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(54) **Berendezés mikroszálás és nanoszálás kétdimenziós vagy háromdimenziós szál as anyagok előállítására**

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(54) **APPARATUS FOR PRODUCTION OF TWO-DIMENSIONAL OR THREE-DIMENSIONAL FIBROUS MATERIALS OF MICROFIBRES AND NANOFIBRES**

VORRICHTUNG ZUR HERSTELLUNG VON ZWEIDIMENSIONALEN ODER DREIDIMENSIONALEN FASERMATERIALIEN AUS MIKROFASERN UND NANOFASERN

APPAREIL POUR LA PRODUCTION DE MATÉRIAUX FIBREUX DE MICROFIBRES ET DE NANOFIBRES BIDIMENSIONNELS OU TRIDIMENSIONNELS

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Description

TECHNICAL FIELD

[0001] The present invention refers to an apparatus for a production of two-dimensional and three-dimensional fibrous materials of microfibers and nanofibers comprising a set of spinning nozzles attached to a first potential, a first set of electrodes facing the set of nozzles which are arranged having regular mutual spacing and attached to a second potential, and a collecting plate for collecting microfibers or nanofibers settled between couples of adjacent electrodes.

BACKGROUND OF THE INVENTION

[0002] Hitherto known apparatuses for production of microfibers and nanofibers working on principle of electrostatic field of very high intensity, the effects of which form melt or solution of polymers into fibrous structures, use plate collecting electrodes most frequently. The first methods of polymers spinning have been patented as far back as at the beginning of the 20th century - US0705671 (1900), US0692631 (1902), US2048651 (1934) [1]. Individual fibers deposited onto such a plate electrode are placed at random, i.e. they are not placed in any preferred direction. It is because of an unstable phase of a moving polymer jet, the trajectory of which is very complicated and spatially chaotic before its incidence onto the collecting electrode.

[0003] If the material produced is composed of regularly arranged microfibers or nanofibers, applications of such materials can spread boundlessly also in many new modern fields and branches. Their promising potential consists in substantial improvement of their morphological properties and consequently mechanical, physiological, biological, physical, optical and chemical properties, namely in particular thanks to their internal regularly oriented structure.

[0004] Several publications deal with principals of providing the arrangement of fibers deposited in this way. Two basic methods are known. The first one utilizes a mechanical principle of winding fibers onto a cylinder, bar or disc, rotating at high revs. The second principle, which this invention also refers to, utilizes static gathering collector divided into two or more conductive parts, separated from each other by a non-conductive gap of a definite size. The collector shapes the lines of force of an acting electrostatic field. The trajectory of the polymer jet is determined by these electrostatic forces and fibers falling onto the gathering collector are deposited parallel to each other in preferred direction in the non-conductive areas of the divided collector. The structure of the conductive and non-conductive areas of the collector defines the acting electrostatic forces, influencing hitherto random flight of the polymer jet, and thus it controls its movement. The mechanism of the ordered depositing of fibers onto the collector can be deduced from systematic ex-

perimental studies or numerical simulations of a physical model. In principal these methods work successfully. In 2003 - 2005, Dan Li et al. published the principle discussed above in professional journals [2-4].

[0005] The production of planar (2D) or voluminous (3D) materials using similar apparatuses is significantly limited and it is not possible to produce larger 2D and thicker 3D materials having regular structure. Thus the production is restricted to manufacturing of individual oriented fibers only. Ordered micro- or nanofibers are deposited onto non-conductive areas of the divided collector, where they form a fine regular layer. The divided collector consists of conductive usually metallic links separated by non-conductive backplate having high resistivity (higher than $10^{16} \Omega \cdot \text{cm}$). Fibers deposited onto such gathering collector are mechanically connected with it, so that any further independent practical use of them is limited. Positioning of an underlying substrate on the divided collector, or rather between emitter and collector, leads to a degradation of the structured electrostatic forces, the effects of which take part in the formation of fibers orientation. For an application of materials produced by this method, the resulting layer has to be taken from the collector first and transferred.

[0006] Rouhollaha Jalili et al. [5] described a simple collector for an accumulation of several oriented fibers into a common bundle. The result of it is not a planar structure but the bundle of fibers, only. Such fiber sample was prepared solely for the purpose of subsequent X-ray and mechanical analyses of the bundle properties. Practical use of the several fibers bundle is not mentioned in [5] and due to the achieved dimensions (length of 30 mm and diameter of about 0.08 mm), it may be assumed that it is not significant.

[0007] Patent applications US2005-0104258A1 and PPVCZ2007-0727A3 discuss a collecting electrode structure generating singular charges, but they do not deal with any ordered formation and orientation of fibers. A divided collector is a part of a US patent US4689186, but it is used for different purposes and it is not directly involved in any formation of oriented fibers. Patent application EP2045375A1 describes an apparatus for production of 2D or 3D materials composed of micro- or nanofibers with regular structure using an electrically divided collector of cylindrical shape, during a rotation of which oriented fibers are collected. By means of the described solution it is possible to produce materials with a restricted dimension that is partly limited by the diameter of the rotating collector. Also an implementation of the apparatus for producing materials of this type with larger area (i.e. multiple repeating of the proposed solution) is practically complicated, line restricted and therefore ineffective.

[0008] Micro- or nanofibers of lower strength, especially fibers made of biopolymers, are being torn by their own gravity between the collector electrodes when thicker layers (2D or 3D) are to be formed and thus the whole structure is being impaired. This is limiting for any production

technology and for getting applicable materials having desired parameters.

[0009] When depositing fibers in thicker layers, a degradation of an orientation level occurs and fibers arrangement becomes more random again. It is caused by a progressive increase of electric charge in the formed layers of fibers, i.e. in those collector parts that should remain non-conductive and without electric charge, to enable correct functioning of the fiber orienting principle. This negative effect brings about depositing of oriented fibers in lower layers of material only, i.e. in those layers which were deposited first at the beginning of the deposition; on the other hand fibers with random arrangement prevail in the higher layers. For that reason a structure of a gathering collector and an automatic mechanism were designed, where the automatic mechanism withdraws thin deposited layers of micro- or nanofibers and superimposes them in thicker layers (2D or 3D) simultaneously with the spinning process.

SUMMARY OF THE INVENTION

[0010] It is an object of the present invention to enable a control of morphological properties and other properties resulting from them of produced micro- or nanofibrous materials, and thereby to get better, also anisotropic, properties of these new materials. Resulting properties of the produced fibrous materials, especially the degree of fibrous structures orientation, morphology, density, porosity, and mechanical, physical, biological and chemical properties, are influenced by means of the process parameters. The new materials have large macroscopic dimensions in the form of planar (2D) or voluminous (3D) objects. Various starting materials, preferably polymers, namely synthetic or natural, can be used for a spinning process leading to the production of micro- or nanofibers.

