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(54) Title: THERMALLY BONDABLE FABRICS, PREPARATION AND USE

#### (57) Abstract

Thermally bonded fabrics having high normalized tensile strength are disclosed. Blends of high molecular weight linear ethylene polymers and low molecular weight linear ethylene polymers provide improved staple fiber-forming capabilities over that found with either polymer taken alone. Staple fibers formed from the blends have Q values above 4.5 and/or  $I_{10}/I_2$  values above 7. The staple fibers formed from the blends of high molecular weight linear ethylene polymers and low molecular weight linear ethylene polymers have a broader thermal bonding window than either linear ethylene polymer taken alone. At least one of the linear ethylene polymers useful for blending for forming staple fiber can be a higher alpha-olefin in the  $C_3$ - $C_{12}$  range, preferably in the  $C_3$ - $C_8$  range.

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### THERMALLY BONDABLE FABRICS, PREPARATION AND USE

Improvements are made in the making of staple fibers and fibrous products from linear ethylene polymer by melt spinning a blend comprising at least one high molecular weight linear ethylene polymer having a melt index less than 25 grams/10 minutes and at least one low molecular weight linear ethylene polymer having a melt index greater than 25 grams/10 minutes at blend ratios sufficient to form linear ethylene polymer staple 10 fibers having a "Q" value above 4.5 and/or a melt index ratio  $I_{10}/I_2$  of at least 7.0.

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Linear low density polyethylene (LLDPE) is an ethylene polymer prepared using a coordination catalyst 15 in the same manner used in preparing linear high density polyethylene (HDPE), and is actually a copolymer of ethylene and at least one higher alpha-olefin. expression "linear ethylene polymers" includes those linear ethylene polymers which have from 0 percent to about 30 percent of at least one higher alpha-olefin of 3 to 12 carbon atoms copolymerized with the ethylene.

According to Modern Plastics Encyclopedia, as 25 defined today, linear high density polyethylene

generally has a density in the range of about 0.941 gm/cm<sup>3</sup> to about 0.965 gm/cm<sup>3</sup>. Skilled artisans realize that the density can vary in accordance with the reaction conditions and type of catalyst used. It is also known that densities a little above that range can be achieved by special techniques, such as by annealing the polymer. It is known that polymerization conditions which will produce a homopolymer having a density of about 0.965 gm/cm<sup>3</sup> will produce a copolymer of ethylene 10 and higher alpha-olefin having a density lower than the said 0.965 gm/cm<sup>3</sup>, the extent of the lowering of the density being directly related with the amount of the higher alpha-olefin used.

15 It is known from EPO 85 101380.5 (U.S. Patent 4,830,907) that linear low density polyethylene (LLDPE) can be made into fibers by melt-spinning and that even very fine fibers can be produced.

20 Whereas LLDPE has been introduced in the market place as a polymer suitable for making fibers, it is also known that not all versions and varieties of LLDPE are entirely adequate for commercial production of staple fibers. In particular, the fabric strength of 25 carded staple-based fabrics has generally been significantly less than fabrics made from carded staplebased polypropylene fibers.

It is recognized in the art of making LLDPE 30 polymers that the density of the LLDPE is affected by the amount and kind of olefin comonomer which is copolymerized with the ethylene and, to some extent, by the process conditions and catalyst used. A given mole percent of, e.g., propylene in the copolymer will reduce the density of the polyethylene less than the same mole

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percent of a higher olefin comonomer. The melt index or MI (sometimes called melt flow rate or MFR) as measured by ASTM D-1238 (E) conditions  $190^{\circ}$ C/2.16 kg, is also affected to some degree by the kind and amount of olefin comonomer in the copolymer, and is also affected to some extent by the coordination catalyst used, the polymerization conditions, and/or by any telogens or chain regulators or other reactants which may be present during polymerization.

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It is also recognized in the art that there are important distinctions between linear polyethylenes (which includes LLDPE polymers), and branched-chain ethylene polymers, which are made using a free-radical catalyst and are generally referred to as LDPE (low density polyethylene), and were also known in the past as ICI-type polyethylene and as HPPE (high pressure polyethylene). Our invention deals with linear polyethylenes.

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U.K. Patent Application GB 2,121,423A teaches away from our invention claimed hereinafter, because the U.K. patent application teaches away from the use of "linear" polyethylene staple fibers having polymer "Q" values of more than 4 in order to prepare strong, thermally bonded fabrics, for the following reasons. In particular, the patent application relates to hot-melt adhesive binder fibers comprising specific polyethylene 30 resin compositions. Its Example 1 discloses cut short fibers of linear polyethylene homofilaments wherein the polyethylene has a density of 0.92; an MI of 25; and a "Q" value of "2.82". It also discloses the use of those homofilaments (having a Q value of 2.82) in preparing

hot-calendered non-woven fabrics from blends of such fibers with poly(ethylene terephthalate) staple fibers.

adhesive fibers comprising a polyethylene resin composition (C) alone, consisting of 50 to 100 percent by weight of a polyethylene (A) having a density of 0.910 to 0.940 g/cm<sup>3</sup> and a Q value (Q=Mw /Mn) of 4.0 or less and 50 to 0 percent by weight of a polyethylene (B) having a density of 0.910 to 0.930 g/cm<sup>3</sup> and a Q value of 7.0 or more based on said composition, or composite fibers which contain said composition (C) as one of the composite components of said composite fibers and in which said composition (C) forms continuously at least a part of the fiber surface of said composite fibers."

Polyethylene B, however, is not a "linear"
ethylene polymer. Polyethylene A is a linear ethylene
polymer. However, when the polyethylene resin
composition (C) consists of 100 percent polyethylene
(A), the claim expressly requires that the Q value be
"4.0 or less". Further, the British patent application
teaches that one advantage of the claimed invention
(when used as a binder fiber) is "superior strength" of
the non-woven fabric. Accordingly, it clearly teaches
away from the invention described hereinafter.

published May 3, 1989 relates to polymers used in making thermally bonded fabrics, but has no working examples directed to discontinuous staple fibers. All the working examples of invention of the European patent application are directed to the use of "continuous" filaments prepared by melt-spinning at linear spinning velocities of at least 3,500 meters/minute. Its

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teachings are, therefore, much less relevant than the aforementioned U.K patent application.

blends or in-situ polymerized blends, of linear ethylene polymers, especially LLDPE, having certain properties, are surprisingly well suited for making staple fibers and yield products having strengths more competitive with those attainable in carded, thermally bonded webs of polypropylene. These blends have a broader molecular weight distribution than a sole linear ethylene polymer resin produced at the same MI (melt index).

process for preparing a fabric, which fabric is either thermally bonded or bondable, from filaments including thermoplastic fine denier discontinuous staple fibers having an average denier per filament in a range of from 0.1 to 15 d.p.f., and having filament lengths up to 30 cm., and in which the staple fibers are prepared from a meltspun blend of linear ethylene polymers characterized in that the polymer blend in the staple fibers has a Q value above 4.5, wherein Q is defined as weight average molecular weight, as determined by gel permeation chromatography.

