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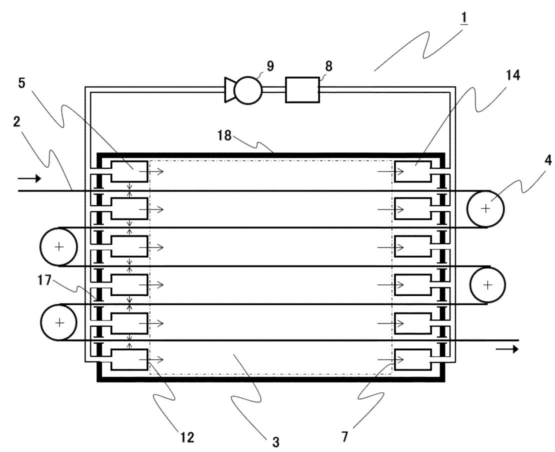
(54) FLAME RESISTANT FIBER BUNDLES, CARBON FIBER BUNDLE PRODUCTION METHOD, AND FLAME RESISTANT FURNACE

(57) The present invention aims at efficient production of high quality oxidized fiber bundles and carbon fiber bundles. A production method for an oxidized fiber bundle comprising a step for heat-treating aligned acryl based fiber bundles in an oxidizing atmosphere while turning them back on guide rollers installed on both ends outside the furnace body of a hot gas heating type oxidation furnace wherein: supply nozzles for supplying hot gas into a heat treatment chamber are installed at an end in the traveling direction of the acryl based fiber bundles; a fiber bundle traveling passage(s) exists above and/or below each nozzle; hot gas is supplied from the supply face(s) located above and/or below the acryl based fiber bundle; and the requirements (1) and (2) are satisfied where Vf is the gas flow speed in a substantially parallel direction to the traveling direction of the acryl based fiber bundle in the fiber bundle traveling passage and V is the gas flow speed in a substantially parallel direction to the traveling direction of the acryl based fiber bundle in the heat treatment chamber.

$$(1)\ 1.5\ \text{m/s} \leq Vf \leq 15\ \text{m/s}$$

$$(2)\ 1.5\ \text{m/s} \leq V \leq 10\ \text{m/s}$$

[Fig. 1]



EP 4 123 065 A1

Description

TECHNICAL FIELD

[0001] The present invention relates to an oxidized fiber bundle and a production method for a carbon fiber bundle. More specifically, the present invention relates to an oxidized fiber bundle useful for efficient production of an oxidized fiber bundle having high quality, a production method for a carbon fiber bundle, and an oxidation furnace.

BACKGROUND ART

[0002] Being high in specific strength, specific elastic modulus, heat resistance, and chemical resistance, the carbon fiber is useful as a reinforcing material for various materials and has been used in a wide range of fields such as aerospace applications, leisure applications, and general industrial applications.

[0003] In a generally known process for producing a carbon fiber bundle from an acryl based fiber bundle, an acryl based fiber bundle containing several thousands to several tens of thousands of aligned single fibers of an acrylic polymer is fed to an oxidation furnace and subjected to heat treatment (oxidation treatment) by exposing it to a flow of hot oxidizing gas such as hot gas heated at 200°C to 300°C supplied from a heated gas supply nozzle (hereinafter referred to simply as supply nozzle) installed in the furnace body, and the resulting oxidized fiber bundle is sent to a carbonization furnace where it is subjected heat treatment (precarbonization treatment) in an inert gas atmosphere maintained at 300°C to 1,000°C. Finally, it is subjected to heat treatment (carbonization treatment) in a carbonization furnace filled with an inert gas atmosphere at 1,000°C or more. The oxidized fiber bundle obtained as an intermediate material is so high in fire retardance that it has been also used widely as material for fire retardant woven fabrics.

[0004] In the carbon fiber bundle production process, the oxidation step is the longest in treatment time and the largest in energy consumption. In this aspect, productivity improvement in the oxidation step is the most important point in the production of a carbon fiber bundle.

[0005] In order to permit prolonged heat treatment in the oxidation step, it is common to use an oxidation apparatus (hereinafter referred to as oxidation furnace) that has turn-around rollers disposed outside the furnace body of the oxidation furnace to allow the acryl based fibers to be turned around and moved to and fro many times in the horizontal direction in the furnace body of the oxidation furnace. The method in which hot gas is supplied in a substantially parallel direction to the traveling direction of the acryl based fiber bundle in the furnace body of such an oxidation furnace is called the parallel flow method, whereas the method in which hot gas is supplied in the orthogonal direction to the traveling direction of the acryl based fiber bundle is called the orthogonal flow method. An apparatus to work according to the parallel flow method has either an end-to-end (hereinafter simply referred to ETE) hot gas heating structure in which a supply nozzle is installed at one end of a parallel flow furnace with a nozzle for discharging gas out of the furnace body (hereinafter referred to simply as discharge nozzle) installed at the other end or a center-to-end (hereinafter simply referred to CTE) hot gas heating structure in which a supply nozzle is installed in the central portion of the parallel flow furnace with discharge nozzles installed at both ends thereof. It is noted that apparatuses of the ETE hot gas heating structure is generally is lower in equipment cost than apparatuses of the CTE hot gas heating structure.

[0006] Among other methods for improving the productivity of an oxidation process, it is effective to feed a large number of acryl based fiber bundles at the same time to achieve a higher density of acryl based fiber bundles in the furnace body of the oxidation furnace. It is also effective to increase the traveling speed of the acryl based fiber bundle. However, with an increasing mass per unit volume of the acryl based fiber bundle supplied to the furnace body, an increasing quantity of heat per unit volume will be required to add heat to or remove heat from the acryl based fiber bundle, and this makes temperature control difficult and leads to oxidized fibers with deteriorated quality.

[0007] Furthermore, in the case of increasing the traveling speed of the acryl based fiber bundle, it will be necessary to increase the size of the oxidation furnace in order to maintain the same heat treatment rate. In particular, when the size in the height direction is increased, it will be necessary to divide a building floor into multiple subfloors or increase the load capacity per unit area of the floor face, which may lead to an increase in equipment cost. Therefore, to increase the size of the oxidation furnace while avoiding an increase in equipment cost, it will be effective to reduce its size in the height direction while increasing the distance of one horizontal pass (hereinafter referred to as the oxidation furnace length). However, as the oxidation furnace length is lengthened, the heat treatment length is lengthened accordingly, which makes it difficult to control the temperature of the acryl based fiber bundle. In particular, this occurs in a noticeable manner in a furnace of the ETE hot gas heating structure.

[0008] Therefore, when an attempt is made to improve the productivity of an oxidation process that uses a furnace of the ETE hot gas heating structure, there occurs the problem of necessity to improve the efficiency in heating of and heat removal from the acryl based fiber bundle traveling in the furnace body of the oxidation furnace.

[0009] As a means of solving this problem, Patent document 1 describes a method in which the discharge faces of the discharge nozzles are provided at a distance from the heat treatment chamber to allow the hot gas in the heat treatment chamber to be sucked so that flows are formed in the gaps between the discharge nozzles, thereby facilitating the heating of and heat removal from the acryl based fiber bundle.

[0010] Furthermore, Patent document 2 describes a heat treatment method in which hot gas is supplied to the space located between supply nozzles that are installed at the center of the furnace body of a furnace having the CTE hot gas heating structure so that the space located between the supply nozzles and the space inside the furnace body have roughly the same temperature.

[0011] In addition, Patent document 3 describes a method, which was originally designed to serve as a means of improving the sealing property of an oxidation furnace, that uses a heating device having a supply face to emit hot gas in order to heat the acryl based fiber bundle in a flow channel gap through which the acryl based fiber bundle travels from outside the furnace body of the oxidation furnace into the furnace body.

PRIOR ART DOCUMENTS

PATENT DOCUMENTS

[0012]

Patent document 1: Japanese Patent No. 5856081
Patent document 2: Japanese Patent No. 5856082
Patent document 3: Japanese Patent No. 4796467

SUMMARY OF INVENTION

PROBLEMS TO BE SOLVED BY THE INVENTION

[0013] However, findings obtained so far by the present inventors suggest that the control of the flow formed in the gap between discharge nozzles proposed in Patent document 1 alone cannot serve enough to change the gas flow morphology in the furnace body of the oxidation furnace and will sometimes fail in sufficiently achieving the heating of or heat removal from the acryl based fiber bundle. Since the gas flow morphology (speed and direction of the hot gas flow) near the acryl based fiber bundle in the oxidation furnace is the dominant factor in the heat transfer to the acryl based fiber bundle, it is considered that the above gas flow control near the discharge nozzles can work effectively only in the space between the gas discharge nozzles, leading to a failure in having an sufficient effect.

