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(54) **HEAT-RESISTANT FIBER STRUCTURE**

(57) A heat resistant fiber assembly contains heat resistant fibers having a glass transition temperature of 100°C or more, the heat resistant fibers being bonded together.

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Description

TECHNICAL FIELD

[0001] The present invention relates to a heat-resistant fiber assembly comprised of heat resistant fibers and used as a thermal insulator or an acoustic absorber, and a method for producing the same.

BACKGROUND ART

[0002] Fiber materials using heat resistant fibers have been used as a thermal insulator or an acoustic absorber in various fields such as vehicles, aircrafts, and building constructions.

[0003] The fiber materials used for such applications, such as fiber structure bodies, are required to be low in density in view of lightness in weight, and to have tenacity such as flexural stress and tensile strength, in particular, tenacity under a high temperature condition. The tenacity at high temperature has been longed for in the fields of, for example, acoustic thermal insulators to be built in a wall surface of an aircraft, filters to be built in an engine compartment of an automobile.

[0004] It has also been proposed an acoustic thermal insulator made of a matted cotton-like material prepared by blending cotton and a binder. More specifically, for example, Patent Document 1 discloses an acoustic thermal insulator prepared by uniformly blending high heat-resistant inorganic fibers with flame-resistant organic fibers having a melting temperature or decomposition temperature of 350°C or more to obtain a cotton-like material, mixing a heat resistant resin binder with the obtained cotton-like material, and matting the whole cotton-like material through a thermal treatment. Patent Document 1 describes that use of this acoustic thermal insulator can provide an acoustic thermal insulator with high safety due to high heat resistance and high acoustic absorption.

CITATION LIST

PATENT DOCUMENT

[0005] Patent Document 1: Japanese Patent No. 4951507

SUMMARY OF THE INVENTION

TECHNICAL PROBLEM

[0006] Patent Document 1 describes that the acoustic thermal insulator can provide a bendable acoustic thermal insulator having high heat resistance and high acoustic absorption properties. However, this acoustic thermal insulator is insufficient in tenacity because the fibers are bonded together with the binder. In particular, the tenacity disadvantageously decreases under a high temperature condition due to melting of the binder.

[0007] Further, in order to give high tenacity to a fiber assembly made of fibers bonded together with a binder, the amount of the binder needs to be increased. However, with the increase in the binder content, the content of the heat resistant fibers decreases, which lowers the heat resistance. Thus, it has been difficult to achieve both of high tenacity and high heat resistance.

[0008] In view of the forgoing background, it is therefore an object of the present invention to provide a heat-resistant fiber assembly which is high in heat resistance, and also in tenacity such as flexural stress and tensile strength.

SOLUTION TO THE PROBLEM

[0009] To achieve the object, the heat-resistant fiber assembly of the present invention is a fiber assembly containing heat resistant fibers having a glass transition temperature of 100°C or more, the heat resistant fibers being bonded together.

ADVANTAGES OF THE INVENTION

[0010] The present invention can provide a heat-resistant fiber assembly which is high in heat resistance, and also in tenacity such as flexural stress and tensile strength.

DETAILED DESCRIPTION

[0011] A heat-resistant fiber assembly of the present invention (will be hereinafter simply referred to as a "fiber assembly") is comprised of a plurality of heat resistant fibers bonded together. Unlike well-known existing fiber structure bodies, the fiber assembly of the present invention does not contain binder fibers having a low melting point, but the heat resistant fibers are directly bonded together to achieve high heat resistance and high tenacity.
[0012] In this context, the fibers being "bonded together" are those which are softened by heat and deformed to engage together by a force generated at intersections between them, or those which are molten and integrated together.

<Heat Resistant Fibers>

[0013] As the heat resistant fibers forming the fiber assembly, fibers having a glass transition temperature Tg of 100°C or more are used.
[0014] In general, the glass transition temperature (at which macromolecules start micro-molecular motions) is used as an index of heat resistance. A resin with a glass transition temperature of 100°C or more is called "engineering plastic," and is suitable for applications which require heat resistance. Fibers made of this resin are called "heat resistant fibers."
[0015] The heat resistant fibers are softened by high-temperature superheated vapor (150°C to 600°C), and capable of self bonding. Examples of such heat resistant fibers include polyamide fibers, meta-aramid fibers, para-aramid fibers, melamine fibers, polybenzoxazole fibers, polybenzimidazole fibers, polybenzothiazole fibers, polyallylate fibers, polyether sulfone fibers, liquid crystal polyester fibers, polyimide fibers, polyetherimide fibers, polyether ether ketone fibers, polyether ketone fibers, polyether ketone ketone fibers, and polyamide imide fibers. These fibers may be used alone, or two or more of them may be mixed together.
[0016] Among these fibers, the polyamide fibers are preferably used in terms of low water absorption properties and high chemical resistance, and the polyetherimide fibers are preferably used in terms of flame resistance and low fuming properties.
[0017] Examples of the polyamide fibers include fibers made of semi-aromatic polyamide, which is polyamide obtained from aliphatic diamine and dicarboxylic acid containing an aromatic component as a main ingredient. Aliphatic diamine is represented by the following chemical formula (1), where n is preferably 4 to 12, more preferably 6 or 9, particularly preferably 9.
[Chemical Formula 1]