A second broad aspect of the invention is a process for preparing a fabric, which fabric is either thermally bonded or bondable, from filaments including thermoplastic fine denier discontinuous staple fibers having an average denier per filament in a range of from 0.1 to 15 d.p.f., and having filament lengths up to 30 cm., and in which the staple fibers are prepared from a meltspun blend of linear ethylene polymers characterized in that the polymer blend in the staple fibers has an

 $I_{10}/I_2$  value of at least 7, wherein  $I_{10}$  is determined by ASTM D-1238(N) conditions, and  $I_2$  is determined by ASTM D-1238(E) conditions.

A third broad aspect of the invention is a thermally bonded fabric wherein the thermally bonded fabric has a normalized strip tensile strength of at least 3,000 grams.

A fourth broad aspect of the invention is polymeric fibers including discontinuous staple fibers wherein the polymer has an I<sub>10</sub>/I<sub>2</sub> value of at least 7 and the polymer is a blend of (A) at least one high molecular weight linear ethylene polymer having a MI value less than 25 grams/10 minutes and a density above 0.91 grams/cm<sup>3</sup>, and (B) at least one low molecular weight linear ethylene polymer having a MI above 25 grams/10 minutes and a density above 0.91 grams/cm<sup>3</sup>.

A fifth broad aspect of the invention is a 20 polymer blend characterized in that the polymer blend has an I<sub>10</sub>/I<sub>2</sub> value of at least 7 as determined by ASTM D-1238(N) and (E) respectively and further wherein the blend is formed by blending (A) and (B) in appropriate 25 weight ratios wherein (A) is at least one high molecular weight linear ethylene polymer having an MI measured in accordance with ASTM D-1238(E) (190°C/2.16 kg) less than 25 g/10 minutes and a density above 0.91 g/cm $^3$ ; and, (B) is at least one low molecular weight linear ethylene 30 polymer having a MI measured in accordance with ASTM D-1238(E) (190°C/2.16 kg) greater than 25 g/10 minutes and a density above 0.91  $g/cm^3$ .

A sixth broad aspect of the invention is a process for preparing polymeric fibers characterized by

the steps of (A) Melt-spinning the polymer into meltspun filaments; (B) Hauling off the meltspun filaments at a speed in a range of from 60 to 2,000 meters/minute; and, optionally, (C) drawing and/or crimping and/or cutting the hauled-off meltspun filaments by conventional means.

A seventh broad aspect of the invention a process wherein molten linear ethylene polymer fine denier staple fibers are spun at commercially feasible throughput rates, optionally followed by mechanical drawing to produce fiber sizes of from 0.1 to 15 denier/filament and used in making a web or fabric, characterized by spinning a blend comprising (A) at least one high molecular weight linear ethylene polymer having a MI measured in accordance with ASTM D-1238(E) 15 (190°C/2.16 kg) less than 25 g/10 minutes and a density above 0.91 g/cm<sup>3</sup>, and (B) at least one low molecular weight linear ethylene polymer having a MI measured in accordance with ASTM D-1238(E) (190°C/2.16 kg) greater 20 than 25 g/10 minutes and a density above  $0.91 \text{ g/cm}^3$ , forming linear ethylene polymer staple fibers having a Q value above 4.5, wherein Q is defined as weight average molecular weight divided by number average molecular weight, as determined by gel permeation chromatography. 25

An eighth broad aspect of the invention is a blend wherein the ratio of the high molecular weight linear ethylene polymer and low molecular weight linear ethylene polymer is sufficient to provide a blend having a MI value in the range of 0.1 to 40 grams/10 minutes and a density in the range of 0.94 to 0.96 grams/cm<sup>3</sup>.

In still another aspect, the invention is a means for increasing the thermal bonding window of staple fibers made from linear polyethylene by blending

high and low molecular weight linear polyethylenes, spinning into staple fiber and thermally bonding the fiber into fabric.

Preferred aspects of the invention include the 5 following.

The average denier per filament of the fiber is preferably in a range from 1 to 15 d.p.f., and more preferably in a range from 2 to 6 d.p.f. The use of d.p.f.'s below 1 makes silk-like products, but adversely affects productivity and tends to increase downstream problems such as poor cardability.

Q value is preferably in a range from 5.5 to 15 10.

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 $I_{10}/I_2$  value (ratio) is preferably in a range from 10 to 20.

- 20 The polymer blend is usually, preferably formed by blending in-situ during polymerization of the polymer. However, it is sometimes preferred to blend discrete polymers.
- 25 The thermally bonded fabric preferably has a normalized strip tensile strength of at least 3,000 grams; more preferably 3,600 grams; and most preferably 3,700 grams.
- 30 The polymer blend preferably has a density in a range from 0.94 to 0.96 gm/cm<sup>3</sup>.
- It is preferred that at least one of the linear ethylene polymers comprise a copolymer of ethylene with 35

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at least one  $C_3$ - $C_{12}$  olefin (particularly octene, especially octene-1).

It is preferred to use haul-off speeds in a range of 60 to 2,000 meters/minute during the melt spinning step.

It is preferred and that at least one of the linear ethylene polymers by LLDPE.

Polymer blends of our invention wherein a high molecular weight linear polyethylene, especially LLDPE, and a low molecular weight linear polyethylene, especially LLDPE, are uniformly blended and used in making fibers, are found to exhibit not only the good hand, softness, and drape which one might expect of a linear polyethylene, especially the LLDPE variety, but a carded, thermally bonded web (fabric) of surprisingly high strength is produced at spinning rates which are very suitable for commercial operations.

In the presently claimed invention, the important thing is to blend an appropriate amount of the low molecular weight linear polymer with the high molecular weight polymer to produce the improvement in making staple fibers from the high molecular weight polymers. We have found that in obtaining the desired improvement, the blends which are preferred will have a blend density of above 0.91 gm/cm<sup>3</sup>, especially above 0.94 gm/cm<sup>3</sup> and up to 0.96 gm/cm<sup>3</sup>.

The types of "fabric" that may be prepared by our invention are essentially all types of fabric now made and new types of fabric that could be made in the future. Our invention is primarily directed towards

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textile fabrics. It does extend to industrial fabrics as industrial filter cloth.

In this disclosure the expression "discrete blends" is in reference to the mixing of linear polymers which are made separate from each other, each under its own set of reaction conditions, possibly at different times and/or places, the mixing taking place after the polymer has been collected from the respective reactors.

The expression "in-situ polymerized reactor blends" is in reference to blends of at least two linear polymers which have differing properties and which are prepared conjointly, but under differing reaction conditions, whereby the properties of each are varied, one from another, and the so-prepared polymers are immediately and intimately mixed within the reaction system before removal therefrom. There are several ways known by skilled artisans for making in-situ polymerized blends, such as in U. S. Patent 3,914,342, and the invention is not limited to any one of the methods.

The linear ethylene polymer resin used for the

"high molecular" weight portion of the blend of the

present invention can be any which may contain an amount
of a C<sub>3</sub> to C<sub>12</sub> olefin comonomer, copolymerized with the
ethylene, sufficient to yield a density in the range of
0.91 gm/cm<sup>3</sup> to 0.965 gm/cm<sup>3</sup>, and has a MI of less than
25 gm/10 minutes, preferably less than 20 gm/10 minutes.