[0014] Furthermore, when performing the method proposed in Patent document 2, the supply of hot gas between the supply nozzles causes gas turbulence as the hot gas moves across the acryl based fiber bundle, and accordingly, the acryl based fiber bundle will undergo significant thread sway even at a low gas flow speed, possibly resulting in contact with an adjacent acryl based fiber bundle, intermingling, breakage, etc. of the acryl based fiber bundles, or the like. In addition, Patent document 2 only proposes a method for equalizing the temperature of the gas flow between the supply nozzles and in the space inside the furnace body and discloses no techniques to control the temperature of the acryl based fiber bundle inside the furnace body. The parameters required for controlling the temperature of the acryl based fiber bundle in the furnace body include the temperature and speed of the hot gas flow. Although there is a description about the former, i.e. temperature, there is no detailed description about the speed of the hot gas flow, and accordingly, it is impossible in some cases to control the temperature of the acryl based fiber bundle. In addition, the document is limited to the CTE hot gas heating structure based on the parallel flow method, and there is no detailed description about application to the ETE hot gas heating structure, which is smaller in equipment cost.

[0015] In Patent document 3, furthermore, the hot gas supply face is located outside the furnace body of the oxidation furnace, and therefore, it is sometimes impossible to fully improve the performance in heating of or heat removal from the acryl based fiber bundle traveling in the furnace body of the oxidation furnace. In addition, since the method proposed in Patent document 3 is intended to improve the sealing performance of the oxidation furnace, hot gas is supplied in a direction toward outside the furnace body so that the hot gas supplied from the supply face is emitted directly out of the furnace body, and therefore, it is sometimes impossible to form a gas flow in the space between the nozzles through which the acryl based fiber bundle is traveling.

MEANS OF SOLVING THE PROBLEMS

[0016] To solve the above problems, the production method for an oxidized fiber bundle according to the present invention is configured as described below. Specifically, it is a production method for an oxidized fiber bundle comprising a step for heat-treating aligned acryl based fiber bundles in an oxidizing gas atmosphere while turning them back on guide rollers installed on both ends outside the furnace body of a hot gas heating type oxidation furnace wherein: supply nozzles for supplying hot gas into a heat treatment chamber are installed at an end in the traveling direction of the acryl based fiber bundles; a fiber bundle traveling passage(s) exists above and/or below each nozzle; hot gas is supplied from the supply face(s) located above and/or below the acryl based fiber bundle; and the requirements (1) and (2) are satisfied where Vf is the gas flow speed in a substantially parallel direction to the traveling direction of the acryl based fiber bundle in the fiber bundle traveling passage and V is the gas flow speed in a substantially parallel direction to the traveling direction of the acryl based fiber bundle in the heat treatment chamber.

$$(1)\ 1.5\ m/s \le Vf \le 15\ m/s$$

$$(2)\ 1.5\ m/s \le V \le 10\ m/s$$

[0017] It is also preferable for the production method for an oxidized fiber bundle according to the present invention to have the following features.

- The requirements (3) and (4) are satisfied where Vf is the gas flow speed in a substantially parallel direction to the traveling direction of the acryl based fiber bundle in the fiber bundle traveling passage and V is the gas flow speed in a substantially parallel direction to the traveling direction of the acryl based fiber bundle in the heat treatment chamber.

$$(3)\ 1.5\ m/s \le Vf \le 10\ m/s$$

$$(4)\ 1.5\ m/s \le V \le 6\ m/s$$

- At a supply face, the requirement (5) is satisfied where Vn is the gas flow speed in the orthogonal direction to the traveling direction of the acryl based fiber bundle.

$$(5)\ 0.1\ m/s \le Vn \le 5\ m/s$$

- The hot gas supplied from a supply face has a temperature of 210°C or more and 295°C or less.
- The acryl based fiber bundle before heat treatment has a single fiber fineness of 0.05 to 0.22 tex.

[0018] In addition, the method for producing a carbon fiber bundle according to the present invention is configured as described below. Specifically, it is

a production method for a carbon fiber bundle comprising a step for subjecting an oxidized fiber bundle produced by the above production method for an oxidized fiber bundle to precarbonization treatment at a maximum temperature of 300°C to 1,000°C in an inert gas atmosphere to produce a precarbonized fiber bundle and a subsequent step for subjecting the precarbonized fiber bundle to carbonization treatment at a maximum temperature of 1,000°C to 2,000°C in an inert gas atmosphere.

[0019] Here, a "substantially parallel direction to the traveling direction of the acryl based fiber bundle" referred to for the present invention means a direction within ±0.7°of the reference horizontal line, which connects the tops of a pair of mutually opposed turn-around rollers located at either end outside the furnace body.

[0020] Here, a "fiber bundle traveling passage" referred to for the present invention means a space around the acryl based fiber bundle formed along the traveling direction of the acryl based fiber bundle, and it is actually the space between a supply nozzle and another supply nozzle that are adjacent to each other in the vertical direction, the space between the supply nozzle and the upper face of the furnace body, or the space between the supply nozzle and the bottom face of the furnace body.

[0021] In addition, the oxidation furnace according to the present invention also has the following features. Specifically, it is

an oxidation furnace designed to perform heat treatment of an acryl based fiber bundle including:

(i) a furnace body having slits though which aligned fiber bundles can enter and exit the furnace body,
(ii) a plurality of supply nozzles disposed at intervals along a vertical line located at an end in the traveling direction of the fiber bundles in the heat treatment chamber so that hot gas is supplied into the furnace body,

(iii) a plurality of discharge nozzles disposed at intervals along a vertical line located at the other end in the traveling direction of the fiber bundles in the furnace body so that the hot gas supplied from the supply nozzles is discharged out of the heat treatment chamber,

(iv) at least one gas blowing device designed to circulate hot gas through the supply nozzles and the discharge nozzles,

(v) at least one heating device disposed in a flow path of the circulating hot gas, and

(vi) guide rollers disposed at both ends outside the furnace body to turn back the fiber bundles so that they travel to and fro a plurality of times in the heat treatment chamber while passing through the spaces between mutually adjacent supply nozzles and between mutually adjacent discharge nozzles, wherein

(vii) each of the supply nozzles has a supply face(s) at the top and/or bottom so that a first hot gas stream(s) is supplied to the fiber bundle traveling passage(s) running above and/or below the supply nozzle and also has an auxiliary supply face on a side of the supply nozzle facing the interior of the heat treatment chamber so that a second hot gas stream is supplied, and

(viii) adjusting devices to adjust the gas flow speed of the first hot gas stream and the gas flow speed of the second hot gas stream supplied from the supply nozzle.

ADVANTAGEOUS EFFECTS OF THE INVENTION

[0022] The production method for an oxidized fiber bundle according to the present invention serves to improve the performance in the heating of and heat removal from an acryl based fiber bundle passing through the furnace body of an oxidation furnace, thereby achieving efficient production of an oxidized fiber bundle and carbon fiber bundle having high quality.

BRIEF DESCRIPTION OF THE DRAWINGS

[0023]

[Fig. 1] This is a schematic cross-sectional view of an oxidation furnace used in an embodiment of the present invention.

[Fig. 2] This is a partial enlarged cross-sectional view of a region extending from around the supply nozzles to around the discharge nozzles used in an embodiment of the present invention.

[Fig. 3] This is a schematic view of the gas flow morphology in the region extending from around the supply nozzles to around the discharge nozzles adopted in an embodiment of the present invention.

[Fig. 4] This is a partial enlarged cross-sectional view of a region extending from around the supply nozzles to around the discharge nozzles used in another embodiment of the present invention.

[Fig. 5] This is a schematic view of the gas flow morphology in the region extending from around the supply nozzles to around the discharge nozzles in a conventional apparatus.

DESCRIPTION OF PREFERRED EMBODIMENTS

[0024] Embodiments of the present invention are described in detail below with reference to Fig. 1 to Fig. 4. Fig. 1 is a schematic cross-sectional view of an oxidation furnace used in an embodiment of the present invention and Fig. 2 is a partial enlarged cross-sectional view of a region extending from around the supply nozzles to around the discharge nozzles. In addition, Fig. 4 is a partial enlarged cross-sectional view of a region extending from around the supply nozzles to around the discharge nozzles in an oxidation furnace used for another embodiment of the present invention. Fig. 3, furthermore, is a schematic view of the gas flow morphology in the region extending from around the supply nozzles to around the discharge nozzles adopted in an embodiment of the present invention. It should be noted that these figures provide only schematic views designed for accurate understanding of important points of the present invention and that they are simplified drawings. There are no specific limitations on the oxidation furnace to use for the present invention and various embodiments may be modified in size etc.

[0025] The present invention provides a production method for an oxidized fiber bundle that is designed to perform heat treatment of an acryl based fiber bundle 2 in an oxidizing gas atmosphere and it is carried out in an oxidation furnace in which oxidizing gas is flowing. As shown in Fig. 1, the oxidation furnace 1 has a heat treatment chamber 3 provided with guide rollers 4 positioned outside the furnace body 18, wherein an acryl based fiber bundle 2 is subjected to oxidation treatment by blowing hot gas against it as it travels to and fro in multi-stage traveling areas in the furnace body 18 while turning around on the guide rollers 4. The acryl based fiber bundle 2 is sent into the furnace body 18 through a slit 17 provided in a side wall of the furnace body 18. Then, after traveling substantially straight in the heat treatment chamber 3, it is once sent out of the furnace body 18 through a slit 17 provided in the opposite side wall. Subsequently, it is turned around on a guide roller 4 provided on either side of the furnace body 18 and sent back into the furnace body 18. In this way, the acryl based fiber bundle 2 is repeatedly turned around on a plurality of guide rollers 4 so that it is sent in and out of the heat treatment chamber 3 a plurality of times. Thus it moves from top to bottom as a whole in the heat treatment chamber 3 while traveling through multi-stage areas as illustrated in Fig. 1. Here, it may move from bottom to top and the number of times the acryl based fiber bundle 2 is turned around in the heat treatment chamber 3 is not particularly limited. These features may be appropriately designed depending on the size of the oxidation furnace 1 etc. In addition, although the guide rollers 4 are installed outside the furnace body 18 in Fig. 1, the guide rollers 4 may be installed inside the furnace body 18.

