(19) Europäisches Patentamt
European Patent Office
Office européen des brevets

(11) EP 3 518 621 A1

(12) EUROPEAN PATENT APPLICATION

(43) Date of publication:
31.07.2019 Bulletin 2019/31

(51) Int Cl.:
H05B 6/78 (2006.01) ***H05B 6/80*** (2006.01)
D01F 9/32 (2006.01) ***H05B 6/64*** (2006.01)

(21) Application number: **18168096.8**

(22) Date of filing: **18.04.2018**

(84) Designated Contracting States:
AL AT BE BG CH CY CZ DE DK EE ES FI FR GB GR HR HU IE IS IT LI LT LU LV MC MK MT NL NO PL PT RO RS SE SI SK SM TR
Designated Extension States:
BA ME
Designated Validation States:
KH MA MD TN

(30) Priority: **29.01.2018 TW 107103129**

(71) Applicant: **UHT Unitech Co., Ltd**
32061 Taoyuan City (TW)

(72) Inventor: **WANG, CHIH-YUNG**
32061 TAOYUAN CITY (TW)

(74) Representative: **Lang, Christian**
LangPatent Anwaltskanzlei
Ingolstädter Straße 5
80807 München (DE)

Remarks:
A request for correction of the drawing has been filed pursuant to Rule 139 EPC. A decision on the request will be taken during the proceedings before the Examining Division (Guidelines for Examination in the EPO, A-V, 3.).

(54) FIBER PRE-OXIDIZATION DEVICE

(57) A fiber pre-oxidization device of the present disclosure basically has a transmitting unit and a microwave processing unit. The microwave processing unit is installed with at least one magnetron and a gas supplying unit, wherein the magnetron is disposed at an oven body of the transmitting unit, and the gas supplying unit is connected to the oven body. By focusing the microwave, an ultra-fast pre-oxidization process is applied on a fiber yarn bunch which continuously passes the oven body, and thus the fiber yarn bunch is processed to form an oxidation fiber yarn bunch. Thus, not only an oxidization time of an oxidation fiber can be reduced, but also the shell-core structure of the oxidation fiber can be reduced. Even, the oxidation fiber has no obvious shell-core. Accordingly, relatively positive and reliable means for increasing the performance of carbon fiber are provided.

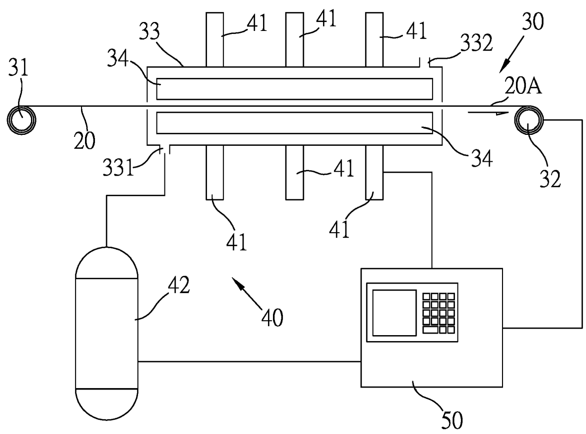


FIG. 2

EP 3 518 621 A1

Description

Technical Field

[0001] The present disclosure relates to a carbon fiber pre-oxidization technology, in particular to fiber pre-oxidization device of helping to enhance the performance of the carbon fiber.

Background Art

[0002] The carbon fiber is a new carbon material with 90 % carbon concentration, in which the organic fiber is performed with sequential thermal processes to transform to such carbon fiber. The carbon fiber has advantages of the high specific strength, the high specific modulus, the high conductivity and the thermal conductivity, the low thermal expansion coefficient, the low density, the high temperature resistance, the fatigue resistance, the creep resistance and the self-lubrication, and is an ideal function and structure material being widely used in the aerospace, civil aviation and transportation and other fields, thus having wide application prospects.

[0003] The carbon fiber preparing process using polyacrylonitrile (PAN) as the raw silk comprises polymerization, spinning, pre-oxidization and carbonization processes, wherein the pre-oxidization process is the key structure transformation stage of in the carbon fiber preparing process, and is the most time consuming stage in the thermal processing processes, which has the objective of transforming the linear macromolecular chains of polyacrylonitrile to the oxidation fiber with the thermal resistance structure, such that the oxidation fiber in the next carbonization will not burned and melted, and can maintain the fiber shape.

[0004] The structure transformation of the raw silk in the pre-oxidization process mainly determines the structure and the performance of the carbon fiber. During the industrial production, the pre-oxidization process with gradient temperature increasing manner is mostly used, and the proper gradient temperature range in the process is required. If the initial temperature is too low, it will not contribute to the pre-oxidization process, and the consuming time will be increased to cause the large cost. By contrast, if the initial temperature is too high, the heat emission of the severe reaction will make the macromolecular chains of polyacrylonitrile be melted, wherein the macromolecular chains of polyacrylonitrile have no thermal resistance. In addition, if the termination temperature is too high, the concentrated heat emission destroys the structure of the pre-oxidization silk, and makes the pre-oxidization silk over oxidized, and thus it is hard to prepare the carbon fiber with high strength. However, if the termination temperature is too low, the raw silk is not pre-oxidized sufficiently.

[0005] Moreover, when the pre-oxidization process is performed by heating, accompanying with the progressing of the pre-oxidization, since the heat is transmitted from the outer layer of the raw silk to the inner layer of the raw silk, the outer layer of the raw silk is firstly formed with an oxidization layer (i.e. the shell portion) having compact trapezoidal structure, and this prevents the oxygen from diffusing to the core portion of the raw silk. As such, as shown in FIG. 1, this causes an obviously differential shell-core structure between the oxidation layer 111 (shell portion) generated by oxidizing one fiber 11 in the oxidation fiber 10 and the core portion 112 being not oxidized, and a shell-core interface 113 exists between the oxidation layer 111 and the core portion 112. To check the shell-core structure, the scanning electron microscope (SEM) can photograph the substantial image to observe the cross section of the oxidation fiber and to respectively calculate the cross section areas of the oxidation layer, the core portion and the oxidation fiber. The core portion degree (%) can be used to evaluate the degree of the shell-core structure, wherein the core portion degree is the value that the cross section area of the core portion divides the summation of the cross section areas of the oxidation layer and the core portion, i.e. the value that the cross section area of the core portion divides the cross section area of the oxidation fiber. In addition, the physical properties of the oxidation fiber 10 and its manufactured carbon fiber, such as the tensile strength and the tensile modulus, are further determined by the oxidization degree and the cyclization degree of the oxidation fiber 10 or the oxidation layer 111. That is, the higher the oxidization degree and the cyclization degree of the oxidation fiber 10 or the oxidation layer 111 are, the higher the tensile strength and the tensile modulus of the carbon fiber manufactured by the oxidation fiber 10 are. The oxidation layer 111 presents the oxidation status and the structure thereof is compact, such that the manufactured carbon fiber thereof has the high tensile strength and the high tensile modulus. The core portion 112 presents the non-complete oxidation status or the non-oxidation status, and the structure thereof is loose, such that the manufactured carbon fiber thereof has the low tensile strength and the low tensile modulus. Since the oxidation layer 111 and the core portion 112 have the different oxidization degrees, the resulted shell-core structure is one important factor of lowering the tensile strength of the carbon fiber. Thus, how to shorten the pre-oxidization time in the pre-oxidization reaction process and how to simultaneously increase the pre-oxidization degree and eliminate the shell-core structure have importance of decreasing the carbon fiber manufacturing cost and increasing the performance (such as the tensile strength and the tensile modulus).

Summary of invention

[0006] Accordingly, the present disclosure provides a fiber pre-oxidization device which has the main objectives of shortening the oxidization time of the oxidation fiber, efficiently eliminating the shell-core structure of the oxidation fiber, and even making the oxidation fiber have no obvious shell-core structure.

[0007] A fiber pre-oxidization device of the present disclosure is used to pre-oxidize a fiber yarn bunch to form an oxidation fiber yarn bunch. The fiber yarn bunch is formed by merely one fiber, or alternatively, the fiber yarn bunch is formed by binding a plurality of fibers. The oxidation fiber yarn bunch is formed by merely one oxidation fiber, or alternatively, the oxidation fiber yarn bunch is formed by binding a plurality of oxidation fibers. The fiber pre-oxidization device basically comprises a transmitting unit and a microwave processing unit. The transmitting unit is installed with a feeding unit, an oven body and a winder unit, wherein the feeding unit is used to provides the fiber yarn bunch, the oven body is used to pre-oxidize the fiber yarn bunch which passes the oven body to form the oxidation fiber yarn bunch, and the winder unit is used to drag the fiber yarn bunch for continuous transmission and to receive the oxidation fiber yarn bunch. The microwave processing unit is disposed at the oven body and used to generate a microwave in interior of the of the oven body.

[0008] According to the above structure features, the microwave processing unit is further installed with a magnetron at the oven body for generating the microwave.

[0009] According to the above structure features, the microwave processing unit is further installed with a gas supplying unit for injecting gas with oxygen into the oven body.