Preferably, the comonomer is a C<sub>3</sub> to C<sub>8</sub> olefin, such as
propylene, butene-1, hexene-1, 4-methyl pentene-1,
octene-1, and the like, especially octene-1, and can be
a mixture of olefins such as butene/octene or
hexene/octene. The above stated MI and density ranges

include linear polyethylenes which contain no comonomer as well as those which contain at least one comonomer.

The linear ethylene polymer resin used for the "low molecular" weight portion of the present blend can 5 be any which contains an amount of C3 to C12 olefin comonomer, copolymerized with the ethylene, sufficient to yield a density in the range of 0.91  $\mathrm{gm/cm^3}$  to 0.965 gm/cm<sup>3</sup>, and has a MI of greater than 25 gm/10 minutes preferably greater than 40 gm/10 minutes. Preferably, the comonomer is a C3 to C8 olefin, such as propylene, butene-1, hexene-1, 4-methyl pentene-1, octene-1 or the like, especially octene-1, and can be a mixture of olefins such as butene/octene or hexene/octene. The 15 above stated MI and density ranges also apply to linear polyethylenes which contain no comonomer as well as those which contain at least one comonomer.

The melt index (MI), also known as  $I_2$ , of the 20 polymers is measured in accordance with ASTM D-1238 using Condition E (also known as 190°C/2.16 kg) unless otherwise specified and is a measurement of the amount (grams) of melted polymer which is extruded from the orifice of the melt index barrel in 10 minutes. 25 melt index (MI) is an indication of relative molecular weight, with a given MI numerical value indicating a higher molecular weight than a greater MI numerical value.

"Gel permeation chromatography" (herein called "GPC"), also known as "size exclusion chromatography", is a measurement made to characterize molecular weight distribution of a polymer and is well known in the 35 industry. Data reported by this GPC technique includes weight average molecular weight  $(MW_{wa})$ , number average

molecular weight (MWna), and weight average molecular weight divided by number average molecular weight (MWwa/MWna). Of these, the  $\text{MW}_{\text{Wa}}/\text{MW}_{\text{na}}$  is of the most interest, indicating broadness of molecular weight distribution. The higher the MWwa/Mana ratio, the 5 broader the molecular weight distribution (sometimes called polydispersity) of the resin. Molecular weight distribution, indicated by MWwa/MWna, or "Q", or by the ratio of  $I_{10}$  (as measured by ASTM D-1238 (N) conditions 10  $190^{\circ}\text{C}/10.0 \text{ kg}$ ) divided by  $I_2$  has been found to influence thermal bonding of staple fiber. We find that resins having Q values above 4.5, or  $I_{10}/I_2$  values above 7 have shown extreme utility in broadening the thermal bonding window of staple fibers made from linear polyethylene. 15 (The "bonding window" is the temperature range over which staple fiber can be satisfactorily thermally bonded.) For example, a single linear ethylene polymer having a melt index of 17 grams/10 minutes, a density of 20 0.950g/cc and a Q value of from 4 to 4.5 has a thermal bonding window of 5°C. A discrete polymer blend of the present invention having a melt index of 17 grams/10 minutes, a density of 0.950 g/cc and a Q value of 5.75to 6 has a thermal bonding window of 10°C. Thus staple 25 fibers made from the polymer blends of the present invention have broader thermal bonding windows.

The normalized fabric tensile strength (per 2.54 cm width) of a thermally bonded web (fabric) is measured on 1-inch wide by 4-inch long (2.54 cm by 10.16 cm) samples by measuring conventionally the total breaking load, and then normalizing it to 1-ounce/yard² (about 33.9 gm/m²) and measured in grams. The tenacity of fibers is measured as "grams/denier".

Prior to our invention it has been found to be difficult to make fine denier staple fibers of linear ethylene polymer for conversion into carded thermally bonded webs, especially at the high production rates and broad thermal bonding windows normally desired in commercial operations, which result in fabrics having typically more than about 50 percent of the normalized fabric tensile strength of fabrics obtained for carded, thermally bonded polypropylene staple fiber at 10 comparable MI's. Greater strength of thermally bonded linear ethylene polymer staple fiber, including LLDPE, is desirable in various products, such as, for example, diaper cover stock, medical garments and feminine hygiene products. 15

Since the formation of staple fibers includes such complex and varied operations as melt drawing, mechanical stretching, crimping, and cutting, the requirements for the polymer are rigorous. The first 20 requirement of the polymer is to withstand the melt drawing of the spinneret extrudate to a filament size ranging from approximately 0.1 to 15 denier at commercially feasible throughputs, especially throughputs ranging from 0.1 to 0.67 grams/minute/hole (gm/min./hole). These throughput variations are dependent not only upon desired production rates, but also equipment design limitations (such as compact melt spinning as compared to conventional melt spinning). 30 Subsequently, this resultant filament can be stretched at draw ratios ranging up to 6:1 to produce filament at the final desired fiber size, typically ranging from 0.5 to 6.0 denier.

35 After stretching (or drawing) the fiber to the appropriate denier size the fiber is usually crimped,

via a stuffer box, air texturizer or other device, and then cut to the desired length. Crimping imparts a thermo-mechanical deformation to the fiber, causing it to have numerous bends and greater entanglement. These bends, or crimps, are useful when carding fiber in order to create some degree of web cohesion prior to bonding. Staple fiber, especially for non-woven applications is usually carded into a web prior to being bonded.

10 Typical bonding techniques include hydrodynamic entanglement (commonly referred to as spunlacing), chemical bonding, and most frequently, thermal bonding. Thermal bonding is typically accomplished by passing carded web through heated calender rolls, infrared oven, ultrasonic bonding device, or through-air bonder. Most frequently, thermal bonding of these carded webs is achieved via heated calender bonding. The technique used to differentiate these various linear ethylene polymers will be described farther on in this disclosure, but it employs essentially all of these processing steps.

good melt draw-down of the filaments into small diameter fibers under the desired production conditions makes staple fiber spinning with a relatively high molecular weight linear ethylene polymer very difficult. While it is already realized that increasing molecular weight of a polymer results in an increased strength (tenacity) of articles formed from that polymer, the increased molecular weight also results in much greater staple spinning problems. That is, the higher molecular weight linear ethylene polymers (including LLDPE) are not well suited for spinning at commercially viable and economical rates. What has not been known is that there

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are unexpected benefits obtained by blending a low molecular weight linear ethylene polymer, especially LLDPE, with a high molecular weight linear ethylene polymer, especially LLDPE, especially in spinning, then carding and bonding staple fiber made from these polymers.

