(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
International Bureau

(43) International Publication Date
29 September 2016 (29.09.2016)

WIPO | PCT

(10) International Publication Number
WO 2016/154252 A1

(51) International Patent Classification:
D04H 1/435 (2012.01) *B68G 1/00* (2006.01)
D04H 1/4391 (2012.01) *A47G 9/00* (2006.01)
D04H 1/541 (2012.01)

(21) International Application Number:
PCT/US2016/023684

(22) International Filing Date:
23 March 2016 (23.03.2016)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:
201510134344.6 25 March 2015 (25.03.2015) CN

(71) Applicant: **3M INNOVATIVE PROPERTIES COMPANY** [US/US]; 3M Center, Post Office Box 33427, Saint Paul, Minnesota 55133-3427 (US).

(72) Inventors: **XIANG, Hong Bing**; Room 901, Building No. 620, Guang Fuling Road No. 1518, Shanghai 201620 (CN). **XU, Feng**; General Office, 8 Xing Yi Road, Maxdo Centre 38/F, Shanghai 200336 (CN). **TUMAN, Scott J.**; 3M Center, Post Office Box 33427, Saint Paul, Minnesota 55133-3427 (US). **ZHAO, Guo Tong**; General Office, 8 Xing Yi Road, Maxdo Centre 38/F, Shanghai 200336 (CN).

(74) Agents: **GALLAGHER, Ann K.** et al.; 3M Center, Office of Intellectual Property Center, Post Office Box 33427, Saint Paul, Minnesota 55133-3427 (US).

(81) Designated States *(unless otherwise indicated, for every kind of national protection available)*: AE, AG, AL, AM, AO, AT, AU, AZ, BA, BB, BG, BH, BN, BR, BW, BY, BZ, CA, CH, CL, CN, CO, CR, CU, CZ, DE, DK, DM, DO, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GT, HN, HR, HU, ID, IL, IN, IR, IS, JP, KE, KG, KN, KP, KR, KZ, LA, LC, LK, LR, LS, LU, LY, MA, MD, ME, MG, MK, MN, MW, MX, MY, MZ, NA, NG, NI, NO, NZ, OM, PA, PE, PG, PH, PL, PT, QA, RO, RS, RU, RW, SA, SC, SD, SE, SG, SK, SL, SM, ST, SV, SY, TH, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, ZA, ZM, ZW.

(84) Designated States *(unless otherwise indicated, for every kind of regional protection available)*: ARIPO (BW, GH,

[Continued on next page]

(54) Title: A THERMAL INSULATION FILLING MATERIAL, A PREPARATION METHOD THEREOF, AND A THERMAL INSULATION ARTICLE

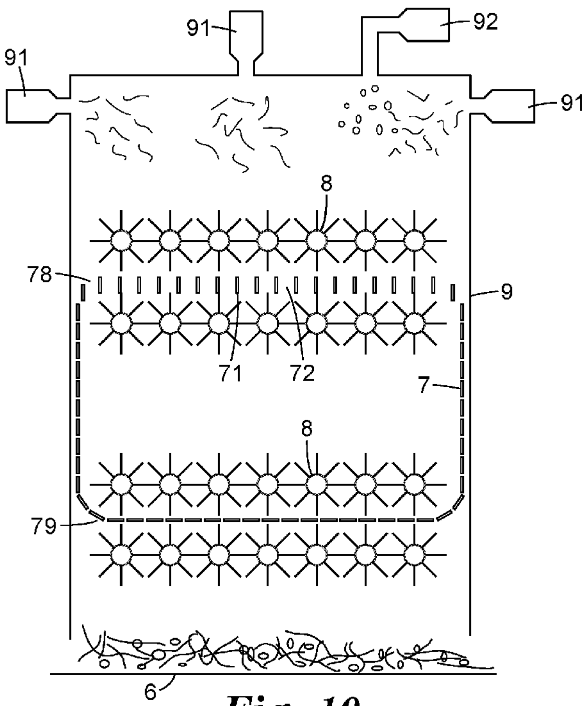

Fig. 10

(57) Abstract: The present invention provides a thermal insulation filling material. The thermal insulation filling material provided by the invention comprises loose fibers and spherical fiber assemblies, wherein a weight ratio of the loose fibers to the spherical fiber assemblies ranges from about 20:80 to about 80:20.

WO 2016/154252 A1

GM, KE, LR, LS, MW, MZ, NA, RW, SD, SL, ST, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, RU, TJ, TM), European (AL, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HR, HU, IE, IS, IT, LT, LU, LV, MC, MK, MT, NL, NO, PL, PT, RO, RS, SE, SI, SK, SM, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, KM, ML, MR, NE, SN, TD, TG).

Published:

— *with international search report (Art. 21(3))*

A THERMAL INSULATION FILLING MATERIAL, A PREPARATION METHOD THEREOF, AND A THERMAL INSULATION ARTICLE

Background

Thermal insulation filling materials are often utilized in articles such as clothes and quilts to improve the heat retention property of these products. The thermal insulation filling materials are primarily loose and fluffy materials formed of fine fibers, which are mainly classified into natural and artificial types.

Natural thermal insulation filling materials include natural fibers such as duck down, goose down, camel hair and cotton. However, due to their high cost and unstable resourcing, natural fibers are liable to be affected by epidemic diseases of animals and production declines of plants. In addition, people may be allergic to natural fibers. Therefore, the application scope of natural fibers is limited.

For the reasons described above, the application of artificial thermal insulation filling materials is growing in popularity. Artificial thermal insulation filling materials are made of artificial fibers. In terms of different forms, the artificial fibers for thermal insulation filling materials can include loose fibers (i.e. fluffy fibers) and spherical fiber assemblies. Loose fibers are loose and fluffy and can be irregular in shape. Spherical fiber assemblies are fluffy and are comprised of loose fibers crimped to be essentially spherical, i.e., comprising a plurality of small "fiber balls".

Due to the intricate structure of natural fibers, it is difficult for artificial fibers to completely simulate the structure of natural fibers. Therefore, it is difficult for artificial fibers to achieve performances similar to those of natural fibers, such as heat retention, fluffiness, and filling ability. After being washed with water, artificial fibers are also likely to be lumped together and entangled to form compact fiber agglomerates (unlike the spherical fiber assemblies), thus resulting in an uneven distribution of the thermal insulation filling materials and a sharp degradation in performance.

Summary

One aspect of the present invention is to provide a thermal insulation filling material having an improvement in wash durability while maintaining heat retention and fluffiness. The thermal insulation filling material includes loose fibers and spherical fiber assemblies, wherein a weight ratio of the loose fibers to the spherical fiber assemblies ranges from about 20:80 to about 80:20.

Another aspect of the present invention is to provide a method of preparing a thermal insulation filling material, which includes mixing loose fibers and spherical fiber assemblies, wherein a weight ratio of the loose fibers to the spherical fiber assemblies ranges from about 20:80 to about 80:20.

In one embodiment, mixing the loose fibers and the spherical fiber assemblies includes employing a licker-in mixing machine to mix the loose fibers and the spherical fiber assemblies. The licker-in mixing machine includes a mixing box having a bottom and at least one material inlet, a plurality of rotating spike rollers arranged in the mixing box and located between the material inlet and the bottom of the mixing box, and a rotating belt screen which comprises an up-stroke adjacent to the rotating spike roller and a down-stroke located between the up-stroke and the bottom of the mixing box. At least parts of the rotating spike rollers are located between the up-stroke and the down-stroke.

Another aspect of the present invention is to provide a thermal insulation article including a cover for forming an enclosed internal space and a thermal insulation filling material filled in the enclosed internal space formed by the cover. A weight ratio of the loose fibers to the spherical fiber assemblies ranges from about 20:80 to about 80:20.

Brief Description of the Drawings

Fig. 1 is a photograph of a Cotton pad 1 filled with thermal insulation filling material of the present invention.

Fig. 2 is a comparison diagram of thickness performances of Cotton pad 1 filled with thermal insulation filling materials of Example 1, Control 1 and Control 2, respectively.

Fig. 3 is a comparison diagram of Clo values of Cotton pad 1 filled with thermal insulation filling materials of Example 1, Control 1 and Control 2, respectively.

Fig. 4 is a comparison diagram of compression resilience ratios of Cotton pad 1 filled with thermal insulation filling materials of Example 1, Control 1 and Control 2, respectively.

Fig. 5 is a local nonopaque image of a washed Cotton pad 1 filled with thermal insulation filling material of Control 1.

Fig. 6 is a local nonopaque image of a washed Cotton pad 1 filled with thermal insulation filling material of Control 2.

Fig. 7 is a local nonopaque image of a washed Cotton pad 1 filled with thermal insulation filling material of Example 1.