[0010] According to the above structure features, the oven body further comprises a gas inlet and a gas outlet, wherein the gas supplying unit is connected to the gas inlet.

[0011] According to the above structure features, the oven body comprises a thermos unit.

[0012] According to the above structure features, two thermos units are respectively disposed at top and bottom sides of the interior of the oven body in respective to a transmission path of the fiber yarn bunch.

[0013] According to the above structure features, a thermos unit is disposed in the interior of the oven body for covering a transmission path of the fiber yarn bunch.

[0014] According to the above structure features, the winder unit, the magnetron and the gas supplying unit are electrically connected to a control unit.

[0015] According to the above structure features, microwave processing unit is installed with a plurality of magnetrons at the oven body for generating the microwave.

[0016] According to the above structure features, the magnetrons are disposed at single one side of the oven body.

[0017] According to the above structure features, the magnetrons are disposed at top and bottom sides of the oven body, and the magnetrons disposed on top and bottom sides of the oven body are arranged corresponding to each other

[0018] According to the above structure features, the magnetrons are disposed at top and bottom sides of the oven body, and the magnetrons disposed on top and bottom sides of the oven body are arranged in an offset manner

[0019] According to the above structure features, the magnetrons are disposed at top, bottom, left and right sides of the oven body.

[0020] According to the above structure features, the fiber yarn bunch is one of a polyacrylonitrile (PAN) fiber, a pitch fiber and other one organic fiber.

[0021] The fiber pre-oxidization device disclosed by the present disclosure mainly uses the microwave processing unit to focus the microwave to apply the ultra-fast pre-oxidization process on the fiber yarn bunch, so as to process the fiber yarn bunch to form the oxidation fiber. Thus, not only the oxidization time of the oxidation fiber is reduced, but also the oxidation layer in the oxidation fiber occupies more than 50 % of the cross section area of the oxidation fiber to efficiently reduce shell-core structure of the oxidation fiber. When the oxidation layer in the oxidation fiber occupies more than 80 % of the cross section area of the oxidation fiber, the oxidation fiber even has no obvious shell-core structure. Accordingly, relatively positive and reliable means for increasing the performance of carbon fiber are provided.

Brief description of drawings

[0022] The accompanying drawings are included to provide a further understanding of the present disclosure, and are incorporated in and constitute a part of this specification. The drawings illustrate exemplary embodiments of the present disclosure and, together with the description, serve to explain the principles of the present disclosure.

FIG. 1 is a schematic diagram showing a shell-core structure of the conventional oxidation fiber.
FIG. 2 is schematic diagram showing a structure of a fiber pre-oxidization device of the present disclosure.
FIG. 3 is a schematic diagram showing an oven body of the present disclosure.
FIG. 4 is a basic flow chart of an oxidation fiber manufacturing method by using the fiber pre-oxidization device of the present disclosure.

FIG. 5 is an oxidization degree curve diagram of oxidation fibers associated with fiber yarn bunches on which microwave focusing processes of 12 kW/m^2, 16 kW/m^2, 20 kW/m^2, and 24 kW/m^2 and the conventional heating process are performed.

FIG. 6 is a cyclization degree curve diagram of oxidation fibers associated with fiber yarn bunches on which the 24 kW/m^2 microwave focusing processes are respectively performed for 2 minutes, 4 minutes, 5 minutes, 10 minutes and 15 minutes.

FIG. 7 is a substantial cross section image of the oxidation fiber of the fiber yarn bunch on which the 24 kW/m^2 microwave focusing process is performed for 5 minutes.

FIG. 8 is a substantial cross section image of the oxidation fiber of the fiber yarn bunch on which the 24 kW/m^2 microwave focusing process is performed for 10 minutes.

FIG. 9 is a substantial cross section image of the oxidation fiber of the fiber yarn bunch on which the 24 kW/m^2 microwave focusing process is performed for 15 minutes.

FIG. 10 is a schematic diagram showing a structure of an oxidation fiber of the present disclosure.

Description of embodiments

[0023] The present disclosure mainly provides fiber pre-oxidization device which can shorten the oxidization time of the oxidation fiber, efficiently eliminate the shell-core structure of the oxidation fiber, and even make the oxidation fiber have no obvious shell-core structure. Please refer to FIG. 2 and FIG. 3 simultaneously. As shown in FIG. 2, the fiber pre-oxidization device of the present disclosure is adapted to pre-oxidize a fiber yarn bunch 20 to form an oxidation fiber yarn bunch 20A. The fiber yarn bunch 20 is formed by merely one fiber (not shown in the drawings), or alternatively, the fiber yarn bunch is formed by binding a plurality of fibers. The oxidation fiber yarn bunch 20A is formed by merely one oxidation fiber 21, or alternatively, the oxidation fiber yarn bunch is formed by binding a plurality of oxidation fibers 21. The fiber pre-oxidization device comprises a transmitting unit 30 and a microwave processing unit 40.

[0024] The transmitting unit 30 is installed with a feeding unit 31, an oven body 33, a winder unit 32, a gas inlet 331 and a gas outlet 332. The feeding unit 31 is used to provide the fiber yarn bunch 20. The oven body 33 is used to pre-oxidize the fiber yarn bunch 20 which passes the oven body 33 to form the oxidation fiber yarn bunch 20A. The winder unit 32 is used to drag the fiber yarn bunch 20 for continuous transmission and to receive the oxidation fiber yarn bunch20A. The gas inlet 331 is disposed at a front end of the oven body 33, and the gas outlet 332 is disposed at a back end of the oven body 33.

[0025] The microwave processing unit 40 is installed with a magnetron 41 and a gas supplying unit 42, wherein the magnetron 41 is disposed at the oven body 33 for generating a microwave, and the gas supplying unit 42 is used to inject gas with oxygen into the oven body 33. The gas supplying unit 42 is connected to the gas inlet 331 of the oven body 33, the gas with oxygen is injected into the oven body 33 via the gas inlet 331, and the gas outlet 332 of the oven body 33 is used to exhaust the gas.

[0026] The winder unit 32, the magnetron 41 and the gas supplying unit 42 are electrically connected to a control unit 50. Operations of the winder unit 32, the magnetron 41 and the gas supplying unit 42 are controlled by the control unit 50, and the spinning speed of the winder unit 32, the power of the magnetron 41 and flux of the gas supplying unit 42 are determined according to the property of the processed fiber yarn bunch 20 or the product specification.

[0027] Preferably, the microwave processing unit 40 is installed with the magnetrons 41 at the oven body 33, wherein the magnetrons 41 are disposed at top and bottom sides of the oven body 33, and the magnetrons 41 disposed on the top and bottom sides of the oven body 33 are arranged corresponding to each other or in an offset manner, or alternatively, the magnetrons 41 disposed at single one side (top or bottom side) of the oven body 33. As shown in FIG. 2, the magnetrons 41 are disposed on top and bottom sides of the oven body 33, and the magnetrons 41 disposed on the top side are arranged corresponding to the magnetrons 41 disposed on the bottom side. Optimally, as shown in FIG. 3, the magnetrons 41 disposed on the top side are arranged corresponding to the magnetrons 41 disposed on the bottom side, such that by simultaneously and uniformly irradiating the microwave on upper and lower portions of the fiber yarn bunch 20 which passes the oven body 33, the length of the oven body 33 can be further reduced, and the process time can be shortened to increasing the production speed.

[0028] The transmitting unit 30 of the present disclosure can be further installed with a thermos unit 34 in the interior of the oven body 33, and as shown in FIG. 2, the thermal storage effect of the thermos unit 34 can be utilized, such that the interior of the oven body 33 can be keep at the predetermined operation temperature to achieve the objective of power saving.

[0029] When the transmitting unit 30 of the present disclosure is implemented, as shown in FIG. 2, the two thermos units 34 are respectively disposed at top and bottom sides of the interior of the oven body 33 in respective to a transmission path of the fiber yarn bunch 20; or alternatively, as shown in FIG. 3, the thermos unit 34 is disposed in the interior of the oven body 33 for covering the transmission path of the fiber yarn bunch 20, such that the fiber yarn bunch 20 is heated uniformly. According to the above possible embodiments, the thermos unit 34 can be selected from at least one of a metal oxide, a carbide and a high microwave sensitive material. In FIG. 3, the feeding unit 31 provides the parallel arranged fiber yarn bunches 20 into the oven body 33.

[0030] When the microwave processing unit 40 of the present disclosure is implemented, as shown in FIG. 2, the magnetrons 41 are respectively disposed at top and bottom sides of the transmission path of the fiber yarn bunch 20; or alternatively, the magnetrons 41 are disposed for covering the transmission path of the fiber yarn bunch 20, such that the microwave focusing process is uniformly performed on the fiber yarn bunch 20. That is, the magnetrons 41 are disposed at the top, bottom, left and right sides of the oven body 33.

[0031] When the fiber pre-oxidization device is used, the fiber pre-oxidization device can execute an oxidation fiber manufacturing method to efficiently shorten the oxidization time of the oxidation fiber, efficiently to eliminate the shell-core structure of the oxidation fiber, and even to make the oxidation fiber has no obvious shell-core structure. The oxidation fiber manufacturing method can be referred in FIG. 4, and comprises steps as follows.