[0011] This object is achieved by an apparatus for production of two-dimensional or three-dimensional fibrous materials of microfibers or nanofibers comprising a set of spinning nozzles connected to a first potential, a set of electrodes facing the set of the nozzles arranged at regular spacing and connected to a second potential, and a collecting plate for collecting microfibers or nanofibers settled between couples of adjacent electrodes, where the substance of the invention is as follows: the set of the electrodes comprises at least two electrodes arranged in a plane and the collecting plate and the plane of the electrodes form an angle α , the size of which ranging between 0° and 90° , the collecting plate being, in relation to the electrodes, supported movably in the direction lying in that plane perpendicular to the plane of the electrodes, in which the axis of the electrode lies, the direction of the collecting plate movement forming with this electrode axis an angle β , the size of which ranging between 0° and 90° .

[0012] In an advantageous embodiment of the apparatus for the production of two-dimensional or three-dimensional fibrous materials of micro- or nanofibers ac-

cording to the present invention, the collecting plate bears on the electrodes with an edge provided with a blade.

[0013] In another advantageous embodiment of this apparatus the collecting plate is provided with open parallel gaps, each of them being arranged facing one of the electrodes, whereas the collecting plate parts between two adjacent gaps are inserted into a space between two adjacent electrodes.

[0014] In a further advantageous embodiment of this apparatus, the set of the electrodes arranged at regular spacing contains at least three parallel electrodes.

[0015] In yet another advantageous embodiment of this apparatus, the collecting plate is covered with a removable substrate on its surface turned away from the electrodes to enable the nanofiber layer being enfolded with this substrate.

[0016] Finally in yet another advantageous embodiment of this apparatus, the collecting plate is provided with recess on its surface turned away from the electrodes for placing the nanofiber layers collected by the collecting plate.

BRIEF DESCRIPTION OF THE DRAWINGS

[0017] The present invention will now be explained in more detail with reference to the accompanying drawings, wherein:

Fig. 1 is a schematic drawing of the first exemplary embodiment of an apparatus for a production of two-dimensional or three-dimensional fibrous materials of microfibers or nanofibers according to the present invention, with collector electrodes in the form of linear parallel guide bars;

Fig. 2 is a schematic drawing of the second exemplary embodiment of an apparatus for production of two-dimensional or three-dimensional fibrous materials of microfibers or nanofibers according to the present invention, with the collector electrodes in the form of concentric circular guide bars arranged in a plane;

Fig. 3 is a schematic side view of a collecting mechanism with a planar collecting plate;

Fig. 4 is a schematic side view of a collecting mechanism with a collecting cylinder;

Fig. 5 is a schematic side view of a collecting mechanism with a direct collection of fibers from the surface of the conductive bars by means of an inclined blade;

Fig. 6 is a photo of fibers deposited in orderly manner between the bar electrodes, separated by an air-gap, before their removal by a collecting plate from the apparatus according to the present invention;

Fig. 7 is a photo of randomly arranged fibers deposited on the plate collector;

Fig. 8 is a photo of partially oriented fibers deposited on an electrically divided collector;

Fig. 9 is a photo of oriented fibers being consecutively withdrawn from the divided collector in accordance with the present invention;

Fig. 10 is an angular spectrum representing fibers orientation corresponding to Figs. 7, 8 and 9, and Fig. 11 is an example of a material made of polyvinylalcohol fibers using the apparatus according to the present invention, magnified 70x, 350x and 3700x, respectively.

DETAILED DESCRIPTION OF THE DRAWINGS

[0018] Reference is now made to Fig. 1 wherein the first exemplary embodiment of the apparatus for the production of two-dimensional or three-dimensional fibrous materials of microfibers or nanofibers is schematically depicted. A nozzle emitter 2 is filled with a polymer 1 solution and one pole of a DC voltage source 4 is connected to its metal nozzle 3, wherein the other pole of the source 4 is connected to conductive bar electrodes 6 of a collector. The conductive bars of the electrodes 6 of the collector pass through gaps provided in a collecting plate 7 which is inclined with respect to an x - axis by angle α . The conductive bars of the electrodes 6 of the collector are arranged in x - y plane and are linear and parallel to each other.

[0019] When the apparatus is in operation, the polymer solution 1 is extruded by a mechanical piston through the metal nozzle 3. High DC voltage from the source 4 supplied between the nozzle 3 and the electrodes 6 of the collector (the electrodes being in a form of conductive bars) directs a polymer jet as a fiber 5 which moves from the nozzle 3 in the direction towards the collector (i.e. in the direction of z-axis) on a random trajectory. This fiber 5 solidifies into a form of a micro- or nanofiber prior to its impact on the collector. Electrostatic forces acting on the fiber 5 will influence its deposition in a preferred direction 8 which is in this case the direction of y - axis, the y - axis direction being perpendicular to the conductive bars of the electrodes 6 of the collector arranged in x - y plane. The collecting plate 7, inclined by an angle α relative to the x - axis, performs translational movement in a direction $v(t)$ during defined time intervals, the direction $v(t)$ forming an angle β with x - axis. During the movement of the collecting plate 7, the fibers 5 are spontaneously deposited onto areas 9 having sizes $S_i = l_i \cdot w_i$. The oriented fibers 5 form a new planar (2D) or voluminous (3D) material 10.

[0020] Reference is now made to Figure 2 wherein the second exemplary embodiment of the apparatus for a production of two-dimensional or three-dimensional fibrous materials of microfibers or nanofibers according to the present invention is schematically depicted with collector electrodes 6 in the form of concentric circular guide bars arranged in a plane. A nozzle emitter 2 is filled with a polymer solution 1 and one pole of a DC voltage source 4 is connected to its metal nozzle 3. The other pole of the source 4 is connected to the electrodes 6 of the col-

lector. The conductive bars of the electrode 6 of the collector pass through gaps provided in the collecting plate 7 which is inclined by an angle α relative the x - axis. The conductive bars of the electrodes 6 of the collector are arranged in the x - y plane and they have the form of concentric circles.