Fig. 8 is a thermal image of a washed Cotton pad 2 filled with thermal insulation filling material of Example 5.

Fig. 9 is a thermal image of a washed Cotton pad 2 filled with thermal insulation filling material of Control 3.

Fig. 10 is a structure diagram of a licker-in mixing machine for preparing the thermal insulation filling material of the present invention.

Detailed Description

To facilitate a person skilled in the art to better understand the technical scheme of the present invention, the present invention will be further described in details with the figures and specific embodiments.

Interpretation of Terms

In the present invention, the following terms or descriptions are elaborated as follows:

The description "range from A to B" includes value A, value B and any value larger than A but smaller than B. For instance, "range from 1 to 10" includes 1, 10, and any value larger than 1 but smaller than 10, such as 2, 3, 4, 5, 6, 7, 8, 9, 2.3, 3.516, 5.26, 7.1, 9.999.

The description "A is essentially B, A is similar to B" refers to that A meets the characteristics of B in general, but unavoidable small differences are allowed between A and B. For instance, "A is essentially spherical" refers to that A is spherical in general, but the spheres are not required to be strictly spherical, and may not possess a solid structure and may possess some changes (i.e. becoming ellipsoidal).

For the term "amount", the amount or amount ratio involved in the present invention refers to weight or weight ratio unless otherwise specified.

The description "weight percentage of A in B" refers to the weight fraction of A as the weight of B is 100% provided that A is a part of B.

The term "loose material" refers to a material formed of a large number of tiny parts (fibers) accumulated by loose connection (with allowing a little action force among the parts), which is deformable, compressible, and divisible on the whole.

The term "fiber" refers to a material composed of continuous or discontinuous fine filament, with the size in the length direction greatly longer than that in any of the cross section directions.

The term "staple fiber" refers to short fibers, and usually refers to the fibers with the length of about 20-150 mm.

Thermal Insulation Filling Material

The present invention provides a thermal insulation filling material including loose fibers and spherical fiber assemblies, wherein a weight ratio of the loose fibers to the spherical fiber assemblies ranges from about 20:80 to about 80:20.

As discovered by the inventor, applying a definite proportion of the loose fibers and the spherical fiber assemblies at the same time in a thermal insulation filling material may greatly enhance the wash durability while maintaining good heat retention, fluffiness property, and filling capability.

The reasons why the thermal insulation filling material provided by the present invention possesses a good wash durability may be as follows (the following sentences do not restrict the principles of the present invention): individual loose fibers or spherical fiber assemblies are in poor contact or not in contact with each other; once encountering water flows, the fibers are liable to move with the flows to convolve with other fibers and to form compact fiber aggregations, thus resulting in the degradation of fluffiness, heat retention property and filling ability; however, in the thermal insulation filling material provided by the present invention, the loose fibers are connected on the spherical fiber assemblies, and different spherical fiber assemblies are connected by the loose fibers (the connection as described above is not firm or tight connection); therefore, each part of the thermal insulation filling material is connected quite compactly, and the fibers are more likely to move integrally without being lumped or entangled when suffering a force from the flows, thus the wash durability of the thermal insulation filling material provided by the present invention is good.

In one embodiment, in the thermal insulation filling materials, the weight ratios of the loose fibers to the spherical fiber assemblies range from about 40:60 to about 60:40, and particularly is about 50:50. The amounts of the loose fibers and the spherical fiber assemblies has an important influence on the performance of the loose fibers and the spherical fiber assemblies. As found by studies, the wash durability of the thermal insulation filling material is greatest when the amount of the loose fibers and the spherical fiber assemblies meets the proportion range as described above.

In one embodiment, the thermal insulation filling material is composed essentially of the loose fibers and the spherical fiber assemblies. In other words, the thermal insulation filling material does not contain any other component except the loose fibers and the spherical fiber assemblies. In this case, the loose fibers account for about 20wt% to about 80wt% of the thermal insulation filling material, particularly about 40wt% to about 60wt%, and more particularly about 50%; with the balance being the spherical fiber assemblies.

Of course, other ordinary components in addition to the loose fibers and the spherical fiber assemblies may be used in the thermal insulation filling material if necessary.

In one embodiment, the fibers for forming the loose fibers and the spherical fiber assemblies are staple fibers, specifically staple fibers having a length ranging from about 20mm to about 150mm. Particularly, the length of the staple fibers ranges from about 32mm to about 72mm and more particularly from about 38 mm to about 51 mm.

In other words, in one embodiment, the staple fibers having a definite length are employed to form the loose fibers and the spherical fiber assemblies. Fibers that are too short to form loose materials are ineffective in keeping the heat, while fibers that are too long are liable to be entangled with each other. Studies have found that the length range as described above is the optimal.

In one embodiment, the fibers for forming the loose fibers and the spherical fiber assemblies meet one or more of the following properties:

(1) The fiber fineness ranges from about 0.3 Denier to about 7 Denier, particularly from about 2 Denier to about 7 Denier. The term “fiber fineness" refers to the size of fibers; and “Denier” refers to the unit of fineness, which is also known as “D”, representing the gram weight of fibers with the length of 9,000 m based on official regain.

(2) The fibers have a three-dimensional crimped hollow structure. The term “three-dimensional crimp” means that the length direction of the fibers is curved rather than distributed along a straight line, and the curvature may occur in each direction rather than only in a particular plane, thus the fibers are liable to form a non-planar stereo structure.

(3) The fibers are siliconized fibers, particularly siliconized polyester fibers.

Studies have found that loose fibers and spherical fiber assemblies which are formed of fibers whose performance does not meet the requirements as described above, may not be applicable to the thermal insulation filling material.

In one embodiment, the particle size of the spherical fiber assemblies in the thermal insulation filling material ranges from about 2 mm to about 15 mm.

The spherical fiber assemblies having the particle size range as described above may best improve the wash durability of the thermal insulation filling material.

Preparation Method for the Thermal Insulation Filling Material

The present invention also provides a method of preparing the thermal insulation filling material, which includes: mixing loose fibers and spherical fiber assemblies, wherein a weight ratio of the loose fibers to the spherical fiber assemblies ranges from about 20:80 to about 80:20.

As one embodiment of the present invention, the spherical fiber assemblies may be formed of raw material fibers in advance and then mixed with the loose fibers. One specific embodiment of preparation steps includes respectively weighing the raw material fibers for forming the loose fibers and the spherical fiber assemblies [S101]. Conducting rough opening and precision opening of the raw material fibers in a CKSC-130 fiber opener (purchased from Jiangsu Changsheng Co., Ltd.) are carried out to form the loose fibers, wherein the processing parameters are as follows: cylinder diameter: 500 mm, rotation speed of opening roller: 800 r/min, installed capacity: 6.95 kW, fan: 2.2 kW, and motor equipped with 2.5# impeller; then in the same opener as described above, conducting rough opening of the raw material fibers, wherein the processing parameters are the same as the parameters as described above; and then processing the raw fiber materials subjected to rough opening into fiber balls in a granulator (purchase from Jiangsu Changsheng Co., Ltd.), wherein the processing parameters are as follows: motor power: 7.5 kW, diameter of opening roller: 550 mm, length of opening roller: 1.8 m, and rotation speed of opening roller: 800 r/min [S102]. The loose fibers and the spherical fiber assemblies are fed into a CSKS-200 large-scale cotton mixer (purchased from Jiangsu Changsheng Co., Ltd.) through an airflow mixing pipe (purchased from Jiangsu Changsheng Co., Ltd.) to mix the two components to form the thermal insulation filling material, wherein the processing parameters are as follows: evener roller: 260 mm, rotation speed of evener roller: 310r/min, diameter of beater: 400mm, speed of beater: 300 r/min, beater motor: Y112M-4-2.2kW, roller motor: 1.5 kW, and spike lattice motor: 2.2 kW [S103].

The steps as described above for preparing the loose fibers and the spherical fiber assemblies are in no particular order. For instance, opening may be conducted in advance of weighing, or weighing may be conducted after the loose fibers and the spherical fiber assemblies are prepared. In addition, the preparation is not limited to the particular devices and processing parameters as described above.

It should be understood that, although the spherical fiber assemblies in the method as described above are self-prepared, commercially available spherical fiber assemblies are also usable as a raw material of the spherical fiber assemblies, such as the spherical fiber assemblies produced by Stein Fibers Co., Ltd. in Albany, New York, U.S.

As another preferred embodiment of the present invention, a licker-in mixing machine may be employed to mix the loose fibers and the spherical fiber assemblies, wherein the licker-in mixing machine includes: a mixing box having a bottom and at least one material inlet, a plurality of rotating spike rollers arranged in the mixing box and located between the material inlet and the bottom of the mixing box, and a rotating belt screen which comprises an up-stroke adjacent to the rotating spike roller and a down-stroke located between the up-stroke and the bottom of the mixing box, wherein at least parts of the rotating spike rollers are located between the up-stroke and the down-stroke.