[0032] Step a: providing the transmitting unit 30 and the microwave processing unit 40.

[0033] Step b: providing the fiber yarn bunch 20, disposing the fiber yarn bunch 20 on the transmitting unit 30, and making the transmitting unit 30 drive the fiber yarn bunch 20 to pass the microwave processing unit 40. For example, the winded fiber yarn bunch 20 can be disposed at the transmitting unit 30, so as to be continuously driven by the transmitting unit 30 to pass the operation region of the microwave processing unit 40. In the embodiment, the winded fiber yarn bunch 20 is disposed at the feeding unit 31, and the tail end of the fiber yarn bunch 20 is guided to pass the oven body 33 and then fixed on the winder unit 32, wherein the fiber yarn bunch 20 can be is one of a polyacrylonitrile (PAN) fiber, a pitch fiber and other one organic fiber.

[0034] Step c: activating the microwave processing unit 40, and using the microwave processing unit 40 to generate a microwaving condition. The microwaving condition comprises: a microwave frequency being 300 MHz through 300,000 MHz; a microwave power being 1 kW/m^2 through 1000 kW/m^2; an operation temperature being 100 °C through 600 °C; and gas atmosphere being at least one of oxygen, air and ozone. The gas atmosphere is the above gas with oxygen. In the embodiment, the gas supplying unit 42 is used to inject the gas with oxygen into the interior of the oven body 33.

[0035] Step d: activating the transmitting unit 30, using the transmitting unit 30 to drive the fiber yarn bunch 20 to be exposed in the microwaving condition for a processing time, so as to transform the fiber yarn bunch 20 to the oxidation fiber yarn bunch 20A. For example, the fiber yarn bunch 20 is driven by the transmitting unit 30 to pass the operation region of the microwave processing unit 40 at the speed which the microwave focusing process is continuously applied for 1 minute through 40 minutes, and that is the processing time is 1 minute through 40 minutes. In the embodiment, the fiber yarn bunch 20 is driven by the transmitting unit 30 to pass the oven body 33 to form the oxidation fiber yarn bunch 20A at the speed which the microwave focusing process is continuously applied for 1 minute through 40 minutes. In addition, the fiber yarn bunch 20 in the oven body 33 is winded and repeated to pass the oven body 33 to the oxidation fiber yarn bunch 20A at the speed which the microwave focusing process is continuously applied for 1 minute through 40 minutes. For example, the fiber yarn bunch 20 at the front end of the oven body 33 enters the interior of the oven body 33, and then is transmitted to the back end of the oven body 33. Next, the fiber yarn bunch 20 is transmitted from the back end of the oven body 33 to the front end of the oven body 33, and then is transmitted from the front end of the oven body 33 to the back end of the oven body 33 again. The manner is used to repeat and wind the fiber yarn bunch 20 until the requirements is satisfied, and then the fiber yarn bunch 20 is sent out from the back end of the oven body 33 to form the oxidation fiber yarn bunch 20A. The above used repeating and winding manner can sufficiently reduce the required length of the oven body 33.

[0036] Accordingly, under the operation of the transmitting unit 30, the fiber yarn bunch 20 is driven to pass the operation region of the microwave processing unit 40 at the predetermined speed. During the progress which the fiber yarn bunch 20 passes the operation region of the microwave processing unit 40, the microwave focusing process is continuously used to apply the ultra-fast pre-oxidization process on the fiber yarn bunch 20, so as to process the fiber yarn bunch 20 to form the oxidation fiber yarn bunch 20A. The fiber yarn bunch 20 is formed by merely one fiber, or alternatively, the fiber yarn bunch 20 is formed by binding a plurality of fibers. The oxidation fiber yarn bunch 20A is formed by merely one oxidation fiber, or alternatively, the oxidation fiber yarn bunch 20A is formed by binding a plurality of oxidation fibers. The fiber pre-oxidization device of the present disclosure can be used to pre-oxidize the fiber o the fiber yarn bunch 20 to form the oxidation fiber 21.

[0037] Referring to FIG. 5, the fiber yarn bunches 20 are respectively applied without the microwave and with the microwave focusing processes of 12 kW/m^2, 16 kW/m^2, 20 kW/m^2 and 24 kW/m^2 microwave powers, and it can obtain the result that the microwave focusing process of 24 kW/m^2 is applied to the fiber yarn bunch 20 for 10 minutes to make the oxidization degree of the oxidation fiber 21 in the oxidation fiber yarn bunch 20A reach 100%. Corresponding to the fiber yarn bunch 20, the oxidation fiber yarn bunch 20A is formed by merely one oxidation fiber, or alternatively, the oxidation fiber yarn bunch 20A is formed by binding a plurality of oxidation fibers. Similarly, the microwave focusing process of 20 kW/m^2 is applied to the fiber yarn bunch 20 for 15 minutes to make the oxidization degree of the oxidation fiber 21 in the oxidation fiber yarn bunch 20A reach 100%. The microwave focusing process of 16 kW/m^2 is applied to the fiber yarn bunch 20 for 25 minutes to make the oxidization degree of the oxidation fiber 21 in the oxidation fiber yarn bunch 20A reach 100%. However, even the microwave focusing process of 12 kW/m^2 is applied to the fiber yarn bunch 20 for 40 minutes, the oxidization degree of the oxidation fiber 21 in the oxidation fiber yarn bunch 20A still cannot reach 100%, but can reach 89%. If the conventional heating process at 270 °C without the microwave is applied to heat the fiber yarn bunch 20 for 40 minutes, the oxidization degree of the oxidation fiber 21 can merely reaches 70%. Thus, compared the microwaving process provided by the present disclosure and the conventional heating process, the present disclosure can efficiently increase the oxidization degree of the oxidation fiber 21 and shorten the process time. Especially, when the microwave focusing process of 24 kW/m^2 is applied to the fiber yarn bunch 20 for 10 minutes, the oxidization degree of the oxidation fiber 21 in the oxidation fiber yarn bunch 20A can reach 100%, and thus the 24 kW/m^2 and 40 minutes are the best process conditions of the oxidization stage.

[0038] Referring to FIG. 6, the microwave focusing processes of 24 kW/m^2 are performed on the fiber yarn bunch 20 respectively for 2 minutes, 4 minutes, 5 minutes, 10 minutes and 15 minutes for checking the cyclization degrees of the formed oxidation fibers 21. The cyclization degree of the oxidation fiber 21 reaches 100 % after 5 minutes are elapsed. Thus, the required time of 5 minutes that the cyclization degree reaches 100 % is less than the required time of 10 minutes the oxidization degree reaches 100 %. Referring to FIG. 7, FIG. 8 and FIG. 9 simultaneously, the cross sections of oxidation fibers 21 associated with the oxidation fiber yarn bunches 20A formed by being processed with the microwave focusing processes of 24 kW/m^2 respectively for 2 minutes, 4 minutes, 5 minutes, 10 minutes and 15 minutes 4 kW/m^2 are photographed by the scanning electron microscope to obtain the substantial cross section images. It is found that the oxidation layer 211 occupies more than 99.0 % of the oxidation fiber 21, or cross section area of the oxidation layer 211 occupies more than 99.0 % of that of the oxidation fiber 21, and no obvious shell-core structure exists.

[0039] Refer to Table 1 and Table 2 simultaneously. Table 1 is a comparison table showing the measured tensile strengths of the fiber yarn bunches 20, the oxidation fiber yarn bunches 20A and the carbon fiber yarn bunches formed by the next carbonization, wherein two sets of the fiber yarn bunches 20, the oxidation fiber yarn bunches 20A and the carbon fiber yarn bunches are respectively processed by the conventional electro thermal tube heating process and the microwaving process of the oxidation fiber manufacturing method associated with fiber pre-oxidization device of the present disclosure. Table 2 is a comparison table showing the measured tensile moduli of the fiber yarn bunches 20, the oxidation fiber yarn bunches 20A and the carbon fiber yarn bunches formed by the next carbonization, wherein two sets of the fiber yarn bunches 20, the oxidation fiber yarn bunches 20A and the carbon fiber yarn bunches are respectively processed by the conventional electro thermal tube heating process and the microwaving process of the oxidation fiber manufacturing method associated with fiber pre-oxidization device of the present disclosure. Regarding the conventional electro thermal tube heating process, the processing condition is the oven body temperature of 270 °C and the processing time of 40 minutes, and the obtained results of the physical properties are called "comparative example 1". Regarding the microwaving process of the present disclosure, the processing condition is the oven body temperature of 220 °C, the microwave frequency of 2450 MHz, the microwave power of 24 kW/m^2 and the processing time of 10 minutes, and the obtained results of the physical properties are called "embodiment 1". In both of the comparative example 1 and the embodiment 1, the fiber yarn bunches 20 are made of polyacrylonitrile.