$$NH_2\text{-}(CH_2)_n\text{-}NH_2 \qquad (1)$$

[0018] The "dicarboxylic acid containing an aromatic component as a main ingredient" contains at least 60 mol% or more of aromatic dicarboxylic acid. Preferred examples thereof include terephthalic acid, isophthalic acid, and naphthalenedicarboxylic acid. A preferred combination of aliphatic diamine and aromatic dicarboxylic acid may be a combination of aliphatic diamine (of the chemical formula (1) where n is 9) and terephthalic acid.
[0019] Examples of the polyetherimide fibers include amorphous polyetherimide fibers having no melting point. The polyetherimide fibers may have a glass transition temperature Tg of 200°C or more, and preferably remains resistant to heat at a high temperature of 200°C, for example, even if its fineness is low. The heat resistance can be determined in accordance with dry heat shrinkage at 200°C. The amorphous polyetherimide fibers used as the heat resistant fibers of the present invention may have a dry heat shrinkage of 5.0% or lower at 200°C, more specifically, a dry heat shrinkage is preferably -1.0% to 5.0% at 200°C.
[0020] The amorphous polyetherimide fibers have good flame resistance derived from a polymer thereof, and may have a limiting oxygen index (LOI) of, for example, 25 or more, preferably 28 or more, more preferably 30 or more.
[0021] In addition, the amorphous polyetherimide fibers may have a single fiber fineness of 15.0 dtex or less. The single fiber fineness is preferably 0.1 dtex to 12.0 dtex, more preferably 0.5 dtex to 10.0 dtex in terms of production costs and ease of handling.
[0022] Further, the amorphous polyetherimide fibers preferably have a fiber strength of 2.0 cN/dtex or more at room temperature. The fiber strength of less than 2.0 cN/dtex is not preferable because the passage of the fibers may become less smooth during a process of making the fibers into paper, or fabric such as nonwoven fabric and woven fabric, and the applications of the fibers are limited. The fiber strength is more preferably 2.3 cN/dtex to 4.0 cN/dtex, much more preferably 2.5 cN/dtex to 4.0 cN/dtex.
[0023] The transverse cross-section (a cross section perpendicular to the fiber length direction) of the heat resistant fibers is not limited to have a general shape, such as round and various other shapes (flat shape, oval shape, polygonal shape, 3- to 14-foliate shape, T-shape, H-shape, V-shape, dog bone shape (I-shape)), and may have a hollow shape.
[0024] A mean fineness of the heat resistant fibers can be selected from a range of, for example, 0.01 dtex to 100 dtex, depending on the applications, and is preferably 0.1 dtex to 50 dtex, more preferably 0.5 dtex to 30 dtex (in particular

1 dtex to 10 dtex). The mean fineness in this range can keep the fiber strength and the adhesion in balance.

[0025] A mean fiber length of the heat resistant fibers can be selected from a range of, for example, 10 mm to 100 mm, preferably 20 mm to 80 mm, more preferably 25 mm to 75 mm (in particular 35 mm to 55 mm). The mean fiber length in this range allows the fibers to entangle sufficiently, which improves the mechanical strength of the fiber assembly.

[0026] The resistant fibers have a percentage of crimp of, for example, 1% to 50%, preferably 3% to 40%, more preferably 5% to 30% (in particular 10% to 20%). The number of crimp is, for example, 1/inch to 100/inch, preferably 5/inch to 50/inch, more preferably 10/inch to 30/inch.

<Fiber Assembly>

[0027] The fiber assembly of the present invention has a structure containing the above-described heat resistant fibers which are bonded together, and is generally in the shape of a sheet or a plate, although the shape can be selected depending on the application.

[0028] In order to provide the fiber assembly of the present invention with a nonwoven fiber structure having high surface hardness and high flexural rigidity, and keeping lightness in weight and air permeability in balance, fibers forming a nonwoven fiber web need to be arranged and bonded together in a suitably controlled state. Specifically, the fibers forming the fiber web are desired to be arranged substantially parallel to a surface of the fiber web (nonwoven fiber structure) and intersect with each other.

[0029] In the fiber assembly of the present invention, the fibers are preferably bonded together at intersections between them. In particular, in a fiber assembly (compact) which is required to have high hardness and high strength, the fibers, except for their intersecting portions, are arranged substantially parallel to each other, and several or several tens of them may be bonded into bundles to form bonded fiber bundles. These fibers form a structure, in some portions of which the single fibers are bonded together at their intersections, the fiber bundles are bonded together at their intersections, or the single fiber and the fiber bundle are bonded together at their intersections. That is, a "scrummed" structure is formed (in which the fibers being bonded together at their intersections are entangled like a mesh, or the fibers are bonded together at their intersections and confine adjacent fibers). This structure can express intended bending behaviors and surface hardness. In the present invention, it is desired that such bonding portions are substantially uniformly distributed in the structure in a direction parallel to the surface and a thickness direction of the fiber web.

[0030] Note that the expression that the fibers are "arranged substantially parallel to the surface of the fiber web" means that fibers continuously arranged in the thickness direction of the web are not locally present in the web. More specifically, when a certain cross section of the fiber web of the fiber assembly is microscopically observed, a ratio of fibers (number of fibers) continuously extending in the thickness direction of the web over a region of 30% or more of the fiber web thickness to the whole fibers in the cross section is 10% or less (in particular, 5% or less).

[0031] If multiple fibers are arranged in the thickness direction (a direction perpendicular to the fiber web surface), the fibers around such fibers become loosely arranged to generate voids larger than necessary in the nonwoven fiber structure, thereby lowering the flexural strength and surface hardness of the fiber assembly. This is why the fibers are arranged parallel to the fiber web surface. Therefore, it is preferred to reduce the voids as much as possible, and for this purpose, the fibers are desired to be arranged parallel to the fiber web surface as much as possible.

[0032] In particular, if a load is applied in the thickness direction to the fiber assembly of the present invention formed as a sheet- or plate-like compact, large voids, if presented in the fiber assembly, are crushed under the load, and the surface of the compact is easily deformed. In addition, the load easily reduces the whole thickness of the compact when applied to the entire surface of the compact. Such a problem can be solved if the fiber assembly itself is formed as a filled resin having no voids. However, this results in low air permeability, and makes it difficult to ensure resistance against bending (folding strength) and lightness in weight.

[0033] In order to make the fiber assembly less deformed in the thickness direction even under a load, a possible solution is to arrange thin fibers more densely. However, use of the thin fibers only to ensure the lightness in weight and the air permeability leads to low rigidity of each fiber, and eventually lowers the flexural stress. To ensure the flexural stress, the fibers need to have a somewhat large diameter. However, simply blending thick fibers may easily generate large voids around the intersections between the thick fibers, and the resulting fiber assembly may be easily deformed in the thickness direction.

[0034] In view of the foregoing, the fibers in the fiber assembly of the present invention are arranged such that their longitudinal direction is parallel to the direction of the web surface, and dispersed (or oriented at random) so that the fibers intersect with each other, and are bonded together at the intersections. Thus, small voids are generated, thereby ensuring the fiber assembly lightweight. Further, such fiber arrangement is continuous, thereby ensuring suitable air permeability and surface hardness. In particular, if some of the fibers arranged substantially in parallel without intersecting with each other are parallelly bonded in the fiber length direction to form a fiber bundle, the obtained fiber assembly can ensure higher flexural strength than that made of single fibers only. If a fiber assembly with high hardness and high strength is required, it is preferred that the fibers be bonded one by one at their intersections, while the fibers between the intersections are arranged in a bundle to form several fiber bundles. Such fiber arrangement can be seen from the state of single fibers observed on a cross section of the compact.

[0035] In the fiber assembly of the present invention, the heat resistant fibers are preferably bonded to have a fiber bonding ratio of 10% to 85%, more preferably 25% to 75%, much more preferably 40% to 65%.