The licker-in mixing machine as described above has been described in U.S. Patents Applications US2005/0098910 and US2010/0092746 and PCT application WO2011/133396A. As shown in Fig. 10, the licker-in mixing machine includes a mixing box 9 which is provided with one or more material inlets 91, 92 for feeding in raw materials. The mixing box 9 is provided with a space at the bottom for collecting mixed materials; alternatively, the bottom of the mixing box 9 is open so that a conveying belt 6 may take the mixed materials away. The mixing box 9 is internally provided with a plurality of rotating spike rollers 8 capable of tearing apart the fibers to make the fibers loose and mixed with each other. The mixing box 9 is also internally provided with a rotating belt screen having a circular rotating belt screen 7. The belt screen 7 has supporting parts 71 capable of supporting the fibers and a net opening 72 between the supporting parts 71. In one embodiment, the area of the supporting part 71 and that of the net opening 72 are adjustable (i.e. changing the area of the area for the supporting part 71 and that of the net opening 72 through rotating the supporting part 71). The belt screen 7 has an up-stroke 78 and a down-stroke 79. The up-stroke 78 is lower than the material inlet 91 and close to at least one spike roller 8, and the down- stroke 79 is lower than the up-stroke 78 but higher than the bottom of the mixing box 9. At least parts of the spike rollers 8 are located between the up-stroke 78 and the down-stroke 79.

After entering the material inlets 91 and, 92, raw material fibers pass through the upper spike roller 8, the up-stroke 78 of the belt screen 7, the lower spike roller 8 and the down-stroke 79 of the belt screen 7 in sequence, and are torn apart and mixed by the spike rollers 8. The fibers not fully torn or mixed are unable to drop from the net opening 72 of the down-stroke 79 of the belt screen 7 due to being too large and are liable to be retained on the upper surface of the down-stroke 79 of the belt screen 7 and driven to the lower surface of the up-stroke 78 along with the motion of the belt screen 7. Thereby, the fibers drop again to be torn apart by the spike rollers 8 until the fibers are small enough to pass through the net opening 72 to the bottom of the mixing box 9.

In one embodiment, the loose fibers and the spherical fiber assemblies can be fed through different material inlets 91 and 92. For instance, as shown in Fig. 10, the loose fibers are fed through three material inlets 91 on the top side of the mixing box 9 and lateral sides of the top side and the spherical fiber assemblies are alone fed through the material inlet 92 which is also arranged on the top side of the mixing box 9. As described in the PCT application WO2011/133396A, the material inlet 92 is suitable for feeding chemically active particles and the spherical fiber assemblies are similar to the particles in shape, thus the spherical fiber assemblies can be fed through the preceding material inlet.

Using the spike roller as described above, the components of the thermal insulation filling material can be mixed more sufficiently and the resulting mixed product is fluffier, thus improving performance of the product.

A Thermal Insulation Article

The present invention also provides a thermal insulation article including a cover for forming an enclosed internal space and thermal insulation filling material filled in the internal space formed by the cover. The thermal insulation filling material includes loose fibers and spherical fiber assemblies, wherein a weight ratio of the loose fibers to the spherical fiber assemblies ranges from about 20:80 to about 80:20.

In other words, the thermal insulation filling material as described above can be filled in the cover to form the thermal insulation article suitable for actual application.

In one embodiment,, the cover is a flexible cover. In other words, the cover may be a flexible sheet such as fabric or leather, thus the enclosed internal space may be formed by sewing and the thermal insulation filling material may be filled in the enclosed internal space.

In one embodiment, the thermal insulation article may be bedclothes and clothes, including, but not limited to: shoes, hats, clothes (such as garments, trousers, underwear and coats), pillows, quilts, mats and sleeping bags.

EXAMPLES

Materials

The raw materials used in the Examples and Controls of the present invention were as follows:

Siliconized polyester fibers 1: 3 Denier, 38 mm long, hollow structure, purchased from Sinopec Yizheng Chemical Fiber Company Limited.

Siliconized polyester fibers 2: 3 Denier, 60 mm long, hollow structure, purchased from Sinopec Yizheng Chemical Fiber Company Limited.

Siliconized polyester fibers 3: 7 Denier, 38 mm long, hollow structure, purchased from Sinopec Yizheng Chemical Fiber Company Limited.

Siliconized polyester fibers 4: 7 Denier, 60 mm long, hollow structure, purchased from Sinopec Yizheng Chemical Fiber Company Limited.

Siliconized polyester fibers 5: 0.9 Denier, 32 mm long, purchased from Stein Fibers Co., Ltd. in Albany, New York State, U.S.

Siliconized polyester fibers 6: 6 Denier, 51 mm long, purchased from Stein Fibers Co., Ltd. in Albany, New York State, U.S.

Spherical fiber assemblies 1: 4 Denier, purchased from Stein Fibers Co., Ltd. in Albany, New York State, U.S.

Performance Testing

In the present invention, the thermal insulation filling material of each of the Examples was subjected to performance testing. The test methods are detailed in the following sections.

Preparation of Cotton Pad

Certain performance tests performed on the thermal insulation filling material were possible only after the thermal insulation filling material was filled. Thus the thermal insulation filling material was processed into a cotton pad (thermal insulation article) as follows:

Cotton pad 1: A piece of 190T blue nylon fabric was sewed to form a cover having a size of 19.7 in x 10.7 in (50 cm × 27 cm), a required amount of the thermal insulation filling material was weighed to fill into the cover, and then the cover was sewed in a manner of forming three lattices in any direction (i.e. evenly subdividing the cover into nine lattices) to form Cotton pad 1 with the required filling content. The filling content refers to the content of the thermal insulation filling material filled in the cover, expressed in grams per square meter of the cotton pad, with the unit of "g/m^2".

Cotton pad 2: A standard sewing machine was employed to sew a piece of blue nylon fabric to form a cover having a size of 12 in x 12 in (30.5 cm x 30.5 cm), a required amount of the thermal insulation filling material was weighed to fill into the cover, and then the cover was evenly sewed into three strips (respectively being 4 in wide) to form Cotton pad 2, wherein the filling content of the Cotton pad 2 was 1.9 oz/yd^2 (64.1 g/m^2).

Thickness Test

Generally speaking, for the test on thickness of the cotton pad, provided that the filling content is the same, the thicker the cotton pad is, the better the fluffiness of thermal insulation filling material is.

Different cotton pads were tested by different methods specifically comprising:

Cotton pad 1: An ID-U1025E thickness testing gauge (purchased from Mitutoyo Corporation) was employed to test the thickness of Cotton pad 1. The size of the presser foot of the thickness testing gauge was 30 cm x 30 cm. During the test, 190 gram weights were placed on the presser foot.

Cotton pad 2: The thickness of Cotton pad 2 was measured as per the ASTM 5736 Standard (Test Method for Thickness of Nonwovens with High Filling Content). The size of the presser foot of the thickness testing gauge was 30 cm x 30 cm, the levelness difference between the anvil and the presser foot was within 0.13 mm, the size of the anvil in each direction was 10 mm larger than that of the presser foot, and the measurement of the thickness testing gauge was accurate to at least 0.02 mm. During the test, Cotton pad 2 was placed gently on the anvil during the test, 288 gram weights were placed on the presser foot to steadily lower the presser foot to contact with Cotton pad 2, and then the thickness of Cotton pad 2 was recorded after the reading remained steady for 9-10 seconds.

Fill Power Test

The fill power of the unfilled thermal insulation filling material was tested directly. The fill power refers to the volume of the un-pressed thermal insulation filling material per unit weight, which is influenced by many factors such as the environment, the material form and the treatment of the material. Generally speaking, the higher the fill power is, the better the fluffiness of thermal insulation filling material is.

The fill power test was conducted as follows: Approximately 2 ounces (56.7 plus or minus 0.5 grams) of the thermal insulation filling material was placed into a measuring cylinder having a diameter of 9.5 in (24.1 cm) and a height of 20 in (5.8 cm) by tearing the thermal insulation filling material into small pieces with a diameter of about 1 in and feeding them in one by one in to the cylinder if the thermal insulation filling material is compressed. The thermal insulation filling material was then stirred in the measuring cylinder using a stick, thus loosening the thermal insulation filling material and enabling the thermal insulation filling material to reach a maximum volume. A cover weighing about 68 grams was then gently placed onto the upper surface of the thermal insulation filling material in the measuring cylinder and was held in place for 30 seconds. The cover was then removed and the volume at half level of the thermal insulation filling material in the measuring cylinder (i.e. at the level corresponding to 1 ounce) was recorded to obtain the fill power of the thermal insulation filling material. The fill power was expressed in cubic inches per ounce (in^3/oz).

