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(54) **Title**: COMPOSITE LAYER FOR REINFORCEMENT OF OBJECTS SUCH AS TIRES OR BELTS

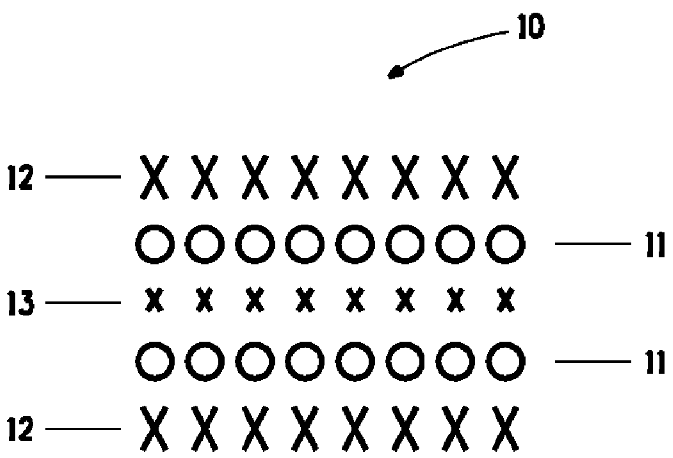

FIG. 1

(57) **Abstract**: The present invention pertains to a fibrous cord comprising yarns twisted together wherein the cord has a twist multiplier of from 5.0 to 12.0, and comprises a blend of aromatic polyamide or aromatic copolyamide yarns and polyester yarns. The invention further pertains to a composite layer comprising an elastomer and a fabric wherein the fabric comprises from about 25 to 60 weight percent of the total composite layer, and comprises a plurality of inventive cords.

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TITLE OF THE INVENTION

COMPOSITE LAYER FOR REINFORCEMENT OF OBJECTS SUCH AS TIRES OR BELTS

BACKGROUND

1. Field of the Invention

The present invention concerns a composite layer suitable as a reinforcement means against heat and fatigue for use in products such as tires or belts

2. Description of Related Art

In Ultra-High Performance (UHP) or Run-on-Flat (RoF) tires, the carcass reinforcement is usually based on polymeric multi-filament yarns with good temperature stability, meaning that the modulus of the yarn does not deteriorate (or show a tendency for deterioration) at the high operating temperatures of the tire under high speed or run-flat conditions. The most commonly used material is rayon. Because of the relatively low strength of rayon, many tires are built with dual-carcass layers, i.e. two carcass reinforcement layers, in order to achieve the required mechanical strength.

When reinforcement materials are used in elastomeric components of tires, the following advantages and disadvantages occur:

Rayon requires a two-ply carcass construction, therewith increasing tire weight and rubber consumption. Further, elastomeric components incorporating rayon have low retained strength after fatigue and low strength retention following high temperature exposure.

Polyester can only be used for lower performance tires because of insufficient strength retention following high temperature exposure. However polyester yarns do provide components having high retained strength after fatigue.

Para-aramid is strong and thermally very stable permitting a single ply construction. However, compression fatigue and riding comfort are sometimes considered insufficient.

There remains a need, therefore to provide an elastomeric cord component that can offer both good retained strength after fatigue and good strength retention following high temperature exposure.

It is also known to make hybrid cords for reinforcing tires comprising steel strands wound with p-aramid strands, both with a steel core and a p-aramid core.

US patent 5,551,498describes hybrid cords having a core of two p-aramid strands twisted together and an outer layer or sheath of six steel strands or filaments surrounding the core. Also described are hybrid cords consisting of a core consisting of three steel strands and a layer consisting of four p-aramid strands surrounding the core.

US patent 4,176,705 describes wire reinforcement cords for tires. The cords consist of a p-aramid core, and steel strands comprised of steel filaments twisted together are disposed about the core.