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For purposes of describing the present invention, a linear ethylene polymer, (including HDPE and LLDPE) having a MI value of less than 25, preferably less than 20, especially less than 5, and, optionally, as low as 0.1, is considered to be in the high molecular weight range; the lower the MI value, the higher is the molecular weight. Linear ethylene polymer having a MI 15 value in the range of 25-40 may, in some aspects, be considered to be an "intermediate" molecular weight range, but in relating the present invention, it is considered as being on the "high" end of the low molecular weight range. Linear ethylene polymer having 20 a MI in the range above 40, especially above 45, is considered to be in the low molecular weight range and is not considered (in relating the present invention) to be in an "intermediate" molecular weight range. Whereas MI values exceeding 300 can be used as the low molecular weight polymer, especially if the high molecular weight portion of the present blend has a MI value below 1 or 2, it is preferred that the MI values of the low molecular weight polymer be not more than 600, 30 preferably not more than 500. Above 500-600 MI, one might encounter problems such as diminished melt strength properties. In a general sense, one should consider that the lower the MI value of the high molecular weight resin, the greater the need to blend it 35 with an off-setting amount of a linear ethylene polymer

having a high MI value as the low molecular weight polymer.

one can calculate the MI values and the density values of the polymers used in the blends of the present invention and obtain values which are reasonably close to the actual values obtained by actual measurement of the blend.

The following formula may be used to calculate the melt index of polymer blends:

ln blend = (fraction A)ln A + (fraction B) ln B

The following formula may be used to calculate the density of polymer blends:

pblend = (fraction A)ρA + (fraction B)ρB

The present invention employs, in a blend, an amount of low molecular weight linear ethylene polymer 20 which is effective in overcoming the deficiencies of high molecular weight linear ethylene polymer in the making of staple fiber based webs or fabrics and enables one to utilize the high molecular weight linear ethylene 25 polymer in the demanding processing conditions of staple fiber spinning, carding and bonding, while substantially maintaining the inherent strength of the high molecular weight linear ethylene polymer. This strength, when evaluated in bonded fabric form, improves by as much as 30 165 percent or more at a comparable MI's. The strengths of thermally bonded fabric made from these higher molecular weight blended polymers, approach the strength of typical commercial polypropylene-based fabrics by as much as 75 percent or more.

the present invention.

The thermally bonded webs or fabrics prepared using the blends of the present invention can be made wettable by incorporating into one or both of the polymers certain additives such as in U.S. 4,578,414. Furthermore, the addition of minor amounts of additives, such as colorants and pigments is within the purview of

The webs or fabrics made using the present

10 blends exhibit excellent softness, good gamma
irradiation stability, high strength, and good thermal
bondability to itself and to other thermoplastic films
or webs, such as other polyolefins.

15 The ratio of high molecular weight linear ethylene polymer to low molecular weight linear ethylene polymer in the blend is largely dependent on the MI of each. Generally, the amount of low molecular weight polymer used in modifying the high molecular weight 20 polymer is desirably about the minimum amount needed to render the high molecular weight polymer processable at the desired spinning rate and denier size and to improve thermal bonding strength of fabrics made therefrom. Conversely, the amount of high molecular weight polymer 25 added to the low molecular weight polymer is desirably an amount needed to render the low molecular polymer processable at the desired spinning rate and denier size and to improve thermal bonding strength of fabrics made 30 therefrom.

It is obvious that "trial and error" and
"successive approximation" techniques can be used to
determine the particular "weight ratios of the unblended
components" that are needed in order to make a blended
polymer having a Q value of at least 4.5 and/or an

 $I_{10}/I_2$  value of at least 7. Further, it is obvious that, if so desired, the polymer may be formed from more than two unblended components.

The examples hereinafter illustrate some embodiments of the present invention, but the invention is not limited to these specific embodiments.

It should be noted that in all the following examples the molecular weights were determined on a 10 Water's Associates Model No. 150C GPC. The measurements were made by dissolving polymer samples in hot, filtered, 1, 2, 4 trichlorobenzene (TCB). The GPC (Gel Permeation Chromatography) runs were made at 140°C in TCB. A flow rate of 1.0 ml/min was used and the columns used were 3 Polymer Laboratories 10 micron linear columns. Column performance was typically around 30,000 plates/meter (see Yau, W. W., J. J. Kirkland, D. D. Bly and H. J. Stoklosa, Journal of Liquid Chromatography, 20 125, 219 (1976)) as determined using 0.02 grams eicosane in 50 milliliters of TCB. Columns were disposed of if the plate count was below 20,000 plates per meter. Column performance was also monitored using the multiplied product of the spreading factor " $\sigma$ " and the 25 slope of the calibration curve "D" (see Yau, W. W., J. J. Kirkland, D. D. Bly and H. J. Stoklosa, Journal of Liquid Chromatography, 125, 219 (1976)). This value was typically around 0.081. Columns with values above 0.09 30 for  $D\sigma$  were not employed. The antioxidant butylated hydroxytoluene was added at a concentration of 250 parts per million to the TCB. The system was calibrated using narrow molecular weight polystyrene standards. following formula (see Williams, T. and I. M. Ward, J. 35 Polymer Sci, Polymer Letters, 6, 621 (1968)) was used to transform polystyrene molecular weights to polyethylene molecular weights:

Mw polyethylene = 0.4316 (Mw polystyrene)

The polyethylene samples were prepared at a concentration of 0.25 grams of polyethylene in 50 milliliters of TCB. The volume injected was 100 microliters.

## 10 Example I

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A discrete polymer blend comprising 40% by weight high molecular weight linear ethylene polymer (ethylene/octene, 2.3 MI, 0.917  $\rm g/cm^3$ ) and 60% low 15 molecular weight linear polyethylene (ethylene/octene, 105 MI, 0.953 g/cm<sup>3</sup>), a blended density of 0.939 g/cm<sup>3</sup>, a blended MI of 23, and a Q-value of about 6.82 is formed into fiber at about 0.4 g/min./hole and 1,200 meters/minute haul-off speed and collected on spool. 20 These filaments had average d.p.f of about 3.0. spooled yarn was then unwound and conventionally cut into 1.5 inch (3.81 cm) staple fibers. Fabric made from the resulting fiber has a maximum 1" (2.54 cm) normalized fabric strip tensile strength at 2686 grams, which is more than 55% of commercially available fibergrade polypropylene and over 140% of a single commercially available polyethylene at a comparable MI. Data are shown in Table I (shown after Example III).

A test on the above polymer blend is performed as follows: A one inch diameter extruder, 24 inches in length, containing a high shear type screw was used to melt and convey polymer to a positive displacement gear pump, which accurately metered polymer to the spin pack. Many different screw designs may be used, including one

commonly used in the extrusion industry for polyethylene, i.e., the low shear or barrier type screw. Spin pick configuration may vary considerably, but that used in tests described herein had, as its major components, a 40 micron sintered metal filter media and 5 a spinneret which had hole size of 600 microns, with length-to-diameter ratio of 4:1. Fibers are collected continuously onto spools using godet speeds necessary to achieve the desired denier per filament (dpf), relative 10 to the throughput. For example, a throughput of about 0.4 gms/min./hole requires a corresponding 6 inch (15.24 cm) diameter godet speed of about 2600 revolutions per minute to achieve about 3.0 dpf. at 1,200 meters/minute. For all the tests herein, fibers were melt spun with no 15 additional drawing. After collection of sufficient sample size, the fibers are cut off of the collection spool and cut into 1.5-inch (3.81 cm) staple fibers. Samples of these staple fibers (each 1.25 gm) are 20 weighed out and formed into slivers using a Roto Ring (manufactured by Spinlab, Inc.); a sliver is an ordered collection of fibers such that the fiber ends are randomized while the fibers themselves are all paralleled. The structure is about 10 cm wide by about 25 25.4 cm long after gently opening the sliver tow. opened sliver tow (simulating a carded web) is then fed into a Beloit Wheeler calender bonder for thermal tie down of the filaments where pressure and temperature are adjusted for optimal bonding conditions and fabric 30 strength.