[0036] The above ratio is preferred because the fiber bonding ratio less than 10% may disadvantageously lower the hardness, flexural stress, and tensile strength of the fiber assembly. Further, the fiber bonding ratio more than 85% shrinks the voids between the fibers and increases an apparent density too much, thereby disadvantageously impairing the lightness in weight. Specifically, setting the fiber bonding ratio within the range of 10% to 85% can further improve the tenacity, such as the flexural stress and tensile strength, of the fiber assembly, without impairing the lightness in weight.

[0037] The fiber bonding ratio can be measured by the method described later in the following examples, and indicates a ratio of the count of the sections of two or more fibers bonded together to the count of the sections of all fibers in a cross section of a nonwoven fiber structure. Thus, if the fiber bonding ratio is low, the ratio of the bonding of two or more fibers (the ratio of fibers bundled and bonded) is low.

[0038] The heat resistant fibers forming the nonwoven fiber structure are bonded together at their points of contact. In order to express high flexural stress while reducing the bonding points as much as possible, the bonding points are preferably distributed uniformly in the thickness direction from the front surface to inside (middle region), and to rear surface, of the fiber assembly. If the bonding points are concentrated on the surface or inside of the fiber assembly, sufficient flexural stress cannot be ensured easily, and a portion of the fiber assembly with less bonding points decreases in the shape stability. Therefore, in terms of further improvement in flexural stress without lowering the shape stability, suppose that a cross section of the fiber assembly cut along the thickness direction is divided into three equal regions arranged in the thickness direction, the fiber bonding ratio of the middle region (central region) is preferably within the above range (10% to 85%).

[0039] Further, the uniformity of the fiber bonding ratio of each region (i.e., a difference between the maximum and minimum values of the fiber bonding ratio) is preferably 20% or less (e.g., 0.1% to 20%), more preferably 15% or less (e.g., 0.5% to 15%), much more preferably 10% or less (e.g., 1% to 10%).

[0040] The fiber assembly of the present invention has the fiber bonding ratio which is uniform in the thickness direction, and therefore, exhibits hardness, flexural strength, folding resistance, and toughness which are all good.

[0041] In the context of the present invention, the "three equal regions arranged in the thickness direction" are obtained by slicing the plate-like fiber assembly in a direction perpendicular to the thickness direction thereof into three equal regions.

[0042] Thus, according to the fiber assembly of the present invention, the bonding points of the heat resistant fibers are uniformly dispersed, and in addition, the bonding points are present at a small distance from each other (e.g., several ten to several hundred μm) to form a dense network structure. Being configured in this manner, the fiber assembly of the present invention can follow strain, if caused by an external force acted thereon, due to the flexibility of the fiber structure, and the external force is reduced as it is dispersed to the bonding points of the fibers that are finely dispersed. Thus, it is estimated that the fiber assembly has high flexural stress and high tensile strength. In contrast, a common fiber assembly in which the heat resistant fibers are bonded together with the binder fibers has a small number of bonding points because the amount of the binder fibers is limited to ensure the heat resistance. Even if the amount of the binder fibers is increased to increase the bonding points, the heat resistance cannot be sufficient, and the bonding points cannot be easily dispersed uniformly in the thickness direction of the fiber assembly. Therefore, it is estimated that such a common fiber assembly is easily strained, and its flexural stress and tensile strength are lowered.

[0043] In the fiber assembly of the present invention, the frequency of presence of single fibers (single fibers' end faces) in the cross section cut along the thickness direction is not particularly limited. For example, in an area of 1 mm^2 of the cross section, the frequency of presence of the single fibers may be 100 fibers/mm^2 or more (e.g., 100 fibers/mm^2 to 300 fibers/mm^2). In particular, if mechanical properties are required in preference to the lightness in weight, the frequency of presence of the single fibers may be, for example, 100 fibers/mm^2 or less, preferably 60 fibers/mm^2 or less (e.g., 1 fibers/mm^2 to 60 fibers/mm^2), more preferably 25 fibers/mm^2 or less (e.g., 3 fibers/mm^2 to 25 fibers/mm^2). If the frequency of presence of the single fibers is too high, the bonding of the fibers is insufficient, and thus, the strength of the compact made of the fiber assembly is lowered. If the frequency of presence of the single fibers exceeds 100 fibers/mm^2, the bonding of the fibers into bundles is insufficient, and thus, the high flexural strength cannot be ensured easily. Further, in the case of a plate-like compact, it is preferred that the fibers bonded into bundles be thin in the thickness direction of the compact, and wide in the surface direction (length or width direction) of the compact.

[0044] In the present invention, the frequency of presence of the single fibers is measured in the following manner. Specifically, a region corresponding to 1 mm^2 selected from a scanning electron microscopic (SEM) image of the cross section of the compact is observed to count the number of the sections of the single fibers. Several regions are optionally selected from the image (e.g., ten regions are selected at random), and observed in the same manner. Then, a mean value of the single fibers' sections per unit area is determined to be the frequency of presence of the single fibers. In this measurement, the fibers each being a single fiber on the cross section are all counted. Specifically, in addition to the sections of the fibers which are definitely single fibers, the sections of the fibers bonded together are also counted if they are separated from a bonded portion and appear as the single fibers on the cross section.

[0045] The heat resistant fibers in the fiber assembly do not extend across the fiber assembly in the thickness direction (do not penetrate the fiber assembly in the thickness direction). This can avoid a failure of the fiber assembly due to falling of the fibers. A production method for arranging the heat resistant fibers in this manner is not particularly limited, but it is simple and reliable to stack a plurality of fiber compacts prepared by entangling the heat resistant fibers, and bond the stacked compacts together using superheated vapor. Controlling the relationship between the fiber length and the thickness of the fiber assembly can significantly reduce the number of fibers extending across the fiber assembly in the thickness direction. From this viewpoint, the thickness of the fiber assembly with respect to the fiber length is 10% or more (e.g., 10% to 1000%), preferably 40% or more (e.g., 40% to 800%), more preferably 60% or more (e.g., 60% to 700%), much more preferably 100% or more (e.g., 100% to 600%). The thickness of the fiber assembly and the fiber length within the range can avoid the failure of the fiber assembly due to the falling of the fibers without lowering the mechanical strength, such as flexural stress, of the fiber assembly.

[0046] As can be seen, the density and mechanical properties of the fiber assembly are influenced by the ratio and state of the bonded fiber bundles. The fiber bonding ratio, indicating the degree of bonding, can be easily measured based on the count of the sections of the bonded fibers in a certain region of an image of the cross section of the fiber assembly taken in an enlarged scale using an SEM. However, in the bonded fiber bundles, the fibers are bonded in bundles or at their intersections. Therefore, if the density is particularly high, the fibers cannot be easily observed as the single fiber alone.

