

Oct. 15, 1968

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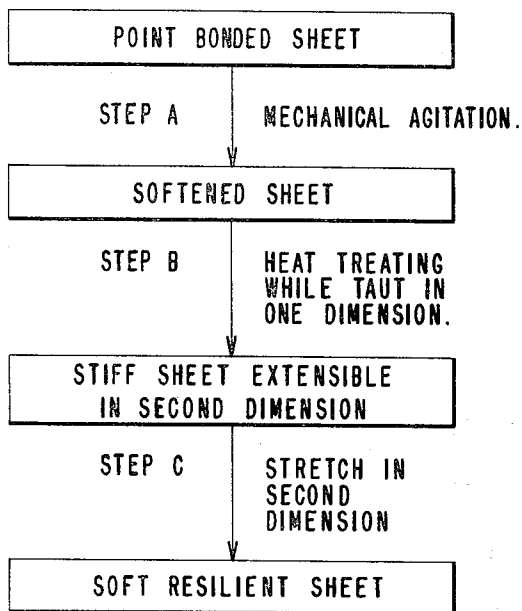
3,406,033

METHOD FOR TREATMENT OF FILM-FIBRIL SHEETS

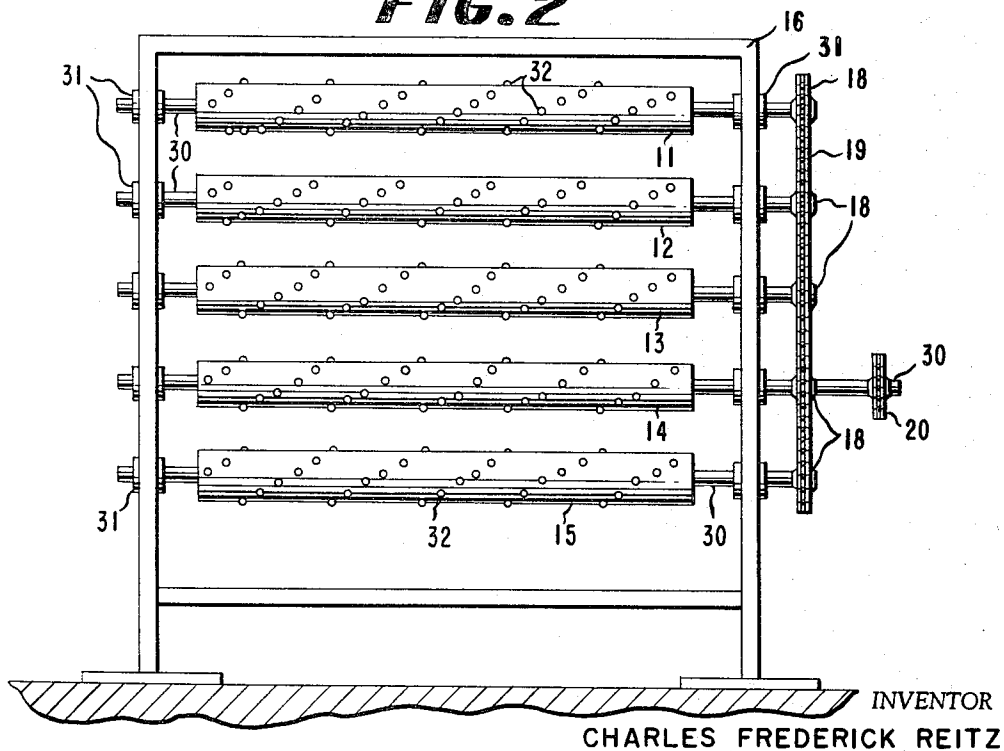
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3 Sheets-Sheet 1

**FIG. 1**



**FIG. 2**



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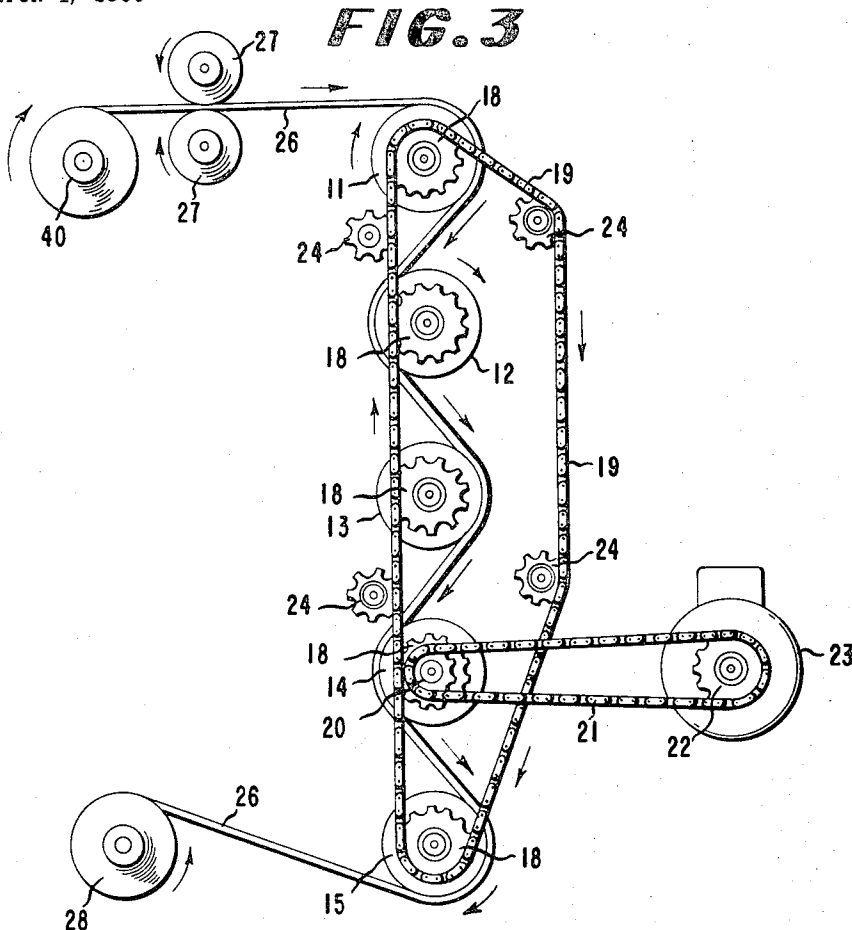
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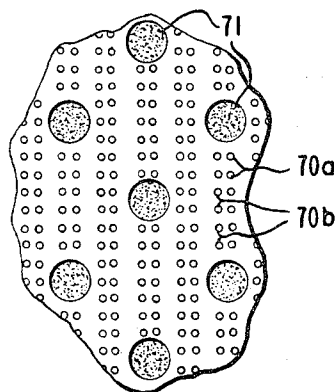
METHOD FOR TREATMENT OF FILM-FIBRIL SHEETS