[0026] While the acryl based fiber bundle 2 turns around repeatedly and travels to and fro in the heat treatment chamber 3, it is gradually oxidized as it is heated by hot gas flowing from the supply nozzles 5 toward the discharge faces 7 of the discharge nozzles 14, finally resulting in an oxidized fiber bundle. As described above, this oxidation furnace 1 is a parallel flow ETE hot gas heating type oxidation furnace. Here, a plurality of aligned acryl based fiber bundles 2 are arranged in the perpendicular direction to the figure plane to form a wide sheet.

[0027] The oxidizing gas flowing in the heat treatment chamber 3, which may be gas for example, is heated to a desired temperature by a heating device 8 before entering the heat treatment chamber 3 and supplied into the heat treatment chamber 3 from the supply faces 6 and/or the auxiliary supply faces 12 of the supply nozzles 5 with its supply rate controlled by the gas blowing device 9. Here, a supply face 6 of a supply nozzle 5 is a supply face positioned so as to face another supply nozzle 5 located next to the upper or lower face of the former supply nozzle 5, and the auxiliary supply face 12 of the supply nozzle 5 is a supply face positioned on the side of the supply nozzle 5 that faces the corresponding discharge nozzle 14. Then, the oxidizing gas discharged out of the heat treatment chamber 3 through the the discharge faces 7 of the discharge nozzles 14 may be released to the atmosphere after treating unnecessary substances in an exhaust gas treatment furnace (not shown in figures). However, it is not necessary to treat the oxidizing gas completely, and part of the untreated oxidizing gas may be sent through the circulation channel back into the heat treatment chamber 3 from the supply nozzle 5. Hereinafter, a supply face 6 of a supply nozzle 5 is simply referred to as supply face 6, the auxiliary supply face 12 of a supply nozzle 5 referred to as auxiliary supply face 12, and the discharge face 7 of a discharge nozzle 14 referred to as discharge face 7.

[0028] The heating device 8 used in the oxidation furnace 1 is not particularly limited as long as it has a desired heating ability, and for example, a known heating device such as an electric heater may be used. The gas blowing device 9 is not particularly limited either as long as it has a desired gas blowing ability, and for example, a known gas blowing device such as an axial fan may be used.

[0029] In addition, the guide rollers 4 can serve to control the traveling speed and tension of the acryl based fiber bundles 2 when their respective rotation speeds are adjusted appropriately. They can be adjusted on the basis of the required physical properties of the oxidized fiber bundles and the required processing rate per unit time.

[0030] Furthermore, the spaces between and the number of the plurality of acryl based fiber bundles 2 running in parallel can be controlled by engraving an appropriate number of grooves at appropriate intervals on the surface layers of the guide rollers 4 or by providing an appropriate number of comb guides (not shown in figures) at appropriate intervals in the vicinity of the guide rollers 4.

[0031] Conventionally, it has been known that an improved productivity can be achieved by increasing the traveling speed of the acryl based fiber bundle 2 or increasing the number of acryl based fiber bundles per unit distance, that is, the thread density, in the width direction of the oxidation furnace 1. However, if such requirements are satisfied with the aim of improving the productivity, it causes an increase in the supply rate of the acryl based fiber bundles 2 into the furnace body 18 relative to the supply rate of hot gas into the furnace body 18 per unit time. Accordingly, it leads to a relative decrease in the amount of heat of the hot gas that can be used for the heating of or heat removal from the acryl based fiber bundles 2. As a result, the temperature controllability of the acryl based fiber bundles 2 tends to decrease, possibly leading to deterioration in quality. For this, conceivable solutions include providing additional heating or heat removal devices and increasing the supply of heat in order to increase the amount of hot gas available for the heating of or heat removal from the acryl based fiber bundles 2. However, they are disadvantageous because they can cause a significant increase in costs due to increased equipment cost and utilities cost.

[0032] To solve this problem, it is effective to improve the efficiency in heat transfer between the acryl based fiber bundles 2 and the hot gas and good means thereof include increasing the flow speed of the hot gas and supplying hot gas in the orthogonal direction to the traveling direction of the acryl based fiber bundles 2 . Another means is to loosen the fibers in the acryl based fiber bundles 2 in order to increase the surface area to improve the heat transfer efficiency. However, if the fibers are loosened, adjacent acryl based fiber bundles 2 running in parallel tend to be entangled together. As described above, furthermore, an increase in the flow speed of hot gas causes an increase in utilities cost and accordingly an increase in running cost, and a change in the flow direction of hot gas toward the orthogonal direction to the acryl based fiber bundles 2 causes increased sway of the acryl based fiber bundles 2, allowing adjacent acryl based fiber bundles 2 running in parallel to be entangled together more easily. In addition, if the oxidation furnace length is increased in order to construct a larger ETE heating type oxidation furnace, which requires less equipment cost, there occur various disadvantages against productivity improvement such as excessive heat generation from the acryl based fiber bundles 2 in the latter part of each passage, which makes it impossible to control the temperature of the acryl based fiber bundles 2.

[0033] The production method for an oxidized fiber bundle according to the present invention, which was developed as a result of intensive studies on the above problems, serves for efficient production of high-quality oxidized fiber bundles. Specifically, the present inventors have developed a method that can achieve an improved efficiency in heat transfer between the acryl based fiber bundles 2 and the hot gas while preventing an increase in equipment cost and running cost and suppressing the entanglement between acryl based fiber bundles 2. Described in detail below is the principle of improving the efficiency in heat transfer between the acryl based fiber bundles 2 traveling in the heat treatment chamber 3 and the hot gas, which is the most important point of the present invention.

[0034] First, in order to clarify the difference between the conventional technology and the present invention, the gas flow morphology in the furnace body 18 constructed based on the conventional technology will be described with reference to Fig. 5. The lengths of the arrows showing gas flows in Fig. 5 represent the speeds of the gas flows.

[0035] In Fig. 5, the hot gas supplied from the first supply face 19 of a supply nozzle 5 installed at one end in the furnace body 18 passes through the fiber bundle traveling passage 10 between two supply nozzles 5. When it reaches the confluence plane 13 where the fiber bundle traveling passage 10 and the heat treatment chamber 3 come in contact with each other, it merges with the hot gas supplied from the second supply face 20 and continues to flow through the heat treatment chamber 3 while gradually narrowing the difference in speed between them. In this conventional technology, the speed of the gas flow moving in the fiber bundle direction in the fiber bundle traveling passage 10, which originates in the hot gas supplied from the first supply face 19, is smaller than the speed of the gas flow that originates in the hot gas supplied from the second supply face 20. Therefore, the gas flow speed in the vicinity of the acryl based fiber bundles 2 immediately after passing the confluence plane 13 is accelerated as the gas flow moves from the fiber bundle traveling passage 10 into the heat treatment chamber 3 while maintaining its flow speed, and it merges gradually with the gas flow that originates in the hot gas supplied from the second supply face 20. Then, the merged gas flow reaches a discharge nozzle 14 installed at the other end in the furnace body 18 and it is discharged nearly completely through the discharge face 7 while partly passing between two discharge nozzles 14 and flowing out of the furnace body 18.

[0036] Described below is the temperature of the acryl based fiber bundle 2 in the case where the thread density of the acryl based fiber bundles 2 is increased (or the traveling speed of the acryl based fiber bundles 2 is increased) in order to improve the productivity. The acryl based fiber bundles 2 once exit the furnace body 18 and, after being cooled by outside air, enters the fiber bundle traveling passage 10 again and is heated again. If the acryl based fiber bundles 2 have an increased thread density, a larger amount of heat is required for heat transfer and it becomes more difficult to implement the heating of or heat removal from the acryl based fiber bundles 2, making it impossible to heat them sufficiently in the heat treatment chamber 3. In particular, if the gas flow speed Vf in the fiber bundle traveling passage 10 is too small, the acryl based fiber bundles 2 will be fed to the heat treatment chamber 3 without being heated sufficiently, and consequently, the temperature in the heat treatment chamber 3 will drop, making it all the more difficult to raise the temperature of the acryl based fiber bundles 2. As described above, as the flow speed of the gas flow in the vicinity of the acryl based fiber bundles 2 has the greatest influence on the heat transfer, the acryl based fiber bundles 2 near the supply nozzles 5 in the heat treatment chamber 3 will be largely affected by the flow speed Vf of the hot gas passing through the fiber bundle traveling passage 10.