[0021] When the apparatus is in operation, the polymer solution 1 is extruded by a mechanical piston of the nozzle emitter 2 through the metal nozzle 3. High voltage DC between the nozzle 3 and the electrodes 6 of the collector directs a polymer jet of a fiber 5 that moves from the nozzle 3 in the direction to the collector (i.e. in the direction of z - axis) on random trajectory. This jet of polymer fiber 5 solidifies into the form of a micro- or nanofiber before its impact on the collector. The electrostatic forces acting on the fiber 5 influence its deposition in a preferred direction 8, which is radial in relation to the circular conductive bars of the electrodes 6 of the collector, arranged in the x - y plane. The collecting plate 7, which is inclined by an angle α relative to the x - axis, moves in specified time intervals rotating around its vertical axis 11 in a direction $\omega(t)$, whereas the collecting plate mass centre describes a circle 12 which is inclined by an angle β relative to the x - axis. During this movement of the collecting plate, the fibers are spontaneously deposited onto areas 9. The oriented fibers 5 form a new planar (2D) or voluminous (3D) material 10. A schematic side view of the collecting mechanism with a planar collecting plate 7 is schematically depicted in Fig. 3. Fibers 5 are deposited on the conductive bars of the electrodes 6 of the collector by the electrostatic spinning process. Afterwards the fibers are placed on the collecting plate 7 surface whereas their orientation remains preserved. In this exemplary embodiment, the Collecting plate 7 is planar and it is inclined by an angle α with respect to the bars of the electrodes 6 of the collector and it performs a translational movement in a direction which forms an angle β with the x - axis.

[0022] A side view of a collecting mechanism with a collecting cylinder 14 is schematically depicted in Fig. 4. Fibers 5 are deposited on the conductive bars of the electrodes 6 of the collector by the electrostatic spinning process. Afterwards the fibers 5 are placed on the collecting cylinder 14 surface, whereas their orientation remains preserved. The collecting cylinder 14 rotates around its axis and it performs a translational movement along the x - axis at the same time.

[0023] Fig. 5 shows a schematic side view of a collecting mechanism with a direct collection of fibers 5 from the surface of the conductive bars of the electrodes 6 of the collector by means of an inclined blade. Fibers 5 are deposited on the conductive bar electrodes 6 of the collector by the electrostatic spinning process. Afterwards the fibers 5 are placed on a surface of the collecting plate 7, whereas their orientation remains preserved. In this exemplary embodiment the fibers 5 are collected directly from the surface of the conductive bars of the electrodes 6 of the collector by means of an inclined blade 13. The

blade 13 is inclined by an angle α with respect to the conductive bars of the electrodes 6 of the collector and it performs a translational movement along the x - axis.

[0024] Fig. 6 is a photo of fibers deposited in an orderly manner between the conductive bars of the electrodes 6 of the collector separated with an air-gap, prior to their removal by means of the collecting plate. It is evident from the Fig. 6 that the nanofibers are arranged in parallel.

[0025] Figs. 7, 8 and 9 are photos illustrating the importance of the gathering collector design and of the method of a consecutive depositing on nanofibers of polyvinylalcohol. The photos were taken by an electron microscope with magnification approx. 5000x. In Fig. 7, fibers 5 applied onto a plate collector are deposited at random; in Fig. 8, fibers 5 deposited onto electrically divided collector are partly oriented, and Fig. 9 is a photo of oriented fibers 5 which have been consecutively removed from the divided collector according to the present invention.

[0026] Fig. 10 shows an angular spectrum diagram representing the orientation of the fibers 5 of the samples shown in Fig. 7 (sample A), Fig. 8 (sample B) and Fig. 9 (sample C). The spectrum was obtained on the basis of picture analysis by means of a Fourier transformation. The peak in the spectrum of the sample C corresponds to the most important angle of fibers 5 arrangement, in this case to angle of 90° - the vertical direction. The analysis applied is commonly used in professional practice for an automatic evaluation and comparison of fibers 5 orientation, even though the picture analysis works with dots, i.e. with picture pixels, not with individual fibers 5.

[0027] Photos of an exemplary material produced by means of the apparatus in accordance with the present invention are in Figure 11. There are three different magnifications of the material part of polyvinylalcohol fibers 5 in Figure 11, namely magnification 70x in Fig. 11a, magnification 350x in Fig. 11b and magnification 3700x in Fig. 11c.

[0028] Micro- or nanofibers are formed by the method of electrostatic spinning. A single or a multiple nozzle emitter 2 generates a stream of polymer fibers 5 in a form of jets which move towards the second electrode 6 of the collector and uniformly cover the whole area of the collector. Micro- or nanofibers are carried away by electrostatic field forces and are deposited in parallel to each other, because - during their move from the nozzle emitter 2 towards the electrodes 6 - their trajectory is influenced by lines of force of the electrostatic field in vicinity of the collector, which is for these purposes divided in two or more conductive and non-conductive areas. On the basis of numerous experiments a gathering collector was designed and tested wherein the electrodes 6 of the collector are constituted by two or more thin conductive bars, e.g. in the form of wires or strings, that are separated from each other by an air-gap. Neither their number nor their lengths are limited. It was further found that the most suitable shape of the bar section is not circular but an-

gular, namely square or rectangular, having a width of 0.1 mm to 10 mm, preferably of 1 to 5 mm. Individual bars are laterally spaced apart from each other and separated by an air-gap of a specified width, namely 0.1 mm to 200 mm, but more preferably 1 mm to 100 mm. The influence of the air-gap on the formation of ordered fibers 5 was studied systematically and it was found that in case of a short distance the degree of orientation is lowered. On the contrary in the case of a long distance, the fibers 5 are deposited directly onto the conductive electrodes and the number of oriented fibers 5 extended between the conductive bars is lower or the fibers are torn by their gravity. Therefore the most suitable size of the air-gap must be experimentally tested for each type of polymer to provide a successful formation of oriented fibers 5. It was further found that the width of the conductive bars need not necessarily be big, on the contrary, from the design and function points of view an application of thin bars of a square section proves to be advantageous in contrast to wider plates as it is shown in the literature cited. Sizes of the air-gaps were optimized for several sorts of synthetic and natural polymers depending on their mechanical properties.

[0029] The space between the conductive bars of the electrodes 6 of the collector, where the fibers 5 are being arranged longitudinally in one direction or rather perpendicularly to the conductive bars of the electrodes 6 of the collector across the non-conductive area, is gradually filled up during the deposition. The deposition of the fibers 5, oriented in this way, into the thicker layers is not possible for the reasons mentioned above, e.g. because of degradation of the orientation degree etc., and therefore a process has been proposed by which a thin deposited layer was withdrawn in regular time intervals and transferred onto a backplate, preferably simultaneously with the deposition.

