areas are dug away or eaten away from the fiber surface. This is a subtractive effect, wasteful of fiber substance, and always accompanied by a decided degree of fiber weakening, from the very nature of the process.

It will be appreciated that this invention is applicable to a wide variety of fibrous arrays including card webs, needled batts, bonded non-woven fabrics, roving, and other fibrous aggregates wherein the surface friction of fiber against fiber constitutes one of the main elements in the strength of the product. It is also applicable to fabrics made from low twist or "zero twist" yarns, which may be strengthened for the weaving process by use of a sizing agent which may be subsequently removed prior to or during the practise of my invention on such fabrics. The increased strength shown by the process of this invention will vary with the geometry of the fibrous array in question. For example, in an unpressed card fleece, the fibers are normally in engagement only along a small fraction of their length, since the density of such material is very low, being only about 1% of the density of the fiber substance. Under such circumstances, as illustrated in Example I, the fibers make little contact with each other, and the strength increase shown by the practise of this invention, though useful, is only about 100%.

Much higher strength increases, up to twentyfold, are shown by needled batts of suitable fibers, as illustrated by Example II. To some extent, at least, this can be explained by the increased fiber-to-fiber contact shown by needled webs, so that the surface roughness introduced by the process of this invention has a greater chance to take effect.

As illustrated in Example III, the treatment of my invention produces only moderate strength increases in fibrous arrays previously bonded by the use of thermoplastic fibers. This is not surprising in view of the consideration that in such products, the principal source of strength is the bond between the thermoplastic fibers and the non-binder fibers, which is so high as to obscure the effect of slippage between non-binder fibers. Such bonded webs are, of course, much less flexible and conformable than needled batts of the same composition.

Example I

A quantity of 3 denier 1½ inch polyethylene terephthalate fiber of the type known commercially as Dacron 54 (E. I. du Pont Company) was carded on conventional textile carding equipment to form an assembly of superimposed webs weighing 183 grams per square yard. This type of polyester fiber consists principally of highly polymerized polyethylene terephthalate associated with a small amount of a trimer of polyethylene terephthalate in cyclic form. To increase the fiber-to-fiber contact of this rather fluffy assembly, the assembly of webs was passed through a cold textile calender at a pressure of 200 pounds per inch of nip, to form a compressed batt.

Inch-wide strips were cut from this batt and measured in an Instron Tensile Tester using 3-inch jaws 3 inches apart moving at 5 inches per minute. The tensile strength of the strips averaged 0.332 pound.

A part of rest of the batt was subjected to a temperature of 400° F. in an air-circulating oven for 3 minutes. There was less than 10% shrinkage in the batt, no appreciable change in softness and porosity, and no evidence of fiber fusion or retraction. In a tensile test parallel to that set forth above the heated batt showed a strength of 0.697 pound, an increase of 110%.

Example II

A quantity of the same fiber used in Example I was carded to form a batt which was passed through a needle loom at such a rate to impose on that batt about 370 penetrations per square inch, the needling process being carried out on both faces of the batt. The needled fibrous array weighed 100 grams per square yard.

Tensile strength of inch-wide strips of this material averaged 1.45 pounds, as measured on an Instron Tensile Tester with 3-inch jaws 3 inches apart, moving at 5 inches per minute.

A sample of this needled material was heated at 400° F. for 4 minutes in a circulating air oven. Comparable tensile tests on one-inch wide strips of heated material showed a strength of 26.9 pounds, or between 18 and 19 times the strength of the unheated needled batt.

Shrinkage was inconsequential, being less than 15%, and there was no evidence of fiber fusion, or of loss of softness or porosity due to heat treatment.

In a parallel set of tests, a similarly needled batt was heated to 250° F. for 39 hours, and showed a strength increase of 8.8 fold. Another similar batt heated to 300° F. for 3 hours showed a 12.6 fold increase in strength. For the realization of maximum strength in the shortest time, therefore, I prefer to operate in the neighborhood of 400° F. when processing fibers such as Dacron 54. At temperatures much in excess of 400° F., secondary effects of fiber softening and shrinkage begin to occur, and the strength per unit weight falls off rather rapidly. Additionally, heating to above 400° F. for longer periods seems to sublime the surface crystal deposit off the fiber surface, as followed by microscopic examination and tests of tensile strength, which decreases under these conditions.

Although the strength increase shown by treatment at 400° F. for 4 minutes is considerably more than that at 250° F. for 39 hours, this is probably not directly attributable to the higher temperature having caused a higher amount of surface crystal growth. The nature of the growth is important: high temperatures result in numerous small crystals on the fiber surface, while the 250° F. treatment resulted in larger, but fewer, crystals, which

might be expected to show a lower degree of enhancement of fiber-to-fiber friction.