Heat Retention Test

The heat retention property of the thermal insulation filling material was evaluated by testing the Clo value of Cotton pad 1 and Cotton pad 2. Clo value is a metric parameter for the heat retention property. For reference, the Clo value of clothing is equal to 1 when a person (with the calorific value of 209.2 $kJ/m^2 \cdot h$) sitting quietly or taking part in a little activity feels comfortable in an environment having a temperature of 21°C, a relative humidity of lower than 50% and a wind speed slower than 0.1 meters/second. The Clo value substantially refers to a thermal resistance value. The larger the Clo value is, the better the heat retention property of a material is.

Different cotton pads were tested by different methods specifically comprising:

Cotton pad 1: The Clo value of Cotton pad 1 was tested as per the ASTM F1868 Standard (Test Method for Thermal Resistance of Textiles).

Cotton pad 2: The Clo value of the Cotton pad 2 was tested as per the ASTM C518 Standard (Test Method for Steady-state Heat Flux), comprising specific test steps as follows. Cotton pad 2 was clamped between a hot plate and a cold plate so that Cotton pad 2 covered the surface of the hot plate and the cold plate. The distance L between the hot plate and the cold plate was determined using the results of the thickness test as described above. A heat flux transducer was arranged between the hot plate and Cotton pad 2. The hot plate and the cold plate were controlled at a required temperature to realize a temperature difference ΔT between the hot plate and the cold plate, and the and the temperature difference was chosen as per the ASTM C1058 Standard, specifically controlling the hot plate at 37°C and the cold plate at 13°C. The output of the heat flux transducer was observed at an interval of at least 10 minutes until the difference between each value of continuous five output values of the heat flux transducer and an average of the five values was not larger than 0.5%. The thermal conductivity C of Cotton pad 2 was then calculated according to the formula $C = S \times E/\Delta T$, wherein S represented the calibration factor of the heat flux transducer, and E represented the output value of the heat flux transducer. The thermal resistance R of Cotton pad 2 was calculated according to the formula $R = 1/C$. The Clo value substantially referred to the thermal resistance, which was 1.136 times of the thermal resistance value R (the unit of the thermal resistance is $m^2 \cdot K/W$).

Compression Resilience Ratio Test

The resilience ratio of Cotton pad 1 after being compressed (i.e. compression resilience ratio) was tested. The higher the compression resilience ratio, the better the deformation recovery capability of thermal insulation filling material is.

The test was conducted as per Part 6.10 of the FZ/T 64006 Standard, specifically comprising the following steps. Cotton pad 1 was placed on the ID-U1025E thickness testing gauge (purchased from Mitutoyo Corporation), and a light pressure of 0.02kPa was applied to Cotton pad 1 over a surface area of $400cm^2$ and was held for 10 seconds to acquire the initial thickness t0 (mm) of the cotton pad. Then a heavy pressure of 0.1kPa was applied to Cotton pad 1 over a surface area of $400cm^2$ and was held for 1 minute to acquire the initial thickness th of the Cotton pad 1 under heavy pressure. The pressure was removed from Cotton pad 1, and then a light pressure was applied to Cotton pad 1 again 1 minute later as per the method as described above to acquire the thickness of resilience tr (mm) of Cotton pad 1. The compression resilience ratio (%) was then calculated according to the formula $100 \times (tr - th) \div (t0 - th)$. Five samples of each Example were tested, and the average was taken as the compression resilience ratio.

Wash Test

The cotton pads were washed and then subjected to performance testing to evaluate the wash durability of the thermal insulation filling material.

Different cotton pads were tested by different methods specifically comprising:

Cotton pad 1: Cotton pad 1 was washed 10 times as per the GB/T8629-2001:7A Standard. The washing was conducted with an FOM71 CLS horizontal drum-type washer (purchased from Electrolux Co., Ltd.), and the mixing at each step was gentle. Each washing operation was conducted through the following specific processes: washing with the water temperature of 40° plus or minus 3°C and the water level of 13 cm for 3 minutes, without cooling, wherein 20 g of standard washing powder as specified in the GB/T8629-2001:7A was used; rinsing with the water level of 13 cm for the first time for 3 minutes; rinsing with the water level of 13 cm for the second time for 3 minutes, and then dewatering for 1 minute; and rinsing with the water level of 13 cm for the third time for 2 minutes, and then dewatering for 6 minutes.

Cotton pad 2: Cotton pad 2 was washed and dried in cycles for 10 times in a top load washer and dryer, and 20 g of standard washing powder as specified in the ISO6330-2000 was used in each washing operation.

Transmitted-Light Image

Cotton pad 1 was placed on a glass table irradiated by sunshine and then was photographed. The distribution of the thermal insulation filling material was identified upon observation of the light transmitting through the cotton pad.

Thermal Image

Cotton pad 2 was placed on a hot plate at 40°C for about 1 hour until the temperature was distributed evenly. A FLIR thermal imaging camera (purchased from FLIR Co., Ltd. in Wilsonville, Oregon, U.S.) was employed to photograph a thermal image of Cotton pad 2, and the distribution of the thermal insulation filling material was identified upon the temperature distribution.

Examples 1-5

The thermal insulation filling material for each Example and Control was prepared from different raw materials as described above. Specific parameters are provided in Table 1.

Table 1 - Thermal Insulation Filling Materials

Example	Raw Materials of Loose Fibers	Content of Loose Fibers (Part by Weight)	Raw Materials of Spherical Fiber Assemblies	Content of Spherical Fiber Assemblies (Parts by Weight)	Mixing Machine	Form of Cotton Pad

Example 1	Siliconized polyester fibers 1	50	Siliconized polyester fibers 1	50	Large-column cotton blending machine	Cotton pad 1
Example 2	Siliconized polyester fibers 2	50	Siliconized polyester fibers 2	50	Large-column cotton blending machine	Cotton pad 1
Example 3	Siliconized polyester fibers 3	50	Siliconized polyester fibers 3	50	Large-column cotton blending machine	Cotton pad 1
Example 4	Siliconized polyester fibers 4	50	Siliconized polyester fibers 4	50	Large-column cotton blending machine	Cotton pad 1
Example 5	Siliconized polyester fibers 5 + siliconized polyester fibers 6	37 + 13	Spherical fiber assemblies 1	50	Licker-in mixing machine	Cotton pad 2
Control 1	Siliconized polyester fibers 1	100	None	None	None	Cotton pad 1
Control 2	None	None	Siliconized polyester fibers 1	100	None	Cotton pad 1
Control 3	None	None	Spherical fiber assemblies 1	100	None	Cotton pad 2

The Examples represent the invention thermal insulation filling material comprising the loose fibers and the spherical fiber assemblies, and the Controls represent prior thermal insulation filling materials comprising the loose fibers alone or the spherical fiber assemblies alone.

Except for Example 5 and Control 3, the spherical fiber assemblies were formed of the raw material fibers by self-preparation using the preparation method as described above. The spherical fiber assemblies in Examples 5 and Control 3 are the product of Spherical fiber assemblies 1.

Except for Example 5, the two raw materials in the Examples were mixed by a large-column cotton blending machine. The two raw materials in Example 5 were mixed by a spike roller using the preparation method described above.

In Example 5, the loose fibers were composed of 37 parts of Siliconized polyester fibers 5 by weight and 13 parts of Siliconized polyester fibers 6.

Performance Test Results and Analysis

According to the methods as described above, the thermal insulation filling material in each Example and Control was tested.

Fig. 2 shows a thickness test result of the Cotton pads 1 filled with the thermal insulation filling materials of Example 1, Control 1 and Control 2.

Fig. 3 shows a Clo value (heat retention property) test result of the Cotton pads 1 filled with the thermal insulation filling materials of Example 1, Control 1 and Control 2.

Fig. 4 shows a compression resilience ratio test result of the cotton pads 1 filled with the thermal insulation filling materials of Example 1, Control 1 and Control 2.

As shown in Fig. 2 to Fig. 4, each performance of the thermal insulation filling material provided by the present invention is essentially between that of the loose fibers alone and that of the spherical fiber assemblies alone, which indicates that the fluffiness, heat retention property and resilience of the thermal insulation filling material provided by the present invention are unlikely to be degraded, capable of meeting the requirements of a thermal insulation filling material.

Fig. 5 to Fig. 7 respectively show a transmitted light image of the washed Cotton pads 1 filled with the thermal insulation filling materials of Control 1, Control 2 and Example 1. The gram weight of each cotton pad was 100 g/m^2.