[0037] To overcame this disadvantage, in an embodiment of the present invention in which, as shown in Fig. 3, hot gas is supplied from a supply face 6 of a supply nozzle(s) 5 located above and/or below the acryl based fiber bundle 2, the gas flow morphology is designed so that the requirements (1) and (2) are satisfied where Vf is the speed of the gas flow moving in a substantially parallel direction to the traveling direction of the acryl based fiber bundles 2 in the fiber bundle traveling passage 10 and V is the speed of the gas flow moving in a substantially parallel direction to the traveling direction of the acryl based fiber bundles 2 in the heat treatment chamber 3:

$$(1)\ 1.5\ m/s \le Vf \le 15\ m/s,$$

and

$$(2)\ 1.5 m/s \le V \le 10 m/s.$$

[0038] Here, the speed Vf of the gas flow moving in a substantially parallel direction to the traveling direction of the acryl based fiber bundles 2 in the fiber bundle traveling passage 10 is the average of measurements taken at three points that are located on the line intersection between the confluence plane 13 and the acryl based fiber bundles 2 and that are aligned in the width direction and include the width-directional center of the furnace body 3 and the speed V of the gas flow moving in a substantially parallel direction to the traveling direction of the acryl based fiber bundles 2 in the heat treatment chamber 3 is the average of measurements taken at three points that are located on the line intersection between the acryl based fiber bundles 2 and the cross section of the heat treatment chamber 3 at the center in the traveling direction of the acryl based fiber bundles 2 and that are aligned in the width direction and include the width-directional center of the furnace body 3. Here, the above-mentioned measurement at each of the three points aligned in the width direction including the width-directional center of the furnace body 3 is the average of 30 measurements taken at one second intervals using a thermal anemometer. In addition, the line intersection between the confluence plane 13 and the acryl based fiber bundles 2 referred to above is the line intersection between the confluence plane 13 and the virtual plane that contains the plurality of traveling acryl based fiber bundles 2 aligned in parallel in the machine width direction, and the line intersection between the cross section of the heat treatment chamber 3 at the traveling-directional center of the acryl based fiber bundles 2 and the acryl based fiber bundles 2 is the line intersection between the cross section of the heat treatment chamber 3 at the traveling-directional center of the acryl based fiber bundles 2 and the virtual plane that contains the plurality of traveling acryl based fiber bundles 2 aligned in parallel in the machine width direction. Therefore, the measuring points are included in the virtual plane that contains the plurality of traveling acryl based fiber bundles 2 aligned in parallel in the machine width direction. However, Vf and V are indicators representing the speeds of the gas flows in the vicinity of the acryl based fiber bundles 2 in the fiber bundle traveling passage 10 and the heat treatment chamber 3, respectively, and accordingly, the arrows showing Vf and V in Fig. 3 (also in Fig. 5) are in the vicinity of the acryl based fiber bundles 2, rather than overlapping them.

[0039] When these conditions are met for the acryl based fiber bundles 2, high-speed hot gas flows come from a supply face 6 that faces a fiber bundle traveling passage 10 and hit the acryl based fiber bundles 2 to strongly promote the heat transfer between the acryl based fiber bundles 2 and the hot gas. Then, this hot gas turns toward the parallel direction to the traveling direction of the acryl based fiber bundles 2 and flows in the vicinity of the acryl based fiber bundles 2 in the fiber bundle traveling passage 10. Thus, it further accelerates the heat transfer and the temperature of the acryl based fiber bundles 2 rises rapidly. Subsequently, as the hot gas continues to flow in the vicinity of the acryl based fiber bundles 2 while maintaining the speed for the time being even in the heat treatment chamber 3, heat transfer between the acryl based fiber bundles 2 and the hot gas is promoted, thus enabling highly accurate control of the temperature of the acryl based fiber bundles 2. Therefore, the temperature of the acryl based fiber bundles 2 can be controlled even if the speed V of the gas flow passing through the heat treatment chamber 3 is reduced, making it possible to decrease the overall volume of hot gas that is circulating in the oxidation furnace 1. As another technique, hot gas that hits the acryl based fiber bundles 2 may be emitted only from an end portion of the supply nozzle 5 near the guide rollers 4, that is, in an area where the sag in the acryl based fiber bundles 2 is relatively small. This serves to increase the heat transfer without a significant increase in the sway of the acryl based fiber bundles 2.

[0040] Then, after passing through the fiber bundle traveling passage 10, the hot gas reaches the heat treatment chamber 3 and then continues to flow while spreading in the vertical direction. At this time, the additional hot gas supplied from the auxiliary supply face 12 works to reduce gas turbulence that may be caused by the spread of the hot gas flow, thereby serving to reduce fiber commingling that may result from sway of the acryl based fiber bundles 2.

[0041] Here, in order to adjust the flow speed of the hot gas supplied from the supply faces 6 and the auxiliary supply face 12, good methods include installing an adjusting valve such as damper in the circulation flow path that leads to each supply face and providing flow control plates such as perforated panel and honeycomb core plywood with different opening ratios at these supply faces.

[0042] Thus, for the oxidation method according to the present invention, it is extremely important that hot gas flows having high flow speeds be supplied in the orthogonal direction to the acryl based fiber bundles 2 in the fiber bundle traveling passage 10 and also that the requirements (1) and (2) given above be satisfied where Vf is the gas flow speed in a substantially parallel direction to the traveling direction of the acryl based fiber bundles 2 in the fiber bundle traveling passage 10 and V is the gas flow speed in a substantially parallel direction to the traveling direction of the acryl based fiber bundles 2 in the heat treatment chamber. These features are not taken into consideration at all in the conventional technology. In order to maximize the effect of the invention, it is more preferable that the requirements (3) and (4) be satisfied where Vf is the gas flow speed in a substantially parallel direction to the traveling direction of the acryl based fiber bundles 2 in the fiber bundle traveling passage 10 and V is the gas flow speed in a substantially parallel direction to the traveling direction of the acryl based fiber bundles 2 in the heat treatment chamber 3:

$$(3)\ 1.5\ \text{m/s} \le Vf \le 10\ \text{m/s},$$

and

$$(4)\ 1.5\text{m/s} \le V \le 6\text{m/s}.$$

[0043] To give an example where the requirement (1) or (2) is not satisfied, an gas flow speed Vf of less than 1.5 m/s may sometimes cause a failure in achieving sufficient heating of or heat removal from the acryl based fiber bundles 2. If the gas flow speed Vf is larger than 15 m/s, on the other hand, the acryl based fiber bundles 2 may sometimes receive an increased drag from the hot gas, which may lead to increased sway.

[0044] If the gas flow speed V is less than 1.5 m/s, furthermore, it may sometimes lead to a failure in achieving sufficient heating of or heat removal from the acryl based fiber bundles 2 in the heat treatment chamber 3. If the gas flow speed V is larger than 10 m/s, on the other hand, the acryl based fiber bundles 2 may sometimes receive an increased drag from the hot gas, which may lead to increased thread sway. If the gas flow speed V is larger than 10 m/s, furthermore, it may sometimes cause an increase in the amount of hot gas that circulates in the oxidation furnace, leading to an increase in utilities cost.

[0045] In addition, it is more preferable that the requirement (5) be satisfied where Vn is the gas flow speed in the orthogonal direction to the traveling direction of the acryl based fiber bundles 2 at a supply face 6. In this case, the efficiency in the heating of or heat removal from the acryl based fiber bundles 2 can be largely increased while suppressing their sway caused by the drag that the acryl based fiber bundles 2 may receive from the hot gas. Here, if the gas flow speed Vn is less than 0.1 m/s, it may sometimes lead to a failure in achieving sufficient heat transfer to the acryl based fiber bundles 2 and increasing its temperature sufficiently. An gas flow speed Vn of more than 5 m/s may sometimes lead to an increased thread sway. In addition, it is more preferable that the gas flow speed Vn be less than 3.5 m/s because this can maximize the effect of the present invention.

$$(5)\ 0.1\ \text{m/s} \leq Vn \leq 5\ \text{m/s}$$

Here, the speed Vn of the gas flow emitted from a supply face 6 in the orthogonal direction to the traveling direction of the acryl based fiber bundles 2 is the average of measurements taken at three points on the supply face 6 that are located on a line orthogonal to the fiber bundle traveling direction and aligned in the width direction and that include the width-directional center of the furnace body 3. Here, the above-mentioned measurement at each of the three points aligned in the width direction including the width-directional center of the furnace body 3 is the average of 30 measurements taken at one second intervals.

[0046] In addition, if the hot gas supplied from a supply face 6 has a temperature controlled at 210°C or more and 295°C or less, it serves more remarkably to achieve an improvement in the heat transfer performance. In this case, the temperatures of the hot gas supplied from supply faces 6 and auxiliary supply faces 12 may be different from each other, but they are preferably identical from the viewpoint of temperature controllability of the acryl based fiber bundles 2 and equipment cost.