Table 1

[0040]

Table 1

the tensile strength (MPa)	the fiber yarn bunch	the oxidation fiber yarn bunch	the carbon fiber yarn bunch
comparative example 1	865	221	2824
embodiment 1	865	164	3675

[0041] In Table 1, the embodiment 1 shows the tensile strength of the final carbon fiber yarn bunch carbonized by the oxidation fiber yarn bunch processed with the microwaving process of the present disclosure is 1.3 times of that in the comparative example 1 (i.e. 3675 divides 2824), and that is the tensile strength has the improvement of 30 %. The microwaving process can oxidize polyacrylonitrile more complete, and the tensile strength of the oxidation fiber yarn bunch associated with the microwaving process is slightly less than that of the oxidation fiber yarn bunch associated with the conventional electro thermal tube heating process, which is another one evidence that the microwaving process of the present disclosure can further increase the oxidization degree of the fiber yarn bunch.

Table 2

[0042]

Table 2

the tensile modulus (GPa)	the fiber yarn bunch	the oxidation fiber yarn bunch	the carbon fiber yarn bunch
comparative example 1	8.82	6.03	194.4
embodiment 1	8.82	6.92	227.1

[0043] In Table 2, the embodiment 1 shows the tensile modulus of the final carbon fiber yarn bunch carbonized by the oxidation fiber yarn bunch processed with the microwaving process of the present disclosure is 1.17 times of that in the comparative example 1 (i.e. 227.1 divides 194.4), and that is the tensile modulus has the improvement of 17 %.

[0044] Accordingly, compared with the oxidation fiber yarn bunches respectively generated by the fiber yarn bunches on which the conventional heating process and the microwaving process of the present disclosure are performed, the microwaving process of the present disclosure can reduce the required time of the conventional heating process from 40 minutes to 10 minutes, thus the process efficiency is increased with three times, and the process time is reduced. Compared to the conventional heating process, the present disclosure can enhance the 30 % tensile strength and the 17 % tensile modulus of carbon fiber yarn bunch. Compared to the conventional heating process, the present disclosure can further make the cross section area of the oxidation layer 2111 of the oxidation fiber 21 in the oxidation fiber yarn bunch 20A occupy the 99.0 % cross section area of the oxidation fiber 21, such that no obvious shell-core structure exists. The cross section of the oxidation fiber yarn bunch 20A is more uniform, and thus the tensile strength and the tensile modulus of the carbon fiber yarn bunch are increased. The relatively positive and reliable means for enhancing the carbon fiber performance are therefore provided.

[0045] When the present disclosure is implemented, the 24 kW/m^2 microwave focusing process is applied to process the fiber yarn bunch for 5 minutes through 10 minutes, preferably. Accordingly, the present disclosure is adapted to the continuous carbon fiber yarn bunch generation manner that the fiber yarn bunch 20 passes the oven body 33 without the reception and winding of the winder unit 32 and the carbonization is next performed, or alternatively, the present disclosure is adapted to the generation manner that the winded fiber yarn bunch 20 is winded out by the feeding unit 31 and received and winded by the winder unit 32.

[0046] Refer to FIG. 5 again, after the microwave focusing process of the 12 kW/m^2microwave power at 220 °C is applied to the fiber yarn bunch 20 for 40 minutes, the oxidation degree of the oxidation fiber 21 reaches 89 %. However, after the conventional heating process at 270 °C is applied to the fiber yarn bunch 20 for 40 minutes without the microwaving process, the oxidation degree of the oxidation fiber 21 merely reaches 70 %. Therefore, compared to the conventional heating process, the present disclosure can obtain the higher oxidation degree at the lower temperature, thus preventing the thermal waste.

[0047] Refer to Table 3, and Table 3 is a comparison table showing the measured tensile strengths of the fiber yarn bunches 20, the oxidation fiber yarn bunches 20A and the carbon fiber yarn bunches formed by the next carbonization, wherein several sets of the fiber yarn bunches 20, the oxidation fiber yarn bunches 20A and the carbon fiber yarn bunches are respectively processed by the conventional electro thermal tube heating process and the microwaving processes of the oxidation fiber manufacturing method associated with fiber pre-oxidization device of the present disclosure. Regarding the conventional electro thermal tube heating process, the processing condition is the oven body temperature of 270 °C and the processing time of 40 minutes, and the obtained results of the physical properties are called "comparative example 1". Regarding the microwaving processes of the present disclosure, the processing conditions are the oven body temperature of 220 °C, the microwave frequency of 2450 MHz and the processing time of 10 minutes, and the obtained results of the physical properties associated with 24 kW/m^2, 22 kW/m^2, 16 kW/m^2 and 15 kW/m^2 microwave powers are called "embodiment 1", "embodiment 2", "embodiment 3", "embodiment 4" and "embodiment 5". In all of comparative example 1 and embodiments 1 through 5, the fiber yarn bunches 20 are made of polyacrylonitrile. In addition, the cross sections of the oxidation fibers 21 of the oxidation fiber yarn bunches 20A associated with all of the comparative example 1 and the embodiments 1 through 5 are photographed by the scanning electron microscope to obtain the substantial cross section images, and the calculated values that the cross section areas of the oxidation layer 211 respectively divide the cross section areas of the oxidation fiber 21, i.e. the ratios which the oxidation layers 211 occupy the oxidation fiber 21, are also listed in Table 3.

Table 3

[0048]

Table 3

number	the tensile strength of the fiber yarn bunch (MPa)	the microwave power (kW/m^2)	the tensile strength of the carbon fiber yarn bunch (MPa)	the tensile strength ratio	R*
comparative example 1	865	0	2824	1	40%
embodiment 1	865	24	3675	1.30	99.0%
embodiment 2	865	22	3580	1.27	91.3%
embodiment 3	865	20	3486	1.23	82.7%
embodiment 4	865	16	3298	1.17	61.5%
embodiment 5	865	15	3204	1.13	51.2%
R*: the value that the cross section area of the oxidation layer divides cross section area of the oxidation fiber					

[0049] In Table 3, embodiment 5 shows the tensile strength of the final carbon fiber yarn bunch carbonized by the oxidation fiber yarn bunch processed with the microwaving process of the present disclosure is 1.13 times of that in the comparative example 1, and that is the tensile strength has the improvement of 13 %. In embodiment 5, the value that the cross section area of the oxidation layer 211 divides the cross section area of the oxidation fiber 21 is 51.2 %, i.e. the oxidation layer 211 occupies the 51.2 % oxidation fiber 21. Embodiment 4 shows the tensile strength of the final carbon fiber yarn bunch carbonized by the oxidation fiber yarn bunch processed with the microwaving process of the present disclosure is 1.17 times of that in the comparative example 1, and that is the tensile strength has the improvement of 17 %. In embodiment 4, the value that the cross section area of the oxidation layer 211 divides the cross section area of the oxidation fiber 21 is 61.5 %, i.e. the oxidation layer 211 occupies the 61.5 % oxidation fiber 21. Embodiment 3 shows the tensile strength of the final carbon fiber yarn bunch carbonized by the oxidation fiber yarn bunch processed with the microwaving process of the present disclosure is 1.23 times of that in the comparative example 1, and that is the tensile strength has the improvement of 23 %. In embodiment 3, the value that the cross section area of the oxidation layer 211 divides the cross section area of the oxidation fiber 21 is 82.7 %, i.e. the oxidation layer 211 occupies the 82.7 % oxidation fiber 21. Embodiment 2 shows the tensile strength of the final carbon fiber yarn bunch carbonized by the oxidation fiber yarn bunch processed with the microwaving process of the present disclosure is 1.27 times of that in the comparative example 1, and that is the tensile strength has the improvement of 27 %. In embodiment 2, the value that the cross section area of the oxidation layer 211 divides the cross section area of the oxidation fiber 21 is 91.3 %, i.e. the oxidation layer 211 occupies the 91.3 % oxidation fiber 21. Embodiment 1 shows the tensile strength of the final carbon fiber yarn bunch carbonized by the oxidation fiber yarn bunch processed with the microwaving process of the present disclosure is 1.3 times of that in the comparative example 1, and that is the tensile strength has the improvement of 30 %. In embodiment 1, the value that the cross section area of the oxidation layer 211 divides the cross section area of the oxidation fiber 21 is 99.0 %, i.e. the oxidation layer 211 occupies the 99.0 % oxidation fiber 21.

[0050] Thus, the oxidation fiber 21 generated by the present disclosure comprises the oxidation layer 211 and a core portion 212, wherein the oxidation layer 211 covers the outer side of the core portion 212, and the oxidation layer 211 occupy the more than 50 % oxidation fiber 21, or the cross section area of the oxidation layer 211 occupy the more than 50 % cross section area of the oxidation fiber 21. As shown in FIG. 10, the oxidation layer 211 occupy the more than 80 % oxidation fiber 21, or the cross section area of the oxidation layer 211 occupy the more than 80 % cross section area of the oxidation fiber 21.

[0051] Of course, since the oxidation fiber 21 of the present disclosure is formed under the microwaving condition, the oxidation layer 211 is an microwaved oxidation layer, and the oxidation layer 211 of the oxidation fiber 21 in the oxidation fiber yarn bunch 20A occupies the at least 50 % oxidation fiber 21.