[0030] For the oriented fibers 5 collecting, transferring and superimposing, the collecting plate 7 with elongated openings is used, the elongated openings enabling the collected plate 7 to be put on the conductive bars of the electrodes 6 of the collector and to move in translational movement, in lengthwise direction along the conductive bars. The shape of the collecting plate 7 was repeatedly experimentally tested and modified. The resulting optimal design is described in this disclosure. During specified time intervals from 1 s to 1 hour, the collecting plate 7 shifts in a longitudinal direction along the conductive bars whereas it picks up the in orderly manner deposited micro- or nanofibers on its surface. It was found that due to the inclination of the collecting plate 7 by a specific angle relative to the bars of the electrodes 6 of the collector, namely $0^\circ < \alpha < 90^\circ$, the fibers 5 withdrawn in the vicinity of edges of the conductive bars of the electrodes 6 of the collector are mechanically stressed to a lesser extent, and further that the inclination of the collecting plate 7 assists in regular deposition of individual fibers 5 along the whole of their length onto the collecting plate 7. The inclination of the collecting plate further enables

simultaneous withdrawing of the fibers 5 deposited directly onto the conductive bars of the electrodes 6 of the collector. The fibers 5 are deposited in greater quantities in these places as a result of stronger acting electrostatic forces and therefore they increase the mechanical ruggedness of the resulting material. Furthermore the problem of the collection of oriented fibers 5 on a larger area $S = \sum S_i = \sum (l_i \cdot w_i)$ (where l_i is length and w_i is width of area i) has been solved, namely just by the newly designed and Experimentally verified process. The collecting plate performs translational movement (at a speed of 0.001 m/s to 10 m/s) along the conductive bars of the electrodes 6 of the collector, the direction of this movement forming an angle β (at interval $0^\circ < \beta < 90^\circ$) with the conductive bars of the electrodes 6 of the collector. During this movement, the micro- or nanofibers deposited in an orderly manner are superimposed in thick layers (2D) or voluminous (3D) objects while the regular ordered structure of the material 10 is maintained. The value of the angle β determines areal density of fibers 5 in the layer formed from the new material 10 and the length l of the collecting plate part that is covered with the fibers. The areal or voluminous materials 10 are created consecutively depending on an overall time of the process and an overall area of the produced material 10. The process developed enables depositing of micro- or nanofibers into thicker layers while the orientation degree being maintained even in higher layers. By placing on a prepared final backplate, fibers 5 are mechanically strained only to minimum degree and therefore their structure is not disturbed.

[0031] Fibers 5 manufactured of different mixtures, e.g. of synthetic or natural polymers, generally have different mechanical characteristics and materials 10 produced by electrostatic spinning have different morphology as well. On the basis of the examined characteristics, one of the proposed processes of collection and deposition of ordered fibers 5 was selected. It was found that the use of the collecting plate 7 which is inserted between the conducting bars of the electrodes 6 of the collector is suitable for fibers 5 with lower mechanical strength manufactured of natural polymers. Fibers 5 can be that fine that they may tear even by their own weight while being suspended between the conductive bars of the electrodes 6 of the collector. In such a case there is no other possibility than to take fibers 5 away by the apparatus in accordance with the present invention. On the contrary, a collecting plate 7 with a collecting blade 13 which performs translational movement over the surface of the conductive bars is used with more resistant materials 10 like synthetic polymers. The advantage of this process is that the resulting material 10 is not discontinued in any place and is even strengthened in areas on the conductive bars of the electrodes 6 of the collector which substantially increases its resistance in subsequent mechanical stress, e.g. in a specific application.

[0032] Translational movement of the collecting plate 7 along the conductive bars of the electrodes 6 of the

collector is reverse during specific time intervals in order to form a one-sided deposit of the material 10. The new material 10 being created on an arbitrary backplate, the backplate can be designed as a packing material. A practical solution enables a production of ordered materials that will simultaneously be placed into a sterile packing in a deposition chamber "in situ" and thus will be ready for a direct application and use. The apparatus as designed solves a problem of a technically demanding mechanical transfer of fine fiber materials 10 onto another transport substrate and eliminates possible causes of disturbance, damage, pollution and deterioration of the material 10 during the manipulation. The apparatus as designed makes it possible to carry out the production process in the single environment of the deposition chamber and therefore a necessary sterility of materials 10 intended for medicine may be achieved easily.

[0033] In another case, the collecting plate 7 moves always in one direction only after expiration of a time interval. It remains in an end position for the same time interval and then moves back. The divided translational movement results in depositing of micro- or nanofibers from both sides of the collecting plate 7 which is adapted in its shape to attach underlying material. This principle makes it possible to create fiber layers on both sides of the only supporting backplate.

[0034] Further a problem of discrete movement of the collecting plate 7 has been dealt with, the problem being more demanding in terms of design. A centro symmetrical construction uses circular conductive bars of a collector as electrodes 6 of the collector. In this case, the collecting plate 7 rotates around its central axis. In this case the collecting plate moves at an angular velocity $\omega(t)$ ranging between 0.001 and 10 rad/s. Fibers 5 are deposited and layered in the same way as in the preceding embodiment. Here the continuous rotating movement of the collecting plate 7 is of advantage when compared with the discrete translations in the preceding solution.

[0035] Constructional modifications of the collecting plate 7 enable rotation of individual elements of the collecting plate 7 by an angle γ lying in the range of $0 < \gamma < 90^\circ$. After an expiration of a specific time interval (from 1 s to 1 hour) of a fiber material 10 layering, elements of the collecting plate 7, having areas $S_i = l_i \cdot w_i$, are slightly turned and further layers of the material 10 are deposited again. The inner structure of the material 10 formed in this way, has individual layers composed of micro- or nanofibers wherein the layers are slightly turned relative to each other by an adjusted angle γ . This principle makes it possible to produce materials 10 with two or more preferred directions of the anisotropic material 10 and to form an ordered 3D structure as well. The regular structure arises not only on the area but also in a three-dimensional object by the rotation of the collecting plate 7 elements or by multiple repeating of the fibers 5 collection in the process described above.

[0036] Deposited fibers 5 fill up the area between the gaps in the collecting plate 7. A size of the area 9 where

the oriented micro- or nanofibers are layered is not dimensionally limited. The transverse width of the conductive bars of the electrodes 6 (and the width of the gaps in the collecting plate 7 derived from it) is the only important parameter. In these places fibers 5 in resulting material 10 are not deposited in an orderly manner or some spots here are left unfilled. There are maximum 20% of these areas in the resulting material 10.

[0037] Multiple metal nozzles 3 of the emitter are used for the purpose of covering a larger area of the collector with fibers 5 and increasing of the production efficiency. Individual metal nozzles 3 of the emitter are also used for the depositing of fibers 5 of different polymer mixtures. In case that the metal nozzles 3 of the emitter are positioned in line along the conductive bars of the electrodes 6 of the collector, fibers 5 are deposited in layers one after another whereas individual layers are created by the fibers 5 of different polymer. Fiber structure of the resulting material is of a composite type.

[0038] Replacement of the collecting plate 7 by a collecting cylinder 14 of a specific diameter R, in the lateral surface of which the gaps for individual conductive bars of the electrodes 6 of the collector are provided, enables a manufacturing of hollow tubes which walls are composed of fibers 5 arranged regularly in longitudinal direction. The collecting cylinder 14 performs two independent movements: a rotational movement around its longitudinal axis and a translational one in the direction along the conductive bars of the electrodes 6 of the collector (along x-axis). These movements of the cylinder enable collection of micro- or nanofibers onto its surface. The surface of the collecting cylinder 14 with a backplate, where the fibers 5 are deposited into planar (2D) materials 10, is either left in tube shape or is spread out for the purpose of creating areal materials 10 of larger sizes.