It is known that steel wires have a lower elongation at break than filaments of aromatic polyamide (aramid). As a consequence, prior art composite reinforcing cords can be exposed only to loads at which the cord’s extension does not exceed the breaking extension of the steel wire strands. If this limit load is exceeded, the steel wires rupture and the entire load is taken up by the aramid core which in turn immediately exceeds its own breaking elongation and ruptures as well. In other words, known composite cords break when the extension corresponds to the breaking extension of its steel wire component, in spite of the fact that this breaking extension is distinctly below the breaking elongation of the polyamide core.

Premature breakage of the steel wires is described in US patent 4,807,680, which discloses a tire including a hybrid cord consisting of a core of multiple p-aramid filaments surrounded by steel filaments that are wound around the core. Hybrid cords using rectangular cross-section wires, are also described in US patent 4, 878, 343.

US patent application publication 2009/0159171discloses hybrid cords for tire reinforcement which make full use of the properties of the p-aramid component mentioned in the prior art.

US patent application publication 2011/0086224 describes a sheet and method of making a sheet for support structures and tires.

US patent application publication 2005/0017399 discloses a multifilament aramid yarn with high fatigue resistance.

SUMMARY OF THE INVENTION

This invention pertains to a fibrous cord comprising a blend of yarns twisted together wherein the cord

(i) has a twist multiplier of from 5.0 to 12.0, and

(ii) comprises a blend of polyester yarns with either aromatic polyamide or aromatic copolyamide yarns, and,

The invention further pertains to a composite layer comprising an elastomer and a fabric wherein the fabric

(i) comprises from about 25 to 60 weight percent of the weight, equivalent to 5 to 40 volume percent, of elastomer plus fabric, and

(ii) comprises a plurality of cords wherein a cord has a twist multiplier of from 5.0 to 12.0, and comprises a blend of aromatic polyamide or aromatic copolyamide yarns and polyester yarns.

BRIEF DESCRIPTION OF DRAWINGS

Figure 1 shows a cross section of a test sample.

Figure 2 depicts the force-elongation relationship for the Examples 4-6 and G-J.

DEFINITIONS

The following words if and when used in the present specification have the meaning as indicated hereunder.

“Strand” as used herein means a continuous band of materialwhich band of material may comprise either a single filament or multiple filaments twisted together to form a yarn.

“Filament” as used herein means a relatively flexible, macroscopically homogeneous body having a high ratio of length to width across its cross-sectional area perpendicular to its length. The filament cross section can be any shape, but is typically circular. Herein, the term "fiber", with respect to aramid, is used interchangeably with the term "filament". “Yarn” as used herein means a strand comprising multiple filaments twisted together.

“Denier” the weight in grams per 9,000 m length of filament, strand, yarn or cable.

“Tex” the weight in grams of one kilometer of filament, strand, yarn or cable.

“Decitex” is one tenth of a Tex, abbreviated as dTex.

“Cord” as used herein means a product formed by twisting together two or more plied yarns.

“Hybrid cord” as used herein means a cord comprising at least two yarns of different composition or, if of the same composition, having different physical properties. This is sometimes also known as a merge cord.

“Twist multiplier or TM” as used herein is defined as the product of twist level (in turns per meter, according to ASTM D1423) and the square root of the linear density (in dtex), divided by 3000.

“Retained strength” is the strength measured after a particular event such as conditioning at a temperature of 110°C or after a specified number of fatigue cycles. It is frequently reported as a percentage which compares the after event result with the before event result.

Ends Per Inch (EPI) is a measure of cord end-count over the fabric width or length.

DETAILED DESCRIPTION

In particular, in the case of a tire, the present invention aims at replacing a 2-layer carcass construction by a single layer design, hence saving rubber in the carcass layer leading to weight reduction while also achieving good fatigue performance and good strength retention after high temperature exposure.

Cords

A fibrous hybrid cord of this invention comprises a blend of yarns twisted together wherein the cord

(i) has a twist multiplier of from 5.0 to 12.0, and

(ii) comprises a blend of polyester yarns with either aromatic polyamide or aromatic copolyamide yarns.