[0047] According to the present invention, as an index of the degree of the bonding of the fibers, the ratio of an area of the sections of the fibers and fiber bundles on a cross section of the fiber assembly (a cross section cut along a thickness direction thereof), i.e., a fiber filling ratio, may be used. The fiber filling ratio of the cross section in the thickness direction is, for example, 20% to 80%, preferably 20 to 60%, more preferably 30 to 50%. If the fiber filling ratio is too low, too many voids are formed in the fiber assembly, which makes it difficult to ensure the desired surface hardness and flexural stress. If the fiber filling ratio is too high, sufficient surface hardness and flexural stress can be ensured, but the fiber assembly becomes too heavy, which tends to lower the air permeability.

[0048] It is preferred that the fiber assembly of the present invention (in particular, a fiber assembly in which the fibers are bonded into bundles, and the frequency of presence of the single fibers is 100 fibers/mm^2 or less) have surface hardness enough to avoid the fiber assembly from being deformed or recessed under a load applied thereto, even if it is in the shape of a plate (board). As an index of the hardness, the hardness is, for example, A50 or more, preferably A60 or more, more preferably A70 or more when measured by determination of hardness with a type A durometer (in accordance with JIS K6253 "Rubber, vulcanized or thermoplastic - Determination of hardness"). If the hardness is too low, the fiber assembly is easily deformed by a load applied to its surface.

[0049] It is preferred that, in the fiber assembly containing the fibers bonded into bundles, the frequency of presence of the bonded fiber bundles be low, and the fibers (fiber bundles and/or single fibers) be bonded more frequently at their intersections for the purpose of keeping the flexural strength, the surface hardness, the lightness in weight, and the air permeability in balance at a higher level. If the fiber bonding ratio is too high, however, the bonding points get too close to each other, which lowers the flexibility, and makes it difficult to reduce the strain caused by an external force. Therefore, as described above, the fiber assembly of the present invention preferably has a fiber bonding ratio of 85% or less. The fiber bonding ratio being not too high can ensure passages formed by fine voids in the fiber assembly, thereby making the fiber assembly more lightweight and air-permeable. Therefore, in order to express high flexural stress, high surface hardness, and high air permeability while reducing the bonding points as much as possible, the fiber bonding ratio is preferably uniform in the thickness direction of the fiber assembly from the front surface to inside (middle region), and to rear surface, of the fiber assembly.

[0050] If the bonding points are concentrated on the surface or inside of the fiber assembly, it is difficult to ensure, not only the flexural stress and shape stability described above, but also the air permeability. Under these circumstances, if a cross section of the fiber assembly of the present invention cut along a thickness direction thereof is divided into three equal regions arranged in the thickness direction, a middle region of the three regions preferably has the fiber bonding ratio in the above range. More preferably, all the three regions including a front surface region, the middle region, and a rear surface region, have the fiber bonding ratio in the above range. Further, a difference between the maximum and minimum values of the fiber bonding ratio in each region is preferably 20% or less (e.g., 0.1% to 20%), more preferably 15% or less (e.g., 0.5% to 15%), much more preferably 10% or less (e.g., 1% to 10%). The fiber assembly of the present invention has the fiber bonding ratio which is uniform in the thickness direction, and therefore, exhibits flexural stress, tensile strength, folding resistance, and toughness which are all good. The fiber bonding ratio of the present invention is measured by the method described later in the following examples.

[0051] One of the features of the fiber assembly of the present invention is that the fiber assembly shows a bending behavior that cannot be achieved by a common fiber assembly in which the heat resistant fibers are bonded together with binder fibers. To represent the bending behavior, a flexural stress is calculated from a repulsive force and bending amount of a test piece generated when the test piece is gradually bent in accordance with JIS K7171 "Plastics - Determination of Flexural Properties," and used as an index of the bending behavior. Specifically, a higher flexural stress indicates that the fiber assembly is harder. Further, a larger bending amount (displacement) before a measurement target breaks indicates that the compact is more flexible.

[0052] The fiber assembly of the present invention has a flexural stress in at least one direction (preferably in every direction) of 0.05 MPa or more (e.g., 0.05 MPa to 100 MPa), preferably 0.1 MPa to 30 MPa, more preferably 0.2 MPa to 10 MPa. If the flexural stress is too small, the fiber assembly, when used as a board material, would easily break by its own weight or under a small load. If the flexural stress is too high, the fiber assembly would be too hard, and easily break when a force bending the fiber assembly exceeds the peak of the stress. To obtain a hardness higher than 100 MPa, the fiber assembly needs to have a high density, which makes it difficult to ensure the lightness in weight.

[0053] The fiber assembly of the present invention has voids between the fibers, thereby ensuring the lightness in weight. The voids, unlike those in a resin foam such as sponge, are not independent from each other, but are continuous, and thus, allow the air to pass through. This structure is so hard to produce by a common hardening technique, such as impregnation with a resin, or a method of forming a film-like structure by making the fibers near the surface into close contact with each other.

[0054] Specifically, the fiber assembly of the present invention has a low density, and in particular, has an apparent density of, for example, 0.03 g/cm^3 to 0.7 g/cm^3. If the lightness in weight is of importance, the apparent density is, for example, 0.05 g/cm^3 to 0.5 g/cm^3, preferably 0.08 g/cm^3 to 0.4 g/cm^3, more preferably 0.1 g/cm^3 to 0.35 g/cm^3. If the hardness is more necessary than the lightness in weight, the apparent density is, for example, 0.2 g/cm^3 to 0.7 g/cm^3, preferably 0.25 g/cm^3 to 0.65 g/cm^3, more preferably 0.3 g/cm^3 to 0.6 g/cm^3. If the apparent density is too low, the fiber assembly becomes lightweight, but cannot ensure sufficient bending hardness and surface hardness. If the apparent density is too high, the hardness can be ensured, but the weight increases. If the apparent density decreases, the resulting fiber structure becomes similar to a common nonwoven fiber structure in which the fibers are entangled and bonded at their intersections. If the apparent density increases, the fibers are bonded into bundles, forming a structure similar to that of a porous compact.

[0055] The "apparent density" mentioned herein is a density calculated based on the mass per unit area and thickness measured in accordance with JIS L1913 (Test Methods For Nonwovens).