Both Fig. 5 and Fig. 6 show unevenly distributed dark regions and bright regions indicating that the fibers in the cotton pad are distributed unevenly after being washed. In other words, the loose fibers as well as the spherical fiber assemblies alone are liable to be obviously lumped and entangled after being washed. In Fig. 7, the brightness of each part is essentially the same. In other words, even if the thermal insulation filling material provided by the present invention is washed many times, the fibers are still evenly distributed and hardly lumped or entangled, and the performances such as heat retention property and fluffiness are essentially unchanged. Therefore, the thermal insulation filling material provided by the present invention has an improvement in wash durability.

A comparison of the performances of Cotton pads 2 filled with the thermal insulation filling materials of Example 5 and Control 3 is provided in Table 2.

Table 2 - Performance Test Results for Thermal Insulation Filling Materials of Example 5 and Control 3

Example	Fill Power before Washing (in^3/oz)	Thickness before Washing (cm)	Thickness after Washing (cm)	Clo Value before Washing	Clo Value after Washing
Example 5	350	4.4	3.9	4.6	4.3
Control 3	250	3.5	3.4	4.0	3.9

Provided that the same spherical fiber assemblies are used, each performance of the thermal insulation filling material mixed by the licker-in mixing machine (Example 5) before and after washing was better than that of the thermal insulation filling material comprising the spherical fiber assemblies alone (Control 3), which suggests that the mixing approach as described above may further improve the performance of the thermal insulation filling material. Additionally, some performance parameters of the thermal insulation filling material of Example 5 were not as good after washing; however, the performance parameters after washing were still better than those of Control 3, which suggests that the mixing approach as described above may be helpful to improve the wash durability of the product. In other words, the mixing approach as described above does not have a negative impact on the wash durability of the thermal insulation filling material.

Fig. 8 and Fig. 9 respectively are thermal images of Cotton pads 2 respectively filled with the thermal insulation filling materials of Control 3 and Example 5. The temperature distribution in the thermal image of Cotton pad 2 filled with the thermal insulation filling material of Example 5 is more even compared to Control 3, which suggests that compared to the distribution of the thermal insulation filling material of Control 3, the distribution of the thermal insulation filling material of Example 5 was more even after washing, and the heat retention property was better.

The performance data suggests that the mixing approach (mixing with a licker-in mixing machine as described above) that was used to prepare the thermal insulation filling materials of the Examples of the present invention may further improve the product performances before and after the washing.

It should be understood that the Examples as described above are exemplary embodiments only for illustrating principles of the present invention, and the present invention is not limited to the foregoing embodiments. Various other modifications and improvements may be made by one skilled in the art without departing from spirit or scope of the present invention, and those modifications and improvements will also fall within the protection scope of the present invention.

What is claimed is:

1. A thermal insulation filling material, comprising:
loose fibers, and
spherical fiber assemblies,
wherein a weight ratio of the loose fibers to the spherical fiber assemblies ranges from about20:80 to about 80:20.

2. The thermal insulation filling material according to Claim 1, wherein the weight ratio of the loose fibers to the spherical fiber assemblies ranges from about 40:60 to about 60:40.

3. The thermal insulation filling material according to Claim 2, wherein the weight ratio of the loose fibers to the spherical fiber assemblies is about 50:50.

4. The thermal insulation filling material according to Claim 1, wherein the loose fibers and the spherical fiber assemblies are formed from staple fibers having a length ranging from about 20mm to about 150mm.

5. The thermal insulation filling material according to Claim 4, wherein the staple fibers forming the loose fibers and the spherical fiber assemblies have a length ranging from about 32mm to about 72mm.

6. The thermal insulation filling material according to Claim 5, wherein the staple fibers forming the loose fibers and the spherical fiber assemblies have a length ranging from about 38mm to about 51mm.

7. The thermal insulation filling material according to Claim 1, wherein the loose fibers and the spherical fiber assemblies are formed from fibers have a fineness ranging from about 0.3 Denier to about 7 Denier.

8. The thermal insulation filling material according to Claim 7, wherein the fibers forming the loose fibers and the spherical fiber assemblies have a fineness ranging from about 2 Denier to about 7 Denier.

9. The thermal insulation filling material according to Claim 1, wherein the loose fibers and the spherical fiber assemblies are formed from siliconized fibers.

10. The thermal insulation filling material according to Claim 9, wherein the fibers forming the loose fibers and the spherical fiber assemblies are siliconized polyester fibers.

11. The thermal insulation filling material according to Claim 1, wherein the loose fibers and the spherical fiber assemblies are formed from fibers having a three-dimensional crimped hollow structure.

12. The thermal insulation filling material according to Claim 1, wherein the spherical fiber assemblies have a particle size ranging from about 2mm to about 15mm.

13. The thermal insulation filling material according to Claim 1, wherein the thermal insulation filling material is composed essentially of the loose fibers and the spherical fiber assemblies.

14. A method of preparing a thermal insulation filling material, comprising:
mixing loose fibers and spherical fiber assemblies, wherein a weight ratio of the loose fibers to the spherical fiber assemblies ranges from about 20:80 to about 80:20.

15. The method according to Claim 13, wherein mixing the loose fibers and the spherical fiber assemblies comprises:

employing a licker-in mixing machine to mix the loose fibers and the spherical fiber assemblies, wherein the licker-in mixing machine comprises:

a mixing box having a bottom and at least one material inlet,

a plurality of rotating spike rollers arranged in the mixing box and located between the material inlet and the bottom of the mixing box, and

a rotating belt screen which comprises an up-stroke adjacent to the rotating spike roller and a down-stroke located between the up-stroke and the bottom of the mixing box, wherein at least parts of the rotating spike rollers are located between the up-stroke and the down-stroke.

16. A thermal insulation article, comprising:

a cover for forming an enclosed internal space; and

a thermal insulation filling material filled in the enclosed internal space formed by the cover, wherein the thermal insulation filling material comprises loose fibers and spherical fiber assemblies, and wherein a weight ratio of the loose fibers to the spherical fiber assemblies ranges from about 20:80 to about 80:20.

17. The thermal insulation article according to Claim 16, wherein the cover is a flexible cover.

18. The thermal insulation article according to Claim 16, wherein the thermal insulation article is any of shoes, hats, clothes, pillows, quilts, mats and sleeping bags.