Example III

A blend of 80% Dacron 54 and 20% undrawn polyester fiber of a lower softening point was processed on carding equipment to form a batt weighing 25 grams per square yard. This batt was unified by passing through a 4-roll textile calender heated to 380° F. at which point the 20% of undrawn polyester fiber softens and acts as a binder fiber. The resulting unified sheet was two-thousandths of an inch thick and had a tensile strength of 3.18 pounds per inch-wide strip.

Samples of this material were heated at 400° F. for 5 minutes in a circulating air oven, without pressure or dimensional restraint being applied. No dimensional changes were observed, and the heat-treated product had a strength of 4.02 pounds per inch-wide strip, an increase of 25% over the base material.

I have found that the strength-increasing effects of this invention are not confined to fibrous arrays consisting entirely of fibers on the surface of which crystalline growth can be induced, but that such fibers can be mixed or diluted with what may be termed insensitive fibers, to realize strength increases of lesser degree, depending on the amount of dilution. For example, a series of intimate blends of 1½ inch 3 denier rayon was made with 1½ inch 3 denier Dacron 54 fiber, in which the Dacron fiber was present to the extent of 25%, 50%, and 75%. These blends were carded and then needle-loomed under identical conditions to form a series of needled batts of varying fiber composition. All batts, together with a batt of 100% Dacron 54 fiber of comparable weight, were then heat-treated for 3 minutes at 400° F.

The strength of the treated 100% Dacron 54 batt was 20 times the strength of the base needled material: the 75% Dacron, 25% rayon blend was increased 15 fold: the 50% Dacron, 50% rayon blend was between 9 and 10 fold; and the 25% Dacron, 75% rayon blend was between 4 and 5 fold. This indicates that the strength increase is probably a straight-line function of the Dacron 54 content.

It also exemplifies a method of substantially doubling the strength of needled rayon batts by the inclusion of about 10% of Dacron 54 fiber followed by appropriate heat treatment according to this invention. This strength increase, moreover, is effected without shrinking, fusing, or otherwise changing the fiber dimensions, and without the addition of bonding agents or other foreign material to the fibrous array.

As starting fibers for the practice of this invention, I prefer to use synthetic polymeric fibers which do not exhibit strong retractive forces when heated. Although strength increases are shown both by low-temperature long-dwell tests and by high-temperature short-dwell tests, the latter method, as explained above, is generally a more desirable commercial process since it may be made continuous with a set of carding machines or other fiber-arranging devices, a needle loom, and an oven.

By suitable choice of fibers considered commercially as non-retractible, and by operating below the fusing or sticking point of the fibers, I arrive at strength increases which do not involve undesirable shrinkage of the array, so that there is no appreciable change in density due to compacting, nor is there any substantial stiffening or change in hand of the product. Thus the invention is especially adapted to the production of soft, porous, conformable, dimensionally stable fibrous arrays, characterized by a strength which is not attainable by any other process without sacrificing at least some of the desirable properties.

Having thus described my invention, what I claim is:

1. A process for increasing the strength of nonwoven arrays of fibers composed at least in part of highly polymerized polyethylene terephthalate fibers, said polyethylene terephthalate fibers containing within the fiber body a lower molecular weight substance in the form of a cyclized trimer of terephthalic acid and ethylene glycol, which comprises heating said arrays to promote the migration of said trimer to the surfaces of said fibers, said heating being carried out at a temperature of between about 250° F. and about 400° F., continuing said heating until said trimer has migrated through the fiber surfaces and has become adherent thereto in the form of rough projecting protuberances, and discontinuing the heating process and allowing the array to cool before said trimer has sublimed from said surface.

2. A process according to claim 1 wherein the fibrous array is a batt of intermingled fibers.

3. A process according to claim 1 wherein the nonwoven array is a needled batt.

References Cited by the Examiner

UNITED STATES PATENTS

2,200,946	5/1940	Bloch	161—169
3,042,971	7/1962	Wolinski	264—22
3,061,882	11/1962	Wolinski	264—22

OTHER REFERENCES

Du Pont Textile Fibers Technical Information, "Dacron" Polyester Fiber, Bulletin D–67, July 1955, 1 page.

Giuffria, R.: "Microscopic Studies of Mylar Films and Its Low Molecular Weight Extracts," Journal of Polymer Science, vol. XLIX, pages 427–436 (1961).

ALEXANDER H. BRODMERKEL, *Primary Examiner.*

ALFRED L. LEAVITT, *Examiner.*