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3 Sheets-Sheet 2



**FIG. 4**



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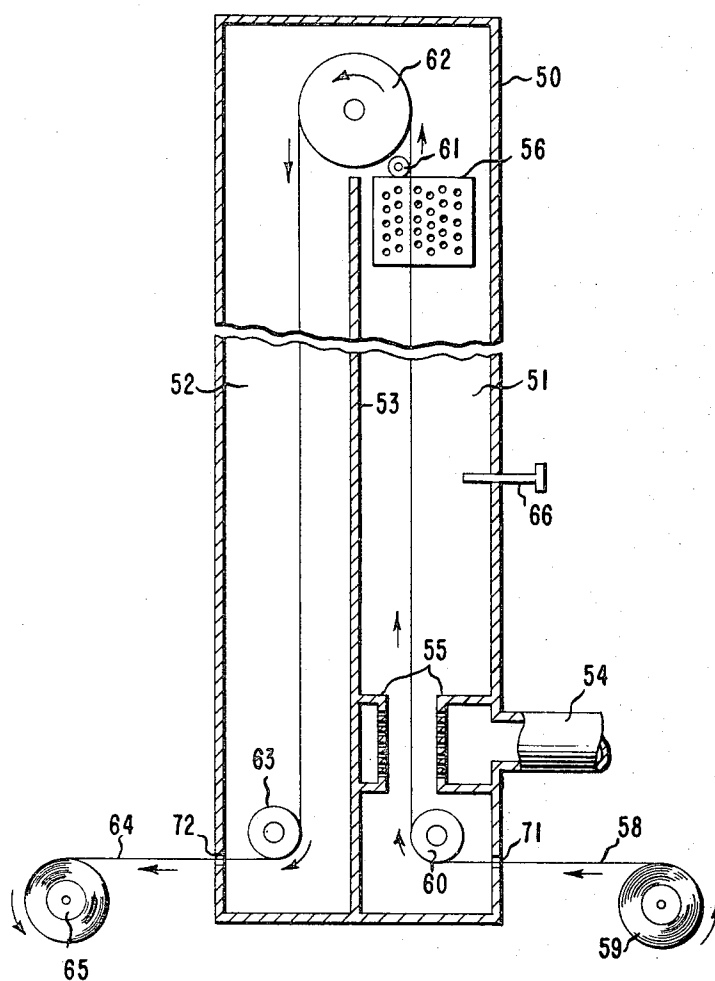
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METHOD FOR TREATMENT OF FILM-FIBRIL SHEETS

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3 Sheets-Sheet 3

**FIG. 5**



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3,406,033

## METHOD FOR TREATMENT OF FILM-FIBRIL SHEETS

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### ABSTRACT OF THE DISCLOSURE

Polymeric nonwoven, point-bonded film fibril sheets are treated to increase their bulk, resilience and recovery and to provide a drier, less slick hand. The treatment involves the steps in order of mechanically agitating the sheet to loosen the film fibril elements, heating the sheet to within 35° C. of the melting point while under tension in one dimension, then stretching the sheet in the second dimension.

#### Specification

This invention concerns a process for improving soft nonwoven fabrics to provide greater resilience and a more pleasing tactile hand.

Recently methods have become available for preparing nonwoven sheets from film-fibril elements. These film-fibril elements are tiny molecularly oriented elements often less than 4 microns thick. They may be prepared by a number of methods such as by fibrillating an oriented film, or by flash-spinning by the methods of Blades and White described in U.S. 3,081,519. In the latter patent the film-fibrils exist as continuous three-dimensional networks along and across a continuous strand. These continuous networks regardless of their origin are herein termed plexifilaments. The plexifilaments may be formed into nonwoven sheets by a variety of methods. The network material for example may be deposited on a moving belt in random intersecting, overlapping layers. After cold rolling, a coherent film-fibril sheet is obtained. The method for preparing a film-fibril sheet by flash-spinning is described in Steuber U.S. 3,169,899 issued Feb. 16, 1965.

The products of the aforementioned Steuber patent may be prepared in the form of stiff structural materials, or as stiff paper-like sheets, or as soft nonwoven fabrics. The soft nonwoven fabrics are of particular interest in the present invention. One process has been developed wherein these soft sheets are point bonded by hot embossing to improve tensile properties and delamination resistance. However, these bonded sheets often tend to be stiff and papery. This fault has been largely corrected by a process and apparatus described in the U.S. patent application of Reitz, Ser. No. 421,902, filed Dec. 29, 1964. In that process a series of button rolls is used to soften the fabric by vigorously stroking the fabric at a multitude of small areas while the sheet is under tension. While the apparatus and process of Reitz provide much softer fabrics, the fabrics still have relatively poor compression recovery, low resilience, and lack certain aesthetic qualities needed for fabrics to be used in garments. In particular they have a slick or waxy feel.

The purpose of the present invention is, therefore, to provide a process for finishing nonwoven film-fibril sheets to obtain soft nonwoven fabrics with increased bulk, porosity, resilience, compressional recovery, and with a drier, less slick hand. It is essential that these improvements be achieved without appreciable loss of porosity, tensile strength, delamination resistance, or abrasion resistance.

