

Sept. 12, 1967

G. A. KINNEY

3,341,394

SHEETS OF RANDOMLY DISTRIBUTED CONTINUOUS FILAMENTS

Filed Dec. 21, 1966

7 Sheets-Sheet 1

FIG. 1

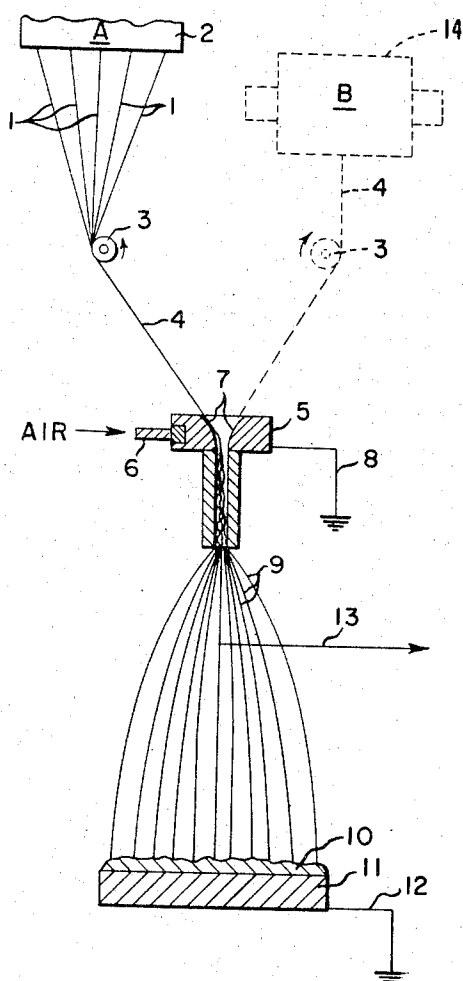


FIG. 2

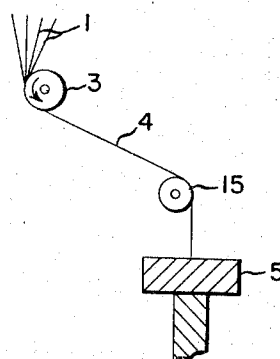
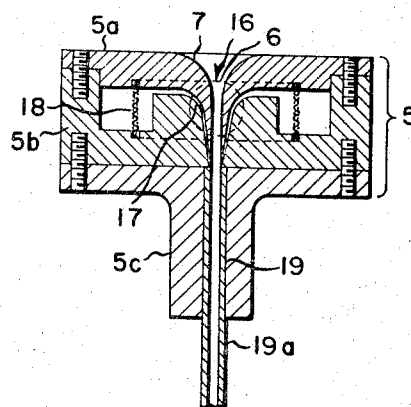
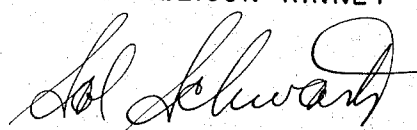


FIG. 3



INVENTOR
GEORGE ALLISON KINNEY

BY 
ATTORNEY

Sept. 12, 1967

G. A. KINNEY

3,341,394

SHEETS OF RANDOMLY DISTRIBUTED CONTINUOUS FILAMENTS

Filed Dec. 21, 1966

7 Sheets-Sheet 2

FIG. 4

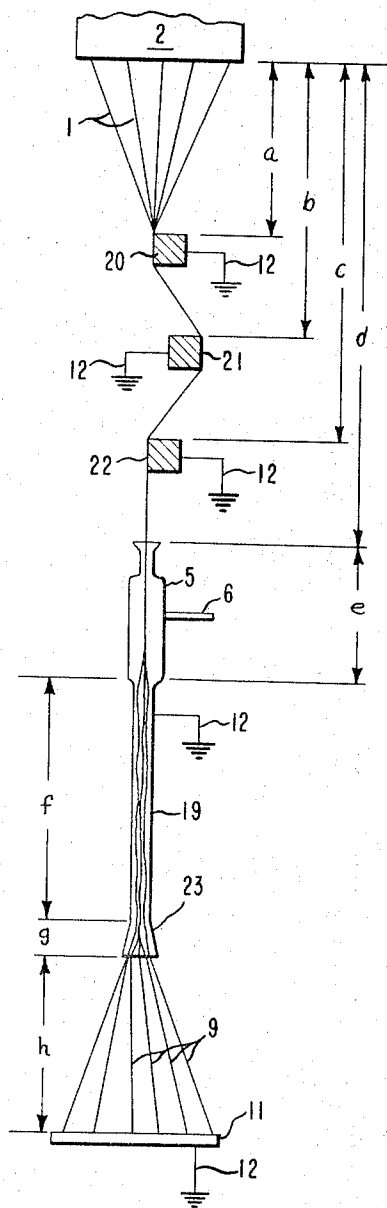
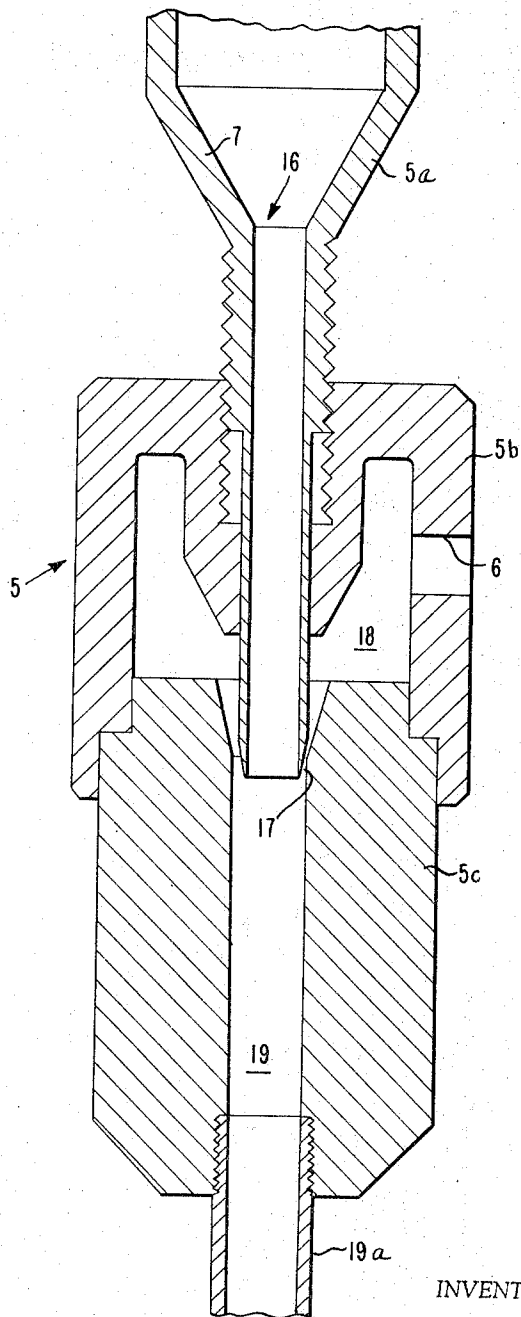


FIG. 5



INVENTOR

GEORGE ALLISON KINNEY

BY

Sol Schwartz

ATTORNEY

Sept. 12, 1967

G. A. KINNEY

3,341,394

SHEETS OF RANDOMLY DISTRIBUTED CONTINUOUS FILAMENTS

Filed Dec. 21, 1966

7 Sheets-Sheet 3

FIG. 7

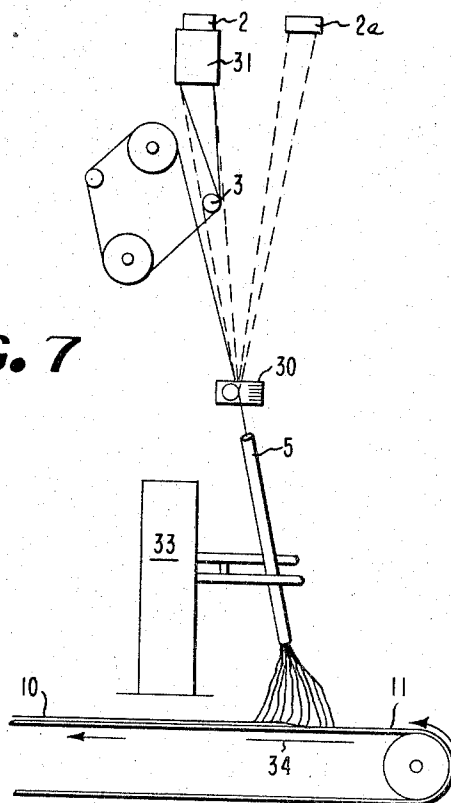


FIG. 8

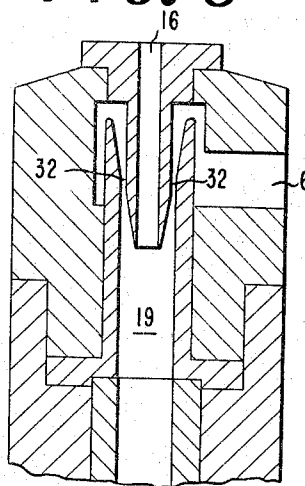
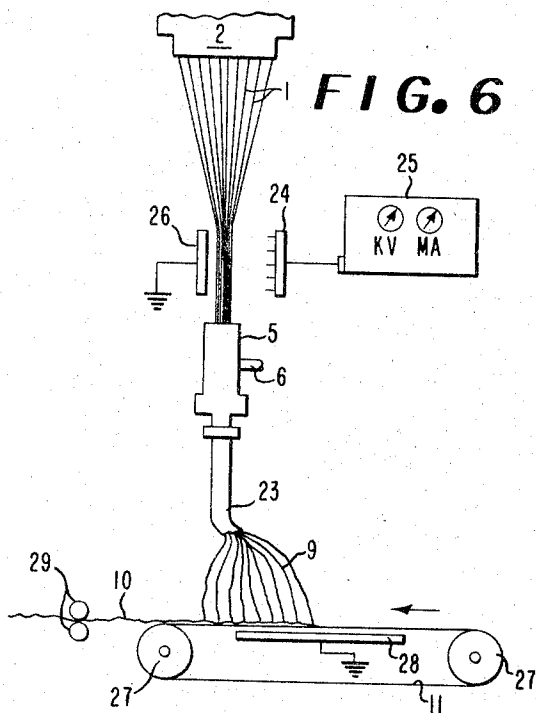