In some embodiments, the cord has a twist multiplier of from 7.0 to 10 or from 7.5 to 10 or even from 8.0 to 10.0 or even from 9.0 to 10.0. If the twist multiplier is greater than 12.0, the cord will not provide adequate strength retention after high temperature exposure although fatigue strength is acceptable. If the twist multiplier is less than 5.0, the cord will not provide adequate fatigue strength although strength retention after high temperature exposure is acceptable.

It has been found that carefully designed hybrid cords having a twist multiplier of from 5.0 to 12.0 and made of a blend of polyester yarns with either aromatic polyamide or aromatic copolyamide yarns show surprisingly high temperature resistant properties as well as acceptable fatigue performance such that a cord of this construction is very suitable for use in a tire carcass, therefore combining the advantage of polyester's fatigue performance and potential riding comfort with the advantage of aramid's high temperature stability.

These hybrid cords have compression fatigue values and thermal strength retention values that reflect the synergy from the combination of the component yarns. That is to say, the hybrid cord balances the best properties of the component yarns and does not exhibit the poorer performance characteristics. These cords are particularly useful for high performance tires and run-flat tire designs with a single carcass ply.

The cord may be stretch broken or texturized.

Component Yarns

In some embodiments, the aromatic polyamide or aromatic copolyamide yarns have a linear density of at least 400 dtex (360 denier) and are pre-twisted individually to a twist multiplier of at least 3.5. In some other embodiments, the linear density is at least 444 dtex (400 denier). Preferably, the aromatic polyamide used in the composition is para-aramid or copolymers of p-phenylene diamine. As an example, this can be the products available under the tradenames Kevlar®, Twaron®, Heracron®, Technora® or Rusar®. In some embodiments, the aromatic polyamide or aromatic copolyamide yarn may be partially or totally replaced by a stretch-broken aromatic polyamide yarn. Exemplary constructions and properties of such stretch-broken yarns are (1) Nm 50/4/2 of dtex 811x2 with a yarn twist multiplier of 5, a yarn tenacity of 125 cN/tex and a yarn elongation at break of 3% or (2) Nm 50/3/2 of dtex 613x2 with a yarn twist multiplier of 5.4, a yarn tenacity of 132 cN/tex and a yarn elongation at break of 3%.

In some embodiments, the polyester yarns have a linear density of at least 444 dtex and are pre-twisted individually to a twist multiplier of at least 1.1. In some embodiments, the twist mutiplier is at least 3.5. Preferably, the polyester used in the composition is polyethyleneterephthalate, polybuthyleneterephtalate (PBT), polyethylenenaphtalene (PEN) or poly(trimethylene terephthalate) based on fermentation derived from 1,3-propanediol (bio-PDO) which is available as Sorona® from E.I. DuPont de Nemours and Company, Wilmington, DE (DuPont). In some embodiments, the polyester yarn may be partially or totally replaced by a yarn comprising aromatic polyamide fiber and aliphatic polyamide fiber wherein the aromatic polyamide is itself a mixture of meta-aramid fiber such as Nomex® and para-aramid fiber such as Kevlar®, both available from DuPont. An example of such a composition is one comprising about 85 weight percent of Nomex®, about 10 weight percent of Kevlar® and about 5 weight percent of aliphatic polyamide such as nylon. An exemplary construction and properties of such a component yarn is Nm 55/2/2 of dtex 728 with a yarn twist multiplier of 5.4, a yarn tenacity of 40 cN/tex and a yarn elongation at break of 10%.

Also, the present invention includes a method of assembling a carcass structure comprising the highly twisted aramid/polyester hybrid cords into carcass plies.

Alternatively, the hybrid cords may be assembled in a single step, combining the individual multifilament twisting and the cord assembling twisting steps

Composite Layer

The composite layer comprises an elastomer and a fabric wherein the fabric comprises

(i) from about 25 to 60 weight percent of the weight, equivalent to 5 to 40 volume percent, of elastomer plus fabric, and

(ii) a plurality of cords, the cords comprising a blend of yarns twisted together wherein the cord has a twist multiplier of from 5.0 to 12.0, and comprises a blend of aromatic polyamide or aromatic copolyamide yarns and polyester yarns.