[0052] When the present disclosure is implemented, the fiber yarn bunch 20 can be one of polyacrylonitrile, pitch and other organic fibers. Certainly, after the microwave focusing process of 24 kW/m^2 microwave power is applied on the fiber yarn bunch 20 for 10 minutes to obtain the oxidation fiber, the oxidation layer 211 occupy the 99.0 % oxidation fiber 21, or the cross section area of the oxidation layer 211 occupy the 99.0 % cross section area of the oxidation fiber 21

[0053] Compared to the prior art, the fiber pre-oxidization device disclosed by the present disclosure mainly uses the microwave processing unit to focus the microwave to apply the ultra-fast pre-oxidization process on the fiber yarn bunch, so as to process the fiber yarn bunch to form the oxidation fiber. Thus, not only the oxidization time of the oxidation fiber is reduced, but also the oxidation layer in the oxidation fiber occupies more than 50 % of the cross section area of the oxidation fiber to efficiently reduce shell-core structure of the oxidation fiber. Even, no obvious shell-core structure exists in the oxidation fiber. Accordingly, relatively positive and reliable means for increasing the performance of carbon fiber are provided.

Claims

1. A fiber pre-oxidization device, used to pre-oxidize a fiber yarn bunch (20) to form an oxidation fiber yarn bunch (20A); the fiber yarn bunch (20) is formed by merely one fiber, or alternatively, the fiber yarn bunch (20) is formed by binding a plurality of fibers; the oxidation fiber yarn bunch (20A) is formed by merely one oxidation fiber, or alternatively, the oxidation fiber yarn bunch (20A) is formed by binding a plurality of oxidation fibers; the fiber pre-oxidization device is **characterized in**, basically comprising:

    a transmitting unit (30), installed with a feeding unit (31), an oven body (33) and a winder unit (32), wherein the feeding unit (31) is used to provides the fiber yarn bunch (20), the oven body (33) is used to pre-oxidize the fiber yarn bunch (20) which passes the oven body (33) to form the oxidation fiber yarn bunch (20A), and the winder unit (32) is used to drag the fiber yarn bunch (20) for continuous transmission and to receive the oxidation fiber yarn bunch (20A); and
    a microwave processing unit (40), disposed at the oven body (33), used to generate a microwave in interior of the of the oven body (33).

2. The fiber pre-oxidization device according to claim 1, wherein the microwave processing unit (40) is further installed with a magnetron (41) at the oven body (33) for generating the microwave.

3. The fiber pre-oxidization device according to claim 2, wherein the microwave processing unit (40) is further installed with a gas supplying unit (42) for injecting gas with oxygen into the oven body (33).

4. The fiber pre-oxidization device according to claim 3, wherein the oven body (33) further comprises a gas inlet (331) and a gas outlet (332), and the gas supplying unit (42) is connected to the gas inlet (331).

5. The fiber pre-oxidization device according to claim 1, wherein the oven body (33) further comprises a thermos unit (34).

6. The fiber pre-oxidization device according to claim 1, wherein two thermos units (34) are respectively disposed at top and bottom sides of the interior of the oven body (33) in respective to a transmission path of the fiber yarn bunch (20).

7. The fiber pre-oxidization device according to claim 1, wherein a thermos unit (34) is disposed in the interior of the oven body (33) for covering a transmission path of the fiber yarn bunch (20).

8. The fiber pre-oxidization device according to claim 3, wherein the winder unit (32), the magnetron (41) and the gas supplying unit (42) are electrically connected to a control unit (50).

9. The fiber pre-oxidization device according to claim 1, wherein the microwave processing unit (40) is installed with a plurality of magnetrons (41) at the oven body (33) for generating the microwave.

10. The fiber pre-oxidization device according to claim 9, wherein the magnetrons (41) are disposed at single one side of the oven body (33).

11. The fiber pre-oxidization device according to claim 9, wherein the magnetrons (41) are disposed at top and bottom sides of the oven body (33), and the magnetrons (41) disposed on top and bottom sides of the oven body (33) are arranged corresponding to each other.

12. The fiber pre-oxidization device according to claim 9, wherein the magnetrons (41) are disposed at top and bottom

sides of the oven body (33), and the magnetrons (41) disposed on the top and bottom sides of the oven body (33) are arranged in an offset manner.

13. The fiber pre-oxidization device according to claim 9, wherein the magnetrons (41) are disposed at top, bottom, left and right sides of the oven body (33).

14. The fiber pre-oxidization device according to claim 1, wherein the fiber yarn bunch (20) is one of a polyacrylonitrile (PAN) fiber and a pitch fiber.