FIG. 6



INVENTOR

GEORGE ALLISON KINNEY

BY

Ed Schwart

ATTORNEY

Sept. 12, 1967

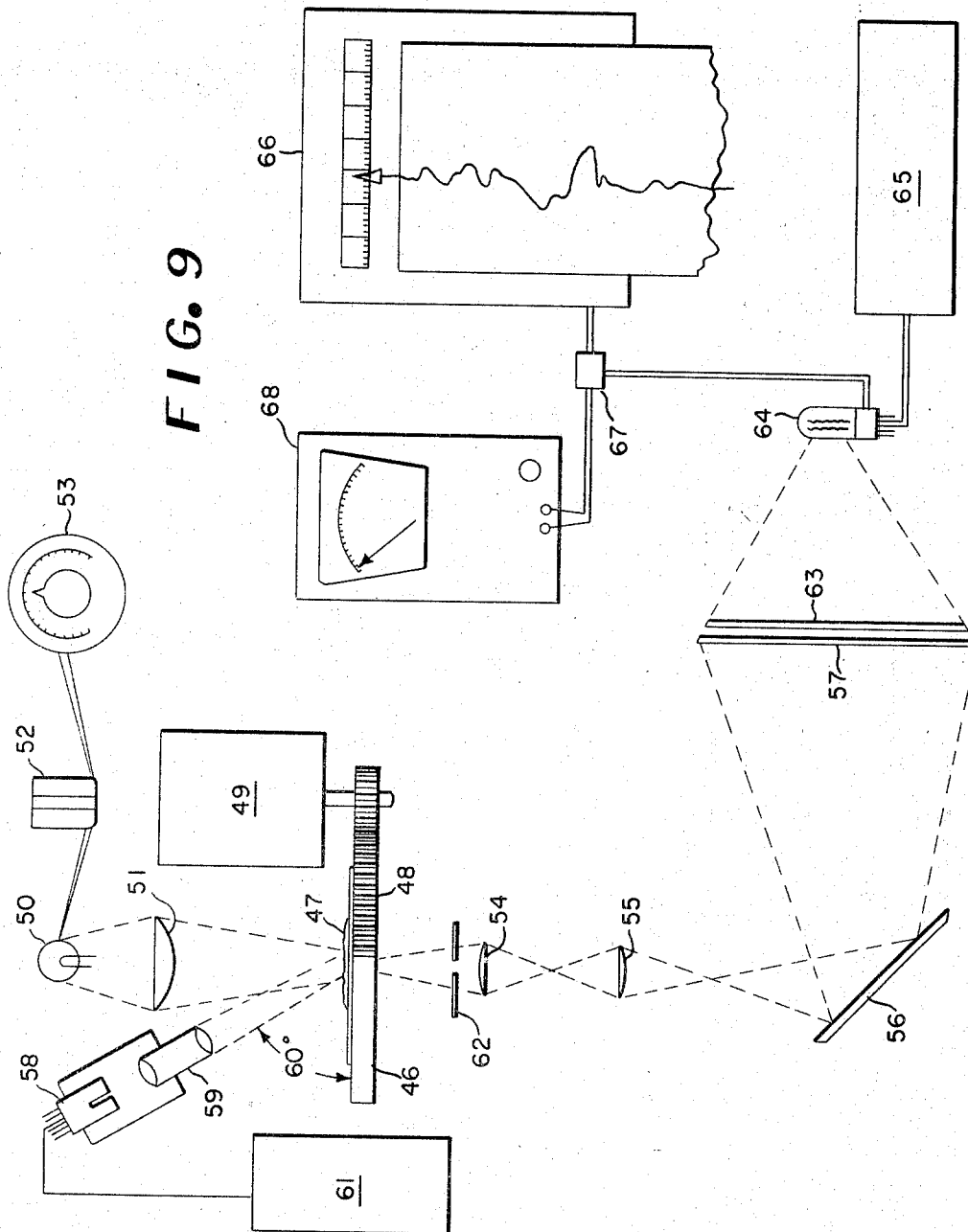
G. A. KINNEY

3,341,394

SHEETS OF RANDOMLY DISTRIBUTED CONTINUOUS FILAMENTS

Filed Dec. 21, 1966

7 Sheets-Sheet 4



INVENTOR

GEORGE ALLISON KINNEY

BY

Del. Howard

ATTORNEY

Sept. 12, 1967

G. A. KINNEY

3,341,394

SHEETS OF RANDOMLY DISTRIBUTED CONTINUOUS FILAMENTS

Filed Dec. 21, 1966

7 Sheets-Sheet 5

FIG. 10A

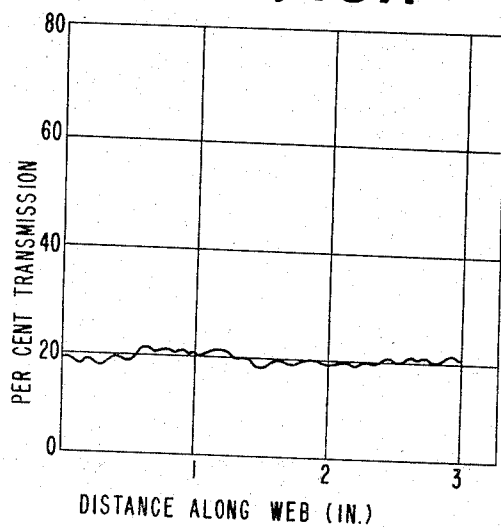


FIG. 10B

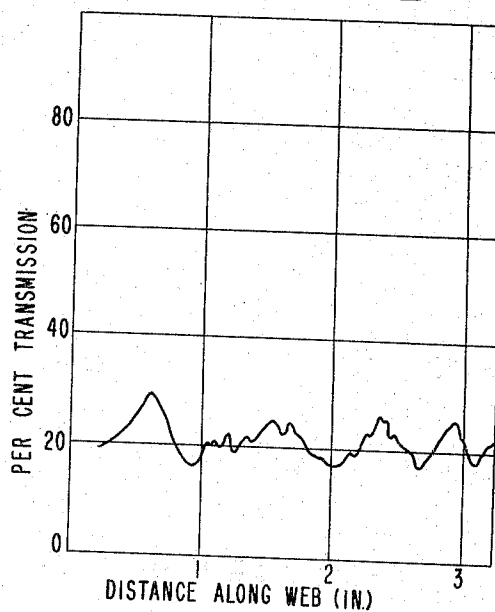
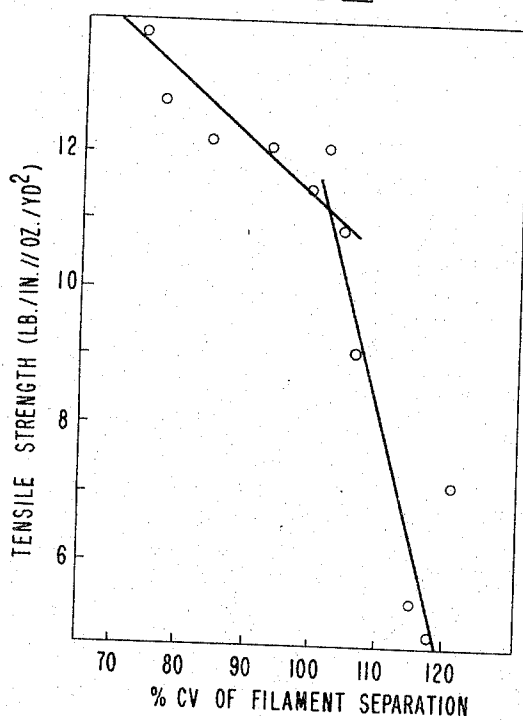


FIG. 12



INVENTOR

GEORGE ALLISON KINNEY

Sol. Schwartz
ATTORNEY

Sept. 12, 1967

G. A. KINNEY

3,341,394

SHEETS OF RANDOMLY DISTRIBUTED CONTINUOUS FILAMENTS

Filed Dec. 21, 1966

7 Sheets-Sheet 6

FIG. IIA

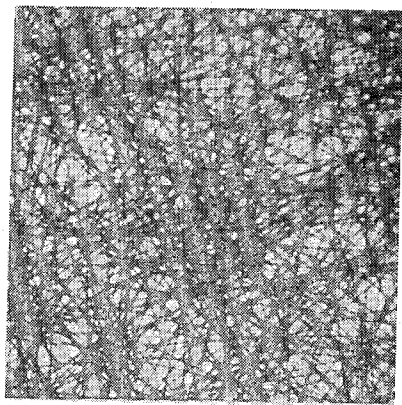


FIG. IIB

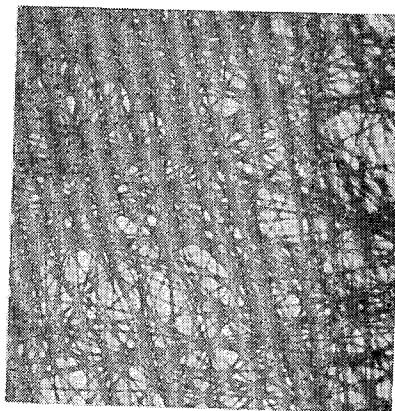


FIG. IIC

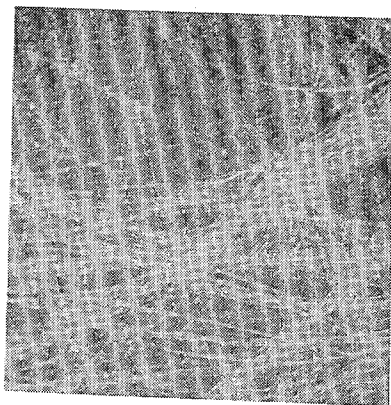
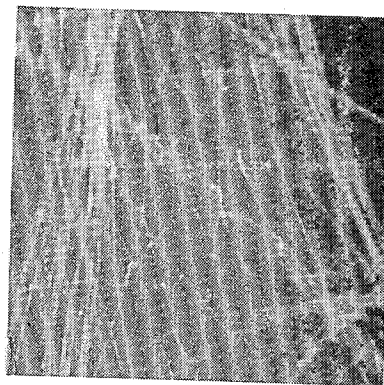


FIG. IID



INVENTOR

GEORGE ALLISON KINNEY

BY

Ed Schwartz

ATTORNEY

Sept. 12, 1967

G. A. KINNEY

3,341,394

SHEETS OF RANDOMLY DISTRIBUTED CONTINUOUS FILAMENTS

Filed Dec. 21, 1966

7 Sheets-Sheet 7

FIG. 13

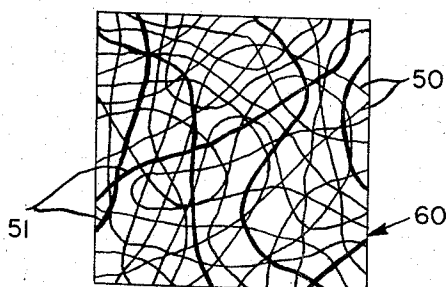


FIG. 14

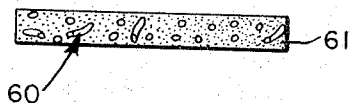
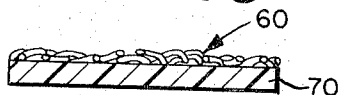
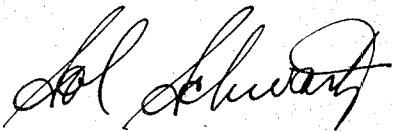


FIG. 15



INVENTOR
GEORGE ALLISON KINNEY

BY 
ATTORNEY

1

3,341,394

SHEETS OF RANDOMLY DISTRIBUTED CONTINUOUS FILAMENTS

George Allison Kinney, West Chester, Pa., assignor to
E. I. du Pont de Nemours and Company, Wilmington,
Del., a corporation of Delaware

Filed Dec. 21, 1966, Ser. No. 613,370

11 Claims. (Cl. 161-72)

This application is a continuation-in-part of copending application Ser. No. 439,361, filed Mar. 12, 1965, which in turn is a continuation-in-part of application Ser. No. 133,736, filed Aug. 24, 1961, and now abandoned, which in turn is a continuation-in-part of application Ser. No. 859,614, filed Dec. 15, 1959, and now abandoned, and is also a continuation-in-part of copending application Ser. No. 515,308, filed Dec. 21, 1965, which in turn is a continuation-in-part of application Ser. No. 345,792, filed Feb. 18, 1964, which in turn is a continuation-in-part of application Ser. No. 200,257, filed June 5, 1962, and now abandoned, which in turn is a continuation-in-part of application Ser. No. 859,661 and Ser. No. 859,614, both filed Dec. 15, 1959, and now abandoned.

This invention relates to nonwoven webs, fabrics and related sheet structures based on continuous filament synthetic organic fibers.

Continuous filament batts of glass fibers are known and are commercially available. The glass fibers are extruded from a melt and laid down in a random mass. The products are especially useful as insulating materials. In some cases, such glass fiber batts are used as reinforcing elements in plastic articles where the fibers provide strength and rigidity. During laydown of the batts, filament aggregation occurs and ropy strands of a plurality of filaments are present in the nonwoven sheet product. Since the continuous filament fibers are usually only a small portion of the final product, the appearance of the fibers and the particular manner of distribution had not been considered important. However, it is the aforementioned structural characteristic of the prior art sheets even apart from the materials employed that is responsible for most of their deficiencies for a variety of applications. Synthetic organic polymer materials are sometimes suggested in patent literature as equivalents for the glass fibers in such batts.

Nonwoven sheet structures derived from staple fiber materials have become increasingly popular for apparel and commercial uses. At the same time, such uses have brought awareness of the deficiencies of the products, in particular their lack of high tensile strength and resistance to tear. It is well-known that felts and papers are in general lacking in these properties. Many ingenious developments have been devised, chiefly, involving preferred binder materials, with the goal of producing nonwoven staple fiber sheet products of higher tensile and tear strength. It has not hitherto been recognized that high quality sheet structures of continuous filament synthetic organic fibers could be prepared so as to give uniform, strong sheet structures of the type desired in many applications now served by woven fabrics. Commercial operations have been restricted in that, except in certain limited applications such as insulating batts, and high tenacity binding tapes, use of continuous filament organic fibers has not been feasible.

Fiber processing techniques which are known in the art will not provide a sheet which is uniform in fiber distribution when continuous filament synthetic organic fibers are employed. Indeed this is no simple problem. Continuous filament synthetic organic fibers are normally prepared at high speeds. They are prepared in large bundles of 30, 50 or even several hundred individual fiber elements all emerging simultaneously from a multi-holed spinneret.

2

Deposition of a filament bundle from such a spinneret in the form of a web inevitably leads to ropy sheet structures composed of overlapping bundles of parallel filaments.

It is an object of the present invention to provide superior continuous filament synthetic organic fiber materials in the form of nonwoven sheet structures to permit greater utilization of the inherent high strength of modern synthetic fiber materials without the necessity of expensive and laborious weaving operations. It is a further object to provide substantially isotropic nonwoven sheet structures of uniform appearance and having superior tensile and tear strength which comprise continuous filament synthetic organic fibers distributed in the sheet in a homogeneous random manner.

Another object is to provide coherent nonwoven continuous filament webs which, even without bonding, are strong enough to permit continuous high-speed processing in such operations as dipping, impregnating, mechanical deforming, laminating and other commercial treatment, without requiring support of special handling of the web.

These objects have now been achieved in nonwoven sheet structures of continuous filament organic fibers in which the individual filament members are disposed in random configuration uniformly throughout the structure and in which the individual filamentary members are so disposed as to be separate, independent, and nonparallel in their relationship to one another. It is only through the utilization of continuous filament nonwoven structures that it is possible to achieve even to an approximate level the full utilization of the inherent tensile strength of the present day synthetic organic filaments in combination with high tear strength and flexibility in the nonwoven structure. This unusual combination of properties is obtainable with continuous filament structures because they do not require a high level of binder to obtain strong fabrics, thus tear resistance and flexibility can be maintained. With staple fiber structures, a higher level of binder is required to obtain strong fabrics and this is detrimental to both tear resistance and flexibility.

The sheet structures of this invention are composed of randomly distributed continuous synthetic organic filaments, preferably of a denier in the range of 0.1 to 30 (0.01 to 3.3 tex.). The individual filaments are separate and independent of each other as defined by a coefficient of variation of filament separation, as hereinafter described, of less than 100%. The filament separation can also be characterized by a bunching coefficient, as hereinafter, described, of 0.7 or greater. Depending on the method of preparation, the individual filaments also may exhibit quite high levels of crimp. Such crimped filaments are highly desirable as components of the nonwoven webs of this invention, and it is a surprising and advantageous feature of these products that even the use of crimped filaments does not interfere with achieving the indicated high level of filament separation.