The fabric may be a woven fabric or unidirectional fabric. A unidirectional fabric is a fabric in which all of the yarns are oriented in the same direction. A woven fabric is any fabric that can be made by weaving; that is, by interlacing or interweaving at least two yarns,typically at right angles. Generally, such fabrics are made by interlacing one set of yarns called warp yarns with another set of yarns called weft or fill yarns. The woven fabric can have essentially any weave, such as, plain weave, crowfoot weave, basket weave, satin weave, twill weave, unbalanced weaves, and the like. Plain weave is the most common and is preferred.

In some embodiments, the fabric has a basis weight of from 64 to 740 g/m^2. In some preferred embodiments the basis weight of the fabric is from 70 to 620 g/m^2. In some most preferred embodiments the basis weight of a fabric is from 140 to 330 g/m^2.

In some embodiments, the fabric yarn count is 20 to 50 ends per inch (80 to 200 ends per decimeter), preferably 22 to 45 ends/inch (88 to 180 ends per decimeter). In some most preferred embodiments, the yarn count is 24 to 40 ends/inch (96 to 160 ends per decimeter) in the warp.

Preferably, the elastomer used in the present composition may be a variety of rubber materials including nitrile butadiene rubber (hydrogenated and nonhydrogenated) (NBR); ethylene-propylene-diene monomer rubber (EPDM) including such dienes as 5-ethylidene-2-norbornene (5-ethylidenebicyclo[2.2.1]hept-

2-ene), dicyclopentadiene(bicyclo[2.2.1]he pta-2,5-diene), and 1,4-hexadiene; ethylene propylene diamine monomer; chlorosulfonyl-polyethylene (CSM); ethylene oxide and chloromethyl oxirane (ECO); hexafluoropropylene vinylidene fluoride (FPM); natural rubber (NR); styrene-butadiene rubber (SBR); and the like and mixtures thereof.

The composite layer has a compression fatigue value or a thermal strength retention value greater than the corresponding values of a similarly constructed composite layer comprising elastomer and either a fabric solely of aromatic polyamide or aromatic copolyamide yarns or a fabric solely of polyester yarns.

Hybrid cords as described above and a composite layer comprising the cords can be utilized in mechanical rubber goods applications such as tires, belts and hoses.

Method of Making a Composite Layer

A method for producing a composite layer comprises at least the steps of preparing a fabric with a plurality of cords having a twist multiplier of from 5.0 to 12, wherein the cords are formed by blending aromatic polyamide or aromatic copolyamide yarns and polyester yarns, wherein the polyamide or copolyamide yarns are pre-twisted to a twist multiplier of at least 3.5 TM before cord assembly and have a linear density of at least 444 dtex, and the polyester yarns have a twist multiplier of at least 1.1 and a liner density of at least 444 dtex.

Examples of possible carcass fabrics made solely of p-aramid cords are also presented. Tires built with rayon or polyester fabrics will require a double-carcass ply construction. All carcass constructions are chosen such that the overall tire carcass strengths are comparable and in the typical range of 7.3 to 8.3 kN/in.

TEST METHODS

All measurements were made at 24°C.

Cord tensile strengths were measured according to ASTM 7269-07 at the respective temperatures of 24°C and 110°C typical for a tire in use. The fabric strengths were calculated taking into account the cord end-counts and the individual cord strengths. The samples were conditioned for 24 hours at 24 degrees C or for 35 minutes at 110 degrees C prior to testing.

Fatigue data was measured using a typical Scott-flex equipment as defined in ASTM D430-06. The pulley size of the Scott-flex machine was 19 mm in diameter. The force applied on the samples during the test was 30 kg for the rayon samples and 60 kg for the aramid and aramid/polyester hybrid samples except for Comparative Example F and Example 3 where the force applied was 30 kg.