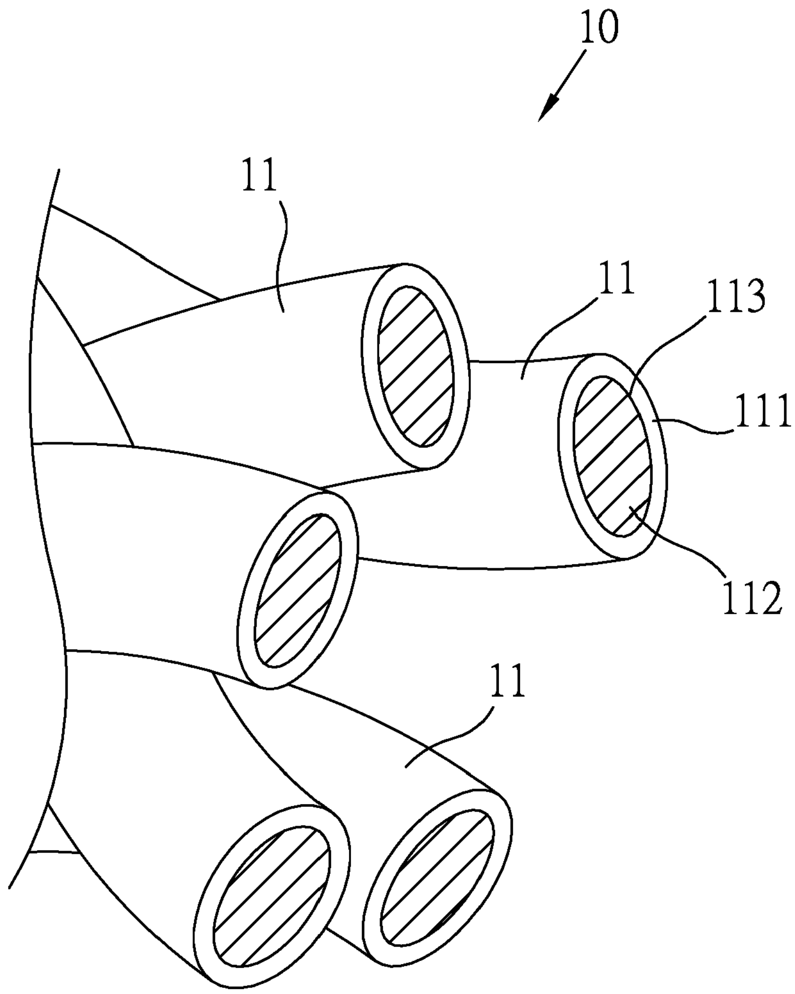


FIG. 1 Prior Art

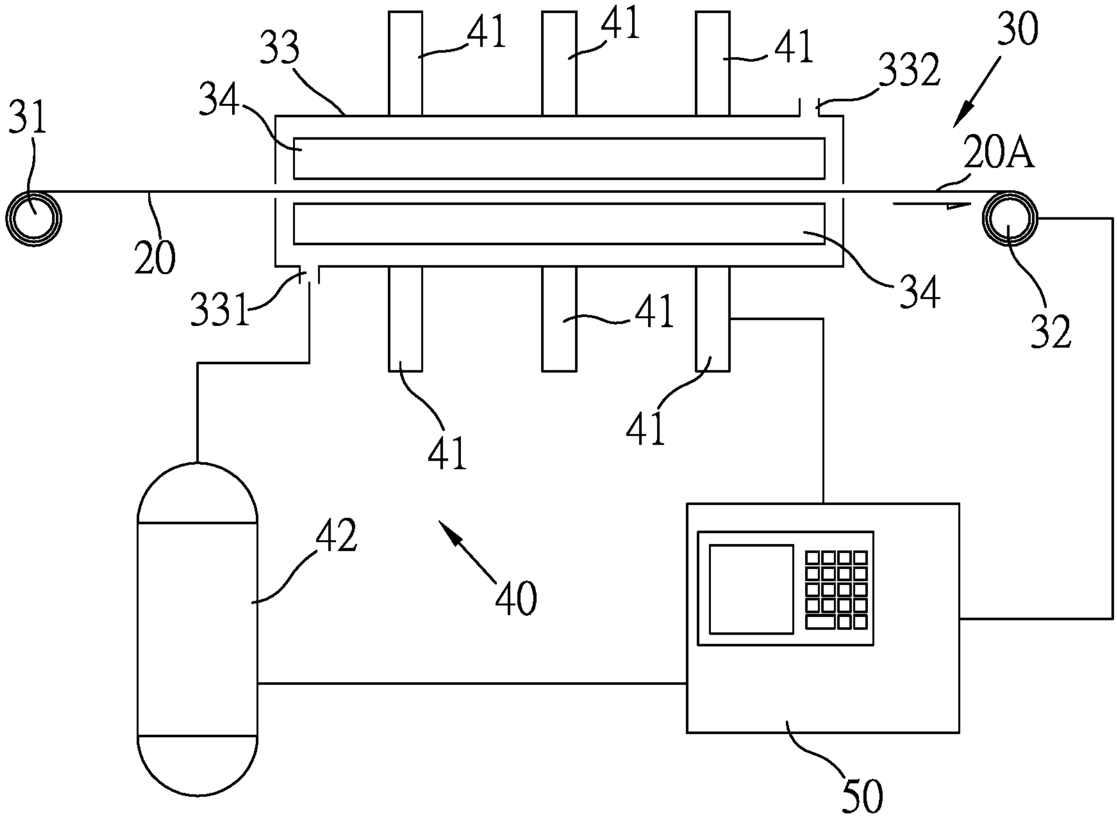


FIG. 2

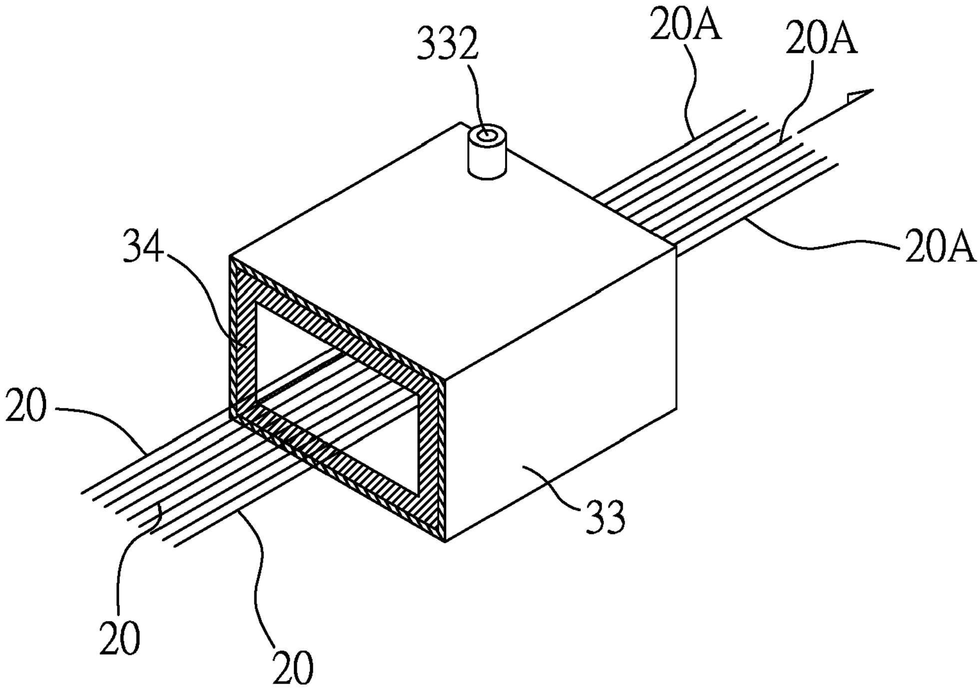


FIG. 3

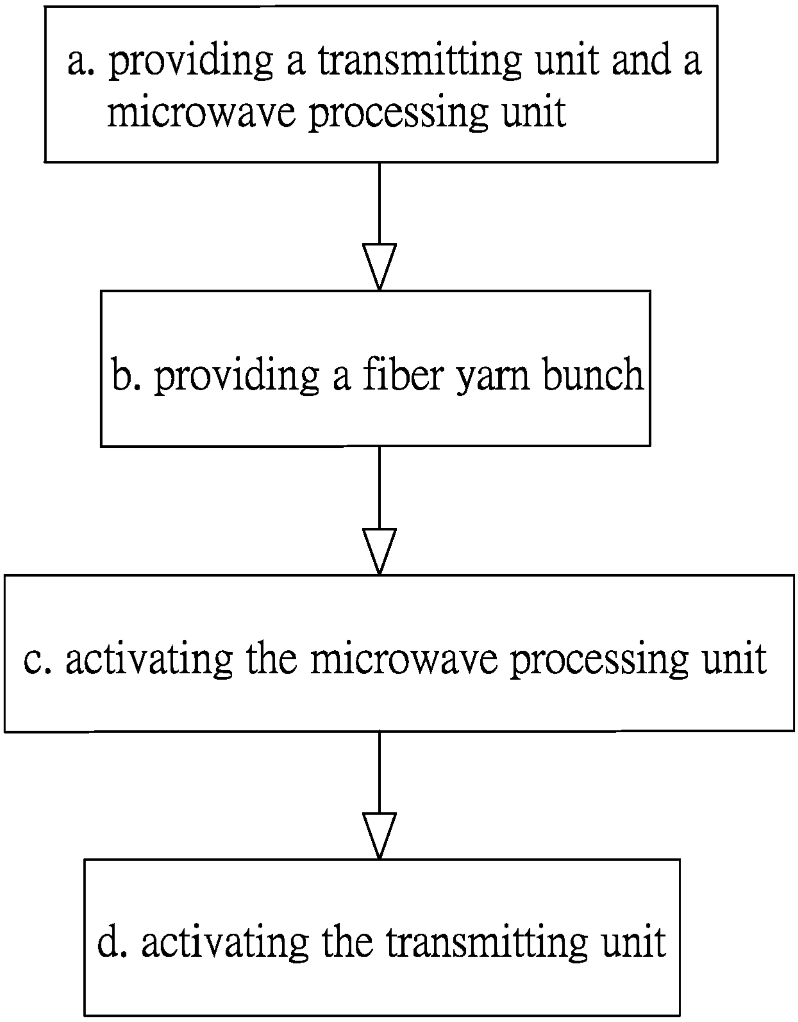

a. providing a transmitting unit and a microwave processing unit
b. providing a fiber yarn bunch
c. activating the microwave processing unit
d. activating the transmitting unit