Because of the individual, random distribution of the filamentary elements of the nonwoven webs of this invention, and the substantial absence of filament aggregates, the sheets are uniform in appearance and in opacity to light. In general, such webs will have a high level of web coherence and sheet strength, i.e., above 0.3 lb./in./oz./yd.², before bonding. Such high strength permits the webs even without bonding to be readily handled at high speeds for any necessary or desired processing. In order to obtain such nonwoven sheet structures it is necessary that individual deposition and distribution of fiber elements be achieved to the greatest possible level.

The crucial structural feature of the continuous filament nonwoven sheet structures of the present invention is a low degree of filament bunching or aggregation. The preferred method of expressing the degree of filament

bunching is by means of the distribution of the filament separation distances. The coefficient of variation of the filament separation distances, hereinafter referred to as CV_{fs} , is used to describe this distribution because it provides a normalization so that webs of different density can be compared.

A web which is formed from bundles of filaments in which there has been no attempt to separate the filaments within the bundles will contain bunches of filaments and the CV_{fs} will be much greater than 100%. Such a web will vary widely in density and appear blotchy. Similarly, webs containing numerous filament entanglements present CV_{fs} values substantially above 100%. On the other hand, if special care is taken to provide for uniform filament separation prior to and during formation of nonwoven webs from continuous filaments, webs with a CV_{fs} of less than 100% can be obtained. A surprising finding is that the nonwoven webs of this invention, which have a CV_{fs} of 100 or less are markedly different from webs having a CV_{fs} greater than 100%. A nonwoven web of this invention, when compared with a nonwoven web of the same type of continuous filaments but having a coefficient of variation greater than 100%, will (1) have much greater strength at equal filament tenacity, (2) have significantly better and more uniform appearance, (3) have greater abrasion resistance, and (4) have improved degree of waterproofness at a given weight of coating.

The bunching coefficient can also be used to describe the degree of filament aggregation. Bunching coefficient, designated BC, is defined as the ratio of the "fiber spaces" occupied by fibers relative to the total number of "fiber spaces" available. In this measurement the term "fiber space" represents the average space occupied by a fiber, and is calculated by dividing a unit distance of the nonwoven sheet structure by the total number of fibers oriented in a single direction in that unit length. The bunching coefficient concept is based on the premise that where the individual fibers disposed in the same direction are uniformly spaced from each other, each "fiber space" will contain one fiber and the bunching coefficient of such a structure will be unity. In a nonwoven which contains bunched fibers, some of the "fiber spaces" will contain bundles of fibers while others will be unoccupied and the bunching coefficient of such a structure will be less than one. Bunching coefficient, however, is insensitive to the location of the filaments within the "fiber space," so that an accurate measure of the structural characteristics of the web is not always obtained. Since the distribution of distances between essentially parallel filament segments does directly describe the structure, CV_{fs} is the preferred measure of filament separation.

A further characteristic which is essential to the structures of the invention is the random disposition of the component filaments. By "random" is implied the substantial absence of any anisotropy in the arrangement of the individual filaments. One test for randomness involves cutting representative square samples one inch or greater from the sheet under consideration and then counting the number of filaments terminating at each side of the square. In a random sheet of uniform basis weight, the number of filaments that will be encountered along any side of the square will vary by less than 20% from the number of filaments terminating at any other side of the square, regardless of the location or orientation of the square within the plane of the sheet. In preferred sheets, substantially the same number of filaments are encountered at each side of the square. A more precise and preferred procedure for measuring randomness than that described above determines the actual orientation or direction in which the component filaments lie within the plane of the nonwoven sheet. For a random sheet, there will be no predominant orientation of the filaments within the sheet, or expressed alternatively, there will be, on the average, as many filaments lying in one direction as in any other direction. This permits maximum utilization of the com-

ponent filaments and leads to a sheet which has essentially equivalent properties in all directions in the plane of the sheet.

The aforementioned sheets are useful in many ways. In general, they are most desirable and most widely used in the form of bonded sheets; that is, fibrous webs in which the continuous filament fiber elements are interconnected to one another with strong physical or chemical bonds. Such bonds may derive from the fiber elements themselves or from some added binder material, or from some second structure or structural elements which are combined with the nonwoven sheet material already described. All of these different embodiments are aspects of the present invention.

The examples which are given below illustrate the production of unbonded webs as well as some of the many modifications of binder technology which may be employed in utilization of the principles of this invention. Bonding procedures which may be employed are summarized here to illustrate the wide range of useful techniques. One desirable embodiment involves nonwoven structures which have been prepared in a form providing co-spun binder fibers. Such binder fibers may consist of continuous filament of a similar chemical nature to the structural filament element but having a lower melting point. In one mode of operation, such binder filaments may be filaments of the same chemical composition but spun with a lower level of orientation or with no orientation. In a second mode of operation, the co-spun binder filaments may be highly oriented but may be of a copolymeric nature or of some other modification which provides a lower melting temperature. Preferred binder fibers for use with poly(hexamethylene adipamide) include polycapraamide filaments or copolymers, melt blends, etc., thereof with poly(hexamethylene adipamide). Preferred binder fibers for use with poly(ethylene terephthalate) include the isophthalate and hexahydro-terephthalate copolymers thereof.

Still another technique is the use of composite filaments as described in Breen, U.S. Patent 2,931,091. The composite filaments may comprise a high-melting structural polymer and a low-melting binder polymer in side-by-side relationship running the length of the filament.

Indeed it is not necessary that any different filaments be employed since the principle of self-bonding may be used, in which the bonds are provided by localized fusion, partial or complete, of individual portions of the fibers. Such fusion may be brought about by spark discharge through the web or the application of heat to highly localized, mechanically isolated portions of the web. Solvent bonding may be employed, using spray techniques or a solution dipping process. The solvent need not be a complete solvent for the polymer but a swelling agent or a wetting agent. It is also possible and within the scope of the present invention to comprehend webs which have been bonded by the application of a vaporous or gaseous material which is not itself chemically reactive with the polymeric compositions of the filaments but which bonds by virtue of having excess heat content. Thus, steam or high temperature air or other vaporous material may be used to implement bonding. Finally, the webs may be rendered more coherent merely by pressing them as freshly prepared. This "self-binding" technique is surprisingly satisfactory and is sufficient to produce a structure useful for numerous applications as such. It is noted in this "self-binding" procedure that the inherent settability and crimp in the component filaments is believed to be in part responsible for the efficacy of the over-all operation. The instant nonwoven structures may also be rendered more stable to delamination by needling techniques (see, for example, Lauterbach & Norton, U.S. Patent 2,908,064).

In further modifications, it is possible to apply or co-deposit resinous thermoplastic binder particles in the form of granules, powders, or fibrils such as those de-

scribed in Morgan, U.S. Patent 2,999,788. In more familiar processing it is equally suitable to employ impregnation techniques using solutions, emulsions, dispersions or melts of resin binders to bring about the desired bond formation. Furthermore, it is well within the scope of this invention to apply to the unbonded webs after formation, intermediates suitable for the formation of polymeric materials to bring about polymerization of a binder polymer within the web, thus in a single step creating the polymeric binder and activating it to give a strong, bonded, highly coherent fabric. It is in the form of bonded fabrics that the nonwoven sheet structures of this invention are most desirable.

Bonding may be applied uniformly over the entire area of the fabric or in closely controlled patterned areas or in random patterned areas. Two or more different bonding techniques may be employed simultaneously or in sequence. In addition to bonding in itself, application of other materials to the nonwoven sheet structures of this invention may be employed for other purposes such as surfacing, modification of visual appearance or opacity or porosity or for providing other physical or chemical properties of a specific desired nature.

It is also possible to laminate the nonwoven structures of the present invention to films or fabrics which are in themselves thermoplastic or may contain thermoplastic elements which can be bonded to the present webs by the application of continuous or localized areas of heat. Within the scope of this aspect of the invention is included the lamination of the nonwoven sheet structures of this invention to metallic foils, and to impervious or pervious films. Such materials are useful for the preparation of protective coverings, vapor seals, conductive materials, dielectrics and other articles of commerce. In all of the sheet structures described herein, it is the critical feature already set forth, that is, the high degree of randomness and uniformity of fiber separation within the web which is the outstanding characteristic of these sheet products and which makes them unique and valuable.

A method by which it is possible to process continuously a bundle of parallel, synthetic organic fibers into a nonwoven sheet structure of the type described in the present invention, has been set forth in applicant's copending application Ser. No. 515,308 filed Dec. 21, 1965. That application describes a process in which a running multi-filament bundle composed of continuous synthetic organic filaments is charged electrostatically under tension to a sufficient level that, when the tension is released, the charge causes each filament to separate from adjacent filaments, and thereafter the filaments are collected on a receiver to form a nonwoven sheet product.

The filaments may be charged by a corona discharge maintained in the vicinity, by triboelectric contact with a suitable guide means or by other suitable electrostatic methods. The charging is accomplished while the filaments are under sufficient tension that they do not separate until such tension is released, i.e., after they have been urged toward the receiver, whereupon they immediately separate and are then collected. In one embodiment, freshly formed melt-spun synthetic organic filaments are charged and are simultaneously oriented with a pneumatic jet, the action of which also serves to forward the charged filaments to the receiver.

The invention will be more readily understood by referring to the attached drawings, wherein

In FIGURE 1, A and B show schematically alternative apparatus assemblies useful in producing webs of the invention from freshly spun and lagged yarn, respectively;

FIGURE 2 shows a modification of the FIGURE 1 apparatus;

FIGURE 3 shows in longitudinal section a pneumatic jet which may be used in combination with the apparatus of FIGURES 1 and 2;

FIGURE 4 shows another modification of the apparatus of FIGURE 1;

FIGURE 5 shows in longitudinal section the pneumatic jet used with the apparatus of FIGURE 4;

FIGURE 6 shows a modification of the apparatus of FIGURE 1 using an alternative charging means;

FIGURE 7 shows schematically further alternative apparatus assemblies wherein the filaments are drawn either mechanically with draw rolls or by a pneumatic jet and wherein the filaments are electrostatically charged with a corona discharge device;

FIGURE 8 shows schematically in longitudinal section, the nozzle portion of a pneumatic jet which may be used with either embodiment of the apparatus in FIGURE 7;

FIGURE 9 shows schematically an optical apparatus suitable for the determination of randomness of nonwoven sheets;

FIGURES 10A and 10B are densitometer traces respectively of webs A and E made as described below in Examples III and VII respectively; and

FIGURES 11A and 11B are enlarged photographs of webs whose traces are shown in FIG. 10A and FIG. 10B. FIGS. 11C and 11D are enlarged photographs of webs G and F, respectively, prepared as described below in Examples IX and VIII respectively; and

FIGURE 12 shows graphically the relationship between the tensile strength and the coefficient of variation of filament separation distances for a series of nonwoven webs having the same filament strength.

FIG. 13 is a schematic representation of a web of the invention 60 containing both matrix filaments 50 and binder filaments 51.

FIG. 14 is a side view schematic representation of a web of the invention 60 impregnated with a waterproofing composition 61.

FIG. 15 is a schematic representation of a web of the invention 60 laminated to a self-supporting film 70.

Referring to embodiment A of FIGURE 1, freshly formed filaments 1 are spun through spinneret 2 and pass freely rotatable, i.e., nonsnubbing idler roll 3, whereupon the filaments 1 are converged into yarn or bundle 4. Yarn 4 then is pulled through pneumatic jet 5, which is continuously supplied with air under pressure through air inlet 6, making triboelectric contact with the tapered inlet section or throat 7 thereof. Optionally, the filaments 1 may pass directly to pneumatic jet 5 without prior convergence to a yarn provided that they (the filaments) make sufficient triboelectric contact with throat 7 of pneumatic jet 5 (i.e., provided that the spinneret 2 and pneumatic jet 5 are not disposed in-line with respect to one another). The pneumatic jet 5 (and hence the throat 7 portion thereof) is electrically grounded through lead 8. The charged filaments 9 issuing from pneumatic jet 5 are collected as sheet 10 on receiver 11 which, in this embodiment, is grounded through lead 12. The repelling effect due to the charge on the filaments 9 exiting pneumatic jet 5 is indicated diagrammatically by the arrow 13 emanating from within the filament region.

Alternatively, as shown in embodiment B of FIGURE 1, yarn 4 may be supplied to pneumatic jet 5 from a package 14, prior to which the yarn has been rendered receptive to charging (i.e., in a relatively anhydrous condition free from charge-diminishing contaminants or finishes). Preferably, the yarn is taken off the side of the package to minimize twisting of the yarn, which otherwise would inhibit subsequent filament separation. In either embodiment (FIGURE 1; A or B), roll 3 need not be freely rotatable, rather it might be fixed to provide a degree of snubbing. The roll 3 also might be replaced by a bar or the like for the same purpose.

FIGURE 2 shows a modification of the FIGURE 1 apparatus wherein the yarn is charged triboelectrically by contact with guide 15 located intermediate roll 3 and pneumatic jet 5 (shown fragmentarily). Guide 15 is com-

posed of a material which is capable of producing sufficient charge on the filaments in yarn 4 to separate the filaments from each other and maintain that separation until the strand strikes the receiver. Guide 15 is located above jet 5 so that the as-charged yarn enters the jet axially. Guide 15 may be slowly rotated and/or traversed to reduce surface wear; it can be a circular pin as shown or may be a bar or the like. A certain degree of snubbing takes place on passing guide 15, depending on the coefficient of surface friction and the angle of wrap made by the yarn over the surface thereof. Additional snubbing would result from fixing roll 3 or its equivalent as earlier described.