[0047] Next, another embodiment of the present invention is described below with reference to Fig. 4. The disposition position of each supply face 6 in a supply nozzle 5 is not limited to both surfaces of the supply nozzle 5 but may be disposed only at the bottom (not shown) or only at the top (not shown) thereof. If a supply face 6 is disposed only at the top, it serves to support the weight of the acryl based fiber bundles 2 and therefore a reduction in their sway can be expected. On the other hand, if supply faces 6 are disposed at both surfaces to supply an gas flow with a constant speed Vf in the fiber bundle traveling passage 10, the gas speed can be reduced by half and the gas turbulence around the acryl based fiber bundles 2 can be decreased. This is preferable because their sway can be suppressed.

[0048] In addition, the disposition position of each supply face 6 of a supply nozzle 5 is not limited to a part nearer to the outer wall of the furnace body (Fig. 2), but may be on a part nearer to the interior of the furnace boy, or may be divided into several parts that are disposed at different positions, or may cover the entire surface (Fig. 4).

[0049] Furthermore, the supply nozzle 5 may have no auxiliary supply face 12 but may be designed to supply hot gas only from supply faces 6. In this case, where there is no auxiliary supply face 12, a flow control plate 16 may be provided to divide and minimize the heat treatment chamber 3 to work only around the traveling path of the acryl based fiber bundles 2 (Fig. 4) in order to prevent gas turbulence from being caused by the rapid spreading of gas flows that occurs as they move from the fiber bundle traveling passage 10 into the heat treatment chamber 3.

[0050] In addition, various effects can be created by changing the angle between the main flow direction of the hot gas supplied from a supply face 6 and the traveling direction of the acryl based fiber bundles. For example, if the angle is not orthogonal, it serves to suppress hot gas turbulence that may be caused by collisions between the acryl based fiber bundles and the supply nozzle 5. Here, if the main flow direction of the hot gas is inclined toward the heat treatment chamber 3, it serves to allow part of the hot gas to move more easily into the heat treatment chamber 3, thereby suppressing its leakage out of the oxidation furnace 1. If the main flow direction of hot gas is designed to be orthogonal to the traveling direction of the acryl based fiber bundles, it serves to improve the efficiency in heat transfer to the acryl based fiber bundles 2. In this way, the main flow direction of hot gas may be set appropriately taking into account the performance required of the acryl based fiber bundles 2 and the oxidation furnace.

[0051] Furthermore, it is preferable that the flow rate of the hot gas removed through a discharge face 7 be larger than the total flow rate of the hot gas supplied from the supply faces 6 and the auxiliary supply face 12 of a supply nozzle 5. This allows the hot gas supplied from a supply face 6 to flow easily into the heat treatment chamber 3, making it possible to suppress the leakage of hot gas from the heat treatment chamber 3 and improve the sealing performance.

[0052] Here, the processing rate of the acryl based fiber bundles 2 is preferably 0.14 to 11 kg/min per meter of the width of the oxidation furnace. As the processing rate increases, a larger heat transfer improving effect can be realized.

[0053] For the production method for an oxidized fiber bundle according to the present invention, furthermore, it is preferable for the acryl based fiber bundles 2 to have a single fiber fineness of 0.05 to 0.22 tex, more preferably 0.05 to 0.17 tex. If it is in the preferable range, it serves to prevent the single fibers in adjacent acryl based fiber bundles 2 from being entangled when the bundles come into contact with each other, thereby effectively avoiding the commingling of fibers between acryl based fiber bundles and it also serves to allow heat to reach the inner single fiber layer thoroughly in the furnace body of the oxidation furnace, prevent the acryl based fiber bundles 2 from undergoing fluffing easily, and ensure effective prevention of large-scale fiber commingling. Accordingly, this ensures that an oxidized fiber bundle with higher quality will be produced with higher operability. In this way, a higher single fiber fineness ensures a higher heat transfer efficiency as an effect of the present invention, thereby allowing heat to reach the inner single fiber layer thoroughly.

[0054] It is preferable that an oxidized fiber bundle produced by the method described above be precarbonized at a maximum temperature of 300°C to 1,000°C in an inert gas atmosphere to produce a precarbonized fiber bundle and that it be carbonized at a maximum temperature of 1,000°C to 2,000°C in an inert gas atmosphere to produce a carbon fiber bundle.

[0055] The maximum temperature of the inert gas atmosphere used for the precarbonization treatment is more preferably 550°C to 800°C. The adoptable gasses to form such an inert gas atmosphere that fills the precarbonization furnace include known inert gasses such as nitrogen, argon, and helium, of which nitrogen is preferable from the viewpoint of economy.

[0056] The precarbonized fiber produced by the above precarbonization treatment is then sent to a carbonization furnace where it is subjected to carbonization treatment. To produce a carbon fiber with improved mechanical properties, it is more preferable to carry out carbonization treatment at a maximum temperature of 1,200°C to 2,000°C in an inert gas atmosphere.

[0057] The adoptable gasses to form such an inert gas atmosphere that fills the carbonization furnace include known inert gasses such as nitrogen, argon, and helium, of which nitrogen is preferable from the viewpoint of economy.

[0058] The carbon fiber bundle thus obtained may be provided with a sizing agent in order to improve the handleability, affinity with the matrix resin, etc. The type of the sizing agent to use is not particularly limited as long as the desired characteristics can be realized, and good examples thereof include sizing agents containing, as main component, an epoxy resin, polyether resin, epoxy-modified polyurethane resin, and polyester resin. A generally known method may be used to apply such a sizing agent.

[0059] In addition, if necessary, the carbon fiber bundle may be subjected to electrolytic oxidation treatment or surface oxidation treatment in order to improve the affinity and adhesiveness with the matrix resin to use for producing a fiber-reinforced composite material.

[0060] For the production method for an oxidized fiber bundle according to the present invention, the acryl based fiber bundle to use as the fiber bundle to be heat-treated is preferably made of an acryl fiber containing 100 mol% acrylonitrile or an acrylic copolymer fiber containing 90 mol% or more acrylonitrile. Preferable copolymerization components for use in the acrylic copolymer fiber include acrylic acid, methacrylic acid, itaconic acid, alkali metal salts thereof, ammonium metal salts thereof, acrylamide, and methyl acrylate. Here, but there are no specific limitations on the chemical properties, physical properties, size, etc. of the acryl based fiber bundle.

EXAMPLES

[0061] The present invention will be illustrated below in greater detail with reference to examples, but the invention should not be construed as being limited thereto. Here, measurements of the gas flow speed and the thread sway used in the examples and comparative examples were taken by the methods described below.

[0062] (1) Measuring method for single fiber fineness of acryl based fiber bundle A specimen was taken from an acryl based fiber bundle before feeding it into an oxidation furnace and measurement was perform according to JIS L 1013 (2010-06-21 revised edition).

(2) Measuring method for gas flow speed

[0063] An Anemomaster Model 6162 high temperature gas flow speed meter, manufactured by Kanomax Japan Inc., was used as a thermal type anemometer and the average of 30 measurements of instantaneous gas flow speed taken at one second intervals was adopted. A measuring probe was inserted through a measuring hole (not shown in figures) provided in the side wall of a furnace body 18. Then, the average of measurements taken at three points that were located on the line intersection between the confluence plane 13 and the acryl based fiber bundles 2 and that were aligned in the width direction and included the width-directional center was determined as Vf; the average of measurements taken at three points that were located on the line intersection between the acryl based fiber bundle 2 and the cross section of the heat treatment chamber 3 at the center in the traveling direction of the acryl based fiber bundle 2 and that were aligned in the width direction and included the width-directional center was determined as V; and the average of measurements taken at three points on a supply face 6 that were located on a line orthogonal to the traveling direction of the acryl based fiber bundles 2 and that were aligned in the width direction and included the width-directional center was determined as Vn.

(3) Measuring method for temperature of acryl based fiber bundle

[0064] A K-thermocouple was fastened to a traveling acryl based fiber bundle 2 and the temperature of the acryl based fiber bundle 2 in the heat treatment chamber 3 was measured at one second intervals, followed by calculating the thread temperature uniformity I (%).

$$I(n) = (\text{time period from when reading on thermocouple rises to T°C till when it lowers to (T-5)°C / transit time through heat treatment chamber}) \times 100\ (\%)$$

[0065] Here, T is the temperature T of the hot gas supplied from a supply nozzle 5, and I is the arithmetic average of five measurements I(n).

(4) Operability and quality

[0066] The criteria for them were as described below.

(Operability)

[0067] Operation was performed continuously for 10 days and the daily frequency of troubles such as fiber commingling and fiber bundle breakage was used for the criterion.

Excellent: The average frequency is zero (extremely good level).
Good: The average frequency is about 1 to 9 (sufficiently good level for performing continuous operation).
Fair: The average frequency is about 10 to 19 (barely satisfactory level for performing continuous operation).
Poor: The average frequency is 20 or more (unsatisfactory level for performing continuous operation).

(Quality)

[0068] A 10 m portion of the oxidized fiber bundle was visually observed after leaving the oxidation process, and the number of 10 mm or more fluff spots detected per meter of the oxidized fiber bundle was used for the criterion.