above are found to have optimum bonding conditions at a top roll (or embossed roll with about 20 percent land area) temperature of about 115°C and a bottom roll

(smooth roll) temperature of about 118°C. The bonding pressure is typically found to be optimal at about 700 psig (4927.9 kPa) or about 199 pli (pounds per linear inch) (90.3 kgs/linear 2.54 cm or 35.6 kgs/linear cm). After forming a sufficient number of thermally bonded 5 fabrics under the same bonding conditions, a single sample is cut out of each bonded strip which measures one inch by four inches (2.54 by 10.16 cm), with the four inch dimension cut such that it is in the machine 10 direction. These samples are individually weighed and then tensiled by use of an Instron tensile tester affixed with a data systems adapter for measuring and recording load and displacement. The fabric samples are loaded into the Instron such that the one inch section 15 of each sample is held by the Instron jaws during the test, thus pulling the fabric in the machine direction. The mean value of the force required to break this fabric strip, normalized to one ounce per square yard 20 weight for every test described herein, is then recorded. For this particular example, the normalized fabric tensile strength (or bonded fabric tenacity) is about 2686 grams.

The above procedure is also carried out for Examples II-XIII.

Example II (for comparison; not example of invention)

A commercially available LLDPE, ethylene/octene copolymer, having a MI of 26, a density of 0.940 g/cm<sup>3</sup>, and a Q-value of 3.74 is formed into fiber at about 0.4 g/min./hole. Fabric made from the fibers has a maximum 1" (2.54 cm) normalized fabric tensile strength of 1855 g-to-break which is less than 40% of commercially

available fiber-grade polypropylene. Data are shown in Table I.

### Example III

A blend comprising 50 percent by weight of low molecular weight linear ethylene polymer (ethylene/octene, 52 MI, 0.953 density) and 50 percent by weight of high molecular weight linear ethylene polymer (ethylene/octene, 12 MF, 0.936 density) is found to have a peak normalized fabric tensile strength of about 2400 gms. Blend MI is 25 and blend density is 0.945 g/cm<sup>3</sup>. See Table I below.

15		Normalized Fabric Tensile Strength, (grams)				
	Example	Type	MI	Density	Q-value	
	I	discrete blend	23	0.939	6.82	2686
	II	LLDPE, single	26	0.940	3.74	1855
	III	discrete blend	25	0.945	NM*	2400
20		*NM=not meas	ured			

# Example IV (for comparison; not example of invention)

A commercially available LLDPE, ethylene/octene copolymer, having a MI of 12, a density of 0.935 g/cm<sup>3</sup>, and a Q-value of 4.36 is formed into fiber at about 0.4 g/min./hole. Fabric made from the resulting fiber has a maximum 1" (2.54 cm) normalized fabric tensile strength of about 2700 grams-to-break which is less than 60% of commercially available fiber grade polypropylene. Data are shown in Table II.

## Example V

An in-situ polymerized reactor blend (ethylene/octene copolymer) having a blended MI of 11, a blended density of 0.934 g/cm³, and a Q-value of 13.6 is formed into a fiber at about 0.4 g/min./hole. Fabric made from the resulting fiber has a maximum 1" (2.54 cm) normalized fabric tensile strength of about 3700 grams which is about 80% of commercially available fiber-grade polypropylene and about 140% of commercially available single linear ethylene polymer at a comparable MI. Data are shown in Table II.

#### TABLE II

15

10

Normalized Fabric Tensile Strength (grams)

	<u>Example</u>	Type	MI	Density Q-value	
	IV	LLDPE, single	12	0.935 4.36	2700
20	V	reactor blend	11	0.934 13.6	3700

## Example VI

An in-situ polymerized reactor blend

(ethylene/octene copolymer)having a blended MI of 10, a blended density of 0.955 gm/cm<sup>3</sup> and a Q-value of 8.87 is formed into a fiber at a throughput of about 0.4 gm/min./hole. Fabric samples of 1" (2.54 cm) made from the resulting fiber has a maximum normalized fabric tensile strength of about 3614 grams which is about 78% of that of commercially available fiber grade polypropylene and 129% of commercially available single linear ethylene polymer at a comparable MI (see Example VII for comparison). The following data indicates

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tenacity and bonding temperature for 6 tests. Also, some data are shown in Table III.

5		1	2	3	4	5	6
	Bonding Temp.°C (embossed/smooth)	119 121	1 <u>20</u> 122	121 123	122 124	123 126	124 127
	PLI (kgs/linear cm)	200 (35.7)	200 (35.7)	200 (35.7)	200 (35.7)	200 (35.7)	200 (35.7)
1	Normalized Fabric Tensile Strength (grams)	2260	2427	3341	3614	3494	3246

15 Example VII (for comparison: not example of Invention)

A commercially available high density single linear, ethylene/propylene copolymer, having a MI of 12, a density of 0.95 gm/cm<sup>3</sup>, and a Q value of 3.92 is

- formed into fiber at about 0.4 gm/min./hole. Fabric made from the resulting fiber has a maximum 1" (2.54 cm) normalized fabric tensile strength of about 2794 gramsto-break which is less than 60% of commercially available fiber grade polypropylene. The following
- 25 chart of data indicates normalized fabric tensile strength and bonding temperature for 4 tests. Also see Table III.

2 3 4 1 121 119 120 Bonding Temp.°C 118 (embossed/smooth) 123\* <u>120</u> <u>121</u> <u>122</u> 200 PLI 200 200 200 (35.7)(35.7)(35.7)(kgs/linear cm) (35.7)2481 2794 1836 2647 **Normalized Fabric Tensile Strength** (grams)

10 \*stick point

TABLE III

Normalized Fabric Tensile Strength (grams)

15

<u>Example</u>	Type	MI	Density Q-va	<u>alue</u>	
	reactor blend	10	0.955 8	.87	3614
VII	sole linear PE	12	0.950 3	.92	2794

20

Example VIII (for comparison; not claimed invention)

A commercially available fiber-grade polypropylene (PP) is spun into fibers and made into a heat-bonded fabric. The PP had a 15.6 MI (@190°C) and 0.91 density. The following data indicates normalized fabric tensile strength and temperature for 5 tests:

30

25

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		1	2	3	4	5
5	Bonding Temp.°C (embossed/smooth)	138 140	138 140	140 142	142 144	144 147*
	PLI (kgs/linear cm) -	75 (13.4)	224 (40)	200 (35.7)	200 (35.7)	200 (35.7)
10	Normalized Fabric Tensile Strength (grams)	2980	- 3485	4699	4307	3881

\*stick point

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## Example IX

A discrete blend comprising 50 percent by weight of high molecular weight linear ethylene polymer (ethylene/octene, 12 MI, 0.935 density) and 50 percent by weight of low molecular weight linear ethylene polymer (ethylene/octene, 105 MI, 0.953 density) is spun into fibers and a bonded web (fabric) is obtained. The blend has a calculated MI of 35.5 and density of 0.944. Bonding temperature and normalized fabric tensile strength is shown below at different bonding pressures. Also see Table IV.