#### Summary of invention

The process of the invention comprises applying the following succession of process steps to a polymeric non-

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woven film-fibril sheet which has been point-bonded at a multiplicity of points throughout the sheet:

(a) Mechanically agitating the fibrils in the sheet while applying tension to the sheet, the agitation and tension being less than would cause rupture of the sheet and substantial separation of fibers in the point-bonded areas, the overall sheet density of the sheet in this step preferably being reduced to less than 20 lbs./ft.<sup>3</sup> (0.32 g./cm.<sup>3</sup>), as measured with a 2.81 p.s.i. compressive load,

(b) Then continuously heating the sheet while applying sufficient tension in one dimension of the plane of sheet to at least prevent shrinkage in that dimension while substantially avoiding restraint in the second dimension of the plane of the sheet, the temperature of the sheet being below the melting point of the polymer but within 35° C. thereof, the applied tension and heat in combination being such as to cause 5 to 60% shrinkage of the sheet in the unrestrained second dimension, and

(c) Then stretching the sheet in the second dimension to restore at least 25%, preferably at least 50%, of the loss incurred in that dimension during the preceding hot stretching operation.

The polymeric material comprising the initial film-fibril sheet is formed of a thermoplastic polymer, preferably a hydrocarbon polymer such as polyethylene, polypropylene or ethylene/propylene copolymers. The sheets may be modified to contain various pigments, dyes, and other agents and additives depending upon the intended use.

#### Figures

FIGURE 1 is a flow sheet showing successive steps in the process and the effect of each of these treatments on the treated sheet.

FIGURE 2 is a front view of an arrangement of rolls suitable for the mechanical working step (a) of the process.

FIGURE 3 is an elevational view of the rolls of FIGURE 2 but further including web feeding and web wind-up rolls.

FIGURE 4 shows an enlargement at about 4.5× of a suitable point bonding design for film-fibril sheets used in the process of this invention.

FIGURE 5 is a cross-section of a film coating tower which is suitable for Step b of the process of the invention.

Now with reference to FIGURE 1 in detail, a point bonded sheet is mechanically agitated (Step a) as indicated by the arrow a in FIGURE 1 to produce an intermediate product which is a soft waxy fibrous sheet with comparative low resilience. A power-driven "button breaker" of the type described in the Reitz application mentioned above may be used for this operation. The resulting sheet is treated further in Step b, which comprises heat-treating the sheet under tension in one dimension while it is unrestrained in the second dimension in the plane of the sheet. This treatment under tension may be effected by actual hot stretching or it may be effected simply by restraining the sheet in one dimension while heating at a temperature just below the melting point. The increased plasticity of the film-fibrils while heated coupled with tension in one dimension causes the sheet to shrink 5 to 60% in the other dimension of the sheet i.e. the dimension at a substantially right angle to the first. The sheet obtained from tensioned heat treatment is a relatively stiff material which is highly extensible in the dimension of shrinkage. The third step in the process (Step c) as shown in FIGURE 1 comprises stretching in the second dimension. For example when tension has been applied longitudinally in Step b, then tension will be applied transversely in Step c. In Step c the product is stretched in the second dimension until at least 25%,

preferably at least 50%, of the loss in dimension of the preceding step is restored to the sheet. The resulting product is a soft bulky, resilient material with a dry, pleasing hand. The sheet loses much of its slickness and yet has the desirable softness needed for fabric uses.

The following discussion provides additional information on the starting material and on the individual process steps shown in FIGURE 1.

#### *The point-bonded sheet*

The film-fibril sheet used in the process should be point-bonded, i.e. bonded at a multiplicity of discrete points throughout its area to promote delamination resistance and to provide greater stability for the sheet during the subsequent mechanical processing. For the purposes of this invention it should be understood that by "point-bonding" is meant any type of discrete area bonding from resin treatment or from hot fusion, with or without the application of pressure. The fibers in the sheet are firmly attached to one another within the discrete bonding areas, but can often be worked loose from one another. Where film-fibril sheets have been point bonded by heat and fusion, the material is translucent within the bonded areas. About 2 to 50% of the area is bonded. The over-all density of the sheet after point bonding is preferably between 0.19 and 0.40 g./cm.<sup>3</sup> (11.8 to 25 lbs./ft.<sup>3</sup>). The bonded areas may be in the form of various shapes such as circles, lines, or rectangles, and they may be placed in such a way as to provide geometric designs in the sheet. They also may exist as annular bonds surrounding perforated areas in the sheet. The point bonded patterns are advantageously applied to the cold contact bonded sheet by passing the sheet through one or more pair of rolls, one of each pair being a hard rubber roll and the other being an embossed roll with raised points over its entire surface. A variety of specific point bonding procedures are well known in the art.

#### *Step a.—Mechanical agitation*

Step *a*, which comprises mechanically agitating the film-fibrils, may be performed in any apparatus which vigorously agitates the fibrils in the nonwoven material without rupturing the sheet and without breaking the point bonds provided by the preceding point-bonding operation. By agitation is meant working the fibrils to effect relative movement and loosening thereof. An apparatus which is ideally suited for this operation is described in U.S. application S.N. 421,902 of C. F. Reitz. The apparatus is a modification of the well known "button breaker" of the textile trade and includes a series of generally parallel-spaced web-working rolls, means for advancing the web in a path about the rolls in a tensioned condition, and drive means for rotating the rolls at different relative surface speeds than that of the web. The web-working rolls are provided with a pattern of spaced-apart rounded projections about the major portion of their curved surfaces for stroking the web as it passes in contact therewith. The sheet need only be under a nominal tension to facilitate the agitation by providing some resistance to the projections.

The softening and fibril loosening which is accomplished in the modified button breaker is surprisingly effective in combination with the other two steps of the process. Unless Step *a* of the process is performed so as to produce a thoroughly loosened fabric, the succeeding steps will not provide the desired bulk and dry hand. For example, a simple cold stretching process provides a soft fabric, but is unsatisfactory for Step *a*, since the subsequent steps do not then provide the desired dry hand. It is believed that in such a process the sheet stretches only in spots, leaving some of the fibrils untouched. Likewise, conventional paper creping operations are unsatisfactory for Step *a*. Only brisk local mechanical action as described above is suitable in Step *a* if the remaining steps are to produce a fully satisfactory product.