After 600,000 cycles on the Scott-flex tester, the fabric cords were extracted from the rubber and the reinforcement layer that was closest to the Scott-flex pulley was tested for mechanical properties according to ASTM 7269-07. This layer was constantly under compression during the Scott-flex test, which is the critical parameter to be assessed.

Retention percent is the percentage of test coupon strength retained after either high temperature exposure or fatigue cycling compared to the test coupon strength prior to either high temperature exposure or fatigue cycling.

EXAMPLES

The following examples are given to illustrate the invention and should not be interpreted as limiting it in any way. Examples prepared according to the current invention are indicated by numerical values. Control or Comparative Examples are indicated by letters.

Sample Preparation

Yarns used in the Examples were:

Rayon – Rayon 610F, manufactured by Cordenka GmbH, Obernburg, Germany.

Polyester: - HMLS PET - NAA Kordsa 792, manufactured by Kordsa Global, Istanbul, Turkey.

Aramid - Kevlar® K29AP 670dtex and Kevlar® K129 1330dtex were used in the examples of Table 1 and Kevlar® K29 1100 dtex was used in the examples of Table 2. All the fiber was manufactured by DuPont.

Cords were formed from the above yarns having a twist multiplier as shown in Table 1. The cords were then formed into fabrics having structures as described in Table 1.

Samples were prepared by embedding the two layers of reinforcement fabric into natural rubber after adhesion treatment of the fabric in a two-step RFL (Resorcinol, Formaldehyde and Vinyl Pyridine Butadiene-Styrene Latex) dip hot-stretching process as is commonly used to enhance rubber adhesion to polyester, rayon or polyamide yarns. The samples were cured in the mold for 20 min at 168°C and 200 psi. A description of a typical RFL dip process can be found in the DuPont Kevlar® Technical guide and comprises a first step of dipping the yarn through an aqueous epoxy resin subcoat solution under specific tension followed by an oven curing at 243°C. In a second step, the subcoat treated yarn is dipped through an aqueous topcoat of RFL, again followed by an oven curing at a typical temperature of 232°C. This process is required in order to obtain optimal adhesion between the textile fabrics and elastomers/rubber.

The rubber composite samples were belt shaped so that a full cycle travel of 132 mm could be achieved on the Scott-flex machine. As shown generally at 10 in Fig.1, the two fabric layers 11 were covered by 1.14 mm thick natural rubber layers 12 towards the sample surface and were separated by 0.77 mm of rubber 13 between the fabrics.

Mechanical properties and fatigue data for hybrid cords of Examples 1-3 are compared to cords of Comparative Examples A-F comprising only rayon, polyester or para-aramid carcass constructions used in tires and are shown in Table 1.

Table 1

Material (Example Reference)	Cord	Fabric EPI	Construction	Final Cord Twist Multiplier	Cord Strength @ 24°C (N)	Cord Strength @ 110°C (N)	Strength Retention % @ 110°C vs 24°C in N	Fabric Strength @ 24°C (kN)/in	Retained Fatigue Strength % after 600,000 Cycles
Rayon (A)	R-1	28	1840/1/2	8.5	142.8	68.9	48	4.00	75
Rayon (B)	R-2	36	1230/1/2	8.0	100.8			3.63	83
Polyester (C)	PE-1	28	1100/1/2	7.5	135.7	55.2	41	3.80	97*, 99**
Kevlar° (D)	K-1	28	1100/1/2	9.5	296.7	165.9	56	8.31	94
Kevlar° (E)	K-2	45	670/1/2	9.5	186.0			8.37	95
Kevlar® (F)	K-1	28	1100/1/2	7.5			56	8.31	88
Kevlar°/ Polyester (1)	K/P-1	38	1100/1 + 1100/1 (Kev + Poly)	9.0	198.8	107.7	54	7.55	103
Kevlar°/ Polyester (2)	K/P-2	34	1330/1 + 1440/1 (Kev + Poly)	9.0	210.0			7.14	
Kevlar°/ Polyester (3)	K/P-1	38	1100/1 + 1100/1 poly	7.5			54	7.55	99