25

30

	1	2
Bonding Temp.°C (embossed/smooth)	118 120	<u>118</u> 120
PLI (kgs/linear cm)	75 (13.4)	200 (35.7)
Normalized Fabric Tensile Strength (grams)	2355	2297

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## Example X

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A discrete blend comprising 70 percent by weight of high molecular weight linear ethylene polymer (ethylene/octene, 18 MI, 0.93 density) and 30 percent by weight of low molecular weight linear ethylene polymer (ethylene/octene, 105 MI, 0.953 density) and having a calculated MI of 30.5 and density of 0.937, is spun into fibers and bonded as a fabric in 3 tests; data are shown below. Also see Table IV.

		1	2	3
15	Bonding Temp.°C (embossed/smooth)	114 117	<u>116</u> 118	<u>117</u> 119
	PLI (kg/linear cm)	200 (35.7)	200 (35.7)	200 (35.7)
20	Normalized Fabric Tensile Strength (grams)	2190	2243	2586

Example XI (for comparison; not claimed invention)

A single linear ethylene polymer

25 (ethylene/octene) having a MI of 30 and a density of

0.94 g/cm<sup>3</sup> is found to have a maximum normalized fabric
tensile strength of about 1531 gms. See Table IV below.

30

TABLE IV	Normalized
	Fabric Tensile
	Strength (grams)

5	<b>Example</b>	Type	MI	<u>Density</u>	
,	IX	discrete blend	35.5	0.944	2355
	X	discrete blend	30.5	0.937	2586
	XI	LLDPE, single	30 <b>-</b>	0.94	1531

## 10 Example XII

A discrete blend comprising 71% by weight of a high molecular weight linear ethylene polymer (ethylene/propylene, 8 MI, 0.952 density) and 29% by weight of low molecular weight linear ethylene polymer (ethylene/octene, 105 MI, 0.954 density) and having a calculated MI of 17 and density of 0.953 g/cm<sup>3</sup> is spun into staple fibers and bonded into fabric at 200 PLI (35.7 kg/linear cm). Data are shown below. Also see 20 Table V below.

1868 2034 2259 2596 2620 3066 3034

Normalized Fabric Tensile Strength (grams)

\*stick point

5

XIII

## Example XIII (for comparison; not claimed invention)

A single linear ethylene polymer (ethylene/propylene copolymer) having a MI of 17 and a density of 0.95 g/cm<sup>3</sup> is spun into fibers and bonded as a fabric at 200 PLI (35.7 kg/linear cm). Data are shown below. Also see Table V below.

10	Bonding Temp. °C (embossed/smooth)	1 116 118	<u>117</u> 119	3 118 120	<u>119</u> 121	5 120 122*
	Normalized Fabric Tensile Strength	2016	2213	2202	2749	2837*
15	(grams) *stick r	oint				

		TABL	<u>E V</u>		Normalized
					Fabric Tensile
					Strength (grams)
20	Example	Type	MI	Density	
	XII	discrete blend	17	0.953	3066

0.95

sole linear PE 17

Finally, the following is a hindsight partial explanation as to how our claimed invention results in superior products, notwithstanding the strongly negative teachings of the prior art.

Continuous fiber (and subsequently the thermally bonded fabric) made using the spunbonded process differs dramatically from staple fiber spinning, optionally drawing the fiber, crimping the fiber, cutting the fiber making it discontinuous, carding the fiber into fabric and thermally bonding the fiber.

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The spunbonded process can effectively use polyethylene having lower densities (such as about 0.94 g/cc and lower) and the continuous filaments are thermally bonded into fabric. Spunbonding of polymer results in fiber which simulates the shrinkage properties of low draw ratio staple fiber. The thermal bonding window of lower density polyethylene is broader than for higher density polyethylene at equivalent melt indices and molecular weight distributions and the thermal calenders are operated such that the fabric can be bonded. There is no carding step and thus no need to modify the polymer for forming the fiber into fabric.

In the staple process, the discontinuous or cut 15 fiber has to be carded into fabric. Carding can be accomplished with polyethylene fiber by using a high draw ratio during the spinning step. But, this high draw ratio results in fiber having high shrinkage and resultant low thermally bonded fabric strength. 20 heat shrinks the fiber instead of bonding it together. However, the density of the polyethylene can be increased to enable it to card, but then the melting point range of the fiber is too narrow to effectively 25 thermally bond it into fabric. Trying to control a thermal calender within a narrow range either results in an unbonded fabric or fiber/fabric sticking to the calender rolls resulting in equipment shutdown.

30 The present invention solves these staple fiber and fabric forming deficiencies by broadening the thermal bonding window of the polyethylene staple fiber making it useful in forming high strength thermally bonded fabric while maintaining the cardability of the 35

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fiber, especially when higher density polyethylene is used, such as densities between 0.94 and 0.965 g/cc.

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1. A process for preparing a fabric, which fabric is either thermally bonded or bondable, from filaments including thermoplastic fine denier discontinuous staple fibers having an average denier per filament in a range of from 0.1 to 15 d.p.f., and having filament lengths up to 30 cm., and in which the staple fibers are prepared from a meltspun blend of linear ethylene polymers:

characterized in that the polymer blend in the staple fibers has a Q value above 4.5, wherein Q is defined as weight average molecular weight divided by number average molecular weight, as determined by gel permeation chromatography.

2. A process for preparing a fabric, which
fabric is either thermally bonded or bondable, from
filaments including thermoplastic fine denier
discontinuous staple fibers having an average denier per
filament in a range of from 0.1 to 15 d.p.f., and having
filament lengths up to 30 cm., and in which the staple
fibers are prepared from a meltspun blend of linear
ethylene polymers:

characterized in that the polymer blend in the staple fibers has an  $I_{10}/I_2$  value of at least 7, wherein 25

 $I_{10}$  is determined by ASTM D-1238(N) conditions, and  $I_2$  is determined by ASTM D-1238(E) conditions.

- unbonded fabric prepared thereby is capable of being thermally bonded to give a stronger fabric, as determined by normalized strip tensile strength, over a broader range of thermal bonding temperatures, than an otherwise identical process used to prepare a comparative product wherein the polymer blend in the staple fibers in the comparative product has either a Q value in a range of from 3.5 to 4.0 or an I<sub>10</sub>/I<sub>2</sub> value in a range from 6.0 to 6.5.
- 15 4. The process of Claim 1 wherein the polymeric blend in the staple fibers has a Q value in a range from 5.5 to 10.
- 5. The process of Claim 2 wherein the 20 polymeric blend in the staple fibers has an  $I_{10}/I_2$  value in the range from 10 to 20.
- 6. Thermally bonded fabric prepared by the process of Claims 1 or 2, wherein the thermally bonded fabric has a normalized strip tensile strength of at least 3,000 grams.
  - 7. The thermally bonded fabric of Claim 6 wherein the polymeric staple fibers comprise a polymeric blend is an in-situ blend formed during polymerization of the polymer; and the thermally bonded fabric has a normalized strip tensile strength of at least 3,600 grams.