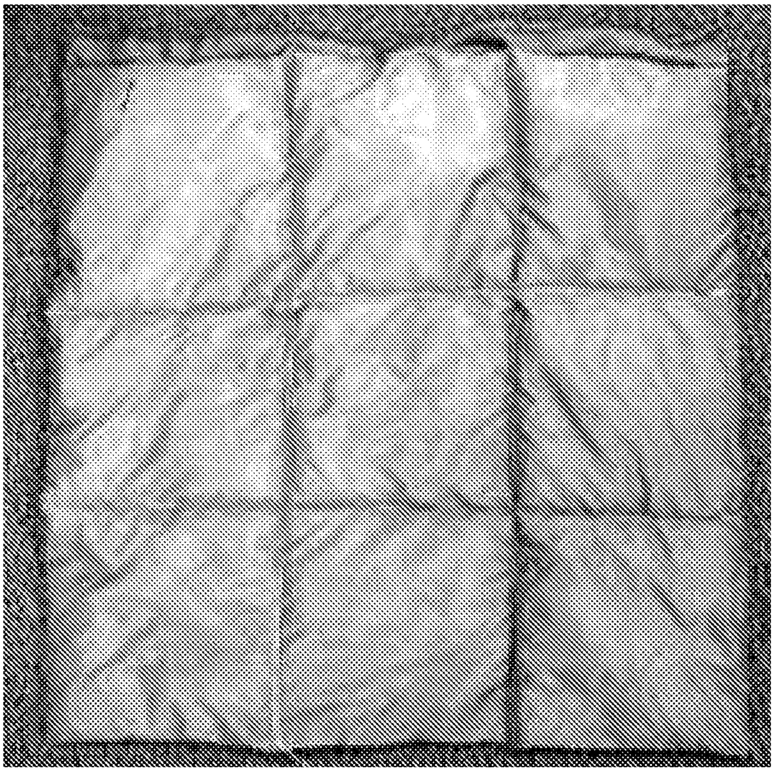

Fig. 1

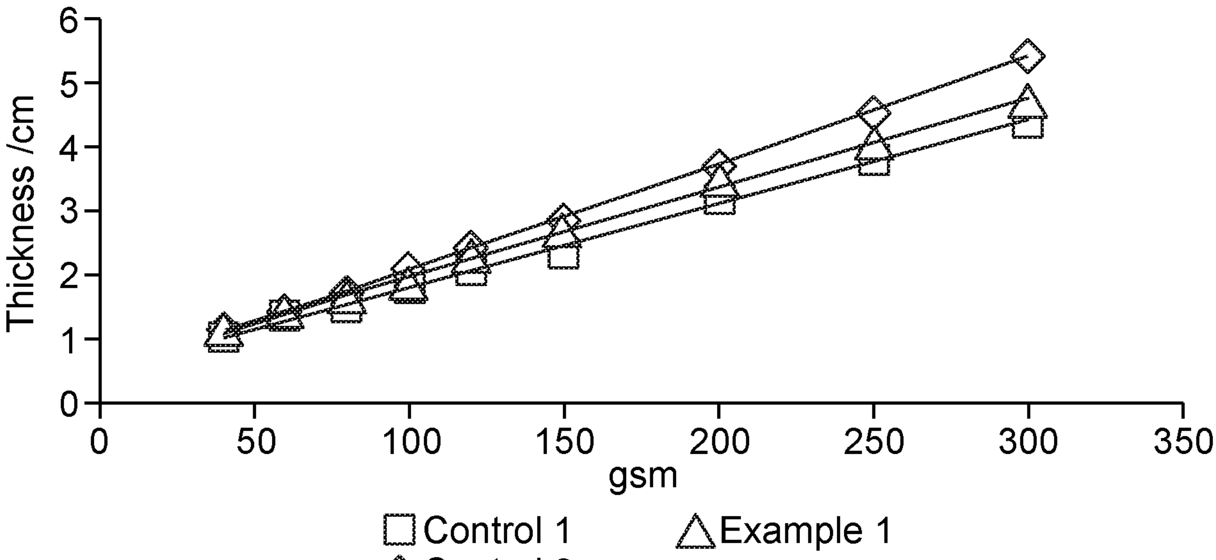
Thickness /cm
0
1
2
3
4
5
6
0
50
100
150
200
250
300
350
gsm
Control 1
Example 1
Control 2

Fig. 2

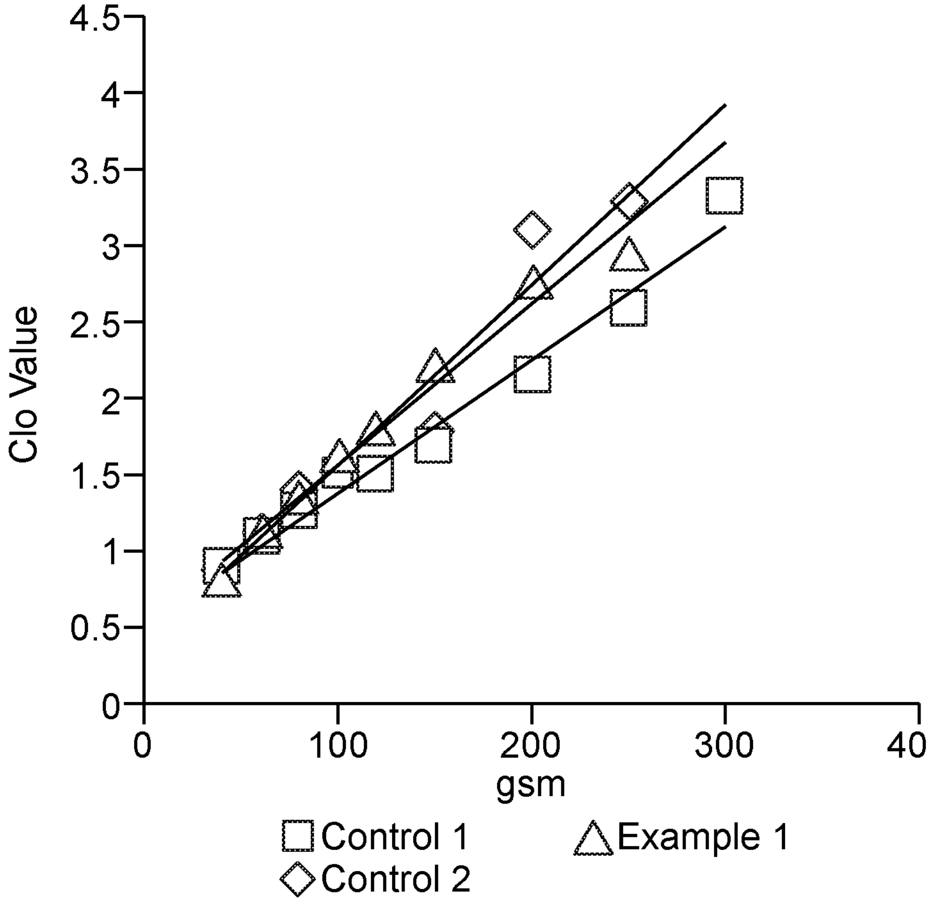
4.5
4
3.5
3
2.5
2
1.5
1
0.5
0
Clo Value
0
100
200
300
400
gsm
Control 1
Example 1
Control 2

Fig. 3

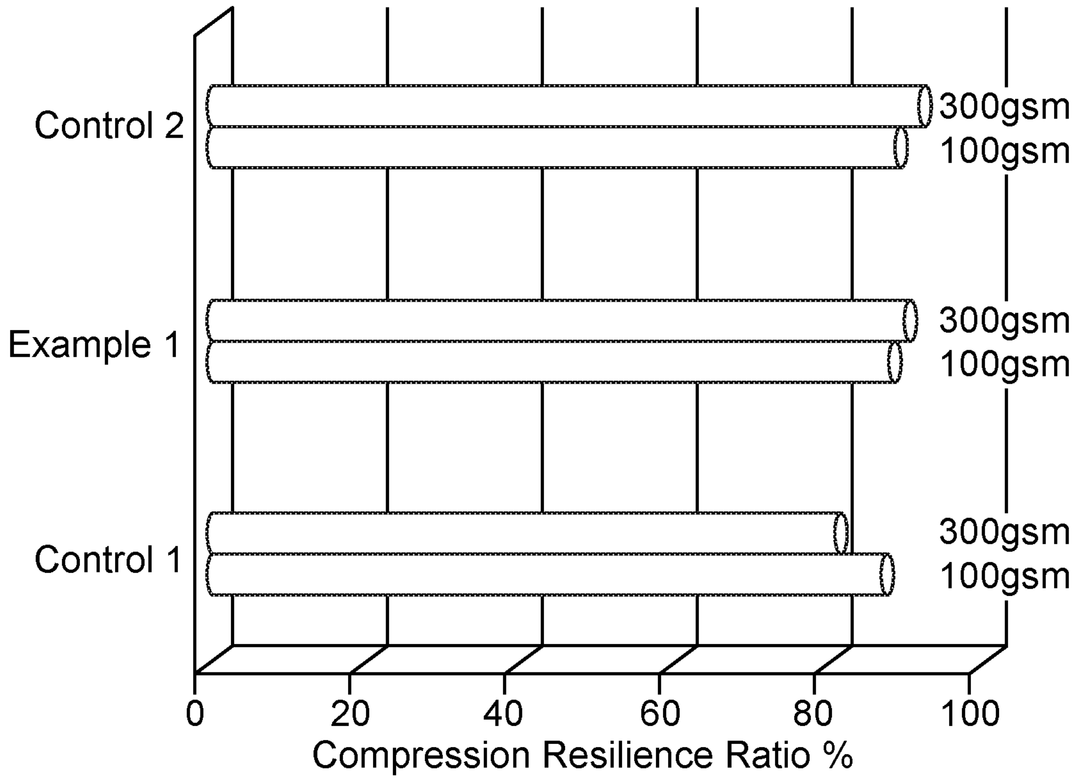
Control 2
Example 1
Control 1
300gsm
100gsm
300gsm
100gsm
300gsm
100gsm
0
20
40
60
80
100
Compression Resilience Ratio %