FIGURE 3 shows in longitudinal section a pneumatic jet which can be used with the apparatus of FIGURES 1 and 2. Jet 5 is assembled from components 5a, 5b, and 5c with cap screws (not shown). The assembled jet consists of essentially cylindrical yarn passageway 19 (the extension 19a of which is shown fragmentarily) which is outwardly flared toward filament inlet 16 in entrance section 5a to form a guide throat 7. Air under pressure is supplied through inlet 6 to the plenum 18 and enters filament passageway 19 through the annular slit 17. In the present embodiment, the air passing through slit 17 encounters the filaments at an angle of about 15° thereto, whereby a forwarding motion is imparted to the filaments. The composition of entrance section 5a (hence guide throat 7) is important to over-all process results; in the present embodiment, entrance section 5a is readily interchangeable.

In operation with any of the above-described apparatus, the yarn, i.e., the filament bundle, is forwarded from the supply means and urged to the receiver means by the action of the pneumatic jet. In the case of freshly spun filaments, the pneumatic jet (or equivalent forwarding means) is located beyond the point where the filaments are substantially completely solidified or "quenched," as are usually the associated guide means unless they are of the non-snubbing variety. These precautions prevent sticking of the individual filaments. Simultaneously, the individual filaments are charged to a high potential, positive or negative, depending on the yarn and guide compositions, by virtue of their triboelectric contact therewith. A similar charging effect results from maintaining a corona discharge in the upstream vicinity of the pneumatic jet. Accordingly, as the filaments issue from the jet and are urged toward the receiver means, they immediately separate, owing to the forces of electrostatic repulsion. Partly due to the impetus received at the jet and partly due to the attraction of the filaments toward the grounded or oppositely charged receiver, they are deposited on the receiver as a compact unitary structure, i.e., as the desired nonwoven batt, sheet, web, or the like.

Referring to FIGURE 4, freshly formed filaments 1 are spun through spinneret 2, pass as shown over bar guides 20, 21 and 22 thence to pneumatic jet 5 supplied with air under pressure through inlet 6. Pneumatic jet 5 embodies extended filament passageway extension 19 flared outwardly at the terminus 23. The charged filaments 9, which separate on exiting the extension of jet 5, are collected on receiver 11, an aluminum plate. The various components downstream from spinneret 2 are grounded through leads 12. The guide bars 20, 21 and 22 are 1 in. x 1 in. with rounded edges and are composed of chromic oxide. Guide bar 21, i.e., the functional surface thereof, is offset from the filament line by 2.5 in. Pneumatic jet 5 is drawn in greater detail in FIGURE 5 wherein the reference numerals have substantially the same significance as those in FIGURE 3.

FIGURE 6 schematically illustrates another and highly desirable embodiment which involves the use of corona discharge as the electrostatic charging means. Molten polymer is extruded through a multi-hole spinneret 2, in the form of fine filaments 1. The continuous filaments pass through an electrostatic charging zone consisting of

a set of corona discharge points 24 supplied with a high voltage by source 25, together with a target electrode 26, which is grounded. The charged filaments pass through a pneumatic jet 5, supplied with air at 6, which forwards them toward a collecting mechanism. The action of the pneumatic jet establishes positive tension on the filaments, causing them to undergo molecular orientation by drawing in the space between the spinneret and the jet. As the filaments emerge from the outlet 23 of the jet, the like electrical charges carried by the individual filaments cause them to repel one another and deposit on the collecting device in random, individual, nonparallel disposition. The collecting device shown consists of an endless belt 11 running over rollers 27, and backed up by a grounded electrode 28. The deposited filaments form a web 10 which passes through consolidating rolls 29 and then on to optional later processing steps, such as bonding, coating, laminating, embossing, etc.

Chargeable continuous synthetic organic filaments which are useful for the purpose of this invention include those comprised of polyamides, such as poly(hexamethylene adipamide), polycaproamide and/or copolymers thereof; polyesters, such as poly(ethylene terephthalate), poly(hexahydro-p-xylylene terephthalate), and/or copolymers thereof; polyhydrocarbons, such as polypropylene and polyethylene; polyurethanes, polycarbonates, polyacetals, polyacrylics, vinyl polymers, vinylidene polymers, and the like. Filaments of different polymers may be charged and laid on the receiver simultaneously. Preferred polymers are the melt-spinnable ones (see FIG. 1A) which can be processed from polymer to nonwoven web in a single continuous operation. Otherwise the filaments usually require preparation prior to charging, etc., by drying, removal of solvent, possible adjustment of finish, and the like.

Depending on the particular mode of filament preparation, the individual filaments may exhibit a high level of crimp superimposed upon the random arrangement of each filament within the sheet. The concept of filament crimp is understood in the art. In a filament crimp the amplitude of the departure from a straight line is less than 3 times the radius of curvature of the crimp, the latter being always less than 0.5 inch. The presence of crimp in the filaments can contribute to the utility of the sheet. For example, finished structures based on sheets wherein the individual filaments exhibit crimp at levels in excess of about 30 crimps per inch are useful in apparel applications, owing to their enhanced softness and drapability. At crimp levels in excess of about 100 crimps per inch, the effect is especially pronounced. At crimp levels less than about 30 crimps per inch, the articles are stiffer, hence are best suited for the more demanding industrial applications, e.g., tarpaulins. Crimp enhances the stability of the sheets and contributes to improved covering power.

Crimped filaments can be obtained by orienting the filaments immediately subsequent to the preparation thereof. Representative of such a process is the one described in Hebeler, U.S. Patent 2,604,689. Variations of this basic procedure are applicable to melt-spun filaments generally; the process is termed "spin-drawing." It is especially useful with filaments of poly(hexamethylene adipamide), polycaproamide, and poly(ethylene terephthalate), including copolymers thereof. In the case of most spun-drawn polyamides, the crimp develops spontaneously after a few minutes standing. The development of crimp is accelerated by heat and/or moisture, i.e., by relaxing the filaments. In the case of poly(ethylene terephthalate), or the like compositions, a distinct relaxation step is required, during which the filaments shrink, crimp develops and, in many instances, the property of spontaneous extensibility is achieved (see Kitson and Reese, U.S. Patent 2,952,879). Relaxation can be effected as a separate operation apart from sheet preparation by heating the sheet, or during sheet formation proper by heat

ing (steam, hot air, or infra-red radiation) the filaments within or downstream from the pneumatic jet. The filaments may be collected on a hot water bath to effect relaxation simultaneous with collection. In the case of filaments supplied in accordance with FIGURE 1B, the filaments may already be crimped, and so long as such crimp does not impede filament separation, the method is a satisfactory one. Crimp can also be obtained in filaments by the process described in Kilian, U.S. Patent 3,118,012, or by the use of two-component filaments as disclosed in Breen, U.S. Patent 2,931,091. Crimp also is obtainable in filaments composed of thermoplastic polymers by the deformation thereof over a sharp surface such as a blade or edge over which the filaments make an acute angular pass. The development of crimp in such products also is enhanced by relaxing conditions. Other crimping procedures may also be employed for other polymer compositions, such as polyhydrocarbons, e.g., polypropylene. The presence of crimp in the filaments tends to cause filament entanglement and may require more careful control.

The sheets and webs of this invention may be made in varying densities. High-bulk, low-density webs, for example, are particularly useful for many nonapparel uses. Fabric densities below 0.1 g./cc. may be prepared, especially when highly crimped fibers are present. Higher density materials, in the range 0.2 to 0.5 g./cc., are useful, while high-density webs 0.5 g./cc. and higher, are also valuable.

DETERMINATION OF COEFFICIENT OF VARIATION OF FILAMENT SEPARATION DISTANCES (CV_{fs})

In order to measure the distance between filaments in a nonwoven web, it is often necessary to section the structure longitudinally. This may be done with unbonded webs by simple delamination; however, with bonded webs, this is not satisfactory since the initial structure is disturbed in the delamination procedure. Satisfactory sections can be obtained by a technique which involves imbedding a 2 in. x 0.5 in. sample of web in a curable epoxy resin composition. After curing overnight, the sample can be sliced longitudinally with a microtome into sections 30 to 40 microns thick. This method has been found to be satisfactory for both bonded and unbonded webs. The distances between the filaments are then measured with a projection microscope set at 100× magnification for filaments having a denier of 4 or less and at 50× magnification for filaments having a denier greater than 4. Separation distances are measured along a line which covers at least 2 in. of web; preferably however, at least 3 in. of web are scanned in which case it is necessary to imbed two samples of the web. The filament segments involved in the count are those which are perpendicular within $\pm 2^\circ$ to the line of count. At least 200, and preferably 400 filaments are counted in order to characterize a given sample. The precision of the coefficient of variation which is calculated from the filament distances is of the order of $\pm 3\%$.

DETERMINATION OF BUNCHING COEFFICIENT (BC)

The bunching coefficient concept is based on the premise that where individual fibers, disposed in the same direction in a nonwoven web, are uniformly spaced from each other, each "fiber space" will contain one fiber. This concept was developed by D. R. Petterson, and is described in his Ph.D. Thesis, "On the Mechanics of Nonwoven Fabrics," presented to the Massachusetts Institute of Technology in 1958.

The basic equation is:

$$BC = \frac{\text{Number of "fiber spaces" occupied by fibers}}{\text{Total number of "fiber spaces" available}}$$

Where all fiber elements are completely parallel, and exactly uniformly spaced, the bunching coefficient is unity. The actual bunching coefficient may be determined by taking a photograph of the web, ordinarily of a sample not greater than 5 mils thick, and counting the number of fibers crossing a given line segment at right angles to that line (using an angular tolerance level of not over 2° in considering or not considering each fiber). The total number of fibers counted is equal to the total number of "fiber spaces" in that line segment. The average "fiber space" width is calculated by dividing the segment length by the number of fibers. A scale is now constructed with unit distances equal to the average "fiber space" width. With this scale, the number of fiber spaces occupied by at least one fiber is determined. For accurate results, measurements are made in several directions, and averaged.

DETERMINATION OF RANDOMNESS

As indicated hereinabove, the most precise and preferred test for randomness will determine the actual orientation or direction in which the component filaments lie within the plane of the nonwoven sheet. The method described by J. W. S. Hearle and P. J. Stevenson in the Textile Research Journal, November 1963, pp. 879-888, determines the randomness of a nonwoven sheet. This method requires the counting and plotting of a large number of filaments in order to obtain accurate and reproducible results and is, therefore, very time-consuming. It is further noted that, whereas the actual visual measurement of filament orientation is readily applicable to nonwoven sheets in which the fibers are predominately straight, it is not as satisfactory for sheets in which the fibers are curved or crimped.

Instead of counting the number of filaments oriented at the various directions within the nonwoven sheet, it has now been found that a randomness measurement can be obtained by determining the total length of the filament segments that are oriented at the various directions throughout the sheet. In a random sheet, the total length of filament segments at any one orientation is the same as at any other orientation. This measurement has the advantage that it is universally applicable to straight, curved, or crimped fibers.

It has been found that the measurement of the length of filament segments at the various orientations can be made rapidly and accurately by an optical method. The method is based on the principle that only the incident light rays which are perpendicular to the fiber axis of a round fiber are reflected as light rays which are perpendicular to the fiber axis. Hence, by focusing a beam of parallel light rays on a nonwoven sheet at an incident angle less than 90° , e.g., 60° , the light which is emitted perpendicular to the plane of the sheet comes only from filaments having an orientation within the plane of the sheet which is perpendicular to the incident light rays. By collecting and measuring photoelectrically the intensity of the light, the total length of the filament segments perpendicular to the light rays, therefore, parallel to each other, can be determined. By rotating the sheet, the parallel filament segments for any given direction can be measured and from this measurement, an analysis of the randomness can be made.

An apparatus suitable for this measurement is shown schematically in FIGURE 9 and will hereinafter be referred to as a randomometer. A detailed description of the components, the method of operation, and the method for standardizing the characterizations are given below.

As shown in FIGURE 9, the apparatus has a revolving stage 46 on which the sample 47 to be examined is placed. Stage 46 is modified by gear 48 which has half the teeth removed so that when driven by synchronous motor 49, it rotates only 180° . Stage 46 rotates at $\frac{1}{4}$ r.p.m., thus the time for rotation of the sample through 180° is 2 minutes. Lamp 50 is located directly over the

sample and in line with magnifying lens system 51. Lamp 50 is a 6-volt lamp and its intensity is controlled through 6-volt transformer 52 and variable-voltage transformer 53. The light from 50 is focused by lens 51 onto the bottom of the sample, and when projected through objective lens 54, eyepiece 55 and reflected from mirror 56, gives a shadow of the sample on ground-glass screen 57 at a magnification of 36 \times . Screen 57 is circular and has a diameter of 6.9 inches.

A second lamp 58 is mounted in a housing with projection lens 59 to focus the light on the sample at an angle of 60°. Lamp 58 is a 25-watt, concentrated arc lamp receiving its power from power supply 61 which is modified to eliminate the A.C. ripple. The filaments or segments of filaments which are perpendicular to the light from lamp 58 reflect the light into the magnifying lens and mirror system to screen 57 for measurement. Optical slit 62 is located between the objective lens 54 and stage 46 and serves to control the limits of the light reflected from the sample. The slit is $\frac{1}{16}$ in. \times $\frac{3}{8}$ in. and is mounted with its long axis parallel to an imaginary line which is perpendicular to the light from lamp 58 and within the plane of the sample.