[0039] The above described construction of the collector and the mechanism of the oriented micro- or nanofibers collection and deposition enable an efficient production of new materials that are areally large or layered in voluminous (3D) forms while their fine and regular fiber structure remains maintained.

INDUSTRIAL APPLICABILITY

[0040] The presented invention may be used for a production of areal (2D) or voluminous (3D) materials which have their inner fiber structure composed of oriented micro- or nanofibers arranged longitudinally in one or more directions.

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[0041]

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Claims

1. An apparatus for a production of two-dimensional or three-dimensional fibrous materials of microfibers or nanofibers comprising at least one spinning metal nozzle (3) connected to a first potential, a set of electrodes (6) of a collector, the set of the electrodes (6) of the collector contains at least two electrodes (6) of the collector arranged in a plane, the set facing the nozzle(s) (3) and being arranged having a constant spacing relative to each other and being connected to a second potential, **characterised in that** it comprises a collecting plate (7) or a collecting cylinder (14) for collecting microfibers or nanofibers settled between couples of adjacent electrodes (6) of the collector, and the collecting plate (7) is provided with gaps, through which the electrodes (6) of the collector pass, while the collecting plate (7) in the line of its intersection or a tangent to the collecting cylinder (14) which is perpendicular to a contact line with the plane of the electrodes (6) of the collector form with the plane of the electrodes (6) of the collector an angle α , the size of which ranges between 0° and 90° , the collecting plate (7) or the collecting cylinder (14) being arranged movably relative to the electrodes (6) of the collector in a direction lying in a plane which is perpendicular to the plane of the electrodes (6) of the collector and in which the axis of the electrode (6) lies, while the direction of movement of the collecting plate (7) or of the collecting cylinder (14) forms with said electrode (6) axis an angle β , the size of which ranges between 0° and 90° .
2. The apparatus for the production of two-dimensional or three-dimensional fibrous materials of microfibers or nanofibers according to claim 1, **characterised in that** the collecting plate (7) is provided with open parallel gaps, each of them being arranged facing one of the electrodes (6) of the collector, whereas the collecting plate (7) protrusions between two adjacent gaps are inserted into a space between two adjacent electrodes (6) of the collector.
3. The apparatus for the production of two-dimensional or three-dimensional fibrous materials of microfibers or nanofibers according to any of claims 1 to 2, **characterised in that** the set of the electrodes (6) of the collector having constant spacing relative to each

other comprises at least three parallel electrodes (6) of the collector.

4. The apparatus for the production of two-dimensional or three-dimensional fibrous materials of microfibers or nanofibers according to claim 1, **characterised in that** the collecting plate (7) comprises a surface which is turned away from the electrodes (6) of the collector, said surface being covered with a removable substrate to enable the microfiber or nanofiber layer being enfolded by the substrate. 5 10
5. The apparatus for the production of two-dimensional or three-dimensional fibrous materials of microfibers or nanofibers according to claim 1, **characterised in that** the collecting plate (7) comprises a surface which is turned away from the electrodes (6) of the collector and which is provided with a recess for placing the microfiber or nanofiber layers collected by the collecting plate (7). 15 20
6. The apparatus for the production of two-dimensional or three-dimensional fibrous materials of microfibers or nanofibers according to claim 1, **characterised in that** the shape of the cross section of the electrodes (6) of the collector is square or rectangular having a width of 0.1 mm to 10 mm. 25
7. The apparatus for the production of two-dimensional or three-dimensional fibrous materials of microfibers or nanofibers according to claim 6, **characterised in that** the shape of the cross section of the electrodes (6) of the collector is square or rectangular having a width of 1 to 5 mm. 30 35
8. The apparatus for the production of two-dimensional or three-dimensional fibrous materials of microfibers or nanofibers according to claim 1, **characterised in that** the electrodes (6) of the collector are separated from each other by an airgap while being laterally spaced from each other 0.1 mm to 200 mm. 40
9. The apparatus for the production of two-dimensional or three-dimensional fibrous materials of microfibers or nanofibers according to claim 8, **characterised in that** the electrodes (6) of the collector are laterally spaced from each other 1 mm to 100 mm. 45

Patentansprüche

1. Ein Gerät zur Herstellung von zweidimensionalen oder dreidimensionalen faserartigen Werkstoffen aus Mikrofasern oder Nanofasern, umfassend mindestens eine metallische Spinnöse (3), die mit einem ersten Potential verbunden ist, und einen Elektrodenatz (6) eines Kollektors, wobei der Elektrodenatz (6) des Kollektors mindestens zwei Elektro-

den (6) des Kollektors enthält, die gegenüber der Spinnöse (den Spinnöse) (3) in einer Ebene und in gleichmäßigen gegenseitigen Abständen angeordnet sind und mit einem zweiten Potential verbunden sind, **dadurch gekennzeichnet, dass** es ferner eine Sammelplatte (7) oder einen Sammelzylinder (14) zum Sammeln der im Bereich der von den benachbarten Elektroden (6) des Kollektors gebildeten Elektrodenpaaren abgelagerten Mikrofasern oder Nanofasern umfasst und die Sammelplatte (7) mit Spalten versehen ist, durch welche die Elektroden (6) des Kollektors durchgehen, wobei die Sammelplatte (7) in ihrer Durchdringungslinie oder eine Tangente des Sammelzylinders (14), die sich senkrecht zu einer in der Ebene der Elektroden (6) des Kollektors liegenden Berührungslinie erstreckt, mit der Ebene der Elektroden (6) des Kollektors einen Winkel α einschließt, dessen Größe zwischen 0° und 90° liegt, wobei die Sammelplatte (7) oder der Sammelzylinder (14) bewegbar gegenüber den Elektroden (6) des Kollektors gelagert sind, nämlich in einer Richtung, die in einer Ebene verläuft, die senkrecht zu der Ebene der Elektroden (6) des Kollektors ist und in der die Achse der Elektrode (6) liegt, wobei die Richtung der Bewegung der Sammelplatte (7) oder des Sammelzylinders (14) mit der genannten Achse der Elektrode (6) einen Winkel β einschließt, dessen Größe zwischen 0° und 90° liegt.