Fig. 4

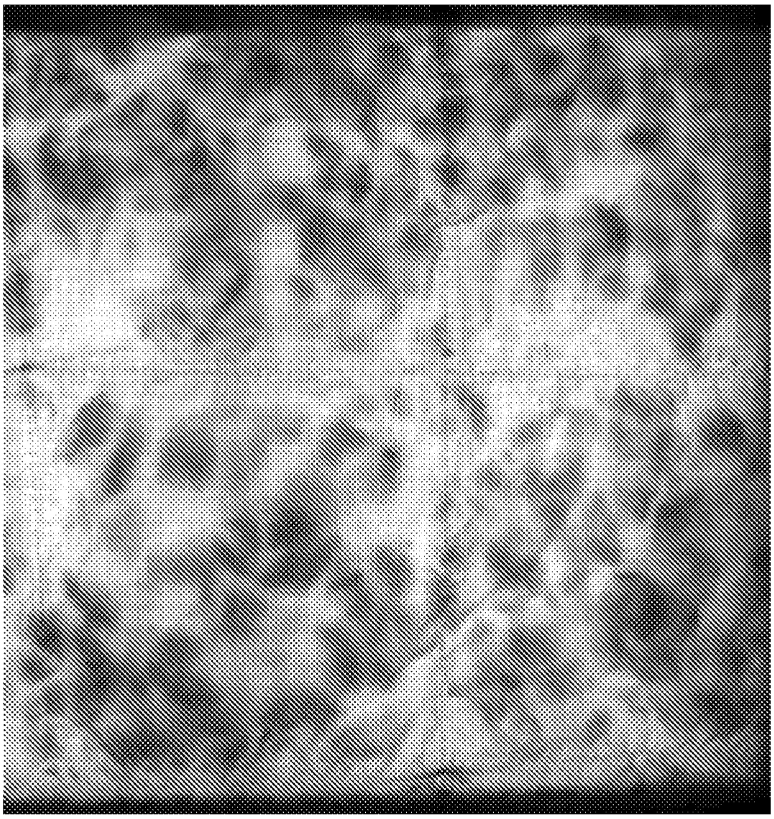

Fig. 5

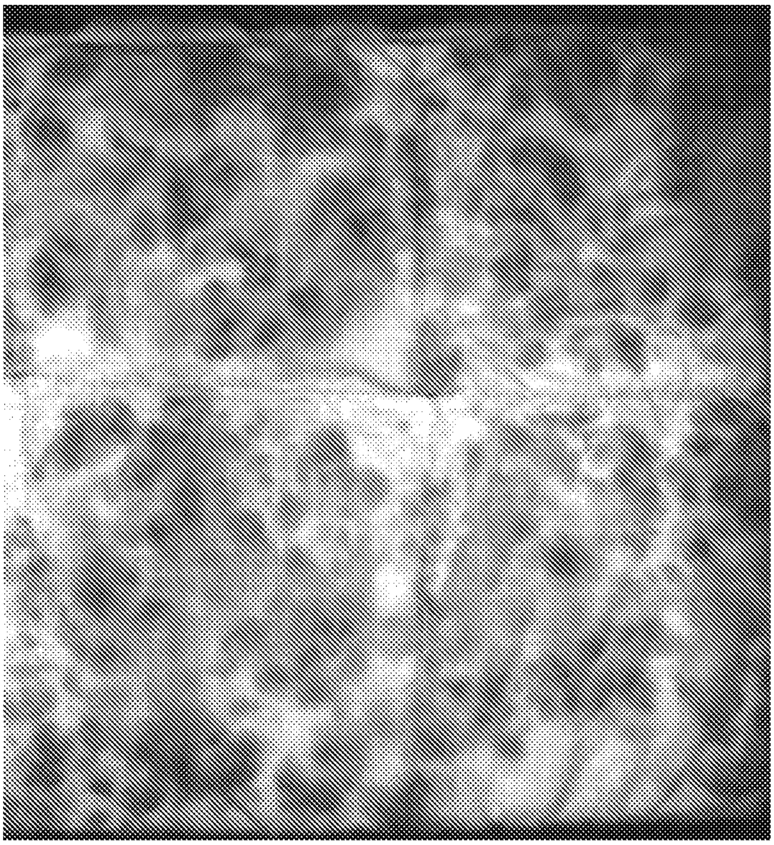

Fig. 6

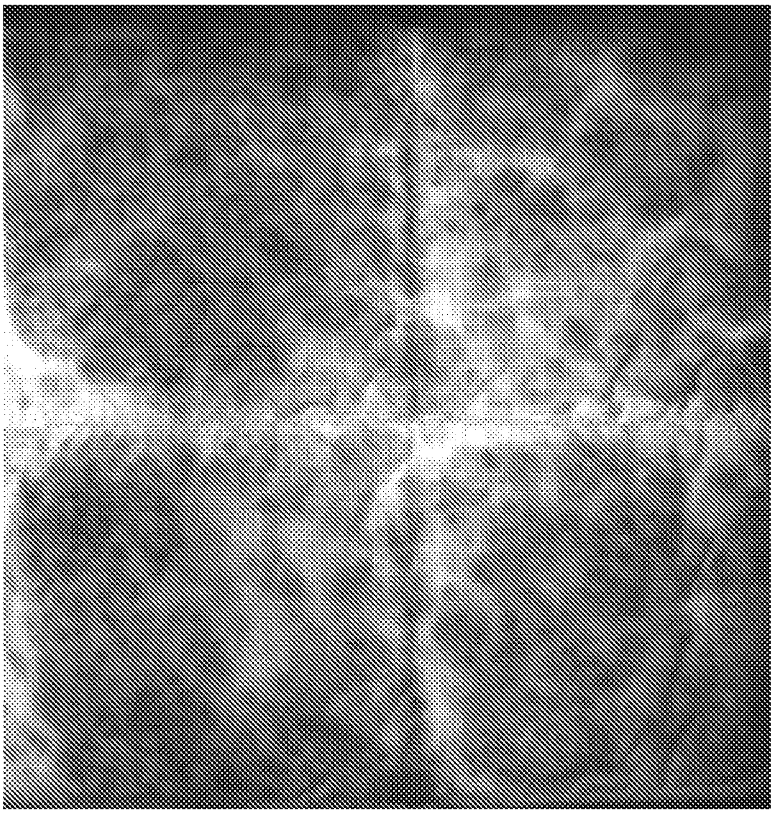

Fig. 7

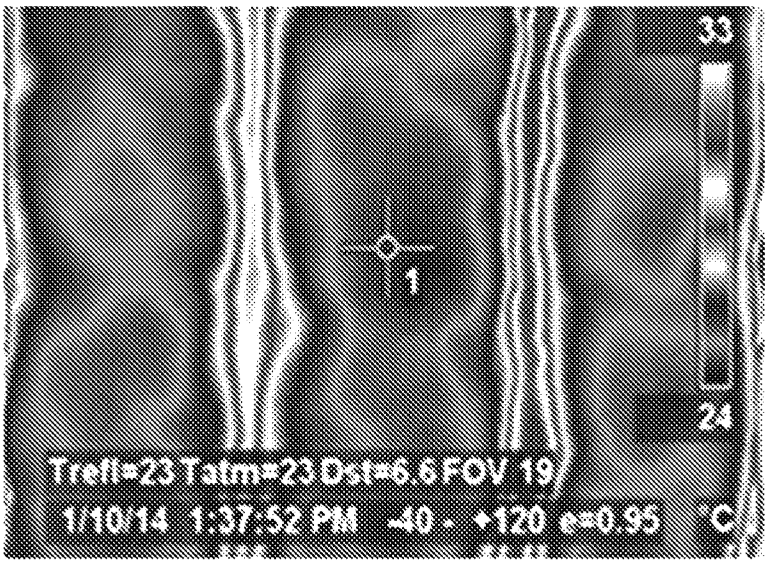
33
1
24
Trefl=23 Tatm=23 Dst=6.6 FOV 19
1/10/14 1:37:52 PM -40 - +120 e=0.95 °C

Fig. 8

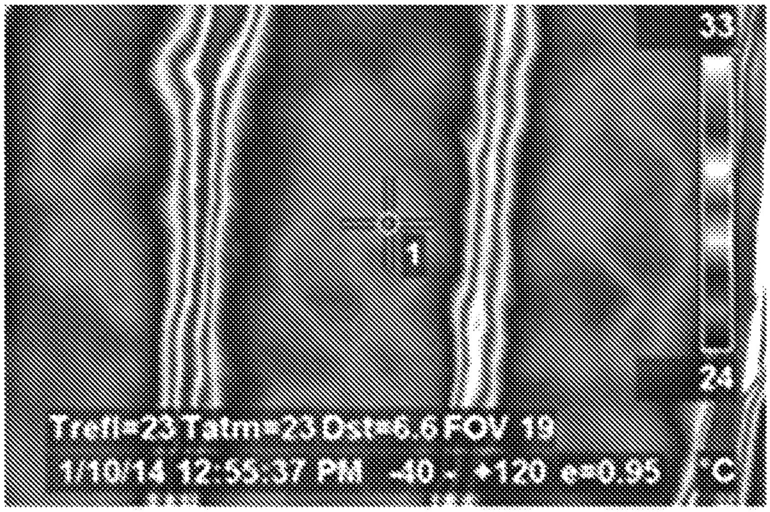
33
24
Trefl=23 Tatm=23 Dst=6.6 FOV 19
1/10/14 12:55:37 PM -40 - +120 e=0.95 °C