Excellent: The average number is 1 or less (the fluffing quality has no influence at all on the processability in production process or higher-order processability of the resulting product).
Good: The average number is more than 1 and less than 10 (the fluffing quality has little influence on the processability in production process or higher-order processability of the resulting product).
Fair: The average number is 10 or more and less than 20 (the fluffing quality has influence frequently on the processability in production process or higher-order processability of the resulting product).
Poor: The average number is 20 or more (the fluffing quality has adverse influence on the processability in production process or higher-order processability of the resulting product).

[Example 1]

[0069] The heat treatment furnace according to the present invention shown in Fig. 1 is used as the oxidation furnace for producing a carbon fiber. At one end of a furnace body 18, a plurality of supply nozzles 5, which serve as supply sources of hot gas, are installed above and below acryl based fiber bundles 2 traveling in the furnace body 18. As illustrated in Fig. 2, supply faces 6 are provided at the top and bottom of each supply nozzle 5, and an auxiliary supply face 12 is provided to supply hot gas in the traveling direction of the acryl based fiber bundles 2. A perforated plate with an opening ratio of 30% is provided on the supply faces 6 and the auxiliary supply face 12 so that the gas flow speed is maintained uniform in the width direction, and a damper (not shown in figures) was provided in the circulation flow path that leads to each supply face in order to control the flow speed of the hot gas supplied from each supply face.

[0070] Regarding the acryl based fiber bundles 2 traveling in the furnace body, 100 acryl based fiber bundles 2 containing 20,000 single fibers with a single fiber fineness of 0.11 dtex were pulled and hannei aligned, and they were heat-treated in the oxidation furnace 1 to produce oxidized fiber bundles. In addition, guide rollers 4 were installed at both ends outside the furnace body 18 of the oxidation furnace 1 with horizontal distance (roller span) L' of 15 m. The guide rollers 4 were grooved rollers with a groove distance (groove pitch) Wp of 10 mm. At this time, the oxidizing gas in the heat treatment chamber 3 of the oxidation furnace 1 had a temperature of 240°C to 280°C. The traveling speed of the acryl based fiber bundles 2 was adjusted in the range of 1 to 15 m/min depending on the oxidation furnace length L to ensure a sufficiently long oxidation treatment time while the process tension was adjusted in range of 0.5 to 2.5 g/dtex.

[0071] The resulting oxidized fiber bundles were then subjected to stabilization and carbonization in a precarbonization furnace at a maximum temperature of 700°C, subjected to stabilization and carbonization in a carbonization furnace at a maximum temperature of 1,400°C. Then, electrochemical treatment of the fiber surface was carried out and a sizing agent was applied to provide carbon fiber bundles.

[0072] Results are given in Table 1 and they show that the thread temperature uniformity was 20% when the gas flow speed Vn on the supply faces 6, the gas flow speed Vf in the fiber bundle traveling passage 10, and the average gas flow speed V in the heat treatment chamber 3 were 8.5 m/s, 11.2 m/s, and 7.0 m/s, respectively. Under the above conditions, the frequencies of fiber intermingling and fiber bundle breakage due to contact between acryl based fiber bundles were small during the oxidation treatment of the acryl based fiber bundles 2, serving to produce oxidized fiber bundles with high operability. In addition, the resulting oxidized fiber bundles were observed visually, and results showed that they had good quality with little fluffing etc.

[Example 2]

[0073] Except that the gas flow speed Vn on the supply faces, the gas flow speed Vf in the fiber bundle traveling passage 10, and the average gas flow speed V in the heat treatment chamber 3 were 6.0 m/s, 3.3 m/s, and 3.0 m/s, respectively, the same procedure as in Example 1 was carried out. In this test run, the thread temperature uniformity was 17%. Under the above conditions, the frequencies of fiber intermingling and fiber bundle breakage due to contact between acryl based fiber bundles were zero during the oxidation treatment of the acryl based fiber bundles 2, serving to produce oxidized fiber bundles with extremely high operability. In addition, the resulting oxidized fiber bundles were observed visually, and results showed that they had good quality with little fluffing etc.

[Example 3]

[0074] Except that the gas flow speed Vn on the supply faces was 3.3 m/s, the same procedure as in Example 2 was carried out. In this test run, the thread temperature uniformity was 16%. Under the above conditions, the frequencies of fiber intermingling and fiber bundle breakage due to contact between acryl based fiber bundles were zero during the oxidation treatment of the acryl based fiber bundles 2, serving to produce oxidized fiber bundles with extremely high operability. In addition, the resulting oxidized fiber bundles were observed visually, and results showed that they had extremely good quality without fluffing etc.

[Comparative example 1]

[0075] Except that the gas flow speed Vf in the fiber traveling passage 10 and the average gas flow speed V in the heat treatment chamber 3 were 1.1 m/s and 6.0 m/s, respectively, for Comparative example 1, the same procedure as in Example 2 was carried out. In this test run, the thread temperature uniformity was 8%, and under the above conditions, fiber intermingling and single fiber breakage due to contact between acryl based fiber bundles occurred frequently during the oxidation treatment of the acryl based fiber bundles 2. In addition, the resulting oxidized fiber bundles were observed visually, and results showed that they had poor quality with significant fluffing etc.

[Table 1]

[0076]

Table 1

		Example 1	Example 2	Example 3	Comparative example 1
Equipment settings	roller span [m]	15.0	15.0	15.0	15.0
	groove pitch [mm]	10.0	10.0	10.0	10.0
Vf [m/s]		11.2	3.3	3.3	1.1
V [m/s]		7.0	3.0	3.0	6.0
Vn [m/s]		8.5	6.0	3.3	3.3
Thread temperature uniformity [%]		20	17	16	8
Operability		good	excellent	excellent	poor
Quality		good	good	excellent	poor

INDUSTRIAL APPLICABILITY

[0077] The present invention relates to a production method for an oxidized fiber bundle and a production method for a carbon fiber bundle and can be applied to members of aircraft, industrial products such as pressure vessels and windmill blade, sporting goods such as golf shafts, and the like, although its scope of application is not limited thereto.

EXPLANATION OF NUMERALS

[0078]

1. oxidation furnace
2. acryl based fiber bundle
3. heat treatment chamber
4. guide roller
5. supply nozzle
6. supply face
7. discharge face
8. heating device
9. gas blowing device
10. fiber bundle traveling passage
12. auxiliary supply face
13. confluence plane
14. discharge nozzle
16. flow control plate
17. slit
18. furnace body
19. first supply face
20. second supply face

Claims

1. A production method for an oxidized fiber bundle comprising a step for heat-treating aligned acryl based fiber bundles in an oxidizing gas atmosphere while turning them back on guide rollers installed on both ends outside the furnace body of a hot gas heating type oxidation furnace wherein: supply nozzles for supplying hot gas into a heat treatment chamber are installed at an end in the traveling direction of the acryl based fiber bundles; a fiber bundle traveling passage(s) exists above and/or below each nozzle; hot gas is supplied from the supply face(s) located above and/or below the acryl based fiber bundle; and the requirements (1) and (2) are satisfied where Vf is the gas flow speed in a substantially parallel direction to the traveling direction of the acryl based fiber bundle in the fiber bundle traveling passage and V is the gas flow speed in a substantially parallel direction to the traveling direction of the acryl based fiber bundle in the heat treatment chamber,

$$(1)\ 1.5\ m/s \leq Vf \leq 15\ m/s,$$

and

$$(2)\ 1.5\ m/s \leq V \leq 10\ m/s.$$

2. A production method for an oxidized fiber bundle as set forth in claim 1, wherein the requirements (3) and (4) are satisfied where Vf is the gas flow speed in a substantially parallel direction to the traveling direction of the acryl based fiber bundle in the fiber bundle traveling passage and V is the gas flow speed in a substantially parallel direction to the traveling direction of the acryl based fiber bundle in the heat treatment chamber,

$$(3)\ 1.5\ m/s \leq Vf \leq 10\ m/s,$$

and

$$(4)\ 1.5\ m/s \leq V \leq 6\ m/s.$$

3. A production method for an oxidized fiber bundle as set forth in either claim 1 or 2, wherein the requirement (5) is satisfied where Vn is the gas flow speed at the supply face in the orthogonal direction to the traveling direction of the acryl based fiber bundle,

$$(5)\ 0.1\ m/s \leq Vn \leq 5\ m/s.$$

4. A production method for an oxidized fiber bundle as set forth in any one of claims 1 to 3, wherein the hot gas supplied from the supply face has a temperature of 210°C or more and 295°C or less.

5. A production method for an oxidized fiber bundle as set forth in any one of claims 1 to 4, wherein the acryl based fiber bundle before heat treatment has a single fiber fineness of 0.05 to 0.22 tex.