* Denotes an applied force of 30 kg. ** Denotes retained strength measured for TM of 9

The hybrid cord comprising Kevlar® and polyester yarns had a retained compressive strength after fatigue cycling comparable to that of a cord comprising only polyester yarns and better than a cord comprising only Kevlar® yarns. The hybrid cord also demonstrated a 110°C thermal strength retention similar to a cord comprising only Kevlar® yarns and better than that of a cord comprising only polyester yarns. The hybrid cord was better in all respects when compared to a cord comprising only rayon yarns. These results confirm the synergistic effect of the described hybrid cord in enhancing certain mechanical properties of cords comprising only one type of yarn.

Further examples are described as follows:

Yarn and cord nomenclature as well as twist designations are well understood by those in the art.

Example 4 (BS-612-126)

In this example, a hybrid cord was made with a p-aramid staple spun yarn component Nm 28/2/2 of dtex 365x2x2 with a yarn twist 600Z/300S/530Z and with a polyester (PET) dtex 1440x1 component with a yarn twist multiplier of 9.3 in the Z direction. The hybrid cord was twisted in the S direction with a cord twist multiplier of 9.3. The hybrid cord has a tenacity of 52.2 cN/tex and an elongation at break of 11.4%.

Example 5 (BS-612-127)
In this example, a hybrid cord was made with a stretch broken p-aramid yarn component Nm 75/3/3 of dtex 133x3x3 with a yarn twist 400Z/400S/530Z and a polyester (PET) component dtex 1440x1 yarn with a twist multiplier of 9.0 in the Z direction. The hybrid cord was twisted in the S direction and had a cord tenacity of 67.3 cN/tex and an elongation at break of 11.9%.

Example 6 (BS-612-128)
In this example, a hybrid cord was made with a texturized p-aramid yarn component of dtex 1200/1 with a yarn twist multiplier of 9.0 in the Z direction and with a polyester (PET) yarn component dtex 1440x1 with a yarn twist multiplier of 9.0 in the Z direction. The hybrid cord was twisted in the S direction with a cord twist multipler of 9.0. the hybrid cord had a cord tenacity of 64.3 cN/tex and an elongation at break of 11%.

Comparative Example G (BS-612-129)
In this example, the final cord was made with p-aramid staple spun yarn component Nm 28/2 of dtex 365x2 with a yarn twist 600Z/300S. The cord had a yarn tenacity of 73.8 cN/tex and an elongation at break of 3.8%.

Comparative Example H (BS-612-130)
In this example, the final cord was made with p-aramid stretch-broken yarn component Nm 75/3 of dtex 133x3 with a yarn twist 400Z/400S. The cord had a tenacity of 126.3 cN/tex and an elongation at break of 3.2%.

Comparative Example I (BS-612-131)
In this example, the final cord was made with p-aramid texturized yarn component dtex 1200/1 with a yarn twist multiplier of 0. The cord had a tenacity of 62.2cN/tex and an elongation at break of 5.7%.

Comparative Example J (612-PET1440)
In this example, the final cord was made with polyester (PET) yarn component dtex 1440/1 with a yarn twist multiplier of 1.1. the cord had a tenacity of 71.3cN/tex and an elongation at break of 10.3%.

Fig. 2 depicts the force- elongation relationship for the examples 4-6 and G-J and clearly shows the benefits of a hybrid cord construction when compared to cords comprising only one type of component yarn.

CLAIMS

1. A fibrous cord, comprising a blend of yarns twisted together wherein the cord

(i) has a twist multiplier of from 5.0 to 12.0, and

(ii) comprises a blend of polyester yarn with either aromatic polyamide or aromatic copolyamide yarns.