The modified "button breaker" is similar to the ordinary "button breaker" but differs most notably in that the button rolls are rotated at a different speed than the sheet which passes into contact with them. In terms of the process this means that the projections on the rolls gently but firmly strike and thrash repeatedly first against one side of the sheet and then against the other to work and partially loosen fibers therein. Instead of portions of the sheet being simply distended and stretched by the buttons, the whole sheet is effectively converted to a higher bulk form by the relative movement against the roll projections.

As shown in FIGURE 2, rolls 11, 12, 13, 14 and 15 are supported in a rigid frame-work 16. The roll axes 30 pass through the frame-work and are supported in bearings 31. Sprockets 18 are provided at one end of each roll, and these are interconnected by means of roller chain 19. A second sprocket 20 is provided on an extension of the axle 30 of roll 14.

As shown in FIGURE 3 the second sprocket 20 is in turn connected by roller chain 21 to the sprocket 22 of motor 23. The motor 23 therefore drives all of the five button rolls in this embodiment with a set speed ratio between each of the rolls and the motor. The ratios can be changed by changing the sprockets to provide ones with greater or lesser number of teeth. Supply roll 40 is used to continuously supply sheet material to the nip of feed rolls 27. Feed rolls 27 are power-driven at constant speed. The winding mechanism of windup roll 28 is provided with a conventional form of constant tension device, not shown. The constant tension windup roll 28 working in conjunction with constant speed feed rolls draws, e.g. extends, the sheet lengthwise during treatment at least 0.5%, the sheet surface speed at the windup being essentially constant. An alternate system may be used whereby the speed of the fabric at the windup roll 28 is controlled by means of a positively driven constant speed roll flexibly mounted above roll 28 and running against the top surface of the accumulated sheet on roll 28, sufficient frictional force being provided to avoid slipping by properly weighting the constant speed roll. A similar device may be used against supply roll 40, thereby making it a feed roll of constant speed. In this case rolls 27 may be omitted. Depending upon the amount of bonding in the sheet, the sheet may be drawn as much as 40% during treatment. The feed and windup speed ratios are adjusted to obtain between 0.5% and 40% stretch.

The pair of feed rolls 27 and windup roll 28 are shown mounted separately from the button rolls in FIGURE 3. However, they could be mounted in the same frame-work, provided means were included for adding or removing completed rolls of wound sheet material. In FIGURE 3 adjustable idler sprockets 24 are provided to allow the chain tension to be adjusted.

Rolls 11, 12, 13, 14 and 15 are each provided with a multiplicity of spaced-apart rounded projections 32 disposed about the major portion of their curved surfaces which independently displace and distend small areas of the fabric which contact them. In operation of the process the rolls move at a different speed from the fabric passing over them. Consequently the knobs on the rolls provide a stroking-action along the face of the sheet in addition to displacing small areas out of the plane of the sheet.

The sprockets 18 on rolls 11, 12, 13, 14 and 15 may all have the same number of teeth or the number of teeth may vary, depending upon the type of action needed at each point in the process. For example if more abrupt action is needed near the end of the process, rolls at the windup end of the apparatus may have sprockets with fewer teeth than the sprockets on any of the preceding rolls, thereby providing relatively faster rotation for the rolls at the windup end of the process.

In operation of the process, sheet material 26 passes

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from feed rolls 27 as shown in FIGURE 3 to button roll 11 at a set speed and then successively to rolls 12, 13, 14 and 15. With all of the rolls (feed rolls 27, button rolls 11, 12, 13, 14, and 15 and windup roll 28), operating in clockwise direction as seen in FIGURE 3 fabric is passed over roll 11 in the direction of its rotation. The fabric then passes to the back side of roll 12 and is pulled over the roll surface in opposition to the surface movement of that roll. It passes then to roll 13 in conformance with roll 13 direction of movement. Then the fabric passes to the back side of roll 14 against the direction of roll movement. Finally the fabric passes around the front side of roll 15 in the same direction as the roll surface is moving and is wound up at roll 28.

As shown in FIGURE 3, rolls 11, 13 and 15 alternately stroke one side of the sheet 26. The force of the roll projections 32 thereon thus is applied in the direction of sheet travel. Rolls 12 and 14 contact the opposite side of the sheet with the force of the roll projections 32 thus stroking the sheet against the direction of sheet travel. The sequence in which the two sides of the sheet are contacted and the direction in which each roll is turning relative to the direction of sheet travel are not critical. It is only essential that the stroking of the sheet be applied sequentially to both sides and that part thereof is applied in the direction of sheet travel and the remainder against the direction of sheet travel.

In operation of the process the forward moving rolls (relative to fabric) have speeds well above that of the fabric (e.g. 2 to 50 times). Reverse turning rolls 12 and 14 may move more slowly, however. The overall fabric speed is controlled by the feed and windup speeds. In order to avoid tearing the fabric or excessive stretching thereof, the tension of the fabric against the button rolls 11 to 15 must be controlled at a suitable level. In the embodiment of FIGURE 3 this can be achieved most readily by control of the surface speed ratio of windup roll to feed roll. A windup/feed speed ratio of about 1.005 is satisfactory for a machine having button rolls of 4 inch diameter with centers  $9\frac{3}{4}$  inches apart and the roll axes parallel as well as lying in a single plane. Other methods of varying tension will be apparent. For example to reduce tension the amount of contact of fabric to roll surface can be adjusted by arranging the rolls to allow the fabric to follow a path more nearly planar. Regardless of the means used for tension control, however, the tension on the sheet as it is wound up on roll 28 is advantageously between .05 and 2.0 lbs./in. of sheet width.

The reversal in stroking direction which is afforded by passage from roll-to-roll gives great extremes in tension within the fabric. It will be apparent from FIGURE 3 that the fabric will be under tension between feed roll 27 and roll 11, will be relatively relaxed between rolls 11 and 12, tensioned between rolls 12 and 13 relatively relaxed between rolls 13 and 14 and tensioned between rolls 14 and 15. It is wound up under tension at roll 28. The alternate tensioning and relaxing in conjunction with the distortion caused by local pressure of the moving projections 32 works, disrupts and relocates the fibers into freer positions to give a very soft fabric structure.