[0056] The mass per unit area of the fiber assembly of the present invention can be selected from a range of, for example, about 50 g/m^2 to 10000 g/m^2, preferably 150 g/m^2 to 8000 g/m^2, more preferably about 300 g/m^2 to 6000 g/m^2. If the hardness is more necessary than the lightness in weight, the mass per unit area is, for example, 1000 g/m^2 to 10000 g/m^2, preferably 1500 g/m^2 to 8000 g/m^2, more preferably about 2000 g/m^2 to 6000 g/m^2. If the mass per unit area is too small, the hardness cannot be ensured easily. If the mass per unit area is too large, the web becomes too thick, and the superheated vapor cannot enter sufficiently inside the web in the vapor treatment, which makes it difficult to provide the fiber assembly with uniformity in the thickness direction.

[0057] If the fiber assembly of the present invention is in the shape of a plate or a sheet, the thickness thereof, which is not particularly limited, can be selected from a range of about 1 mm to 100 mm. For example, the range is, for example, 2 to 50 mm, preferably 3 to 20 mm, more preferably 5 to 150 mm. Reducing the thickness too much makes it difficult to ensure the hardness. Increasing the thickness too much increases the mass, thereby making the sheet difficult to handle.

[0058] The fiber assembly of the present invention has a nonwoven fiber structure, and therefore, has high air permeability. The air permeability of the fiber assembly of the present invention is 0.1 cm^3/cm^2/sec or more (e.g., 0.1 to 300 cm^3/cm^2/sec), preferably 0.5 to 250 cm^3/cm^2/sec (e.g., 1 to 250 cm^3/cm^2/sec), more preferably 5 to 200 cm^3/cm^2/sec. The air permeability is generally 1 to 100 cm^3/cm^2/sec. If the air permeability is too low, an external force needs to be applied to allow the air to pass through the fiber assembly, i.e., the air cannot flow in and out naturally. If the air permeability is too high, the air can easily pass through the fiber assembly, but the voids between the fibers in the fiber assembly becomes too large, resulting in low flexural stress.

[0059] Having the nonwoven fiber structure, the fiber assembly of the present invention exhibits high thermal insulation, and has a thermal conductivity as low as 0.1 W/m·K or less. For example, the thermal conductivity is 0.03 W/m·K to 0.1 W/m·K, preferably 0.05 W/m·K to 0.08 W/m·K.

[0060] A method for producing the fiber assembly of the present invention will be described below.

[0061] According to the method for producing the fiber assembly of the present invention, first, the heat resistant fibers are formed into a web. The web can be formed by a generally known method, for example, a direct method such as spunbonding or meltblowing, and a dry method such as carding or airlaying using meltblown fibers or staple fibers. Among these methods, the carding using the meltblown fibers or the staple fibers, in particular, the carding using the staple fibers, is generally used. Examples of the web formed by using the staple fibers include a random web, a semi-random web, a parallel web, and a crosslapped web. Among these webs, the semi-random web and the parallel web are preferable if the ratio of the bonded fiber bundles needs to be high.

[0062] In a step of bonding the fibers in the obtained fiber web, the fibers may be heated through a general process such as heating with hot air, hot pressing, or a treatment with superheated vapor. If the superheated vapor is used, the fiber web obtained in the above step is sent to a next step by a belt conveyor, and exposed to a superheated vapor (high pressure steam) flow. Thus, the fiber assembly of the present invention is obtained. Specifically, the fiber web conveyed by the belt conveyor passes through the superheated vapor flow injected from a nozzle of a vapor injection system, and the heat resistant fibers are three-dimensionally bonded (thermally bonded) by the injected superheated vapor.

[0063] Through the heating with the superheated vapor (150°C to 600°C), the heat resistant fibers are bonded together to form a fiber network. Thus, the fiber assembly can be provided with a uniform and bulky structure throughout the thickness direction.

[0064] In a preferred embodiment, the superheated vapor injected to the heat resistant fibers has a temperature ranging from 150°C to 600°C. If the temperature is lower than 150°C, sufficient energy cannot be given to the heat resistant fibers, and the fibers may be bonded insufficiently. Further, if the temperature is higher than 600°C, excessive energy is given to the fibers adjacent to the vapor injection system, and the fiber bonding ratio may become less uniform.

[0065] The belt conveyor used is not particularly limited as long as the fiber web to be processed can be compressed to an intended density and treated with the superheated vapor. An endless conveyer is suitably used. A common belt conveyor alone may also be used. Alternatively, two belt conveyors may be used in combination as needed so that the conveyors can transport the web being sandwiched therebetween. Transporting the web in this manner can avoid possible disadvantages, such as the deformation of the web caused by the superheated vapor used for the treatment or an external force generated by the vibration of the conveyor(s). If the clearance between the conveyors is adjusted, the density and thickness of the nonwoven fiber structure after the treatment can be controlled.

[0066] If two belt conveyors are used in combination, the vapor injection system for supplying the superheated vapor to the web is mounted in one of the belt conveyors, and supplies the web with the superheated vapor through a conveyor net. The other belt conveyor may be provided with a suction box. The suction box can suck and discharge an excess of the superheated vapor that passed through the web. Alternatively, in order to simultaneously treat the front and rear surfaces of the web with the superheated vapor, another suction box may be provided for a downstream portion of the one of the belt conveyors provided with the vapor injection system, and another vapor injection system may be provided in the other belt conveyor to be opposite to the suction box. If the vapor injection system and the suction box are not provided for the downstream portion, the fiber web that has been treated once is turned upside down, and allowed to pass through the treatment device again so that both of the surfaces of the fiber web can be treated with the superheated vapor.

[0067] The endless belt used for the conveyor is not particularly limited as long as it does not interfere with the transport of the web and the treatment with the superheated vapor. Note that if the fiber web is treated with the superheated vapor, the shape of the surface of the belt may be transferred to the surface of the fiber web, depending on the treatment conditions. Thus, a belt is suitably selected depending on the applications. In particular, for the production of a fiber assembly with flat surfaces, a net with fine meshes is used. The net is of 90 mesh at maximum. A net with a higher mesh density lowers the air permeability, and interferes with the passage of the vapor. In view of resistance to heat applied during the treatment with the superheated vapor, the mesh belt is preferably made of, for example, metal, or a heat resistant resin treated to be heat resistant such as a polyester-based resin, a polyphenylene sulfide-based resin, a polyallylate-based resin (fully aromatic polyester-based resin), and an aromatic polyamide-based resin.