- 8. The thermally bonded fabric of Claim 7 having a normalized strip strength of at least 3,700 grams.
- 9. Polymeric fibers including discontinuous staple fibers suitable for use in the process of Claims 1 or 2 wherein the polymer has an I<sub>10</sub>/I<sub>2</sub> value of at least 7 and the polymer is a blend of
- (A) at least one high molecular weight linear ethylene polymer having a MI value less than 25 grams/10 minutes and a density above 0.91 grams/cm<sup>3</sup>, and
- (B) at least one low molecular weight
  linear ethylene polymer having a MI above 25 grams/10 minutes and a density above 0.91 grams/cm<sup>3</sup>.
- 10. The staple fibers of Claim 9, having an average denier per filament in a range of from 0.1 to 15 d.p.f., and being homofilaments.
- 11. A polymer blend suitable for use in preparing the polymeric fibers of Claim 9 characterized in that the polymer blend has an I<sub>10</sub> /I<sub>2</sub> value of at least 7 as determined by ASTM D-1238(N) and (E) respectively and further wherein the blend is formed by blending (A) and (B) in appropriate weight ratios wherein
- (A) is at least one high molecular weight linear ethylene polymer having an MI measured in accordance with ASTM D-1238(E) (190°C/2.16 kg) less than 25 g/10 minutes and a density above 0.91 g/cm<sup>3</sup>; and,
- (B) is at least one low molecular weight linear ethylene polymer having a MI measured in

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accordance with ASTM D-1238(E) (190°C/2.16 kg) greater than 25 g/10 minutes and a density above 0.91 g/cm<sup>3</sup>.

- the high molecular weight linear ethylene polymer and low molecular weight linear ethylene polymer is sufficient to provide a blend having an MI value in the range of 0.1 to 40 grams/10 minutes and a density in the range of 0.94 to 0.96 grams/cm<sup>3</sup>.
- 13. The blend of Claim 11 wherein at least one of the linear ethylene polymers comprises a copolymer of ethylene with at least one C3-C12 olefin.
- 14. The blend of Claim 13 wherein at least one of the linear ethylene polymers is a copolymer of ethylene and propylene or octene.
- 15. The blend of Claim 11 wherein the blend is a blend of discrete polymers.
  - 16. The blend of Claim 11 wherein the blend is an in-situ blend formed during polymerization.
- 17. The blend of Claim 15 or 16 wherein the high molecular weight linear ethylene polymer is HDPE, having an MI value within the range between 0.1 and 25 grams/10 minutes and the low molecular weight linear ethylene polymer is HDPE having an MI value within the range between 25 and 300 grams/10 minutes.
  - 18. The polymeric staple fibers of Claim 9 wherein the polymer is the blend of any of Claims 11 to 17.

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- 19. A process for preparing the polymeric staple fibers of Claim 9 characterized by the steps of
- (A) Melt-spinning the polymer of any of Claims 11 to 17 into meltspun filaments;
  - (B) Hauling off the meltspun filaments at a speed in a range of from 60 to 2,000 meters/minute; and, optionally,
- 10 (C) drawing and/or crimping and/or cutting the hauled-off meltspun filaments by conventional means.
- 20. A process wherein molten linear ethylene
  polymer fine denier staple fibers are spun at
  commercially feasible throughput rates, optionally
  followed by mechanical drawing to produce fiber sizes of
  from 0.1 to 15 denier/filament and used in making a web
  or fabric, characterized by:

#### spinning a blend comprising

- (A) at least one high molecular weight linear ethylene polymer having a MI measured in accordance with ASTM D-1238(E) (190°C/2.16 kg) less than 25 g/10 minutes and a density above 0.91 g/cm $^3$ , and
- ethylene polymer having a MI measured in accordance with

  ASTM D-1238(E) (190°C/2.16 kg) greater than 25 g/10

  minutes and a density above 0.91 g/cm<sup>3</sup>, forming linear ethylene polymer staple fibers having a Q value above 4.5, wherein Q is defined as weight average molecular weight divided by number average molecular weight, as determined by gel permeation chromatography.

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- 21. The blend of Claim 20 wherein the ratio of the high molecular weight linear ethylene polymer and low molecular weight linear ethylene polymer is sufficient to provide a blend having a MI value in the range of 0.1 to 40 grams/10 minutes and a density in the range of 0.94 to 0.96 grams/cm<sup>3</sup>.
- 22. The blend of Claim 20 wherein at least one of the linear ethylene polymers comprises a copolymer of ethylene with at least one C3-C12 olefin.
  - 23. The blend of Claim 22 wherein at least one of the linear ethylene polymers is a copolymer of ethylene and propylene or octene.
- 24. The blend of Claim 23 wherein at least one of the linear ethylene polymers is LLDPE.
- 25. The blend of Claim 20 wherein the blend is a blend of discrete polymers.
  - 26. The blend of Claim 20 wherein the blend is an in-situ reactor blend formed during polymerization.
- 27. The blend of Claim 25 or 26 wherein the high molecular weight linear ethylene polymer is HDPE, having a MI value within the range between 0.1 and 25 grams/10 minutes and the low molecular weight linear ethylene polymer is HDPE having a MI value within the range between 25 and 300 grams/10 minutes.
  - 28. The blend of Claim 25 or 26 wherein the high molecular weight linear ethylene polymer is HDPE, having a MI value within the range between 0.1 and 25 grams/10 minutes and the low molecular weight linear

ethylene polymer is HDPE having a MI value within the range between 25 and 300 grams/10 minutes.

#### AMENDED CLAIMS

[received by the International Bureau on 5 April 1991 (05.04.91); original claims 7 and 28 amended; new claims 29-34 added; other claims unchanged (3 pages)]

I<sub>10</sub> is determined by ASTM D-1238(N) conditions, and I<sub>2</sub> is determined by ASTM D-1238(E) conditions.

- unbonded fabric prepared thereby is capable of being thermally bonded to give a stronger fabric, as determined by normalized strip tensile strength, over a broader range of thermal bonding temperatures, than an otherwise identical process used to prepare a comparative product wherein the polymer blend in the staple fibers in the comparative product has either a Q value in a range of from 3.5 to 4.0 or an I<sub>10</sub>/I<sub>2</sub> value in a range from 6.0 to 6.5.
  - 4. The process of Claim 1 wherein the
    polymeric blend in the staple fibers has a Q value in a range from 5.5 to 10.
  - 5. The process of Claim 2 wherein the polymeric blend in the staple fibers has an  $I_{10}/I_2$  value in the range from 10 to 20.
    - 6. Thermally bonded fabric prepared by the process of Claims 1 or 2, wherein the thermally bonded fabric has a normalized strip tensile strength of at least 3,000 grams.
    - 7. The thermally bonded fabric of Claim 6 wherein the polymeric blend is an in-situ blend formed during polymerization of the polymer; and the thermally bonded fabric has a normalized strip tensile strength of at least 3,600 grams.