#### Step b.—Hot tensioning

Step b of the process may be performed with any fabric heating device which permits tension to be applied in one dimension while keeping the other dimension slack. It will be understood that a variety of methods are available for providing the necessary tension in one dimension. The tension may be provided either in the longitudinal or transverse dimension of the sheet. It may be provided mainly by the contractile force developed because the sheet is near its melting point; it may be provided mainly by actual mechanical stretching in one dimension; or it may be derived simultaneously or successively from both of these factors.

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In a preferred form of the invention the sheet is actually stretched 5 to 25% in the heating and tensioning step. This is most easily accomplished by tensioning in the longitudinal direction of a sheet, i.e. in the machine direction of a sheet made in a continuous process. This longitudinal drawing method overcomes certain difficulties which would be normally encountered in attempting by a continuous process to stretch the sheet in the cross-direction. In the latter case the forwarding mechanism would actually tend to provide tension in the longitudinal direction as well as in the traverse direction. This problem, of course, would not exist in piece-work operations and could be over-come in the continuous operation by adequate tension controls on the wind-up machinery.

Because of the ease with which tension can be controlled in longitudinally drawing a sheet, the apparatus of FIGURE 5 is particularly useful in Step b. It is essentially a film coating tower, but is not used for coating in the examples which follow. In FIGURE 5 the web treating mechanism is shown enclosed in a box-like tower 50, which is approximately 50 feet high. The box-like tower 50 is divided into two parts 51 and 52 via partition 53 which rises from the bottom of the tower to a point near the top. Thermostatically controlled hot air is introduced into the bottom of the tower by pipe 54 through distributor 55. A smooth distribution of air is provided by perforations in the distributor. The hot air rises in the tower and is removed from the tower by means of grid 56 through an exhaust pipe or other means, not shown. In operation the point bonded film-fibril sheet 58 which has been mechanically agitated to prepare for this operation, enters tower 50 through slit opening 71, passes around idler roll 60, and then proceeds to the top of the tower. Near the top of the tower the film-fibril sheet passes over spreader roll 61 which is equipped with helical lands (S configuration on one end and Z on the other). The spreader roll serves to level the sheet before it passes over idler roll 62. For the purposes of the present invention the compartment 52 is not used to heat the sheet. In essence the sheet is heated in the 50 foot passage from idler roll 60 to idler roll 62. The sheet then tends to cool as it passes down through compartment 52 around idler roll 63, out slit opening 72 to wind-up roll 65. The heat treated sheet 64 emerging from slit opening 72 is 5 to 60% narrower in width than the feed sheet 58. This contraction in width is caused by the combined heating and longitudinal tension applied to the sheet during treatment. For tensioning, the supply roll 59 is provided with an air brake while the wind-up roll 65 is positively driven at the core.

The air temperature in the enclosure which is needed for establishing that the apparatus is operating in a steady condition is measured half-way between idler rolls 60 and 62 by means of dial thermometer 66. This air temperature may vary widely depending upon the speed of the operation. The temperature within the enclosure is sufficient to bring the sheet temperature within 35° C., preferably within 15° C., of the polymer melting point but insufficient to bring the sheet above the polymer melting point. The actual sheet temperature obtained in this dynamic system may be measured if desired by comparison of the surface area of the finished sheet with a series of sheets heated in a press under known equilibrium temperature. Any surface area/g. measurement is characteristic of a specific sheet temperature reached during treatment. The polymer melting point may be determined by the method described hereinafter.

In the absence of a film coating tower a variety of other film heating and drawing machines can be used for drawing or tensioning in one dimension (Step b). One can, of course, also use standard fabric heat-setting equipment such as the clip tenter or pin tenter if adequate care is taken to avoid tension in one dimension.

#### Step c.—Cross-stretching

The third step of the process which comprises stretching the sheet in the dimension not previously held taut

is essential for obtaining a soft, dry hand. The product from Step *b* is usually quite stiff and has very high stretchability in the second dimension, i.e., the one which was kept slack in Step *b*. In Step *c* the material is stretched in the second dimension to restore at least 25%, preferably at least 50%, of the loss in width which occurred in Step *b*. It should be noted here that the final dimension is measured before removal of the sheet from any supporting frame-work. Some of the stretchability is elastic and some is permanent or non-elastic; consequently the sheet may partially contract when it is removed from the processing apparatus. However, it will then possess the required softness, bulk, resilience, and dry hand or feel. The slick, waxy, or filmy hand will be substantially reduced, and the sheet will more nearly approximate a soft woven fabric in hand.

Step *c* is performed by means of any standard sheet stretching or tentering device. In practice a continuous process fabric clip frame has been very satisfactory. The sheet may be cross-stretched either cold or hot. Excessive heat should be avoided in the step to avoid stiffening.

It will be apparent that a variety of forms of the invention will be possible, and the three essential process steps may be combined with other operations. In one variation of the process, for example, the sheet is impregnated with a resin latex after longitudinal stretching and before the cross-stretching operation. It has been found that the fibrous sheet absorbs the latex much more readily during cross-stretching than if applied in a separate operation, and hence more thorough penetration is achieved.

Still another variation comprises coating the hot stretched sheet with a thermoplastic material prior to cross-stretching. The cross-stretched sheet then has high porosity compared to sheet coated without stretching but also has high surface abrasion resistance.

The following examples further illustrate the practice of the invention. Parts and percentages therein are by weight unless otherwise specified.

#### EXAMPLE 1

A film-fibril sheet weighing about 1.25 oz./yd.<sup>2</sup> was prepared from linear polyethylene. A highly fibrillated network was flash spun by the method of U.S. Patent 3,081,519 using trichlorofluoromethane as solvent. The material was randomly deposited on a moving belt by the method of U.S. Patent 3,169,899. A 52-inch wide sheet was obtained. The sheet was lightly consolidated by passing through a pair of cold rolls which exerted a pressure of about 10 lbs./in. of axial length. The rolls were 12–14 inches in diameter.