The light from the screen is focused by Fresnel lens 63 onto photomultiplier tube 64 RCA type 1P21) having a 2500-volt D.C. power supply 65. The screen, Fresnel lens, and photomultiplier tube are contained in a single light-tight unit, which can, however, be opened for visual observation of the screen. The output from the photomultiplier tube is fed into a microampere recorder 66 having a chart speed of 8 in./min. and a chart 9.5 in. wide. The chart records the light reflected from the parallel filaments at each direction as the sample is rotated through 180°. The sensitivity of recorder 66 should be adjusted so that a current of 6 microamperes gives 100% pen deflection.

A two-way switch 67 is in the line from the photomultiplier tube to the recorder so that the signal can be measured on a sensitive microampere meter 68, if desired. This meter can also be used in conjunction with a 6-volt lamp of fixed intensity to measure the fiber density of the sample so that, if desirable, all samples can be compared on the same basis.

Samples of the nonwoven sheet to be examined are preferably unbonded and should permit clear viewing on the randometer of all the filaments through the thickness of the samples. A preferred basis weight range for sheets of 3 denier filaments is 0.75–1.5 oz./yd.². Samples in excess of 1.5 oz./yd.² should be delaminated to fall within the range stated, but care should be exercised to avoid the introduction of directionality due to the delamination. The delaminated specimen should be representative of the total thickness. The sample is placed between two microscope slides which are then taped together. The slide is placed on the revolving stage so that the light from lamp 58 shows on the sample. The background lamp 50 is then turned on and the filaments are focused as sharply as possible by moving revolving stage 46 up or down, while they are viewed on the screen. Lamp 50 is then turned off. Stage 46, lamp 58 and projection lens 59 are enclosed in a light tight unit. The voltage of power supply 65 is adjusted to give about 5 in. pen deflection and the intensity of the reflected light is recorded on the microampere recorder chart as the sample is rotated through 180°.

The heights of the intensity-orientation curve so obtained are measured in inches from the zero line of the chart at 80 equally spaced orientations and the arithmetic mean of these heights is determined. To standardize the randometer characterization, each of the 80 readings is multiplied by the factor

$$\frac{5}{\text{Arithmetic mean}}$$

to shift the curve to a standard mean (5 in.). The standard deviation of these 80 corrected readings from this standard mean is then calculated. A "perfectly" random sheet would have a standard deviation of zero when the reflected light is measured at all orientations. As used herein, a random sheet is defined as one having a standard deviation of 0.6 in. or less, when determined by the above-described method. To improve the precision of the measurement, several samples selected from throughout the sheet may be examined and the results averaged. The presence of filament bundles in the nonwoven sheet can unduly affect the randomness values and therefore the values lose their significance with sheets having a CV₁₅ above 100%.

The following examples are illustrative of the invention.

Example I

Using an apparatus assembly essentially as shown in FIGURE 1A, omitting idler roll 3, poly(hexamethylene adipamide) (39 relative viscosity) is spun through a 34-hole spinneret (each hole 0.009 inch in diameter) into filaments at a rate of 16 grams total polymer per minute, at a temperature of 290° C. The filaments are spun into a quiescent atmosphere at ambient temperature (25° C.) and relative humidity (70%). Downstream (ca. 30 inches) past the point of solidification and about 6 inches laterally from the normal filament line, a pneumatic jet (see FIGURE 3) of the following dimensions is placed:

- inlet diameter, $\frac{15}{16}$ inch
- filament passageway diameter, $\frac{3}{32}$ inch
- inlet cut-down to minimum diameter occurs over $\frac{3}{4}$ inch
- filament passageway length, $15\frac{1}{2}$ inches
- angle of air entry (below inlet), ca. 15 degrees.

The jet, which is grounded, has inlet section or throat 7 composed of aluminum; the body of the jet is composed of brass. The filaments make triboelectric contact with the throat of the jet. The receiver is a 12 in. \times 12 in. aluminum plate which is manually manipulated (hence grounded). Filaments are collected into hand sheets by interposing the receiver into the filament line and rotating the same until a sheet of the desired thickness and configuration is obtained. The results of several such runs are summarized in Table I.

TABLE I

Run	Air Pressure (P), p.s.i.g.	Filament		
		Denier	T/E ¹	Mi ²
1	5	6.0	1.9/408	6.0
2	15	1.9	2.8/201	6.7
3	25	1.6	3.6/190	9.0
4	35	1.5	3.5/172	9.5
5	45	1.7	3.6/142	8.8

¹ Tenacity (T), grams per denier/Elongation (E), percent.

² Mi equals initial tensile modulus, g.p.d.

In all runs, process operability was very good, uniform sheets with good filament separation being produced. Similar sheets are obtained at good levels of operability when the polymer used in the above runs is polycapromamide.

Example II

Sheets are prepared from poly(ethylene terephthalate) using the apparatus shown in FIGURE 4. Referring to that drawing, filaments 1 are spun from spinneret 2 and pass in the manner shown over the bar guides 20, 21 and 22, thence to the pneumatic jet supplied with air under pressure through inlet 6. The filament passageway extension 19 is flared outwardly (6°) at the terminus 23. The charged filaments 9 which separate on exiting the extension of the jet, are collected on an aluminum plate receiver 11. The various components downstream from

spinneret 2 are grounded through leads 12. The pertinent distances along the filament line are as follows:

$a=17$ inches
 $b=19$ inches
 $c=22.5$ inches
 $d=25.5$ inches
 $e=\text{ca. } 4$ inches
 $f=48$ inches
 $g=7.5$ inches
 $h=12$ inches.

The filaments are quenched with air, applied 6 inches below the spinneret face. The guide bars 20, 21 and 22 are 1 in. x 1 in. with rounded edges and are composed of chromic oxide. Guide bar 21, i.e., the functional surface thereof, is offset from the filament line by 2.5 in. Pneumatic jet 5 is shown in greater detail in FIGURE 5. The important dimensions of the jet are:

inlet diameter, ca. $\frac{3}{4}$ inch
 filament passageway diameter, 0.05 inch
 inlet angle, 60°
 angle of air entry, 5°
 entry $1\frac{3}{8}$ inches below inlet.

The entire jet assembly is fabricated from brass.

In operation, poly(ethylene terephthalate) (34 relative viscosity) is spun through a 30-hole spinneret at a rate of 10 grams (total) polymer per minute. Each spinneret hole is 0.007 inch in diameter. The spinning temperature, measured at the spinneret, is 287°C . The following results are obtained:

TABLE II

Run	Air Pressure (P), p.s.i.g.	Filament Properties			
		Tenacity, g.p.d.	Elong., percent	Mi, g.p.d.	Denier
1.....	40	2.0	185	14.5	1.72
2.....	50	2.4	148	16.2	1.37
3.....	70	3.5	101	29.9	1.10
4.....	80	3.5	104	28.0	1.03
5.....	90	3.1	77	26.7	0.97

In all of the runs reported in Table II, process operability is good, as is sheet formation. The resulting sheets are substantially free from aggregated filaments, i.e., filament separation subsequent to charging is wholly satisfactory. Note that increasing air pressure results in a corresponding increase in the speed at which the filaments are delivered to the receiver; filament speeds increase from ca. 2000 yards per minute in run 1 to ca. 3540 yards per minute in run 5.

When each of the above runs is repeated except that atmospheric steam at about 150°C . is applied to the separated filaments downstream from the pneumatic jet, using a foraminous member disposed annularly with respect to the filaments, the filaments relax up to 20% or more with concomitant development of crimp. Upon later hot calendaring, the filaments in the sheet elongate spontaneously, thereby further contributing to the crimp level in the individual filaments and hence to the properties of the sheet.

When each of the above runs is repeated except that the filaments are collected in 75°C . water, the filaments again relax, leading to the development of crimp up to levels of 50 or more crimps per inch (based on in situ examination). The filaments also spontaneously extend upon subsequent treatment at elevated temperatures. The filaments also may be caused to relax by employing a heated gas in the pneumatic jet or in a relaxing chamber downstream from the jet.

Examples III-VII

In these five examples, nonwoven webs of continuous filament polyester fibers were prepared under varying conditions of web-deposition. Process variables were controlled to give sheets with different degrees of filament bunching, as evidenced by bunching coefficients ranging from high (0.83) to low (0.57) and CV_{fs} of 86 to 122%. Insofar as possible, other variables were eliminated.

After the webs were prepared, characterization measurements of CV_{fs} and bunching coefficient were made. The webs were then bonded by application of a copolyester binder solution, and physical properties of the bonded sheets were measured.

Web preparation followed the same general procedure as that described in the earlier examples, except that corona charging was employed as shown in FIGURE 6. Molten poly(ethylene terephthalate) was spun through a 17-hole spinneret at the rate of 0.88 g./hole/minute into a corona discharging area, through a jet and thence onto a receiver plate. Spinneret temperature was 288°C ., and ambient air temperature was 25°C . The charging area was 72 inches below the spinneret face. The pneumatic jet was substantially the same as that used in the preceding example and was located 4 inches below the charging area. Air at 60 p.s.i. was used. The tailpipe or exit tube was 18 inches long. The receiver was a reciprocating table and was charged opposite to the charge on the fibers. Charging conditions are given in Table III. This table also shows results obtained with webs of Examples VIII and IX, made in the manner described below.

TABLE III.—PREPARATION AND CHARACTERIZATION OF CONTINUOUS FILAMENT WEBS

Web Code.....	Example No.						
	III	IV	V	VI	VII	VIII	IX
	A	B	C	D	E	F	G
Corona Charge Volts, kv.....	30	28	23	14	11	None	None
Bunching Coefficient.....	0.83	0.78	0.73	0.63	0.57	0.47	0.43
CV_{fs} (percent).....	86	90	93	122	121	(1)	(1)
Filament Properties:							
Denier.....	2.4	2.4	2.4	2.3	2.4	8.7	12.9
Tenacity (g.p.d.).....	2.9	2.9	3.0	2.9	3.0	1.2	1.0
Elongation (percent).....	141	122	127	125	117	487	563
Web Properties:							
A 6% binder ²							
Tensile strength ³	12.1	11.4	11.2	10.5	7.9	-----	-----
Elongation (percent).....	78	76	74	72	57	-----	-----
B Percent Binder ⁴	12.2	-----	12.7	12.9	-----	13.4	13.6
Tensile strength ³	10.4	-----	12.6	9.0	-----	1.6	1.8
Elongation (percent).....	88	-----	94	69	-----	2	2

¹ Webs similar to Examples VIII and IX were found to have CV_{fs} of 135 and 186%, respectively.

² Binder applied as a 25% solution in methylene chloride.

³ Lb./in./oz./yd.²

⁴ Binder applied as a 10% solution in a 2/1 dimethylformamide/dioxane mixture.

The examples immediately above are concerned with nonwoven webs prepared by electropneumatic spinning processes. The present examples concern similar studies on webs made by prior art processes.

Web F was deposited by passing a strand of polyester filaments through an air jet and deflecting the fibers onto a collecting table, employing an angularly disposed deflector plate. No electrostatic charge was given to the fibers. Web G was deposited directly from an air jet onto the collecting plate without charging. Table III shows the characterization and testing results of the sheets. FIGURES 11C and 11D are photographs of the webs, G and F, respectively.

After the representative webs were prepared, bunching coefficients were measured. The sheets were bonded by the application of a copolymer of poly(ethylene terephthalate) and poly(ethylene sebacate) (55/45 mole ratio) as binder. The binder was applied by dipping the web into the binder solution, letting the excess binder drip off, and air drying. Physical properties were then measured using an electronic tensile testing machine (Instron tester). Results of the measurements are given in Table III.

The results of Table III are, of course, of the highest significance. Webs A through C were produced under preferred process conditions. They had CV_{fs} of below 100% (bunching coefficients in the range of 0.7 or higher). All of these webs had excellent physical properties when bonded.

In addition to the forgoing tests, evaluations were made of the optical uniformity of the webs before bonding by various techniques. In one method the webs were photographed by transmitted light, and also densitometer traces of the webs were made. FIGS. 11A and 11B are reproductions of enlarged photographs of webs A and E. FIGURES 10A and 10B show densitometer traces of the same webs. The tremendous differences in appearance of these webs show that degree of filament bunching is a significant measure of web uniformity. The densitometer traces serve to emphasize further what is readily apparent to the eye.

The densitometer traces referred to above were obtained using a Leeds and Northrup No. 6700-P.I. Recording Microphotometer. The nonwoven web was mounted between glass plates to constitute the specimen. A record was obtained of the light transmission in several transverse across the web. The figures shown are representative traces replotted to show the light transmission on a linear rather than on a logarithmic scale.

Another method for evaluation of nonwoven webs for optical uniformity yields numerical values thus permitting a more precise comparison among various webs. In this method, an instrument consisting of a light source, sample mount, optical system for focusing the sample image on the aperture of a photo cell, amplifier and recorder is used to measure the intensity of light transmitted by the web. Opacity uniformity is measured by scanning, at a rate of 6 in./min., four 10 in. sides taken from a square sample of unbonded web, using a 0.4 in. diameter aperture, which, because of the optics of the system, is equivalent to a 0.1 in. diameter scan area on the web. The uniformity of transmitted light intensity is characterized by the coefficient of variation in transmitted light intensity (CV_{th}) at 50 equally spaced points on the recorder chart for each 10 in. length of sample scanned. The results obtained when webs A through E in Table III were evaluated by this method are summarized in Table IV.