[0068] The superheated vapor injected from the vapor injection system is an air flow. Thus, unlike hydroentangling and needlepunching, the superheated vapor goes into the web without causing significant movement of the fibers in the web which is a treatment target. The vapor flow goes into, and superheats, the web. Thus, it is presumed that the superheated vapor flow efficiently covers the surfaces of the heat resistant fibers in the web in a superheated state, thereby allowing the fibers to be thermally and uniformly bonded. Since this treatment is performed in a very short time under the high speed flow, heat can be sufficiently transferred from the superheated vapor to the surfaces of the fibers, and the treatment ends before the heat is sufficiently transferred to the inside of the fibers. This can reduce the possibility of disadvantages, e.g., the pressure or heat of the superheated vapor crushes the whole fiber web to be treated, or the fiber web decreases in the thickness due to deformation. As a result, the thermal bonding of the fibers is finished such that the degree of bonding in the surface direction and that in the thickness direction are approximately uniform, without causing significant deformation of the fiber web.

[0069] In order to obtain a fiber assembly with high surface hardness and high flexural strength, what is important is to expose the web to be treated to the superheated vapor with the web being compressed between the belt conveyors or rollers at an intended apparent density (e.g., 0.03 g/cm^3 to 0.7 g/cm^3). In particular, to obtain a fiber assembly with a relatively high density, the fiber web needs to be compressed under a sufficient pressure during the treatment with the superheated vapor. In addition, keeping a suitable clearance between the rollers or the conveyors can control the thickness and the density to the intended ones. Since it is difficult to compress the web in one go with the conveyors, it is preferred to set the tension of the belts as high as possible, and gradually narrow the clearance from the upstream of the point of the vapor treatment. Then, the pressure of the vapor and the speed of the treatment are controlled to process the fiber web into a fiber assembly with desired bending hardness, surface hardness, lightness in weight, and air permeability.

[0070] If higher hardness is required, a rear surface of the endless belt located across the web from the nozzle is made of a stainless steel, for example, so that the vapor does not pass through the endless belt. Thus, the vapor that has passed through the web to be treated is reflected from the stainless steel surface, thereby maintaining heat around the web and bonding the fibers more strongly. Conversely, if moderate bonding of the fibers is required, a suction box may be arranged to discharge an excess of the vapor outside.

[0071] As the nozzle for injecting the superheated vapor, a plate or die provided with predetermined orifices continuously arranged side by side in a width direction may be used. This nozzle may be arranged with the orifices thereof arranged side by side in the width direction of the web. The orifices may be arranged in a single line, or two or more lines parallel to each other. Alternatively, a plurality of nozzle dies each having a single line of orifices may be arranged parallel to each other.

[0072] If a plate-shaped nozzle provided with the orifices is used, the nozzle may have a thickness of 0.5 mm to 1 mm. The orifices may have any diameter and may be arranged at any pitch as long as the intended fiber bonding can be achieved. The diameter of the orifices may is 0.05 mm to 2 mm in general, preferably 0.1 mm to 1 mm, more preferably 0.2 mm to 0.5 mm. The pitch of the orifices is 0.5 mm to 3 mm in general, preferably 1 mm to 2.5 mm, more preferably 1 mm to 1.5 mm. The orifices with a too small diameter may lead to facility problems, e.g., the machining accuracy of the nozzle is lowered to make the machining difficult, and also to operational problems, such as frequent clogging. Conversely, the orifices with a too-large diameter lower the force of vapor injection. If the pitch is too small, the orifices are arranged too densely, which lowers the strength of the nozzle itself. If the pitch is too large, the superheated vapor does not sufficiently collide with the web, which lowers the strength of the web.

[0073] The superheated vapor is not particularly limited as long as it can bond the heat resistant fibers. The pressure of the superheated vapor, which can be set depending on the material and shape of the fibers used, is, for example, 0.1 MPa to 2 MPa, preferably 0.2 MPa to 1.5 MPa, more preferably 0.3 MPa to 1 MPa. If the pressure is too high or excessive, the fibers forming the web are moved and the web becomes less uniform, or the fibers are molten too much and some of the fibers cannot maintain their shape. If the pressure is too low, the amount of heat required for the bonding of the fibers cannot be given to the web, or the superheated vapor cannot pass through the web, and the bonding of the fibers may become uneven in the thickness direction. This makes it difficult to control the uniform vapor injection from the nozzle in some cases.

[0074] The fiber assembly with the nonwoven fiber structure thus obtained has significantly high flexural stress and surface hardness while keeping the density as low as that of common nonwoven fabric, and also has air permeability, acoustic absorption properties, heat insulation properties, and heat resistance. With use of such properties, the fiber assembly can be applied to products which require heat resistance, e.g., interior materials of automobiles, inner walls of aircrafts, and building material boards.

[Examples]

[0075] The present invention will be described below by way of examples. Note that the following examples do not limit the present invention, and may be modified or changed based on the intent of the present invention. Such a change and modification shall not be excluded from the scope of the invention.

[0076] In the examples, the physical property values are measured in the following manner. In the examples, "parts" means parts by mass, and "%" means mass%.

(Example 1)

<Production of Fiber Assembly>

[0077] As the heat resistant fibers, heat resistant fibers (fineness: 1.7 dtex, fiber length: 51 mm) were prepared using a semi-aromatic polyamide resin made of diamine whose carbon number is 9 and terephthalic acid (manufactured by Kuraray Co., Ltd. under the trade name "GENESTAR," melting point: 265°C, glass transition temperature: 125°C, thermal decomposition temperature: 400°C). Then, using the heat resistant fibers, 12 carded webs each having a mass per unit area of 50 g/m^2 were produced by carding, and stacked to obtain a single carded web having a mass per unit area of 600 g/m^2 in total. The carded web was transported onto a belt conveyor provided with a stainless steel endless net of 50 mesh and a width of 500 mm.

[0078] Note that the belt conveyor includes a pair of conveyors including a lower conveyor and an upper conveyor. A vapor injection nozzle is installed on a rear side of a belt of at least one of the conveyors. Superheated vapor can be injected through the belt to the web being transported. A metallic roll for controlling the thickness of the web (may be hereinafter abbreviated as a "thickness control roll") is provided for each conveyor to be located upstream of the nozzle. The lower conveyor has a flat upper surface (a surface on which the web passes), and the upper conveyor has a lower surface curved along the thickness control roll. The thickness control roll of the upper conveyor is arranged to be paired with the thickness control roll of the lower conveyor.

[0079] The upper conveyor can move in the vertical direction, thereby controlling a clearance between the thickness control rolls of the upper and lower conveyors to have a predetermined size. An upstream portion of the upper conveyor is inclined to form an angle of 30 degrees with a downstream portion thereof (a lower surface of the downstream portion of the upper conveyor) about the thickness control roll, and the downstream portion is bent to be parallel to the lower conveyor. The upper conveyor moves up and down while keeping the parallel relationship with the lower conveyor.