The cold consolidated sheet was then passed through three successive pairs of rolls (*a*, *b*, and *c*) to prepare a sheet with a pattern similar to that of FIGURE 4. In each pair, one of the rolls was a smooth, hard rubber backup roll while the other roll had a steel surface which carried an embossing pattern. The embossing pattern on the steel rolls for the first two pair of rolls (*a* and *b*) consisted of tiny raised ridges which followed the circumference of the roll. The circumferential ridges were disposed every 1.60 millimeters (.063 inch) across the axial length of the roll. Within the ridges were individual points which were 5.08 millimeters (0.20 inch) high relative to the roll surface and which were 0.91 millimeter (.036 inch) apart. In preparing the sheet of FIGURE 4 the cold consolidated sheet was passed first through the roll pair *a* and then through pair *b*. Lines of tiny compressed areas were impressed in the sheet, the tiny compressed areas from roll *a* being identified by the number 70*a* in FIGURE 4 and compressed areas from roll *b* by 70*b*. The first two pair of rolls were arranged to emboss first one side of the sheet and then the other as the sheet passed from the first pair to the second pair.

Finally the sheet passed through a third pair of rolls *c*, one of hard rubber, the other an embossed steel roll. The embossed roll had much larger raised points than in

the first two pairs. The points were arranged in a diamond pattern. These points were 1.01 millimeters (.040 inch) high, 7.18 millimeters (.283 inch) apart around the circumference of the roll, and 3.58 millimeters (.141 inch) apart across the roll length. In the above operations the film-fibril sheet was passed successively through the three pair of rolls at 18 yds./min., each set of rolls being internally heated at 158 to 159° C. The pressure between the third set of rolls was low enough to avoid perforation and high enough to give translucent fused points; these being identified as 71 in FIGURE 4.

The pattern-bonded sheet was then softened by passing through a power driven "button breaker" as described in aforementioned Reitz application S.N. 421,902. Tension was provided longitudinally during this operation. The width of the sheet was thereby reduced from 52 inches to 48.5 inches and the density as measured under 2.81 lbs./in.<sup>2</sup> load was reduced from 17.7 to 15.7 lbs./ft.<sup>3</sup>. The resulting sheet was then heat treated and stretched in a heated film tower of the type shown in FIGURE 5 to produce a 12% elongation in the sheet and an additional 25% reduction in width (Step *b*). Heat treating was effected with hot air in a vertical tower between two rolls 50 ft. apart, the air in the tower being maintained at 132° C. in the middle of the vertical section. The feed speed was 32 ft./min. The sheet at this point was stable and lint free. The hand was dry and somewhat papery.

This sheet material was then cross-stretched in a clip tenter without over-feed to provide an area equal to about 77% of the original as-spun area. The sheet width was increased from 34 inches to 40 inches in this operation. The air surrounding the sheet was maintained at 105° C. for a 30 ft. zone. The sheet passed through the zone at a rate of 50 ft. per minute.

A second sample from Step *b* was cross-stretched to 51 inches, this time without the application of heat. The sheet contracted to 39 inches width when removed from the clip tenter. Both cross-stretched sheets were porous, resilient, and had a high compression recovery as compared to the original sheet. The hand was soft and dry, the products had good drapeability and good fiber stability.

#### EXAMPLE 2

The as-spun cold-calendered sheet of Example 1 was point bonded as in Example 1 except that treatment by the second rolls was omitted. Thus a small point pattern was first imposed on one side of the sheet and then a large point pattern was imposed on the other side of the sheet using only the first and third pair of rolls mentioned in Example 1. The patterns were imposed by passing the sheet through the two pair of rolls, at 20 yds./min., the temperature of each of the embossing rolls being 162° C. The patterns of the two embossed rolls were imposed on opposite sides of the sheet.

The bonded sheet was then softened and stretched on a power driven button breaker as in Example 1. The sheet width was reduced from 52 to 48.5 inches by this operation. The sheet was stretched longitudinally 6% in the button breaker whereas its density was reduced from 17.7 lbs./ft.<sup>3</sup> to 15.2 lbs./ft.<sup>3</sup>, as measured under load of 2.81 lbs./in.<sup>2</sup>. After the softening operation on the button breaker, the sheet was heat-treated and stretched to effect a 10% elongation and a 33% reduction in width. The width of the stretched sheet was reduced from 48.5 inches to 32.5 inches. The hot-stretching operation was accomplished in the apparatus of FIGURE 8 with rolls 50 feet apart in the hot air zone. The air temperature was 134° C. at the middle of the tower. The sheet speed was 50 ft. per minute. The sheet density increased from 15.2 lbs./ft.<sup>3</sup> to 16.4 lbs./ft.<sup>3</sup>. The resulting product was stable and lint free. The hand was dry and papery. The structure was still quite extendable in the cross-direction.

This sheet was then finished by cross-stretching on the

clip tenter machine without overfeed in an air atmosphere at 20° C. The product had a soft, dry hand.

#### EXAMPLE 3

A film-fibril sheet of 50-inch width and weighing 1.0 oz./yd.<sup>2</sup> was prepared as in Example 1. The sheet was point-bonded and perforated to obtain a pattern similar to that of FIGURE 4 but without the tiny emboss points. The point perforating operation was accomplished by passing the sheet product between a single pair of rolls, one being a rubber roll and the other a pattern roll with raised points. The points were arranged in a diamond pattern on the roll, the points being located 3.58 mm. (0.141 in.) apart across the roll and 7.18 mm. (0.283 inch) around the circumference. The tip of the points were 1.01 mm. (0.040 inch) above the roll surface. The points were conical in shape. Nip pressure was sufficient to cause the points to perforate the sheet. The sheet speed was 12 yds./min. and the roll temperature was 187° C. The resulting sheet was softened on a power driven button breaker as in Example 1. The width of the sheet was thereby reduced from 50 inches to 45 inches and the density was reduced from 16.5 lbs./ft.<sup>3</sup> to 12.7 lbs./ft.<sup>3</sup>. The softened sheet was then heat treated at various temperatures to effect about 10% elongation and an additional 43–51% reduction in width, based on 45 inches at start. Hot stretching in the apparatus of FIGURE 5 at temperatures of 131°, 132, 133 and 134° C. produced sheets with widths of 25, 24, 23, and 22 inches respectively, measured under the same tension in a frame. The sheets had progressively increasing elongation in the cross-dimension. The products at this point were stable and lint free. The hand was dry and very papery. The hot-stretched sheet was then cross-stretched without over-feed in room temperature air to an area equal to about 78% of the original area before point-bonding. The sheet was fed into the apparatus at 23 inches and was taken out at 39 inches. The resulting product was porous, resilient, had a high compression recovery and a soft dry hand. The sheet also had good fiber surface stability, drapeability, and still had some extendability in the transverse dimension.