The randomness of the unbonded webs A through C in Table III was determined with the optical randomometer and the results are given in Table IV.

TABLE IV

Example	Web Code	Percent CV_{fs}	Percent CV_{th}	Randomness Standard Deviation (in.)
III	A	86	14	0.4
IV	B	90	14	0.4
V	C	93	15	0.5
VI	D	122	33	
VII	E	121	26	

An excellent correlation is shown to exist between optical uniformity, as measured by CV_{th} , and degree of filament bunching, as measured by CV_{fs} . Uniformity of appearance deteriorates rapidly at levels of CV_{fs} above 100%.

Example X

In this example a series of nonwoven webs is produced while maintaining all the operating conditions constant except for the level of electrostatic charge applied to the filaments.

The apparatus assembly used in this example is shown schematically in FIGURE 7, wherein the filaments pass directly, as indicated by the dotted lines, from the spinnerets to the target bar of corona discharge device 30. Quench chimney 31 and guide roll 3 are not used in this apparatus embodiment. Poly(ethylene terephthalate) (27 relative viscosity) is spun through spinneret 2 having 17 holes (0.009 in. diameter x 0.012 in. long) at a total throughput of 18.4 g./min. while an 80/20 copolymer of poly(ethylene terephthalate)/poly(ethylene isophthalate) (29 relative viscosity) is spun through spinneret 2a having 20 holes (0.009 in. diameter x 0.012 in. long) at a total throughput of 13.0 g./min. The spinneret temperatures are 271° C. and 263° C., respectively. Four of the copolyester filaments are used and the other 16 are spun to waste. The filaments are quenched in the ambient air at 27° C. before entrance into a draw jet 5 located about 65 inches below the spinnerets. The 21 filaments from the two spinnerets are combined into a filament bundle at the target bar of corona discharge device 30 which is located about 6 inches from the jet inlet.

The corona discharge device consists of a 4-point electrode positioned 5/8 inch from grounded, 1 1/4 inch diameter, chrome-plated target bar rotating at 10 r.p.m. A negative voltage is applied to the corona points and is varied between 0 and 45 k.v. to vary the level of charge on the filaments. The filament bundle makes light contact with the target bar as it passes between the target bar and electrode. The level of charge is measured by collecting filaments exiting from the jet in a calibrated pail coulometer and is expressed as c.g.s. electrostatic units (e.s.u.) per square meter of filament surface.

The filaments are drawn and forwarded toward the laydown belt by a pneumatic jet 5 having a nozzle section as shown in FIGURE 8 and having the following dimensions:

	In.
60 Over-all jet length	24
Filament inlet diameter (16)	0.062
Filament inlet length (16)	0.55
Filament passageway (19) minimum diameter	0.093
65 Metering annulus 32:	
Inner diameter	0.0750
Outer diameter	0.0930
Length	0.020

70 Air at a pressure of 51 p.s.i.g. is supplied to the jet, which under these conditions applies about 13.5 grams total tension to the filament bundle. Attached to the bottom of the jet is a relaxing chamber (9 1/2 in. long; 3/8 in. inside diameter) which is provided with an annular nozzle for 75 supplying additional air to the relaxing chamber. In this

example, hot air is not supplied to effect heat-relaxation of the filaments, but room temperature air is added at 4.8 s.c.f.m. to maintain nonturbulent flow in the relaxing chamber.

The jet-relaxing chamber unit is positioned at an angle of 82° with the plane of laydown belt 11 and is moved by traversing mechanism 33 so that it generated a portion of the surface of a cone, while the output from the relaxing chamber forms an arc on the laydown belt having a chord length of 36 inches. The traverse speed is 30 passes (15 cycles) per minute. The distance from the exit of the relaxing chamber to the laydown belt is approximately 30 inches. The laydown belt moves at a speed of 12.5 inches per minute. Plate 34 located beneath the belt is charged at +35 kv., to pin the filaments to the laydown belt. The properties of the homopolymer fibers are: denier 2.5 d.p.f. (0.3 tex.); tenacity 3.2 g.p.d.; percent shrinkage (when heated at water at 70° C. with no restraint), 35.9.

The random nonwoven web so prepared is consolidated by passing between a heated roll (80° C.) and an unheated roll under light pressure. Samples of the consolidated but unbonded webs are retained for measurement of CV_{fs} and CV_{th} and other samples (8 in. x 8 in.) of the consolidated web are bonded individually by heating them in a laboratory press at 220° C., 5,000 lbs. total pressure, for 30 seconds between two polytetrafluoroethylene-coated grooved plates. The plates have 24 grooves per inch and are placed with the grooves at right angles to each other. The total pressure area between the land areas of the two plates is 4%.

The tensile strengths and formation values of the bonded sheets are determined. Formation value, designated FV, an alternative to CV_{th} for expressing degree of uniformity of the nonwoven sheets, is measured with a Paper Formation Tester (M. N. Davis et al, Technical Association of the Pulp and Paper Industry, Technical Papers, Series 18, 386-391 (1935)). As a standard for determination of FV, a suitable number of sheets of 1 oz./yd.², onion-skin paper are combined to give a basis weight within 0.5 oz./yd.² of the samples to be examined.

Table V summarizes the charging condition, levels of charge obtained on the filaments and properties of the unbonded webs and bonded sheets.

TABLE V

Sample	Electrostatic charge		Percent CV_{fs}	Percent CV_{th}	FV	Tensile Strength, lb./in./oz./yd. ²	Randomness, Standard Deviation (in.)
	Voltage Applied (kv.)	Charge on Filaments (esu/m. ²)					
A-----	45	76,200	74	15.3	200	13.8	0.5
B-----	40	57,300	84	16.5	160	12.2	0.4
C-----	35	45,000	93	19.4	160	12.1	0.2
D-----	30	33,900	102	23.0	120	12.1	
E-----	25	22,200	104	32.0	85	10.8	
F-----	20	12,900	106	33.4	82	9.1	0.5
G-----	15	1,700	121	38.5	65	7.1	
H-----	10		118	50.7	67	5.0	
I-----	0		115	51.2	70	5.5	
J-----	45	76,200	77	15.6	190	12.8	0.2
K-----	35	37,100	99	17.8	110	11.5	0.5

FIGURE 12 is a graph of the tensile strength and CV_{fs} data in Table V. Since these data are for nonwoven webs having the same filament strengths, the data are highly significant and the graph shows clearly that there is a rapid drop in tensile strength at a CV_{fs} above 100%. The data in Table V also indicate a significant and rapid deterioration of uniformity of appearance, as measured either by CV_{th} or formation value, at values of CV_{fs} greater than 100%, the same critical level as that estab-

lished by the tensile strength data. Wide fluctuation in uniformity and unsatisfactory product appearance are obtained with webs having a CV_{fs} greater than 100%.

Data on randomness of the nonwoven webs are also included in Table V and indicate that the webs of this invention meet the previously defined limit for random webs.

Example XI

The data in Example X, and also in preceding Examples III through VII, were obtained on webs prepared with filaments which shrink during the bonding step. For comparison a series of webs is prepared with spontaneously elongatable poly(ethylene terephthalate) filaments and copun binder filaments of an 80/20 copolymer of poly(ethylene terephthalate)/poly(ethylene isophthalate). The property of spontaneous elongation is obtained by supplying about 5 s.c.f.m. of hot air at a temperature of 500-600° C. to the relaxing chamber of the filament-forwarding jet device in Example X. The walls of the relaxing chamber are cooled by water as described in Cope, U.S. Patent 3,156,752. The webs are bonded as in Example X. The results are summarized in Table VI.

TABLE VI

Sample	Percent CV_{fs}	Tensile Strength lb./in./oz./yd. ²
L-----	88	6.9
M-----	92	4.9
N-----	106	2.8
O-----	131	2.3

These data confirm the importance of having a high level of uniform filament separation ($CV_{fs} < 100\%$) to obtain high strength levels with a given fiber tenacity.

Example XII

The suitability of nonwoven sheet samples A through K in Example X for use in window-shade cloth is determined by making pinhole counts. These counts are made on 3 1-in. square samples of each sheet. The sheets, which have constant filament cross-section and denier, are chosen to have basis weights of 4.5 ± 0.25 oz./yd.² An image of the

1-in. square sample is projected onto a screen at a linear magnification of 7 with a 35-mm. projector. The total number of pinholes (bright spots of light) is then counted. An alternative procedure is to project an image of a 1-in. square of sample onto photographic paper through an enlarger (7×) to obtain a permanent record of the sample. Pinholes lead to blackening of the paper and are counted directly using a Zeiss particle size counter. The data obtained are summarized in Table VII.

TABLE VII

Sample	Percent CV _{fs}	Pinholes
A-----	74	530
B-----	84	460
C-----	93	530
D-----	102	1,080
E-----	104	1,720
F-----	106	1,780
G-----	118	2,580
H-----	115	2,280
I-----	77	390
J-----	99	200
K-----		

Below a CV_{fs} of 100%, the number of pinholes is relatively small and the sheets are useful as shade-cloth materials. Above a CV_{fs} of 100%, the number of pinholes increases three to five-fold and no reasonable amount of masking with opaque coatings will produce a useful shade cloth.

Example XIII

The abrasion resistance of bonded nonwoven sheets from Example X is determined using the C.S.I.A. abrader. The conditions of this test are as follows: 1 in. x 2 in. sample size; abradant, 0; load, 5 lb./in.; 2 cycles/minute. The results are summarized in Table VIII.

TABLE III

Sample	Percent CV _{fs}	Cycles to Failure ¹	Minimum Cycles to Failure
A-----	74	1,160	557
B-----	93	940	427
C-----	102	570	243
D-----	106	270	57
E-----	121	340	37

¹ Average of 8 samples.

The data indicate the poorer abrasion resistance of the nonuniform sheets. The data on minimum cycles to failure are included to point out that certain areas of the non-uniform sheets fail quickly and result in failure for the entire fabric.

Example XIV

Bonded nonwoven sheets of continuous poly(ethylene terephthalate) filaments, having different degrees of uniformity of filament separation because of bunches of filaments in the structure, are imbedded in rubber and then evaluated in the standard air-wick test for acceptability as chafer materials in tubeless tires. The samples for evaluation are prepared by imbedding 1 in. x 3 in. pieces of fabric in rubber by a molding operation. The molded test sample is formed so that fabric ends extend from two edges of the sample. The sample is then mounted in a pressure apparatus which exposes one edge of the sample to pressure. Leaks are detected by coating the other edge with a detergent solution and looking for air bubbles. Three samples from each nonwoven sheet are tested with the following results:

Sheet	Percent CF _{fs}	Results
P-----	92	No failures at 100 p.s.i.
Q-----	106	One failure after 30 min. at 100 p.s.i.
R-----	121	One failure after 10 min. at 100 p.s.i.

Example XV

Nonwoven webs are prepared with poly(ethylene terephthalate) filaments which are spontaneously elongatable and 12% cospun binder filaments as in Example XI. The webs (3.5 oz./yd.²) are bonded by the procedure described in Example X. They are evaluated as substrates

for tenting materials by coating with 68% by weight of a formulation containing:

	Parts by weight
Butyl rubber (81,000–100,000 molecular weight) ---	100
Platy talc -----	67
Zinc oxide -----	5
Rutile titanium dioxide -----	10
Phthalocyanine green pigment -----	3

The coating is applied from a xylene medium at 20% solids. The coated sheets are evaluated for water repellency by a rain-impact test as follows: The sample (6" x 12" or larger) to be tested is placed over a funnel (5" x 5") shaped like a roofless house and having a ridge pole connecting symmetrically located apexes on 2 opposing sides. The apexes are 1 3/8 in. higher than the top edges of the other 2 sides. The sample is held in place by 2 clamps (weight 4 lbs. each) attached to the narrow ends of the sample. In order to prevent wicking of water 1/2 in.-wide vapor-impermeable tape is placed on the sample where it contacts the ridge pole and the edge of the funnel. Water is allowed to fall as drops on the mounted sample from a 5" x 5" tray positioned 60 in. above the sample. The tray has 90 holes positioned in a rectangular arrangement on about 1/2 in. centers. The total rate of flow of the water is 1.5 gal. per min. The water leaking through the sample into the funnel in 30 minutes is measured. The results are summarized below:

Sheet	Percent CV _{fs}	Rain, Impact Test (cc.)
S-----	98	12 (acceptable waterproofing).
T-----	110	17 (poor waterproofing).
U-----	116	111 (very poor waterproofing).

These data indicate the desirability of having uniform filament separation (CV_{fs} < 100%). Higher coating weights are required to obtain acceptable waterproofing when nonuniform sheets are used.

Example XVI

The apparatus of Example II is employed in the preparation of sheets composed of polypropylene filaments. The pertinent distances are the same except for the following (see FIGURE 4):

- a=18 inches
- b=22 inches
- c=30 inches
- d=38 inches

Guide bar 21 is offset from the filament line by 2 in. Polypropylene (10 melt-index) is spun at a rate of 6 grams per minute through a 30-hole spinneret, each hole being 0.009 inch in diameter. The temperature at the spinneret is 190° C. Uniform sheets are obtained. Using 19 p.s.i.g. air, the following properties are obtained in the individual filaments, as spun: tenacity, 2.35 g.p.d.; elongation, 369%; modulus, 17.9 g.p.d.; denier, 1.51.