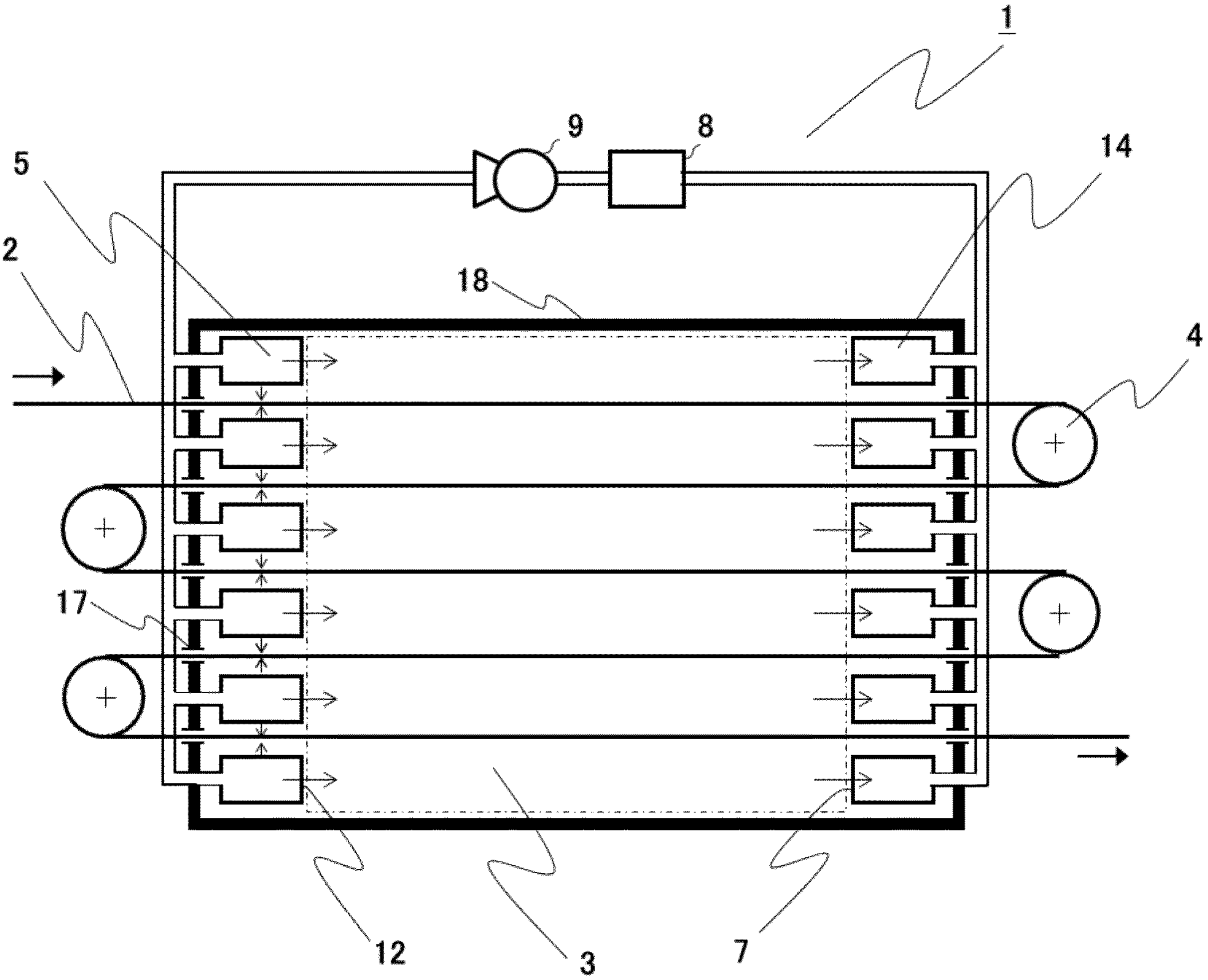
6. A production method for a carbon fiber bundle comprising a step for subjecting an oxidized fiber bundle produced by a production method for an oxidized fiber bundle as set forth in any one of claims 1 to 5 to precarbonization treatment at a maximum temperature of 300°C to 1,000°C in an inert gas atmosphere to produce a precarbonized fiber bundle and a subsequent step for subjecting the precarbonized fiber bundle to carbonization treatment at a maximum temperature of 1,000°C to 2,000°C in an inert gas atmosphere.

7. An oxidation furnace designed to perform heat treatment of an acryl based fiber bundle comprising:

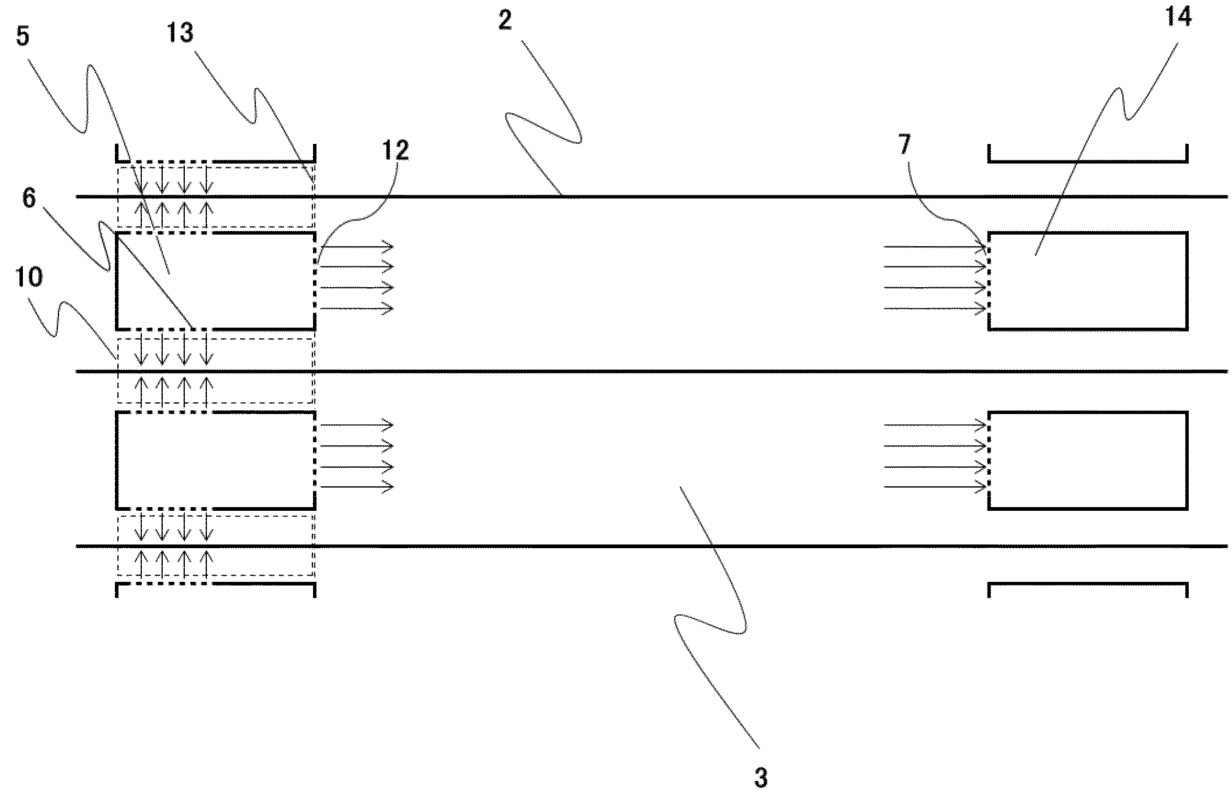
(i) a furnace body having slits though which aligned fiber bundles can enter and exit the furnace body,
(ii) a plurality of supply nozzles disposed at intervals along a vertical line located at an end in the traveling direction of the fiber bundles in the heat treatment chamber so that hot gas is supplied into the furnace body,
(iii) a plurality of discharge nozzles disposed at intervals along a vertical line located at the other end in the traveling direction of the fiber bundles in the furnace body so that the hot gas supplied from the supply nozzles is discharged out of the heat treatment chamber,
(iv) at least one gas blowing device designed to circulate hot gas through the supply nozzles and the discharge nozzles,
(v) at least one heating device disposed in a flow path of the circulating hot gas, and
(vi) guide rollers disposed at both ends outside the furnace body to turn back the fiber bundles so that they travel to and from a plurality of times in the heat treatment chamber while passing through the spaces between mutually adjacent supply nozzles and between mutually adjacent discharge nozzles, wherein
(vii) each of the supply nozzles has a supply face(s) at the top and/or bottom so that a first hot gas stream(s) is supplied to the fiber bundle traveling passage(s) running above and/or below the supply nozzle and also has an auxiliary supply face on a side of the supply nozzle facing the interior of the heat treatment chamber so that a second hot gas stream is supplied, and

(viii) adjusting devices to adjust the gas flow speed of the first hot gas stream and the gas flow speed of the second hot gas stream supplied from the supply nozzle.