Fig. 9

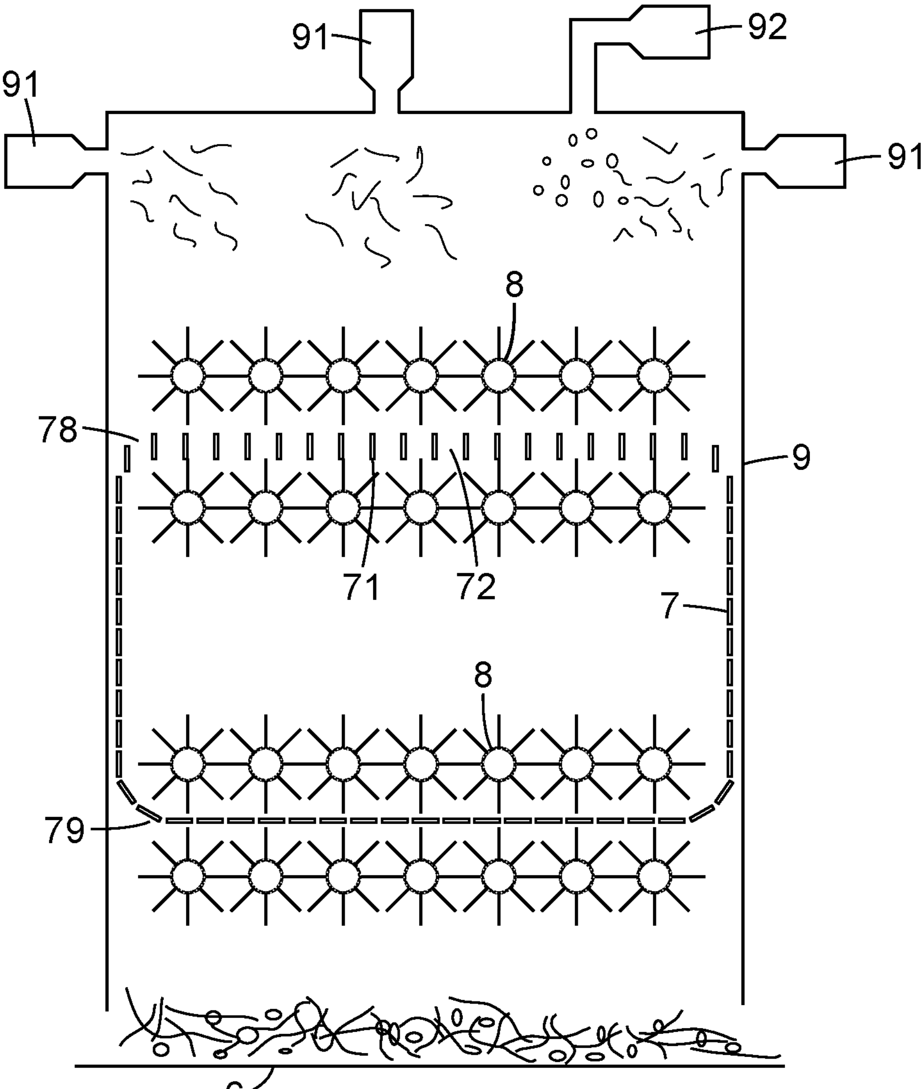
91
91
92
91
8
78
9
71
72
7
8
79
6

Fig. 10

INTERNATIONAL SEARCH REPORT

International application No
PCT/US2016/023684

A. CLASSIFICATION OF SUBJECT MATTER
INV. D04H1/435 D04H1/4391 D04H1/541 B68G1/00 A47G9/00
ADD.

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
D04H B68G A47G

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

EPO-Internal, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 5 683 811 A (HERNANDEZ ISMAEL ANTONIO [US] ET AL) 4 November 1997 (1997-11-04) column 2, line 66 - column 3, line 50; figures 1-2 column 4, line 42 - line 53 column 6, line 59 - line 65; claims 1-13 -----	1-13, 16-18
X	US 2014/206796 A1 (ALLAMPALAYAM JAYARAMAN KANNAN [US] ET AL) 24 July 2014 (2014-07-24) paragraphs [0010] - [0015]; claim all -----	1-13, 16-18
X	US 2003/162020 A1 (RUMIESZ JOSEPH [US] ET AL) 28 August 2003 (2003-08-28) paragraphs [0006] - [0010], [0019] - [0020]; figure 2 -----	1-18
	-/--	

[X] Further documents are listed in the continuation of Box C.

[X] See patent family annex.

* Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier application or patent but published on or after the international filing date

"L" 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

"P" 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 invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"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

Date of the actual completion of the international search	Date of mailing of the international search report
21 June 2016	01/07/2016

Name and mailing address of the ISA/	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Fax: (+31-70) 340-3016	Mangin, Sophie

1

INTERNATIONAL SEARCH REPORT

International application No
PCT/US2016/023684

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	WO 00/77287 A1 (ALBANY INT CORP [US]) 21 December 2000 (2000-12-21) page 3, line 35 - page 4, line 26	1-13, 16-18
X	EP 0 067 498 A1 (KURARAY CO [JP]) 22 December 1982 (1982-12-22) page 3, line 6 - page 4, line 1	1-13, 16-18
X	CN 101 481 851 A (SHANTOU CITY AOSHAN GARMENT LT [CN]) 15 July 2009 (2009-07-15) paragraphs [0011] - [0034]	1-13, 16-18
A	WO 2011/133396 A1 (3M INNOVATIVE PROPERTIES CO [US]; VINCENT BERNARD [FR]; LALOUCH LAHOUS) 27 October 2011 (2011-10-27) cited in the application figure 3	14,15

1

Form PCT/ISA/210 (continuation of second sheet) (April 2005)

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No
PCT/US2016/023684

Patent document cited in search report		Publication date	Patent family member(s)			Publication date
US 5683811	A	04-11-1997	CA	2198223	A1	11-04-1996
			CN	1159839	A	17-09-1997
			DE	69525952	D1	25-04-2002
			DE	69525952	T2	28-11-2002
			DK	0783607	T3	15-07-2002
			EP	0783607	A1	16-07-1997
			ES	2171560	T3	16-09-2002
			JP	3007160	B2	07-02-2000
			JP	H10508507	A	25-08-1998
			US	5458971	A	17-10-1995
			US	5683811	A	04-11-1997
			WO	9610665	A1	11-04-1996
US 2014206796	A1	24-07-2014	CA	2897434	A1	31-07-2014
			CN	104937154	A	23-09-2015
			EP	2948580	A1	02-12-2015
			JP	2016510365	A	07-04-2016
			KR	20150110505	A	02-10-2015
			TW	201446853	A	16-12-2014
			US	2014206796	A1	24-07-2014
			WO	2014116439	A1	31-07-2014
US 2003162020	A1	28-08-2003	AT	326565	T	15-06-2006
			AU	2003211013	A1	09-09-2003
			BR	0303221	A	06-07-2004
			CA	2444695	A1	04-09-2003
			CN	1507509	A	23-06-2004
			DE	60305296	T2	19-04-2007
			EP	1476596	A1	17-11-2004
			ES	2265576	T3	16-02-2007
			HK	1066574	A1	26-02-2010
			JP	2005518483	A	23-06-2005
			KR	20040086505	A	11-10-2004
			MX	PA04001745	A	31-05-2004
			NO	20034710	A	21-10-2003
			NZ	529065	A	24-03-2005
			TW	I229711	B	21-03-2005
			US	2003162020	A1	28-08-2003
			WO	03072865	A1	04-09-2003
			ZA	200307986	A	14-10-2004
WO 0077287	A1	21-12-2000	AT	453005	T	15-01-2010
			AU	760007	B2	08-05-2003
			BR	0011633	A	19-03-2002
			CA	2374799	A1	21-12-2000
			CN	1355865	A	26-06-2002
			EP	1190133	A1	27-03-2002
			ES	2337015	T3	20-04-2010
			HK	1045719	A1	16-04-2010
			JP	2003502516	A	21-01-2003
			NO	20016084	A	13-12-2001
			NZ	515828	A	26-11-2002
			PT	1190133	E	23-02-2010
			US	6329052	B1	11-12-2001
			WO	0077287	A1	21-12-2000
EP 0067498	A1	22-12-1982	DE	3261412	D1	17-01-1985
			EP	0067498	A1	22-12-1982

International application No
PCT/US2016/023684

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
		JP S6241034 B2	01-09-1987
		JP S57205564 A	16-12-1982
		KR 830008920 A	16-12-1983
		US 4418103 A	29-11-1983
CN 101481851 A	15-07-2009	NONE	
WO 2011133396 A1	27-10-2011	CN 102859058 A	02-01-2013
		EP 2561128 A1	27-02-2013
		JP 5866338 B2	17-02-2016
		JP 2013528712 A	11-07-2013
		KR 20130094197 A	23-08-2013
		US 2013030340 A1	31-01-2013
		WO 2011133396 A1	27-10-2011