Example XVII

Following the general teaching of Example XVI, sheets were prepared of continuous filament, predominantly isotactic polypropylene. Triboelectric charging was accomplished with 3 brass bars. Spinning conditions were adjusted to obtain high-tenacity filaments. A 30-hole spinneret with holes 0.015 inch in diameter was used to spin polymer having a melt index of 12.4. A two-stage mechanical drawing process was used. Other spinning variables are shown in Table IX below. After the webs were deposited, they were bonded by the application of 21–24% by weight of polyvinyl chloride in tetrahydrofuran solution. Fiber and fabric properties for two samples made as indicated are shown in Table IX.

TABLE IX

Web	Polymer throughput (g./hole/min.)	Draw Ratio	Fiber Properties				Fabric Properties Tensile Strength	
			Den.	T (g.p.d.)	E. (percent)	Mi	lb./in./oz./yd.	E. (percent)
XVII-1	0.1	5.5X	1.09	4.9	72	25	21	46
XVII-2	0.16	10.5X	1.65	7.8	26	47	33	26

Example XVIII

The following example illustrates co-spinning of poly-(hexamethylene adipamide) (39 relative viscosity) and a 10% (weight) polycapraamide copolymer (relative viscosity 45) thereof. The 66-nylon is spun from a 34-hole spinneret (0.009 inch hole diameter) at 16 total grams per minute at 290° C. The 66/6-nylon copolymer is spun from a 20-hole spinneret (0.009 inch hole) at 255° C. and the output from 2 holes (1.78 grams per minute) is combined with the 34 filaments of 66-nylon. The two spinnerets are located on 5.5 inch centers. The freshly spun filaments are passed over a grounded, polished aluminum bar located 40 inches below, parallel to and offset by 6 inches from the centerline of the spinnerets. A pneumatic jet as shown in FIGURE 3 is located 1 inch below the point where the filaments contact the bar. Air at 25 p.s.i.g. is supplied to the jet. The receiver, a 42 in. x 42 in. grounded aluminum plate, is located 40 inches below the pneumatic jet. The receiver is traversed at a speed of 280 inches per minute below the jet and is further traversed at a speed of 28 inches per minute in a direction perpendicular to the primary traverse. Collecting in this manner for about 8.5 minutes yields a uniform sheet having a 4-ounce per square yard basis weight. The spinning speed during this run, based on polymer throughput and final filament denier, is 2900 yards per minute. Typical properties of 66-nylon filaments prepared by this procedure are: T/E=3.5/165; Mi=7.5; denier per filament, 1.60.

During the process described, the filaments are charged triboelectrically as they pass the aluminum bar; they are oriented upstream of the pneumatic jet, partly at the bar and partly by a spin-draw mechanism upstream from the bar. By virtue of the co-spun "binder fiber," the resultant sheet can be rendered more stable by heating. For example, the sheet is pressed between 50-mesh stainless steel screens at 50 p.s.i. at 200° C. for 1 minute, to yield a tough, drapable fabric which exhibits enhanced resistance to delamination. A typical fabric prepared by this method has a tensile strength of 10 lbs./in./oz./yd.².

Using an apparatus assembly similar to that described in Example II, poly(ethylene terephthalate) is spun through a 68-hole spinneret while a 20% (weight) poly(ethylene isophthalate/terephthalate) copolymer is co-spun through an adjacent 34-hole spinneret, incorporating at least two of the latter filaments in the resulting sheet. A uniform sheet is obtained which, by virtue of the co-spun "binder fiber," can be rendered more stable by subsequent heating. Taking in consideration the different compositions, the instant sheets are comparable to the ones obtained hereinabove as regards uniformity of lay-down, freedom from filament aggregates, and the like; enhanced stability after heating, characteristic of sheets prepared by co-spinning, also is observed.

Example XIX

Polymer flake of poly(ethylene terephthalate) is melted in a melt grid at a maximum temperature of 295° C. and metered at a rate of 15 g.p.m. through a 1 in. layer sand filter bed and a 2 in. spinneret having 23 capillaries (0.009 in. diam. x 0.012 in. long). The pack block is held at 290° C. and the spinneret temperature is controlled at 285° C.

The bundle of filaments is quenched in the ambient air before entrance to a draw jet, located about 70 in. below the spinneret. An operating pressure of 40 p.s.i. in the draw jet would provide a tension of about 3 grams on the threadline, as measured immediately above the jet. A negative corona is formed from a four point source 8 in. above the entrance to the draw jet, and above ¾ in. from the bundle of filaments. A rotating target bar (10 r.p.m., 1¼ in. diam.) makes a slight contact with the filaments and aids in maintaining a uniform distance between individual filaments and the corona source. A negative voltage of 30-40 kv. (200-250 µa.) is applied to the corona points.

The charged poly(ethylene terephthalate) filaments are deposited on a reciprocating table (30 in. square) charged to positive polarity (20 kv.). The speed of the table movement is adjusted to obtain the proper basis weight and satisfactory uniformity. Under the table speed conditions of 29 in./min. in one direction and 580 in./min. in the other direction, the charged fiber is deposited as a web at a rate of 0.8 oz./yd.² per minute. The filament properties are:

Denier	-----d.p.f.---	2.3
Tenacity	-----g.p.d.---	2.9
Elongation	-----%---	183
Mi	-----g.p.d.---	17.2

This web is spun without binder fibers. In the as-deposited state the sheet is strong and coherent, can be readily handled, and is suitable for further processing, such as dipping, coating or laminating. The bunching coefficient of the web is 0.80, and the visual uniformity is excellent.

Example XX

This example illustrates the production of a typical elastomeric polypropylene web. Polypropylene flake ("Profax") at an MI of 10 is screw melted at a maximum temperature of 290° C. and metered at a 12 g./min. rate through a ¼ in. layer sand filter bed and a 2 in. spinneret having 20 capillaries (0.015 in. diam. x 0.020 in. long). The pack block is held at 275° C. and the spinneret temperature is controlled at 260° C.

The bundle of fibers is quenched radially in a 6 in. long quench chimney using 40 c.f.m. air at room temperature. The top of the quench chimney butts against the bottom of the spinning pack to minimize the effect of air flow on the spinneret temperature.

A draw jet operating at 25 p.s.i. provides a tension of about 3 grams on the threadline, measured immediately above the jet. Distance from spinneret to jet entry is 72 in. A negative corona is formed from a four point source at a distance of ¾ in. from a 1¼ in. O.D. bar rotating at 10 r.p.m. The threadline makes light contact with the target bar, the centerline of which is 8 in. above the entrance to the draw jet. A negative voltage of 20-25 k.v. (100-150 µa.) is applied to the corona points.

The elastomeric, charged fibers are deposited on a grounded 47 in. square table which reciprocates in two directions to form a web. Table motion is adjusted to obtain proper web weight and satisfactory web uniformity. Several layers are made for each sheet. For the conditions

above, table speed is 26 in./min. in one direction and 570 in./min. in the other direction, and each layer is 0.5 oz./yd.².

Typical fiber properties are as follows:

Denier	-----	d.p.f.	2.5	5
Tenacity	-----	g.p.d.	2.0	
Elongation	-----	percent	300	
Mi	-----	g.p.d.	14	

The web formed in this manner is coherent and strong, 10 even without bonding. The appearance of the web is uniform. The bunching coefficient of the web is 0.75.

Example XXI

The non-woven webs of this invention, described in 15 examples above, were found to offer advantages as bases for water-proofed materials. Several samples of such fabrics were waterproofed with waxes, wax-resin combinations and resins. In comparison with similar fabrics prepared from woven cotton base material, the novel coated 20 fabrics were found to retain their water-repellency much longer when subjected to scrubbing and flexing action.

The experimental fabrics were similar to those described in Example XIX, except that several different basis 25 weights were used. Wax coatings were applied using commercial wax-solutions and wax-vinyl resin solutions employed as water-proofing compounds. Commercially-coated fabrics were purchased and tested for comparison purposes. Resin coatings employed included both vinyl 30 and chloro-sulfonated polyethylene resins.

Table X lists several experimental and control materials, which were prepared or purchased as indicated. Samples 35 of such material were submitted to the indicated number of scrub/flex cycles using a mechanical flexor and were then tested using an AATCC Hydrostatic Pressure Tester.

As the table shows, improvements over comparable cotton fabrics ranged from 30:1 to 300:1.

The laminated structure, due to the continuous nature of the reinforcing fiber elements, was observed to have greater strength and impact properties than similar staple-reinforced materials. The structure was heat-sealable with no loss in properties at the seal.

Example XXIII

Other laminated combinations are possible using the nonwoven webs of the present invention as well. A web of the type shown in Example XX, but containing highly oriented polypropylene material in continuous filament form, was laminated to a self-supporting film of oriented polypropylene. First the fiber web was deposited, consolidated and thermally self-bonded. The oriented polypropylene film, of the type known in the art, was coated with a low-melting polyethylene adhesive. Then the two components were laminated with the application of heat and pressure to activate the adhesive. The result was a high level of lamination of web to film with a moderate level of fiber-to-fiber bonding. The resulting structure was highly suited to use as an improved industrial film suitable for protective coverings. Equally well, the structure was suited for use as a material for shipping bags. The presence of the film gave a vapor-impermeable barrier while the fiber elements provided high tear resistance and tensile strength.

In a modification of the practice of this example, a portion of the same self-bonded web was melt-coated with an unoriented film of polypropylene applied to one side.

Example XXIV

This example shows the preparation of nonwoven webs of polypropylene and the lamination of such webs to give a highly resilient semi-rigid sheet structure.

Stereo-regular polypropylene having a Melt Index of 12.4 was spun into a web following the general procedure of Examples III through VII. In the present example the

TABLE X.—SCRUB/FLEX RESISTANCE OF WATERPROOFING ON COTTON AND ON CONTINUOUS FILAMENT NONWOVEN FABRICS

Item	Fabric	Gray Weight, oz./yd. ²	Waterproofing		Scrub Flex, Cycles	Hydrostatic Pressure, Cm.		Repellency Retained, percent
			Compound	Operation		Before Scrub/Flex	After Scrub/Flex	
1	Woven Cotton	10	Wax	Commercial	10	78	13	16.7
2	do	12	do	do	10	68	14	20.6
3	do	14.9	do	do	10	100	25	25.0
4	CFNW ¹	4.2	do	Laboratory	3,000	48	23	48.0
5	Woven Cotton	10	Wax-Vinyl	do	10	51	13	26.0
6	do	10	do	Commercial	100	50	19	38.0
7	do	7.7	do	Laboratory	100	35	18	52.0
8	CFNW ¹	4.5	do	do	3,000	48	26	55.0
9	CFNW ¹	5.4	do	do	3,000	31	20	64.5
10	CFNW ¹	3.7	do	do	3,000	22.5	18	80.0
11	Woven Cotton	10	CSPE ²	do	20	54	17	31.5
12	do	7.8	CSPE ²	do	20	67	18	26.9
13	CFNW ¹	4.0	CSPE ²	do	1,200	43	24.5	57.0
14	CFNW ¹	3.0	CSPE ²	do	1,300	25	17.5	70.0
15	Woven Cotton	10	Vinyl	do	5	28.5	14.5	57.0
16	do	7.7	do	do	5	29.0	15.5	53.5
17	CFNW	4.1	do	do	150	28	10	35.8

¹ Continuous filament nonwoven.

² Chlorosulfonated polyethylene.

Example XXII

Continuous filament nonwoven sheets were prepared according to the teachings of Example II. Webs having a basis weight of 1.0 and 2.0 oz./yd.² were prepared. The filaments were all of poly(ethylene terephthalate) and no bonding filaments were included.

After sheet formation, the web structures were individually laminated with polyethylene films by a simple hot pressing procedure. A temperature of 195° C. was used to effect bonding, and a pressure of 125 p.s.i. was employed. The nonwoven web was imbedded in the thermoplastic film which thus acted as a binder for the web. 75

block temperature of the polymer supply was 250–255° C. and the pack temperature was 222–240° C. A 40-hole spinneret was used, each hole being 0.009 inch in diameter.

A supplementary mechanical drawing step was used upstream of the charging stage. The filaments as spun were given a 3× draw, the said rolls operating at 300 y.p.m. and the draw rolls 900 y.p.m. The drawn fibers were 3 denier/filament. Corona charging was employed with a negative voltage of 20–25 kv. applied to the points. The forwarding jet operated at 60 p.s.i.g.

The nonwoven web was collected on an aluminum

tray having an area of 2 sq. ft. The web density was 2 oz./yd.². A number of webs were collected in this way and each web was consolidated by pressing between plates at 115° C. at a pressure of 5 tons/ft.² for 30 seconds.

Six of the consolidated webs were plied together to give a composite laminate weighing 12 oz./yd.². The composite web was needle punched on a needle loom at a density of 740 needles/ft.², the machine being run at 85 strokes/min. The web was run through the loom at a speed of 14 ft./second.

Following the same general procedure described above, a number of these laminated resilient structures is made in varying basis weights. The resulting fabrics are described in Table XI. The resilience and indentation resistance of the sheets are given as well.

Because of their excellent response to physical deformation, these sheets offer advantages for use as floor tiles, stair-step coverings, rug underlay material, counter-top coverings, resilient game-table surfaces, and similar applications. In particular, the high resistance to permanent indentation and the superior resilience indicate excellent behavior as floor tiling materials.