2. Das Gerät zur Herstellung von zweidimensionalen oder dreidimensionalen faserartigen Werkstoffen aus Mikrofasern oder Nanofasern nach Anspruch 1, **dadurch gekennzeichnet, dass** die Sammelplatte (7) mit offenen parallelen Spalten versehen ist, wobei jede der Spalten gegenüber einer der Elektroden (6) des Kollektors angeordnet ist, während die zwischen den benachbarten Spalten der Sammelplatte (7) vorhandenen Vorsprünge in die zwischen den jeweiligen benachbarten Elektroden (6) des Kollektors ausgebildeten Räume eingeschoben sind. 50
3. Das Gerät zur Herstellung von zweidimensionalen oder dreidimensionalen faserartigen Werkstoffen aus Mikrofasern oder Nanofasern nach einem der Ansprüche 1 und 2, **dadurch gekennzeichnet, dass** der Elektrodenatz (6) des Kollektors mindestens drei parallele Elektroden (6) des Kollektors umfasst, die in gleichmäßigen gegenseitigen Abständen angeordnet sind.
4. Das Gerät zur Herstellung von zweidimensionalen oder dreidimensionalen faserartigen Werkstoffen aus Mikrofasern oder Nanofasern nach Anspruch 1, **dadurch gekennzeichnet, dass** die Sammelplatte (7) eine Oberfläche aufweist, die von den Elektroden (6) des Kollektors abgewandt ist und die mit einem abnehmbaren Substrat abgedeckt ist, um die Umhüllung der von Mikrofasern oder Nanofasern gebil-

deten Schicht mit diesem Substrat zu ermöglichen.

5. Das Gerät zur Herstellung von zweidimensionalen oder dreidimensionalen faserartigen Werkstoffen aus Mikrofasern oder Nanofasern nach Anspruch 1, **dadurch gekennzeichnet, dass** die Sammelplatte (7) eine Oberfläche aufweist, die von den Elektroden (6) des Kollektors abgewandt ist und die mit einer zur Unterbringung der von Mikrofasern oder Nanofasern gebildeten und von der Sammelplatte (7) gesammelten Schichten dienenden Aussparung versehen ist. 5
6. Das Gerät zur Herstellung von zweidimensionalen oder dreidimensionalen faserartigen Werkstoffen aus Mikrofasern oder Nanofasern nach Anspruch 1, **dadurch gekennzeichnet, dass** der Querschnitt der Elektroden (6) des Kollektors eine quadratische oder rechteckige Form mit der Breite im Bereich von 0,1 mm bis 10 mm aufweist. 10
7. Das Gerät zur Herstellung von zweidimensionalen oder dreidimensionalen faserartigen Werkstoffen aus Mikrofasern oder Nanofasern nach Anspruch 6, **dadurch gekennzeichnet, dass** der Querschnitt der Elektroden (6) des Kollektors eine quadratische oder rechteckige Form mit der Breite im Bereich von 1 mm bis 5 mm aufweist. 15
8. Das Gerät zur Herstellung von zweidimensionalen oder dreidimensionalen faserartigen Werkstoffen aus Mikrofasern oder Nanofasern nach Anspruch 1, **dadurch gekennzeichnet, dass** die Elektroden (6) des Kollektors voneinander durch einen Lufispalt getrennt sind, wobei der gegenseitige Abstand der Elektroden in Querrichtung im Bereich von 0,1 mm bis 200 mm liegt. 20
9. Das Gerät zur Herstellung von zweidimensionalen oder dreidimensionalen faserartigen Werkstoffen aus Mikrofasern oder Nanofasern nach Anspruch 8 **dadurch gekennzeichnet, dass** der gegenseitige Abstand der Elektroden (6) des Kollektors in Querrichtung im Bereich von 1 mm bis 100 mm liegt. 30

Revendications

1. Un appareil pour production des matières fibreuses bidimensionnelles ou tridimensionnelles en microfibres ou nanofibres comprenant au moins une buse (3) de filage en métal reliée au premier potentiel, un ensemble des électrodes (6) d'un collecteur, l'ensemble d'électrodes (6) du collecteur comprenant au moins deux électrodes (6) du collecteur disposées dans le plan, le dit ensemble se trouvant en face de la buse (3) (des buses (3)) et étant disposé avec un espacement constant par rapport l'une à 35

l'autre et étant relié à un second potentiel, **caractérisé en ce qu'il** comprend une plaque (7) collectrice ou un cylindre (14) collecteur pour collecter des microfibres ou nanofibres déposées entre des couples d'électrodes (6) adjacentes du collecteur, et la plaque (7) collectrice est munie des fentes à travers lesquelles passent les électrodes (6) du collecteur, tandis que la plaque (7) collectrice à la ligne de son intersection ou la tangente au cylindre (14) collecteur qui est perpendiculaire à la ligne de contact avec le plan des électrodes (6) du collecteur fait avec le plan des électrodes (6) du collecteur un angle α , dont la dimension est comprise entre 0° et 90° , la plaque (7) collectrice ou le cylindre (14) collecteur étant disposés de manière mobile par rapport aux électrodes (6) du collecteur dans une direction située dans le plan qui est perpendiculaire au plan des électrodes (6) du collecteur et dans lequel l'axe d'électrode (6) est situé, tandis que la direction de déplacement de la plaque (7) collectrice ou du cylindre (14) collecteur forment avec ladite électrode (6) un angle β , dont la dimension est comprise entre 0° et 90° .

2. L'appareil pour production des matières fibreuses bidimensionnelles ou tridimensionnelles en microfibres ou nanofibres selon la revendication 1, **caractérisé en ce que** la plaque (7) collectrice est munie des fentes ouvertes parallèles, chacune d'elles étant disposée en position frontale contre l'une des électrodes (6) du collecteur, tandis que les saillants de la plaque (7) collectrice entre deux fentes adjacentes sont insérés dans l'espace entre deux électrodes (6) adjacentes du collecteur. 25
3. L'appareil pour production des matières fibreuses bidimensionnelles ou tridimensionnelles en microfibres ou nanofibres selon l'une quelconque des revendications 1 à 2, **caractérisé en ce que** l'ensemble d'électrodes (6) du collecteur ayant un espacement constant l'une par rapport à l'autre comprend au moins trois électrodes (6) parallèles du collecteur. 30
4. L'appareil pour production des matières fibreuses bidimensionnelles ou tridimensionnelles en microfibres ou nanofibres selon la revendication 1, **caractérisé en ce que** la plaque (7) collectrice comprend une surface qui est tournée dans la direction opposée des électrodes (6) du collecteur, ladite surface étant couverte d'un substrat amovible pour permettre à la couche de microfibres ou de nanofibres d'être enveloppé par le substrat. 35
5. L'appareil pour production des matières fibreuses bidimensionnelles ou tridimensionnelles en microfibres ou nanofibres selon la revendication 1, **caractérisé en ce que** la plaque (7) collectrice comprend une surface qui est tournée dans la direction opposée des électrodes (6) du collecteur et qui est munie 40

d'un creux pour placer les couches de microfibres ou de nanofibres collectées par la plaque (7) collectrice.