FIG.4

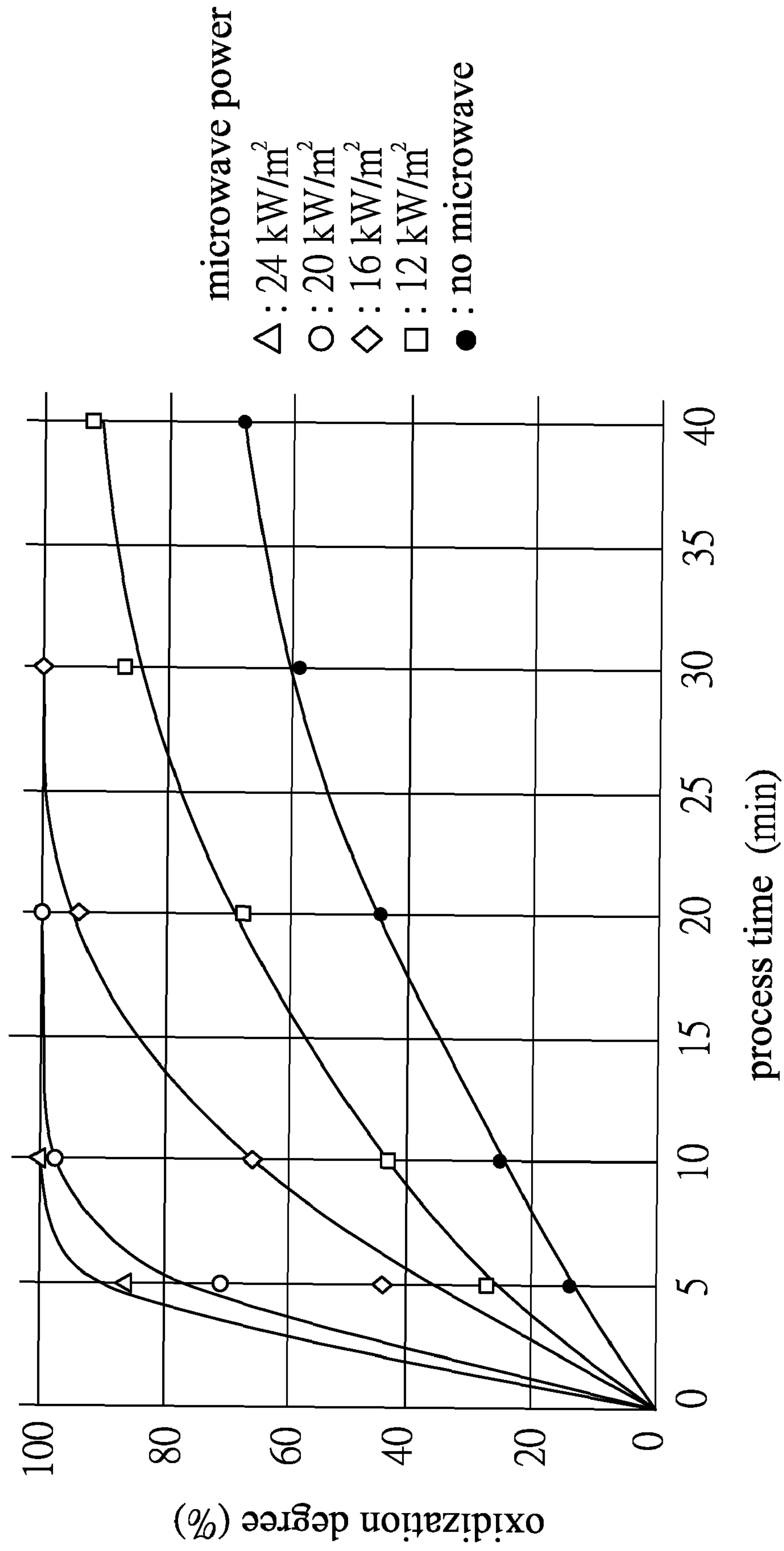
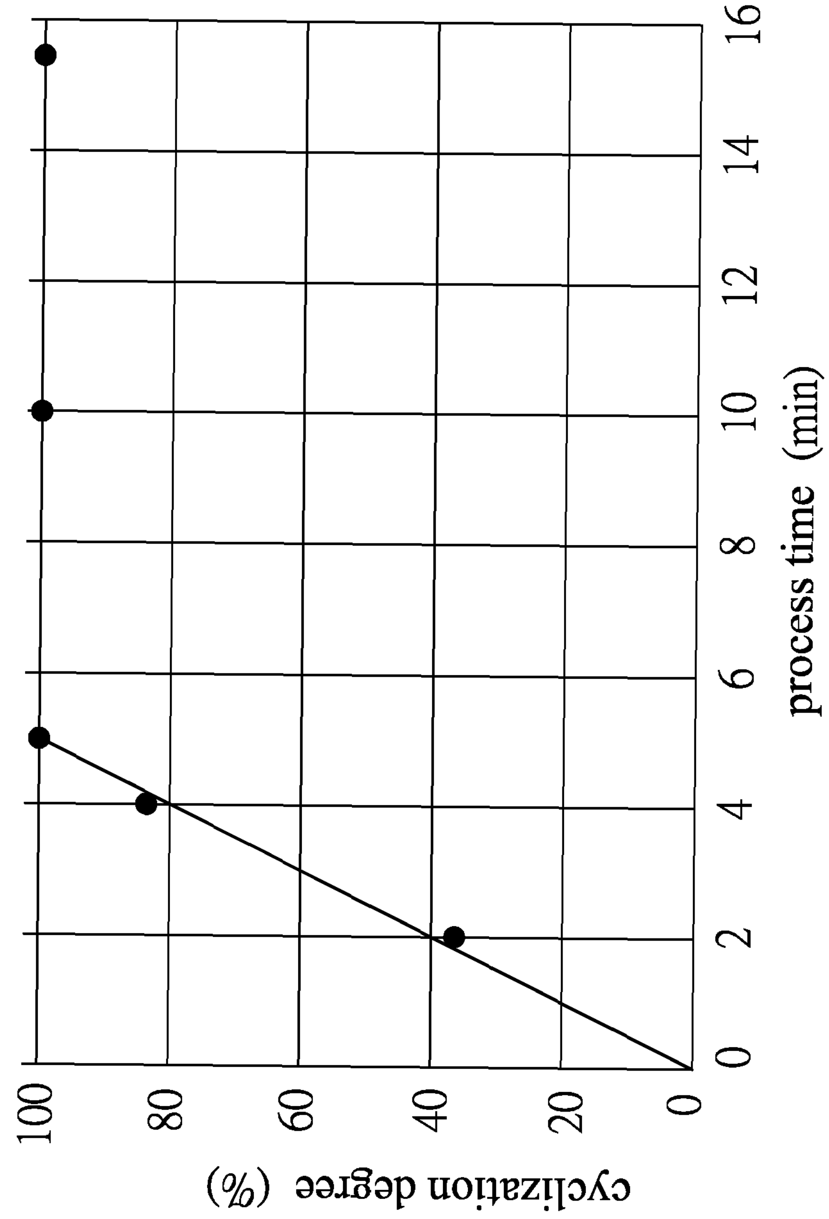

microwave power
△ : 24 kW/m²
○ : 20 kW/m²
◇ : 16 kW/m²
□ : 12 kW/m²
● : no microwave
oxidization degree (%)
100
80
60
40
20
0
0
5
10
15
20
25
30
35
40
process time (min)

FIG. 5



cyclization degree (%)
100
80
60
40
20
0
process time (min)
0
2
4
6
8
10
12
14
16