2. The cord of claim 1, wherein the polyamide or copolyamide yarns are pre-twisted to a twist multiplier of at least 3.5 before cord assembly and have a linear density of at least 400 dtex.

3. The cord of claim 1, wherein the polyester yarns are pretwisted to have a twist multiplier of at least 3.5 and have a linear density of at least 444 dtex.

4. The cord of claim 1, wherein the aromatic polyamide is para-aramid.

5. The cord of claim 1, wherein the polyester is polyethyleneterephthalate, polybuthyleneterephtalate, polyethylenenaphtalene or poly(trimethylene terephthalate).

6. A composite layer, comprising an elastomer and a fabric wherein the fabric

(i) comprises from about 25 to 60 weight percent of the weight of elastomer plus fabric, and

(ii) comprises a plurality of cords of claim1.

7. The composite layer of claim 6, wherein the elastomer is nitrile butadiene rubber (hydrogenated and nonhydrogenated), ethylene-propylene-diene monomer rubber, ethylene propylene diamine monomer,chlorosulfonyl-polyethylene, ethylene oxide, chloromethyl

oxirane, hexafluoropropylene vinylidene fluoride, natural rubber, styrene-butadiene rubber or mixtures thereof.

8. The composite layer of claim 7, wherein the ethylene-propylene-diene monomer is 5-ethylidene-2-norbornene (5-ethylidenebicyclo[2.2.1]hept-2-ene), dicyclopentadiene(bicyclo[2.2.1]hepta-2,5-diene), or 1,4-hexadiene.

9. A composite layer of claim 6 for use in tires or belts.

10. A method for producing a composite layer of claim 6, the method comprising at least the steps of preparing a fabric with a plurality of cords having a twist multiplier of from 5.0 to 12, wherein the cords are formed by blending aromatic polyamide or aromatic copolyamide yarns and polyester yarns.

11. The method of claim 10, wherein the polyamide or copolyamide yarns are pre-twisted to have a twist multiplier of at least 3.5 and have a linear density of at least 400 dtex.

12. The method of claim 10, wherein the polyester yarns are pre-twisted to have a twist multiplier of at least 3.51 and have a liner density of at least 444 dtex.

13. A fibrous cord comprising a blend of yarns twisted together, wherein the cord

(i) has a twist multiplier of from 5.0 to 12.0, and

(ii) comprises a blend of aromatic polyamide and aliphatic polyamide yarns.

14. A fibrous cord, comprising a blend of yarns twisted together wherein the cord

(i) has a twist multiplier of from 5.0 to 12.0, and

(ii) comprises a blend of m-aramid and p-aramid yarns with either aromatic polyamide or aromatic copolyamide yarns or a blend of m-aramid plus p-aramid plus polyester yarns with either aromatic polyamide or aromatic copolyamide yarns.