[0080] These belt conveyors rotate in the same direction at the same speed, and can press the web with a predetermined clearance kept between the conveyor belts, and between the thickness control rolls. The conveyors configured in this manner operate like a calender to control the thickness of the web before the vapor treatment. Specifically, while the carded web sent from the upstream is running on the lower conveyor, the clearance between the upper and lower conveyors is gradually narrowed until the web reaches the thickness control rolls. Then, when the clearance becomes smaller than the thickness of the web, the web is sandwiched between the upper and lower conveyors, and gradually compressed as it runs toward the downstream. The web is compressed to a thickness almost equal to the clearance between the thickness control rolls, and in this state, treated with the superheated vapor. Thereafter, the web runs toward the downstream of the conveyor while keeping the thickness unchanged. In this example, the linear pressure of the thickness control rolls was adjusted to be 50 kg/cm.

[0081] Then, the carded web was introduced to the vapor injection system provided for the lower conveyor, and the vapor treatment was performed such that the superheated vapor of 300°C injected from the system penetrates the carded web in the thickness direction of the carded web (vertically). In this manner, the fiber assembly having the nonwoven fiber structure of this example was obtained. The vapor injection system had a nozzle arranged in the lower conveyor to inject the superheated vapor toward the web via the conveyor net, and a suction device provided for the upper conveyor. On the downstream side of the injection system in the running direction of the web, another vapor injection system including the nozzle and the suction device arranged at reverse positions was provided. Thus, the front and rear surfaces of the web were both treated with the superheated vapor.

[0082] The vapor injection system used had nozzles each having an orifice diameter of 0.3 mm. The nozzle were aligned at a pitch of 1 mm in the width direction of the conveyor. The processing speed was 3 m/min, and a clearance (distance) between the upper and lower conveyor belts between the nozzles and the suction device was 5 mm. The nozzles were arranged to be substantially in contact with the rear side of the conveyor belt.

<Measurement of Mass per Unit Area>

[0083] The mass per unit area (g/m^2) of the produced fiber assembly was measured in accordance with JIS L1913. Table 1 shows the results.

<Measurement of Apparent Density>

[0084] The thickness (mm) of the produced fiber assembly was measured in accordance with JISL 1913, and the apparent density (g/m^3) was calculated based on the thickness and the mass per unit area. Table 1 shows the results.

<Measurement of Fiber Bonding Ratio>

[0085] A 100-times magnified image of a cross section of the fiber assembly was taken with a scanning electron microscope (SEM). Then, the image of the cross section cut along the thickness direction of the fiber assembly was divided into three equal regions arranged in the thickness direction. In each of the regions (a front surface region, an inside (middle) region, and a rear surface region), a ratio of the count of sections (end faces) of the fibers bonded together with respect to the count of sections of all the recognizable fibers was calculated.

[0086] Specifically, in each region, a ratio of the count of the sections of two or more fibers bonded together with respect to the count of all sections of the recognizable fibers was represented in the unit of percentage by the following formula (1).

[Formula 1] Fiber bonding ratio (%) = (count of sections of two or more fibers bonded together) / (count of all sections of fibers)

[0087] The fibers in contact with each other include those which are merely in contact with each other and not bonded together, and those which are bonded together. Through cutting the fiber assembly for taking the microscopic image, the fibers merely in contact with each other were separated by a stress of each of the fibers on the cross section of the fiber assembly. Thus, the fibers in contact with each other in the cross sectional image were regarded as those bonded together.

[0088] In the cross sectional image taken in each example, all the fibers whose sections are recognizable were counted. If the count of the fiber sections was 100 or less, another cross sectional image was used so that the total count of the fiber sections exceeds 100. Further, in each of the three equal regions, the fiber bonding ratio was calculated, and a difference between the maximum and minimum values (i.e., uniformity) was calculated as well. Table 1 shows the results.

<Measurement of Bending Stress>

[0089] A test piece (10 mm wide, 100 mm long) was cut from the produced fiber assembly, and flexural stress (MPa) thereof was measured in accordance with JIS K7171 (Plastics - Determination of Flexural Properties) with the span length set to 80 mm, and the test speed to 10 mm/min. Table 1 shows the results.

<Measurement of Tensile Strength>

[0090] A test piece (30 mm wide, 150 mm long) was cut from the produced fiber assembly, and tensile strength (N/30 mm) thereof was measured in accordance with JIS L1913 (Test Methods for Nonwovens) with the length of the test piece between grips set to 100 mm, and the test speed to 10 mm/min. Table 1 shows the results.

(Example 2)

<Production of Fiber Assembly>

[0091] As the heat resistant fibers, amorphous polyetherimide fibers (manufactured by Kuraray Co., Ltd. under the trade name "KURAKISSS," glass transition temperature: 215°C, thermal decomposition temperature: 540°C, fineness: 8.9 dtex, fiber length: 51 mm) were prepared. With the heat resistant fibers thus prepared, a carded web having a mass per unit area of 100 g/m^2 was formed by carding, and shaped into a sheet by hydroentangling.

[0092] Then, a stack of ten sheets thus prepared was transported to a belt conveyor provided with a stainless steel endless net of 50 mesh and a width of 500 mm.

[0093] Then, in the same manner as in Example 1, the carded web was introduced to the vapor injection system provided for the lower conveyor, and the vapor treatment was performed such that the superheated vapor of 330°C injected from the system penetrates the carded web in the thickness direction of the carded web (vertically). In this manner, the fiber assembly having the nonwoven fiber structure of this example was obtained.

[0094] Then, in the same manner as in Example 1, the mass per unit area, the apparent density, the fiber bonding ratio, the flexural stress, the bending load, and the tensile strength were measured. Table 1 shows the results.

(Example 3)

<Production of Fiber Assembly>

[0095] Nine carded webs used in Example 1 were stacked, and thermally pressed at 260°C for one minute using a hot pressing machine to obtain a fiber assembly.

[0096] Then, in the same manner as in Example 1, the mass per unit area, the apparent density, the fiber bonding ratio, the flexural stress, the bending load, and the tensile strength were measured. Table 1 shows the results.

(Comparative Example 1)

<Production of Fiber Assembly>

[0097] The semi-aromatic polyamide fibers prepared in Example 1 were blended with binder fibers at a mass ratio of 80/20. The binder fibers used were polypropylene/polyethylene sheath-core composite fibers (HR-NTW of Ube Exsymo Co., Ltd., glass transition temperature of the core: -20°C, glass transition temperature of the sheath: -120°C, thermal decomposition temperature of the core: 240°C, thermal decomposition temperature of the sheath: 270°C, fineness: 1.7 dtex, fiber length: 51 mm). Then, using the blended fibers, six carded webs each having a mass per unit area of 50 g/m^2 were produced and stacked to obtain a single carded web having a mass per unit area of 300 g/m^2 in total. This carded web was thermally treated at 150°C for one minute with a hot air dryer to obtain a fiber assembly of this comparative example.