#### EXAMPLE 4

The starting material for this experiment was a 44-inch wide, cold rolled sheet weighing about 1.5 oz./yd.<sup>2</sup>. It was a nonwoven film fibril sheet prepared by flash spinning linear polyethylene according to the method of Blades and White U.S. Patent 3,081,519, using trichlorofluoromethane as a solvent. The sheet was bonded by passing successively through two pairs of rolls. Each pair consisting of a steel roll with raised points working against a rubber covered steel roll of 80 "Shore" A hardness. The bonding patterns employed were the same as in Example 2. The bonding rolls were operated at a surface speed of 18 y.p.m. and at a temperature of 157° and 190° C., for the rib and diamond patterns, respectively. Nip pressure was set at a level sufficient to form a fine rib pattern with the first pair of rolls and at a level sufficient to perforate the sheet with a diamond pattern in the second pair of rolls.

The bonded sheet was then softened and its density reduced on the modified button breaker. The width of the sheet was reduced from 44 inches to 40.5 inches in the operation, and the density as measured under a 2.81 lb./in. load was reduced from 18.4 to 15.8 lbs./ft.<sup>3</sup>. The sheet was then drawn and simultaneously heat-treated to effect a 12% elongation and a 21% further reduction in width (40.5 inches reduced to 32 inches). The hot stretching was effected between two rolls 50 ft. apart in a hot air atmosphere starting at 134° C. at a speed of 50 ft./min. through the zone. Both surfaces of the heat-treated nonwoven structure was stable and lint free. The hand was dry but papery.

The sheet was then cross-stretched to 44 inches wide in a clip tenter at 50 yards/minute at 20° C. After release from the machine the sheet return to a width of 40 inches.

The density was then 14.6 lbs./ft.<sup>3</sup>. The product showed a marked increase in resilience and compression recovery compared to material before hot-stretching and had a dry hand, good fiber stability, pleasing drapeability, and moderate extendability in the transverse direction.

#### EXAMPLE 5

This example shows sheets made without the essential Step *a* of the present invention. The starting material was prepared by flash spinning linear polyethylene according to the method of Blades and White U.S. Patent 3,081,519, using trichlorofluoromethane as a solvent, and then depositing the material to form a sheet. After consolidation, the sheet was bonded by being passed successively through two pairs of rolls, each pair consisting of an embossing roll working against a rubber covered steel roll. The raised point patterns on the two embossing rolls were arranged in diamond patterns both similar in arrangement and dimensions to that described for the diamond pattern in Example 1. The two sets of rolls were operated at a surface speed of 18 y.p.m. the temperature being 200° C. in each case. The rolls were arranged so as to impose a diamond pattern throughout each side of the sheet.

The sheet, having a density 15.40 lbs./ft.<sup>3</sup> under 2.81 lbs./in.<sup>2</sup> load, was then heat treated without a button breaker treatment or other mechanical working. The sheet was heat treated and stretched to effect a 10% elongation and a 27% reduction in width. Heat treating was effected in a tower between two rolls 50 ft. apart in a hot air atmosphere which was at 134° C. half way up the tower, the speed being 38 ft./min. Both surfaces of the heat-treated nonwoven structure were well bonded, lint free, and durable. The hand was dry but papery.

The sheet was then cross-stretched without overfeed to an area equal to about 90% of the original as-spun area. The width was increased from 38 inches to 48 inches in an air atmosphere of 105° C. in a 30 ft. zone, passing through the zone at a rate of 50 ft./min. the porosity was essentially zero. The product was stiff with little or no drapeability. It was concluded that the omission of the mechanical working step (Step *a*) resulted in a sheet of inferior properties.

#### EXAMPLES 6 TO 8

Table 1 shows data obtained from three experiments using the general procedure of Example 1. In each of these the complete process of this invention was used. Example 6 was prepared by imposing a rib pattern on first one side of the sheet and then on the other side. The rib pattern was the same as that obtained from the first pair of rolls in Example 1. The original as-spun sheet had a weight of 1.5 oz./yd.<sup>2</sup> before treatment. The sheet weight in oz./yd.<sup>2</sup> after each step in the process is shown in the table. The changes in weight per square yard were due to changes in the area of the sheet during processing. The density of the sheet at various points in the process is shown also in Table 2. The density values are reported for three different compressive conditions. To determine density a stack of 10 sheets each 2½" x 3" (6.35 cm. x 7.62 cm.) was prepared and weighed. The thickness of this stack was then determined under a load of 0.93 lb./in.<sup>2</sup> after applying for two minutes. After recording the thickness under this load, additional weight was added to provide 2.81 lbs./in.<sup>2</sup> for two minutes. The thickness was again recorded, and the part of the weight was removed to obtain 0.93 lb./in.<sup>2</sup> load. After two minutes the thickness was again recorded. It will be apparent from Table 1 that, regardless of the method of measurement, a real increase in bulk occurs in each successive step of the process, e.g. the density decreases with each successive step.

Similar measurements were made on a sheet processed from 1.00 oz./yd.<sup>2</sup> film-fibril sheet. This is shown for Example 7 in Table 2. The sheet was prepared by passing through two sets of rolls one of which impressed a rib



pattern on one side and the other of which impressed a diamond pattern on the reverse side of the sheet. A substantial increase in bulk was achieved in the process as shown by the density measurements.

In Example 8, as shown in Table 1, a 1 oz./yd.<sup>2</sup> sheet was passed through three successive pairs of rolls to impress a rib pattern on one side as in Example 1, and then a rib pattern plus a diamond pattern on the second side. A substantial increase in bulk was obtained.