15. The cord of claim 1, wherein the cord or the p-aramid component is staple, stretch broken or texturized.

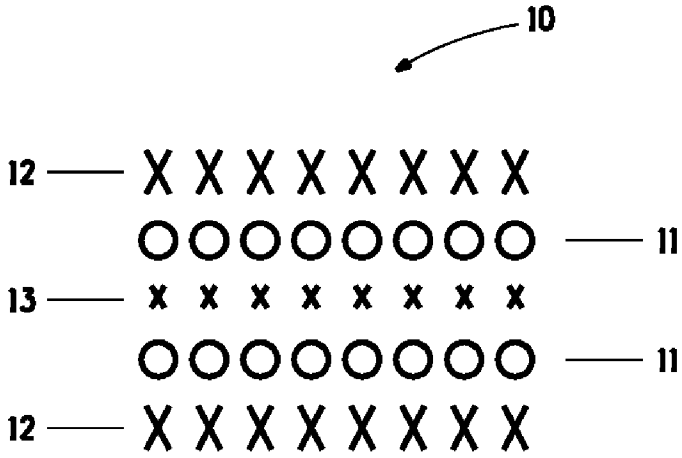

FIG. 1

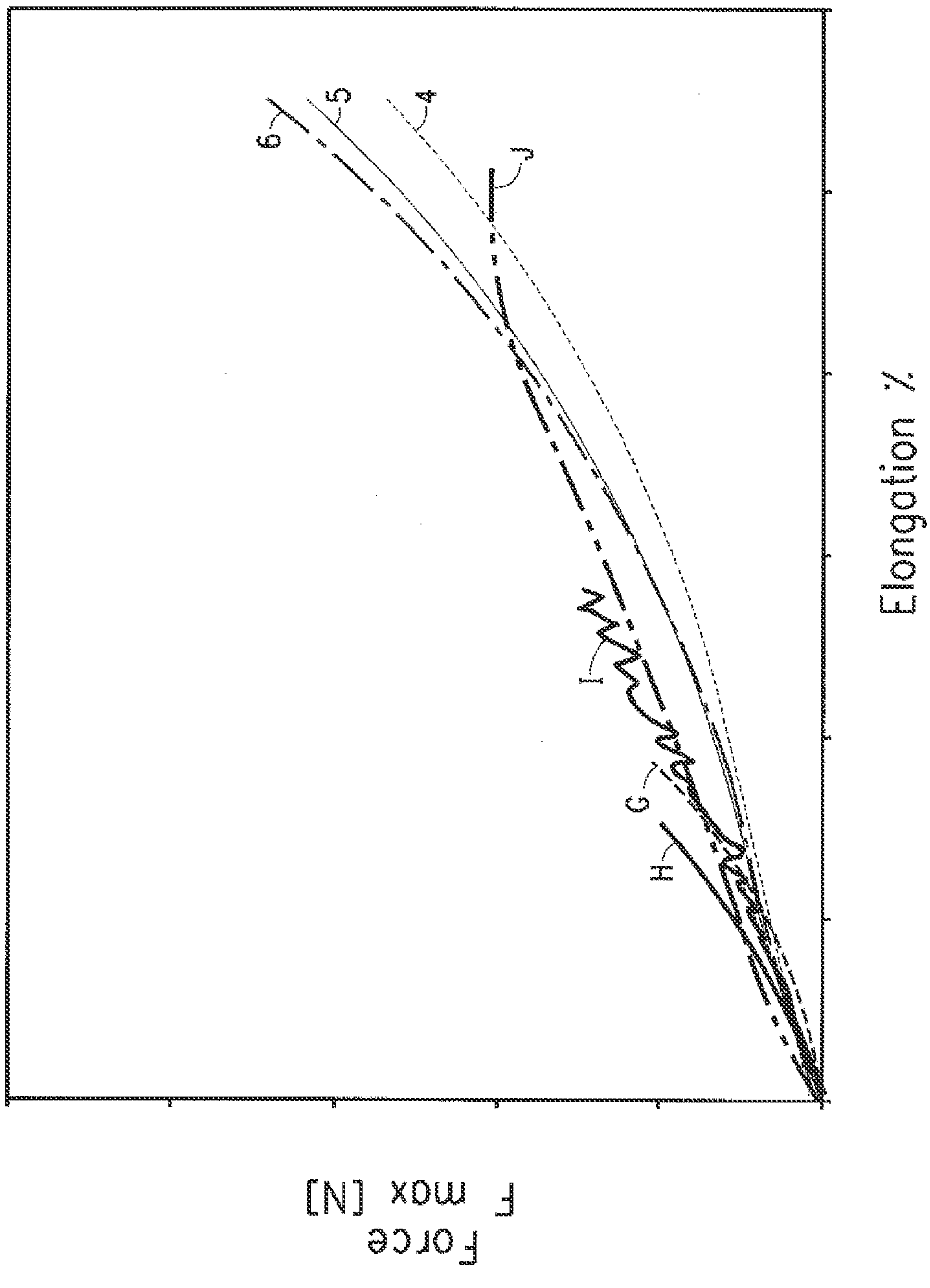

FIG. 2