TABLE XI

Basis Weight, oz./yd. ²	Thickness, mils	Indentation Resistance (mils residual set)					Thickness After 10 Thousand Impacts of 1 ft.-lb.
		200 p.s.i., 24 hr.	600 p.s.i., 1 hr.	3,000 p.s.i., 1 hr.	Resilience		
					Percent Compression	Percent Work Recovery	
18.....	150	29	24	45	58.2	46.7	73.2
24.....	125	2.5	10	20	19.4	72.8	97.5
24.....	150	3	9	22	9.0	90.3	100.0
30.....	175	10	13	28	16.7	69.2	97.0

Example XXV

A web of poly(ethylene terephthalate) fibers and polyester binder fibers was prepared, using the composition described in the second part of Example XVIII. Spinning conditions were the same as those described in Example XIX, except that the binder fibers were added to the bundle of polyester filaments upstream of the charging stage.

Downstream of the drawing jet, the filaments were relaxed and shrunk in a continuous manner by running them through a chamber containing cocurrent-flowing air at 550° C. The walls of the chamber were maintained at room temperature. The air-flow of the hot air was not greatly faster than the entering velocity of the filaments into the relaxing chamber, but substantially faster than the exit velocity of the filaments. During their exposure to the hot air, the filaments shrank continuously in the chamber, losing 25% or more of their original length by relaxation. This relaxation placed the polyester filaments in a state of spontaneous elongatability as already described. Following the relaxation-shrinkage step, the filaments were collected as described in Example XIX to obtain a uniform nonwoven sheet of polyester fibers and co-spun binder fibers.

The web was then placed between a 30-mesh wire screen on one side, and a canvas fabric on the other, and heated to 210° C. at 200 p.s.i. for one minute. The heating caused elongation of the polyester fibers with bonding and embossing taking place simultaneously.

The fabric produced as described was a soft, flexible nonwoven textile-like material, suitable for the same uses as woven fabrics of similar basis weight. The highly crimped fibers rendered the final structure drapable, and gave it a pleasing handle.

Example XXVI

A nonwoven web of 14% low-oriented and 86% high-oriented crystalline polypropylene filaments is prepared

as follows: isotactic polypropylene (melt flow rate MFR 12, by method of ASTM-1238 at 230° C. with a loading of 2.16 kg.) is spun from each of two spinnerets. One spinneret has 30 spinning orifices each 0.015 in. in diameter while the other has 5 orifices each 0.015 in. in diameter. The extrusion rate from the latter is 3 g./min. while polymer is extruded from the 30-hole spinneret at 18 g./min. The temperature of the 30-hole spinneret is 256° C. and that of the 5-hole spinneret, 221° C.

The filaments from the 30-hole spinneret are led to a heated feed roll operating with a surface temperature of 118° C., and advanced by means of an idler roll canted with respect to the heated roll. A total of 5 wraps is used on the heated feed roll, which is operated with a surface speed of 238 yd./min. From the heated feed roll the filaments are then passed 5 wraps around an idler roll/draw roll system operating cold with a surface speed of 842 yd./min. These filaments are drawn 3.54×, are 7.75 denier (0.86 tex.) per filament and have a tenacity of 3.76 g.p.d.

The filaments issuing from the 5-hole spinneret are led to a heated roll operating with a surface temperature

of 95° C. and a surface speed of 667 yd./min. After being in contact with the heated roll for 180°, the filaments leave the heated roll and are then passed to a draw roll operating cold with a surface speed of 858 yd./min. The filaments are in contact with the draw roll for 180° and are drawn 1.28×. As a result of this treatment the filaments are 7.8 denier (0.86 tex.) per filament and have a tenacity of 1.55 g.p.d. The filaments from the two spinnerets meet and are guided so that the low-oriented filaments are dispersed uniformly throughout the high-oriented filaments.

The filaments are stripped from the draw rolls and forwarded by a pneumatic jet having a nozzle section as shown schematically in FIGURE 8 and having the following dimensions:

	Inches
Over-all jet length-----	15¼
Filament inlet diameter (16)-----	0.062
Filament inlet length (16)-----	0.55
Filament passageway (19) minimum diameter--	0.093
Metering annulus (32)	
Inner diameter-----	0.0750
Outer diameter-----	0.0930
Length-----	0.020

The jet is supplied with air at 80 p.s.i.g. and applies a tension of about 36 grams on the filament bundle. The entrance to the jet is 110 inches from the draw roll.

Between the draw roll and the jet and 7½ inches from the latter, the filament bundle is exposed to a corona discharge device to impart an electrostatic charge to the fibers. The corona discharge device consists of a 4-point electrode positioned ⅝ inch from a grounded, 1¼ inch diameter, chrome-plated target bar rotating at 10 r.p.m. A negative voltage of 23 kv. (90 microamperes) is applied to the corona points. The filament bundle passes between the target bar and electrode and makes light contact with the target bar and is charged to a value of 74,100 e.s.u./m.² of filament surface.

The jet is positioned at an angle of 90° with the plane of a moving web laydown belt and is moved back and forth across the web by a traversing mechanism. The traverse speed of the jet is 24 passes (12 cycles) per minute. The distance from the exit of the jet to the laydown belt is approximately 26 inches. The laydown belt moves at a speed of 9 in./min. A plate located beneath the belt is charged at +30 kv. to pin the filaments to the laydown belt. The filaments are laid down in the form of a random nonwoven web that is 30 inches wide.

The web is bonded while passing at a speed of 10 y.p.m. while under restraint between a porous metal belt and a solid metal belt, for a distance of 37 inches through a steam chamber in which saturated steam is maintained at superatmospheric pressure. The steam on the porous belt side is maintained at 74 p.s.i.a. (corresponds to a saturated steam temperature of 153° C.) and on the solid belt side, at 76 p.s.i.a. The difference in pressure serves to restrain the sheet and filaments against shrinkage during bonding.

The properties of the sheet after bonding are as follows:

Basis weight (oz./yd. ²)-----	4.4
Strip tensile (lbs./in./oz./yd. ²)-----	4.6
Elongation %-----	12
Tongue tear (lbs./oz./yd. ²)-----	5.8
Formation value-----	187
Randomness, Standard deviation (in.)-----	0.4
CV _{fs} -----	92%

Thus the present invention encompasses sheets of nonparallel continuous synthetic organic filaments that are randomly distributed throughout. The filaments are disposed as to be substantially separate and independent of each other except at filament cross-over points within the sheet. Since the sheet is formed from continuous filaments, there will be essentially no fiber ends within the sheet. The filaments of the sheet are so separated from each other that the distances of separation have a coefficient of variation, CV_{fs}, of no greater than about 100%. If desired the sheet may be bonded at a plurality of cross-over points as described herein.

The bonded fabrics of this invention are also suitable as substrates for coated fabrics. In one experiment the polyester fabric was coated with a formulation of chlorosulfonated polyethylene resin of the type described in Kelly, U.S. Patent 2,914,496 to give water-repellent but water-vapor permeable protective covering materials, suitable for use as tenting or tarpaulin fabric. The formulation consisted of 100 parts by weight of chlorosulfonated polyethylene, 320 parts of xylene, 60 parts of isopropyl alcohol, 2 parts of silicone flowing agent, 40 parts of tribasic lead maleate, 0.5 part of tetramethyl thiuram disulfide, one part of 2-benzothiazolyl disulfide, 60 parts of calcium carbonate (York Whiting), 60 parts titanium dioxide, one part of phthalocyanine blue pigment, 80 parts of hydrogenated wood rosin, thirty parts of mineral spirits and two parts of synthetic dispersing agent.

The sheet-like structures of this invention may serve a variety of useful purposes. With suitable coating and/or laminations, they may serve in industrial applications, in the stead of conventional woven materials, films and papers. By incorporation of a suitable binder, with or without an additional embossing operation, cloth-like articles are produced. The nonwoven structures of this invention can serve in the preparation of felts, leather-like materials, and suede-like materials. They may be assembled into a layered structure which may then be needed to form a substrate which is suitable for use in poromeric materials. With high binder content and high pressure calendering, paper-like articles are produced. The present structures can serve as interlining or interfacing materials useful in imparting shape and/or stiffness to garments. All of the above-mentioned articles which are

based on the structures of this invention are strong in resistance to tear and have good tensile properties especially in the lateral dimensions. The nonwoven structures of this invention may be modified by stretching to give a material with increased tensile strength and reduced elongation in the direction of stretch. In certain industrial fabrics, such as beltings, hose and bagging, low elongation in one direction is a desirable property. The sheet-like structures function well in such important finishing operations as buffing, felting, shearing, brushing, needling, printing, embossing, napping, sanding and the like.

They are suitable for many different end-uses, including: grinding and polishing cloths, sound absorbent wall coverings, aprons, work aprons, artificial leather, ceiling coverings for automobiles, tea bags, absorbent batts, book-binding, hand towels for hotels, bowling alleys, golf courses, airplanes, etc., tapes and bands, with and without adhesive, chafer, flipper and bead-wrap fabrics for tires, tire and V-belt reinforcement fabrics, V-belt wrappings, covers and reinforcements for mechanical rubber goods, liners for separating in-process rubber; cable insulation and filling materials, electrical insulation; filter material for potable fluids such as beer, wine, lemonade, coffee and milk, filter cloths and fabrics for gas filtration, such as air filters, air conditioner filters, dust filters, gas filters, and flue-gas filters, chemical process filters, such as for oils, dye solutions and as filter elements in filter presses, vacuum and rotary filters; silo covers, portable silos, mulch sheeting; as reinforcement for synthetic resins, over-lays for glass-fiber reinforced plastics, reinforcement for films and papers; bagging material, pond covers, ditch liners; base-fabric for coated adhesives, glass polishing felts, packing felts, packing cushions and pads for sensitive, fragile and expensive medical, optical, mechanical and electronic instruments and for objects of art; upholstery fabrics, linings and stuffings, automobile upholstery, and head liners, luggage lining, lining and inner layers for textiles and apparel garments, coat and suit inner liners and stiffeners, shoulder pads, brassieres, linings for girdles, collars and cuffs; diapers, sanitary napkins, face cloths, dress shields, petticoats and skirt stiffeners, hospital clothing, blankets, napkins, bed spreads, head rests for furniture, clothes bags, laundry bags, ironing board covers, table cloth pads, insoles and linings for shoes, shoe polishing, window polishing and other cleaning, dusting and wiping cloths, carpet underlays, fillings for quilts, sleeping bags, pillows and cushions; post-formed articles such as hats and brassiere cups; felts of all kind including industrial felts, papermakers' felts, apparel felts, felts for game tables, applicator felts and pads for grease, oil, textile sizes and finishes, dye padding, polishing felts; grass-growing mats, lamp shade material, fabric for artificial flowers, tool-bags, office machine ribbons, pipe wrap, journal-box stuffing; and leather replacement substrates, base fabrics, and body armor.

What is claimed is:

1. A substantially uniformly opaque nonwoven sheet of nonparallel continuous synthetic organic filaments, the continuous filaments being randomly distributed throughout said sheet and so disposed as to be substantially separate and independent of each other except at filament cross-over points within the sheet, the filament separation distances having a coefficient of variation, CV_{fs}, of no greater than about 100%.
2. The sheet of claim 1 wherein the filaments lie generally parallel to the plane of the sheet.
3. The sheet of claim 1 in which the filaments comprise poly(ethylene terephthalate).
4. The sheet of claim 1 in which the filaments comprise poly(hexamethylene adipamide).
5. The sheet of claim 1 in which the filaments comprise polypropylene.
6. The sheet of claim 1 in which the filaments comprise poly(ethylene terephthalate) and lower melting binder filaments.

29

30

7. The sheet of claim 1 having a bunching coefficient, BC, of at least 0.7.

8. The sheet of claim 1 impregnated with a water-proofing composition.

9. The sheet of claim 1 laminated to a self-supporting film.

10. The sheet of claim 1 laminated to a polyhydrocarbon film.

11. A nonwoven sheet of nonparallel continuous synthetic organic filaments, the continuous filaments of such sheet being randomly distributed throughout said sheet,

and so disposed as to be substantially separate and independent of each other except at filament cross-over points within the sheet and being bonded at a plurality of such points, the filament separation distances having a coefficient of variation, CV_{ts} , of no greater than about 100%.

No references cited.

A. WYMAN, *Primary Examiner*.

GEORGE D. MORRIS, *Assistant Examiner*.

UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 3,341,394

September 12, 1967

George Allison Kinney

It is hereby certified that error appears in the above numbered patent requiring correction and that the said Letters Patent should read as corrected below.

Column 3, line 49, for "fialment" read -- filament --; line 50, for "described" read -- describe --; column 9, line 51, for "t" read -- at --; column 11, line 25, before "RCA" insert an opening parenthesis; column 14, line 40, for "discharging" read -- charging --; same column 14, TABLE III, fifth column, line 4 thereof, for "2.3" read -- 2.4 --; same table, sixth column, line 4 thereof, for "2.4" read -- 2.3 --; column 15, line 50, for "transverses" read -- traverses --; column 19, line 28, for "TABLE III" read -- TABLE VIII --; columns 21 and 22, TABLE IX, eighth column, sub-heading thereof, for "lb./in./oz./yd." read -- lb./in./oz./yd.² --.

Signed and sealed this 15th day of October 1968.

(SEAL)
Attest:

EDWARD M. FLETCHER, JR.
Attesting Officer

EDWARD J. BRENNER
Commissioner of Patents