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- 21. The blend of Claim 20 wherein the ratio of the high molecular weight linear ethylene polymer and low molecular weight linear ethylene polymer is sufficient to provide a blend having a MI value in the range of 0.1 to 40 grams/10 minutes and a density in the range of 0.94 to 0.96 grams/cm<sup>3</sup>.
- 22. The blend of Claim 20 wherein at least one of the linear ethylene polymers comprises a copolymer of ethylene with at least one C3-C12 olefin.
- 23. The blend of Claim 22 wherein at least one of the linear ethylene polymers is a copolymer of ethylene and propylene or octene.
- 24. The blend of Claim 23 wherein at least one of the linear ethylene polymers is LLDPE.
  - 25. The blend of Claim 20 wherein the blend is a blend of discrete polymers.
- 26. The blend of Claim 20 wherein the blend is an in-situ reactor blend formed during polymerization.
- 27. The blend of Claim 25 or 26 wherein the high molecular weight linear ethylene polymer is HDPE, having a MI value within the range between 0.1 and 25 grams/10 minutes and the low molecular weight linear ethylene polymer is HDPE having a MI value within the range between 25 and 300 grams/10 minutes.
- 30 28. The blend of Claim 25 or 26 wherein the high molecular weight linear ethylene polymer is LLDPE, having a MI value within the range between 0.1 and 25 grams/10 minutes and the low molecular weight linear

ethylene polymer is HDPE having a MI value within the range between 25 and 300 grams/10 minutes.

- 29. A method of increasing the thermal bonding window of staple fibers made from linear polyethylene, characterized by blending high and low molecular weight linear polyethylenes, spinning into staple fiber and thermally bonding into fabric.
- 30. The method of Claim 29 wherein the high and low molecular weight linear polyethylene blend has a 10/12 value above 7.
  - 31. The method of Claim 29 wherein the staple fiber has a thermal bonding window of  $10^{\circ}\text{C}$ .
- 15 32. The method of Claim 29 wherein the staple fiber has an average denier per filament in a range from 1 to 15 denier per filament.
- 33. The method of Claim 29 wherein the high molecular weight linear polyethylene is HDPE and the low molecular weight linear polyethylene is HDPE.
  - 34. The method of Claim 33 further characterized by carding the staple fiber into fabric before thermal bonding.

# STATEMENT UNDER ARTICLE 19(1)

In response to the Transmittal of the International Search Report mailed on 06 February 1991, please add Claims 29-34 and amend Claims 7 and 28 as the enclosed full page replacements (in triplicate) describe.

1

The amendment to Claim 7 deleting the language "staple fibers comprise a polymeric" corrects transcription errors recorded while drafting the application. This correction is supported throughout the disclosure, but is particularly well supported on page 10, lines 9-19 and in Examples V and VI on page 23.

The amendment to Claim 28 deleting the term "HDPE" and replacing with the term "LLDPE" corrects an error discovered by Examiner during the International Search. The amendment to Claim 28 is supported in the disclosure, for example, on page 15, lines 7-9 and demonstrated in Examples I, IX and X on pages 19, 26 and 27, respectively.

New Claim 29 is supported in the disclosure on page 7, line 30 through page 8, line 3, as well as in the Examples of the invention (such as Example VI as contrasted with Comparative Example VII, pp. 23-25).

New dependent Claims 30 and 31 are supported in the disclosure on page 12, lines 11-25.

New dependent Claims 32 is supported in the disclosure on page 8, lines 6-7.

New dependent Claims 33 and 34 are supported in the disclosure on page 30, line 13 through page 31, line 2.

Applicants believe that the new Claims are fully supported by the disclosure and respectfully request that these Claims be added to the above identified application.

#### INTERNATIONAL SEARCH REPORT

International Application 1

		International Application 1 PCT/	′US90/06267					
I. CLASS	SIFICATION	N OF SUBJECT MATTER (if several classification symbols apply, indicate all) 6						
		onal Patent Classification (IPC) or to both National Classification and IPC						
IPC	(5): C	08L 23/06, 23/08, 23/18; D04H 3/03						
II.S.	`cf. 5	25/240; 428/288; 264/210.8, DIG. 26						
	S SEARCH							
		Minimum Documentation Searched 7	- III					
Classification System		Classification Symbols						
		Classification Symbols						
U.S.		525/240; 428/288, 264/210.8, DIG. 26						
	L	Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched 8						
		ONSIDERED TO BE RELEVANT 9	I Polometra Clair No. 12					
ategory *	Citatii	on of Document, 11 with indication, where appropriate, of the relevant passages 12	Relevant to Claim No. 13					
$\frac{X}{Y}$	US, A, 4,438,238 (FUKUSHIMA ET AL) 20 MARCH 1984; See e.g. column 6.  11-17 1-17,20-28							
<u>X</u> Y	US, A, 4,705,829 (KWACK ET AL)  10 NOVEMBER 1987; See e.g. columns 1 and 2  and the claims.  11 & 13-1 20 & 22-26							
<u>X</u> <u>Y</u>	JP, A, 57-59943 (SHOWA DENKO K.K.) 10 APRIL 1982; See e.g. the Abstract.    11 & 13							
Y	EP,	A, 0 154 197 (DOW CHEMICAL COMPANY) SEPTEMBER 1985; See entire document.	1-10, 20-28					
Y	US, 17 H	A, 4,644,045 (FOWELLS) FEBRUARY 1987; See e.g. Abstract	1-10, 20-28					
$\frac{X}{Y}$		A, 4,842,922 (KRUPP ET AL) JUNE 1989; See entire document.	1-17, 20-28 1-17, 20-28					
<u>X</u> Y	US, 24 d	A, 4,461,873 (BAILEY ET AL) JULY 1984; See e.g. column 2 and Table	12 & 17 12,17,21,27					

- Special categories of cited documents: 10
- "A" document defining the general state of the art which is not considered to be of particular relevance
- earlier document but published on or after the international filing date
- document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- "O" document referring to an oral disclosure, use, exhibition or other means
- document published prior to the international filing date but later than the priority date claimed
- "T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the

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- "X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step
- "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.
- "&" document member of the same patent family

#### IV. CERTIFICATION

Date of the Actual Completion of the International Search

15 JANUARY 1991

Date of Mailing of this International Search Report

International Searching Authority

ISA/US

Signature of Authorized Officer Carmen J. Seccuro

III. DOCU	III. DOCUMENTS CONSIDERED TO BE RELEVANT (CONTINUED FROM THE SECOND SHEET)										
Category *	Citation of Document,			f the relevant pas	sages	Relevant to Cla	im No				
Y	US, A, 4,584, 22 APRIL 1986 column 8, lir	347 (HAR)	PELL ET Al	L) 3, line		21, 27,	28				
Y	US, A, 3,914 See e.g. colu	,342 (MIT	CHELL) 21	OCTOBER	1975	12, 17, 27, 28	21,				