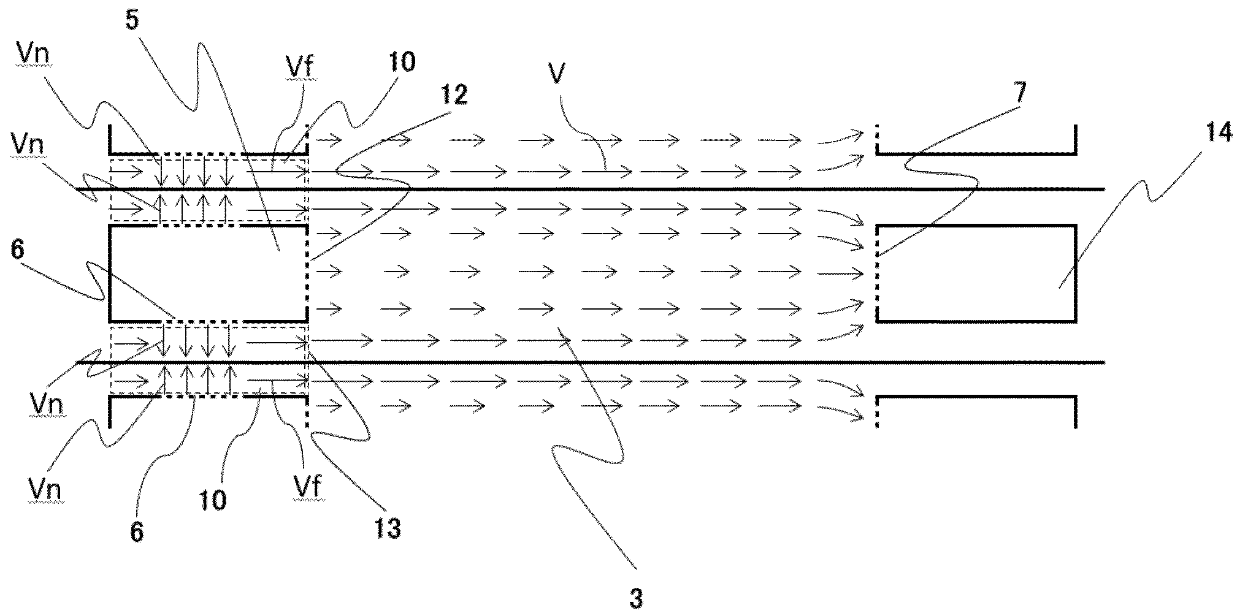
[Fig. 1]



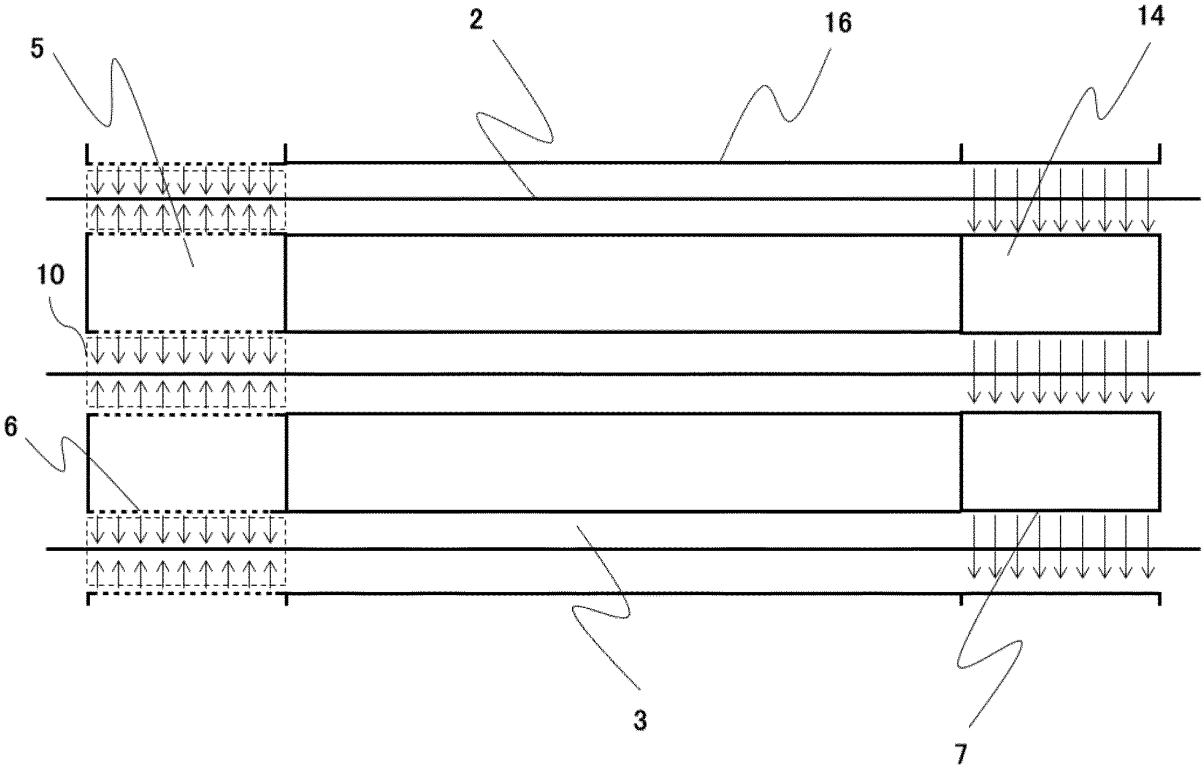
[Fig. 2]



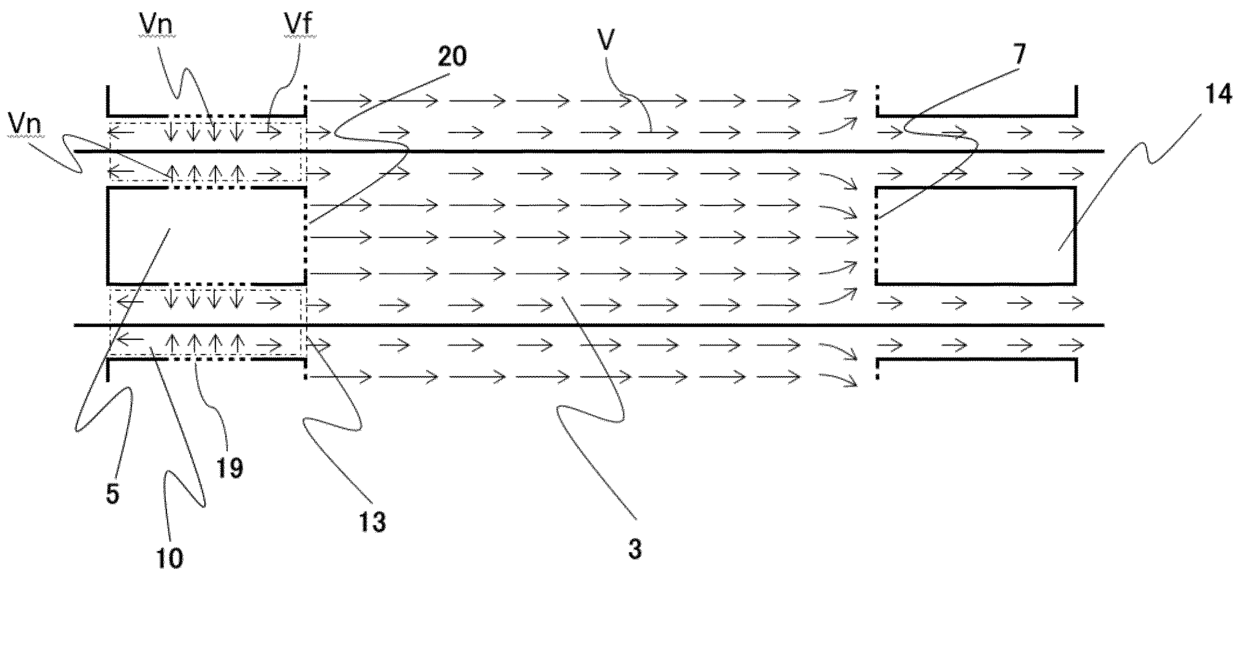
[Fig. 3]



[Fig. 4]



[Fig. 5]



INTERNATIONAL SEARCH REPORT

International application No.
PCT/JP2021/010787

A. CLASSIFICATION OF SUBJECT MATTER
D01F 9/22(2006.01)i
FI: D01F9/22

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)
D01F9/12-9/32

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

Published examined utility model applications of Japan	1922-1996
Published unexamined utility model applications of Japan	1971-2021
Registered utility model specifications of Japan	1996-2021
Published registered utility model applications of Japan	1994-2021

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

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	JP 2000-088464 A (TORAY INDUSTRIES, INC.) 31 March 2000 (2000-03-31) claims 1-8, paragraphs [0005]-[0006], fig. 1-4	1-7
A	JP 2007-247130 A (TORAY INDUSTRIES, INC.) 27 September 2007 (2007-09-27) claims 1-7, fig. 1-6	1-7
A	JP 2005-248339 A (MITSUBISHI RAYON CO., LTD.) 15 September 2005 (2005-09-15) claims 1-2, fig. 1	1-7
A	JP 2017-218720 A (MITSUBISHI CHEMICAL CORPORATION) 14 December 2017 (2017-12-14) claims 1-8, paragraph [0023], fig. 1	1-7

☒ Further documents are listed in the continuation of Box C. ☒ 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)
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"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step 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
26 May 2021 (26.05.2021)	08 June 2021 (08.06.2021)

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Japan Patent Office 3-4-3, Kasumigaseki, Chiyoda-ku, Tokyo 100-8915, Japan	Telephone No.

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INTERNATIONAL SEARCH REPORT

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C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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Form PCT/ISA/210 (continuation of second sheet) (January 2015)

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