[0098] Then, in the same manner as in Example 1, the mass per unit area, the apparent density, the fiber bonding ratio, the flexural stress, the bending load, and the tensile strength were measured. Table 1 shows the results.

[Table 1]

	Mass per unit area [g/m^3]	Thickness [mm]	Apparent density [g/cm^3]	Fiber bonding ratio [%]				Flexural stress [MPa]	Tensile strength [N/30mm]		Maintenance ratio of tensile strength [%]
				Front surface	Middle (center)	Rear surface	Uniformity		Normal temp.	180°C	
Ex. 1	702	2.8	0.26	54.7	60.5	54.4	6.1	1.8	147	34	23.1
Ex. 2	1531	3.9	0.39	59.5	52.3	57.5	7.2	1.1	158	92	58.2
Ex. 3	453	3.6	0.13	53.7	15.8	48.3	37.9	0.4	28	2	7.1
Com. Ex. 1	349	6.7	0.05	8.5	7.4	8.1	1.1	0	13	0.8	6.2

[0099] As shown in Table 1, the fiber structure bodies of Examples 1 to 3, in which the heat resistant fibers having a glass transition temperature of 100°C or higher were thermally bonded, showed higher flexural stress and tensile strength than the fiber assembly of Comparative Example 1 in which the heat resistant fibers were bonded with the binder fibers. In particular, the fiber structure bodies of Examples 1 and 2 with highly uniform fiber bonding ratio had remarkably higher flexural stress and tensile strength than the fiber assembly of Comparative Example 1. This indicates that the fiber structure bodies of Examples 1 and 2 have excellent tenacity.

[0100] Further, the fiber structure bodies of Examples 1 and 2 showed a significantly higher maintenance ratio of the tensile strength (tensile strength at 180°C/tensile strength at room temperature) than the fiber assembly of Comparative Example 1. This indicates that the fiber structure bodies of Examples 1 and 2 have remarkably high heat resistance.

[0101] As shown in Table 1, the flexural stress of the fiber assembly of Comparative Example 1 was zero. Such a fiber assembly is so soft as to be bent by its own weight, and its flexural stress is below the measurement limit. Even if this fiber assembly is used as, for example, a thermal insulator of a construction, it does not stay along the wall surface or the ceiling, but sags, and therefore, is not easy to handle. In contrast, the fiber assembly of Example 3 had a flexural stress of 0.4 MPa, which is higher than that of Comparative Example 1. A fiber assembly having a flexural stress of about 0.4 MPa can stay on the wall surface without sagging. Therefore, the fiber assembly of Example 3 is further improved in ease of handling, for example.

[0102] Further, the fiber assembly of Example 3 showed a higher maintenance ratio of the tensile strength (tensile strength at 180°C/tensile strength at room temperature) than that of Comparative Example 1. This indicates that the fiber assembly of Example 3 has remarkably high heat resistance.

[0103] In contrast, the fiber assembly of Comparative Example 1 containing the fibers bonded together with the binder fibers showed a remarkably low maintenance ratio of the tensile strength as shown in Table 1, as a result of which the fiber assembly of Comparative Example 1 had significantly lower flexural stress and tensile strength than the fiber structure bodies of Examples 1 and 2.

INDU STRIAL APPLICABILITY

[0104] As can be seen, the present invention is suitable for a heat resistant fiber assembly comprised of heat resistant fibers and used as a thermal insulator or an acoustic absorbent.

Claims

1. A heat resistant fiber assembly which is a fiber assembly containing heat resistant fibers having a glass transition temperature of 100°C or more, the heat resistant fibers being bonded together.

2. The heat resistant fiber assembly of claim 1, wherein
the heat resistant fibers have a fiber bonding ratio of 10% to 85%.

3. The heat resistant fiber assembly of claim 2, wherein
if a cross section of the fiber assembly cut along a thickness direction thereof is divided into three equal regions arranged in the thickness direction, a middle region of the three regions has the fiber bonding ratio of 10% to 85%.

4. The heat resistant fiber assembly of claim 2 or 3, wherein
the fiber bonding ratio has a uniformity of 20% or less.

5. The heat resistant fiber assembly of any one of claims 1 to 4, having an apparent density of 0.03 g/cm^3 to 0.7 g/cm^3.

INTERNATIONAL SEARCH REPORT

International application No.
PCT/JP2017/013100

A. CLASSIFICATION OF SUBJECT MATTER
D04H1/54(2012.01)i

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)
D04H1/00-18/04

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

Jitsuyo Shinan Koho	1922-1996	Jitsuyo Shinan Toroku Koho	1996-2017
Kokai Jitsuyo Shinan Koho	1971-2017	Toroku Jitsuyo Shinan Koho	1994-2017

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)
Japio-GPG/FX

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	WO 2014/208671 A1 (Kuraray Co., Ltd.), 31 December 2014 (31.12.2014), claims; 0018, 0025 to 0026 & US 2016/0145782 A1 claims; 0017, 0024 to 0025 & TW 201509652 A & CN 105339541 A & KR 10-2016-0025561 A	1-5
X	JP 3-180588 A (Nitto Boseki Co., Ltd.), 06 August 1991 (06.08.1991), claims; page 3, lower right column, 2nd line from the bottom to the last line (Family: none)	1-5

☒ Further documents are listed in the continuation of Box C. ☐ See patent family annex.

* Special categories of cited documents:
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Date of the actual completion of the international search
01 June 2017 (01.06.17)

Date of mailing of the international search report
13 June 2017 (13.06.17)

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INTERNATIONAL SEARCH REPORT

International application No.
PCT/JP2017/013100

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	CN 103343423 A (Changzhou Institute of Advanced Materials, Beijing University of Chemical Technology), 09 October 2013 (09.10.2013), claims; 0013 (Family: none)	1-5
P,X	WO 2017/002924 A1 (Kuraray Co., Ltd.), 05 January 2017 (05.01.2017), claims; 0014, 0017 (Family: none)	1-5
A	WO 2014/021084 A1 (Kuraray Co., Ltd.), 06 February 2014 (06.02.2014), & US 2015/0140306 A1 & TW 201413084 A & CN 104508018 A & KR 10-2015-0040867 A	1-5

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REFERENCES CITED IN THE DESCRIPTION

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Patent documents cited in the description

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