6. L'appareil pour production des matières fibreuses bidimensionnelles ou tridimensionnelles en microfibres ou nanofibres selon la revendication 1, **caractérisé en ce que** la forme de la section transversale des électrodes (6) du collecteur est de forme carrée ou rectangulaire ayant une largeur de 0,1 à 10 mm 5
10

7. L'appareil pour production des matières fibreuses bidimensionnelles ou tridimensionnelles en microfibres ou nanofibres selon la revendication 6, **caractérisé en ce que** la forme de la section transversale des électrodes (6) du collecteur est de forme carrée ou rectangulaire ayant une largeur de 1 mm à 5 mm. 15

8. L'appareil pour production des matières fibreuses bidimensionnelles ou tridimensionnelles en microfibres ou nanofibres selon la revendication 1, **caractérisé en ce que** les électrodes (6) du collecteur sont séparées les unes des autres par un entrefer, tout en étant espacés latéralement les unes des autres de 0,1 mm à 200 mm. 20
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9. L'appareil pour production des matières fibreuses bidimensionnelles ou tridimensionnelles en microfibres ou nanofibres selon la revendication 8, **caractérisé en ce que** les électrodes (6) du collecteur sont séparées latéralement les unes des autres de 1 mm à 100 mm. 30

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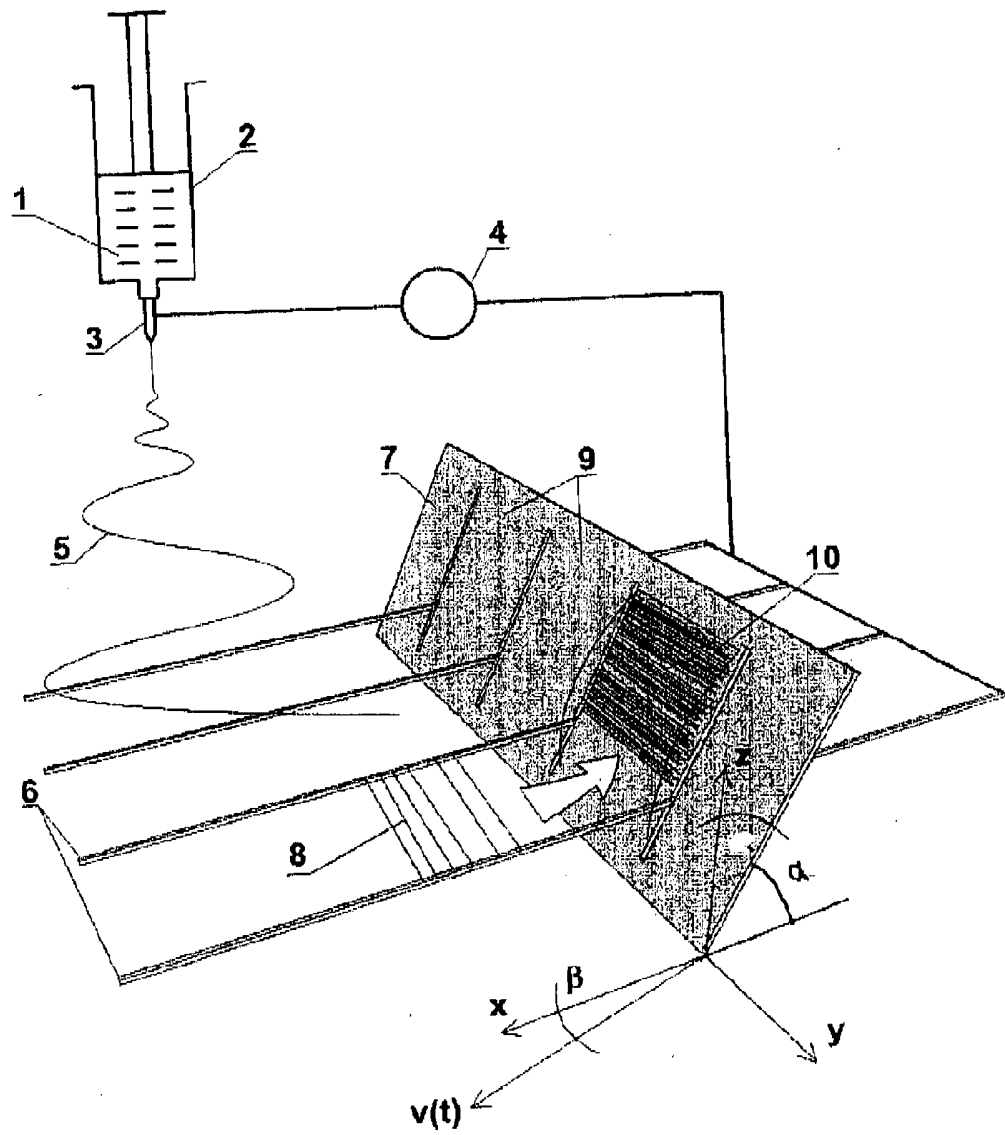