FIG. 6

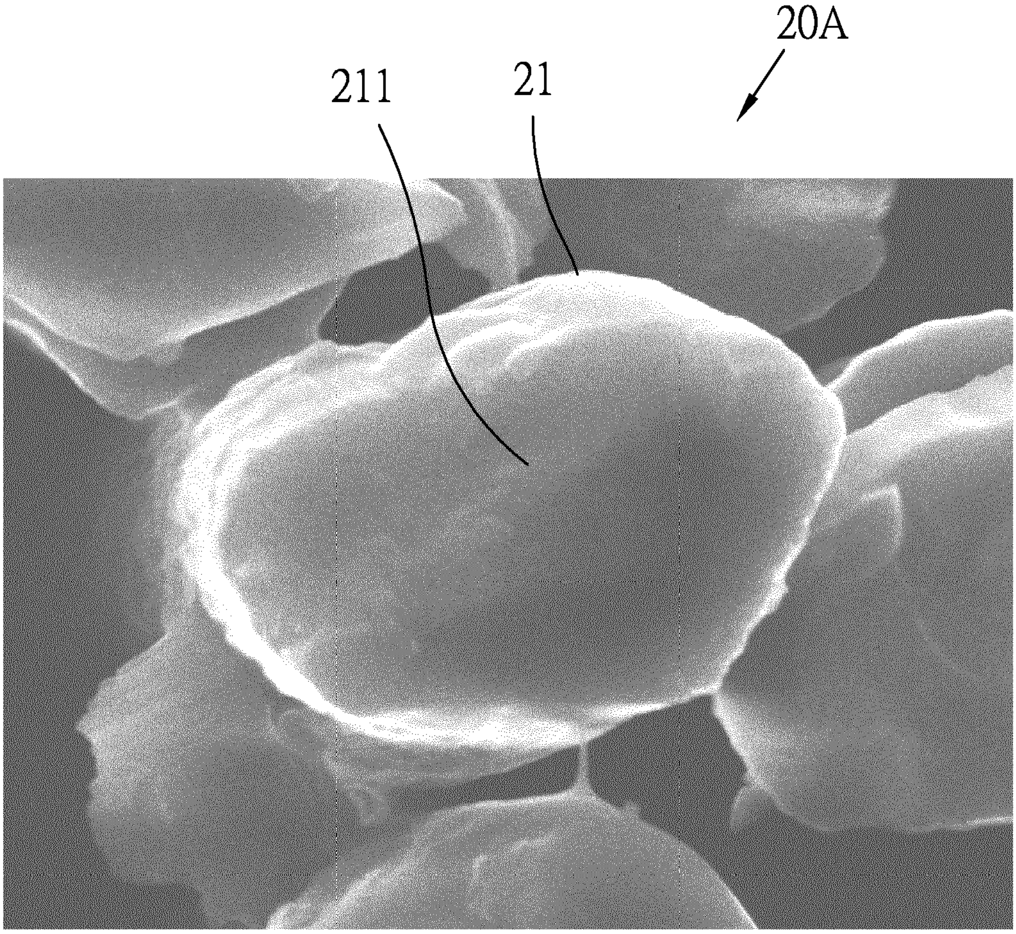


FIG. 7

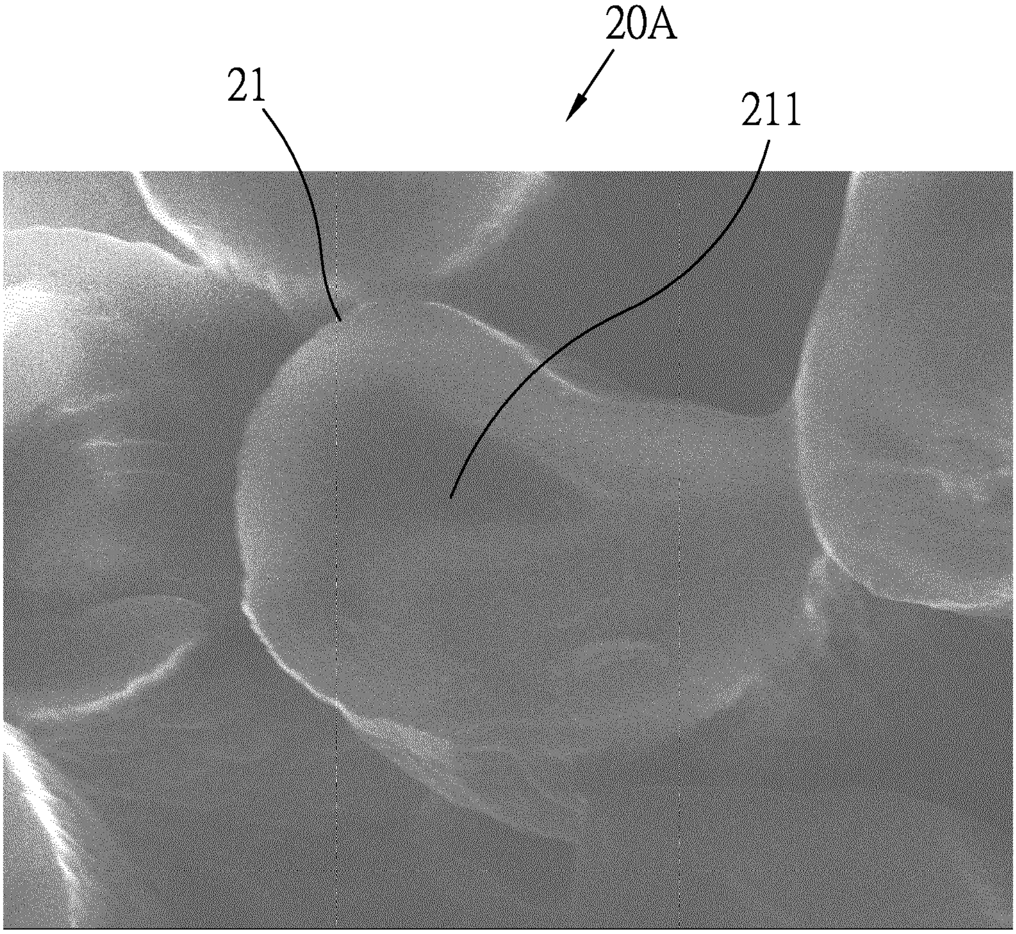


FIG. 8

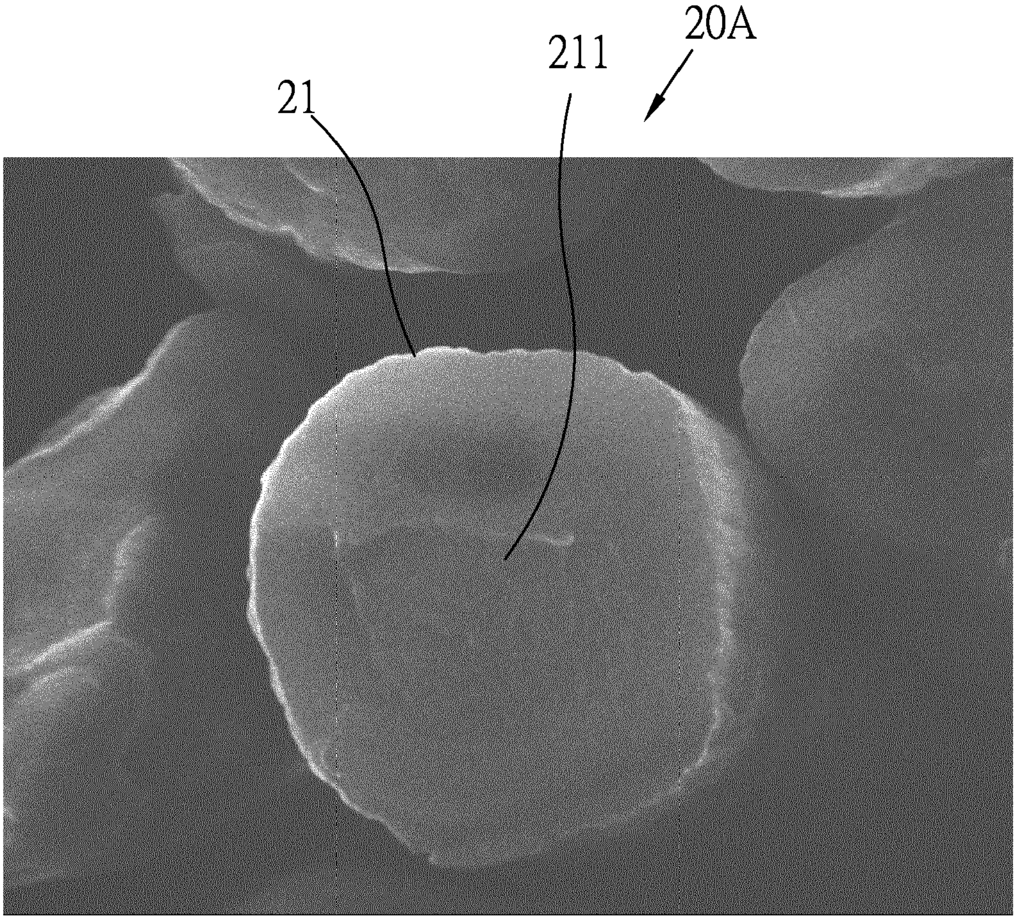


FIG. 9

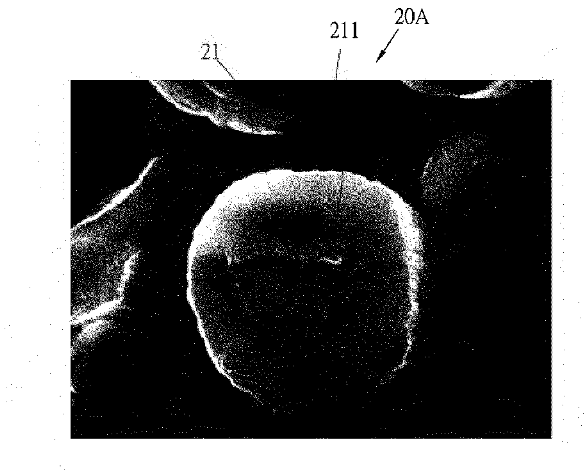


Fig. 10

Europäisches Patentamt
European Patent Office
Office européen des brevets

EUROPEAN SEARCH REPORT

Application Number
EP 18 16 8096

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (IPC)
X	CN 1 442 519 A (CHEN XINMOU [CN]) 17 September 2003 (2003-09-17) * the whole document * -----	1-14	INV. H05B6/78 H05B6/80 D01F9/32 H05B6/64
X	US 2012/137446 A1 (WOHLMANN BERND [DE] ET AL) 7 June 2012 (2012-06-07) * abstract * * paragraph [0001] - paragraph [0002] * * paragraph [0004] - paragraph [0005] * * paragraph [0017] - paragraph [0019] * * paragraph [0021] - paragraph [0022] * * paragraph [0026] - paragraph [0027] * * figure 1 * -----	1-14	
X	CN 105 696 113 A (JIANGXI DAYOU SCIENT AND TECHNICAL CO LTD) 22 June 2016 (2016-06-22) * the whole document * -----	1-14	
X	US 2011/158895 A1 (WANG CHIH-YUNG [TW] ET AL) 30 June 2011 (2011-06-30) * abstract * * paragraph [0005] * * paragraph [0013] * * paragraph [0020] * * paragraph [0032] - paragraph [0043] * * figures 1-6 * -----	1-14	TECHNICAL FIELDS SEARCHED (IPC) H05B D01F
X	CN 102 505 188 A (NINGBO INST MAT TECH & ENG CAS) 20 June 2012 (2012-06-20) * the whole document * -----	1-14	

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
Munich	13 November 2018	Chelbosu, Liviu

2

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
Y : particularly relevant if combined with another document of the same category
A : technological background
O : non-written disclosure
P : intermediate document

T : theory or principle underlying the invention
E : earlier patent document, but published on, or after the filing date
D : document cited in the application
L : document cited for other reasons
& : member of the same patent family, corresponding document

EPO FORM 1503 03.82 (P04C01)

ANNEX TO THE EUROPEAN SEARCH REPORT ON EUROPEAN PATENT APPLICATION NO. EP 18 16 8096

This annex lists the patent family members relating to the patent documents cited in the above-mentioned European search report. The members are as contained in the European Patent Office EDP file on
The European Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

13-11-2018

Patent document cited in search report		Publication date	Patent family member(s)			Publication date
CN 1442519	A	17-09-2003	NONE			
US 2012137446	A1	07-06-2012	AR	078361	A1	02-11-2011
			AU	2010294347	A1	08-03-2012
			BR	112012005159	A2	03-05-2016
			CA	2772580	A1	17-03-2011
			CN	102612576	A	25-07-2012
			DK	2475812	T3	08-09-2013
			EP	2475812	A1	18-07-2012
			ES	2426612	T3	24-10-2013
			JP	5538545	B2	02-07-2014
			JP	2013504696	A	07-02-2013
			PT	2475812	E	03-09-2013
			TW	201129743	A	01-09-2011
			US	2012137446	A1	07-06-2012
			WO	2011029745	A1	17-03-2011
CN 105696113	A	22-06-2016	NONE			
US 2011158895	A1	30-06-2011	TW	201122165	A	01-07-2011
			US	2011158895	A1	30-06-2011
CN 102505188	A	20-06-2012	NONE			

EPO FORM P0459

For more details about this annex : see Official Journal of the European Patent Office, No. 12/82