In Examples 6, 7 and 8 each of the sheets developed a less slick hand during processing, had higher resilience and higher bulk.

The finished sheets had very high porosity compared to the original embossed sheet as shown in Table 2. Porosity or so-called "Frazier Porosity" was measured by the ASTM Method D: 737-46 using the apparatus of Frazier Precision Instrument Company.

TABLE 1.—HEAT SET STUDIES—DENSITY AND POROSITY

	Basis Weight, oz./yd. <sup>2</sup>	Porosity, ft. <sup>3</sup> air/ft. <sup>2</sup> /min.	Density, * g./cm. <sup>3</sup> at—		
			(1) 0.93 p.s.i.	(2) 2.81 p.s.i.	(3) 0.93 p.s.i.
Example 6:					
(a) Embossed only .....	1.18	Negligible	.280	.292	.288
(b) Embossed; agitated .....	1.08	0.65	.243	.266	.280
(c) Embossed; agitated; hot draw .....	1.55	-----	.224	.248	.240
(d) Embossed; agitated; hot draw; reframe .....	1.21	5.29	.203	.229	.222
Example 7:					
(a) Embossed only .....	0.98	Negligible	.267	.289	.282
(b) Embossed; agitated .....	0.87	3.61	.202	.231	.224
(c) Embossed; agitated; hot draw .....	1.68	1.87	.185	.209	.200
(d) Embossed; agitated; hot draw; reframe .....	1.11	19.4	.173	.204	.196
Example 8:					
(a) Embossed only .....	0.98	Negligible	.287	.303	.299
(b) Embossed; agitated .....	0.92	2.19	.234	.259	.253
(c) Embossed; agitated; hot draw .....	1.56	-----	.205	.231	.221
(d) Embossed; agitated; hot draw; reframe .....	1.15	10.33	.200	.230	.219

\* (1) Initial loading; (2) intermediate loading; (3) after partial removal of loading.

## EXAMPLE 9

Part of the sheet material from Example 7c was segregated from the rest and coated before cross-stretching. The segregated portion of the hot-drawn sheet was passed through a gravure coater, which applied a latex coating of a copolymer of ethylene/alpha-beta, beta-unsaturated aliphatic carboxylic acid (about 88/12% by weight) in a liquid vehicle. The latex contained the following:

	Percent
Copolymer	22
Clay pigment	22
Water	56

The wet-coated sheet was passed directly into a tenter frame, where it was dried at 110° C. and simultaneously cross-stretched. The sheet width was increased in this operation from 19 inches to 40 inches in the tenter frame. After standing for several hours off the frame, the sheet retracted in width to 37.3 inches. The sheet weight was 1.68 oz./yd.<sup>2</sup> before coating and 1.23 oz./yd.<sup>2</sup> after coating, cross-stretching, drying, and relaxing. The coating operation stabilized the surface fibers so that the resulting sheet was not abraded easily in a simple pencil-eraser test. The product was especially satisfactory for use in garments because of its high porosity and its soil resistance. The Frazier porosity for the coated, dried, cross-stretched sheet was 16.1 ft.<sup>3</sup> of air/ft.<sup>2</sup> of surface/min., being almost as high as the porosity for the uncoated, cross-stretched sheet (Example 7d, Table 1).

The procedure was repeated with an aqueous dispersion of polyvinyl chloride and other nonelastomeric polymers to obtain essentially the same results.

The polymer melting temperature of the film-fibril materials may be determined as follows: The film-fibril materials are characterized by a melting range that may extend over several degrees C. The upper limit of the melting range of the film-fibrils, as referred to herein, is the temperature at which the highest peak occurs in a differential thermal analysis. The analysis is performed using

a Du Pont 900 Differential Thermal Analyzer and a standard heating block for 2 mm. capillary tubes. A 1-2 mg. sample of polymer is placed in one tube and an equal weight of finely ground glass particles in the other. The block containing the tubes is heated at a rate of 5° C./min. The difference in temperature recorded between the tubes is plotted against the temperature of the polymer sample. The maximum peak of the resultant thermogram is taken as the polymer melting temperature or upper limit of the melting range.

What is claimed is:

1. A method for producing a high bulk, nonwoven fabric of improved resilience and compressional properties and exhibiting a dry, less slick hand, said method comprising applying the following succession of process steps to a polymeric nonwoven film-fibril sheet which has

been point-bonded at a multiplicity of points throughout the sheet:

- (a) applying brisk local mechanical stroking action to spaced-apart areas across the entire width of the sheet while applying tension to the sheet, the agitation and tension being less than would cause rupture of the sheet and substantial separation of fibers in the point-bonded areas,
  - (b) then heating the sheet while applying sufficient tension in one dimension of the plane of the sheet to at least prevent shrinkage in that dimension while substantially avoiding restraint in the second dimension of the plane of the sheet, the temperature of the sheet being below the melting point of the polymer but within 35° C. thereof, the applied tension and heat in combination being such as to cause a 5 to 60% shrinkage of the sheet in the unrestrained second dimension, and
  - (c) then stretching the sheet in the second dimension to restore at least 25% of the loss incurred in that dimension during the preceding hot stretching operation.
2. Method according to claim 1 wherein during step (a), the over-all density of the sheet is reduced to less than 20 lbs./ft.<sup>3</sup>, as measured with a 2.81 p.s.i. compressive load.
  3. Method according to claim 1 wherein the point bonding of the sheet has been effected by fusion of polymer in small discrete areas.
  4. Method according to claim 1 wherein the heating and tensioning of step (b) are effected to stretch the sheet 5 to 25% in the first dimension.
  5. Method according to claim 1 wherein the heating of step (b) is effected at a temperature within 15° C. of the polymer melting point.
  6. Method according to claim 1 wherein the stretching of the sheet in step (c) restores at least 50% of the loss incurred in that dimension during step (b).
  7. Method according to claim 1 wherein the polymer is a polyolefin.

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8. Method according to claim 1 wherein the polymer is polyethylene.

9. Method according to claim 1 wherein intermediate to step (b) and (c) the sheet is provided on at least one surface thereof with a coating of a thermoplastic polymer. 5

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