Fig. 1

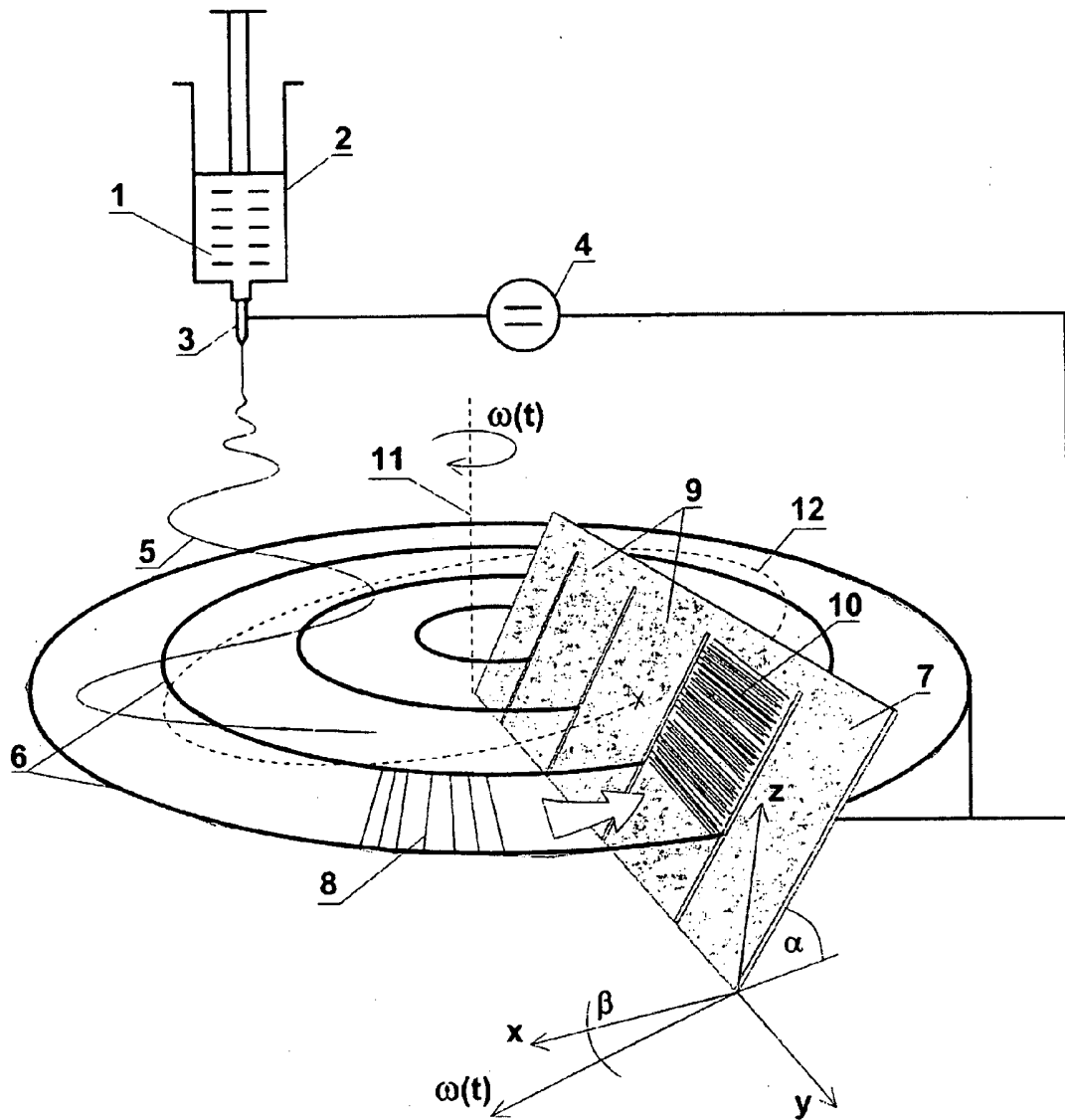


Fig. 2

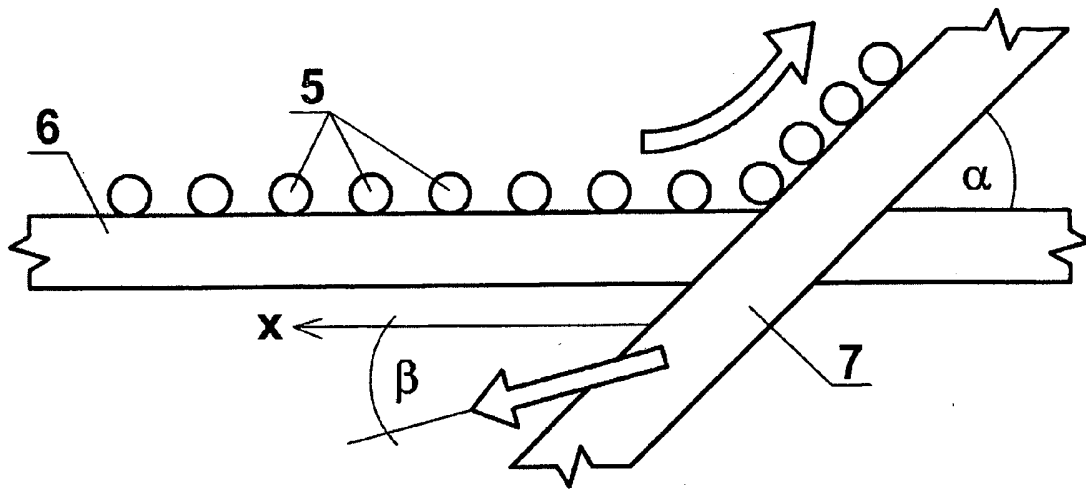


Fig. 3

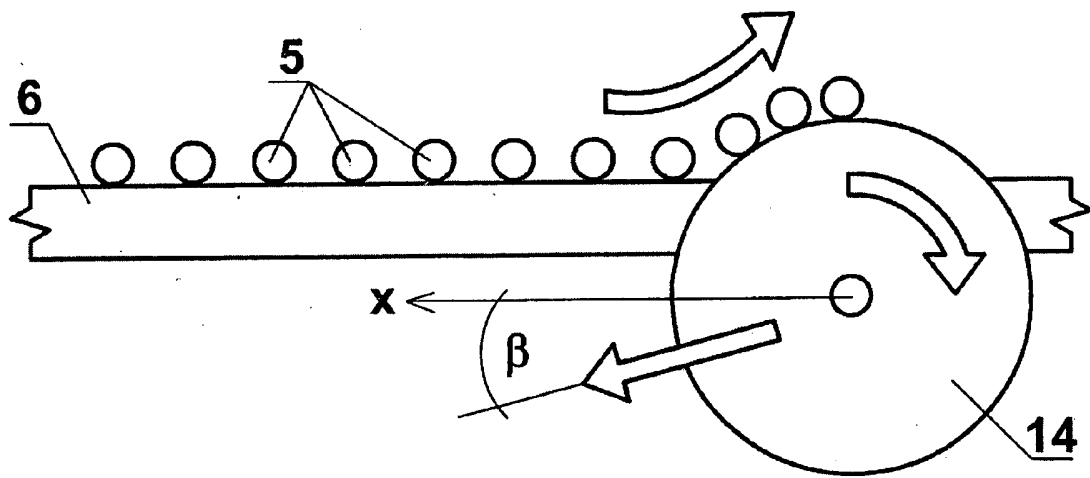


Fig. 4

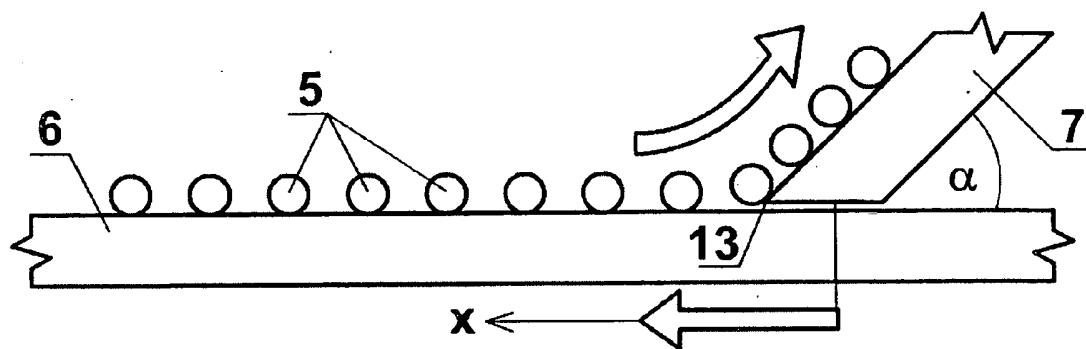


Fig. 5

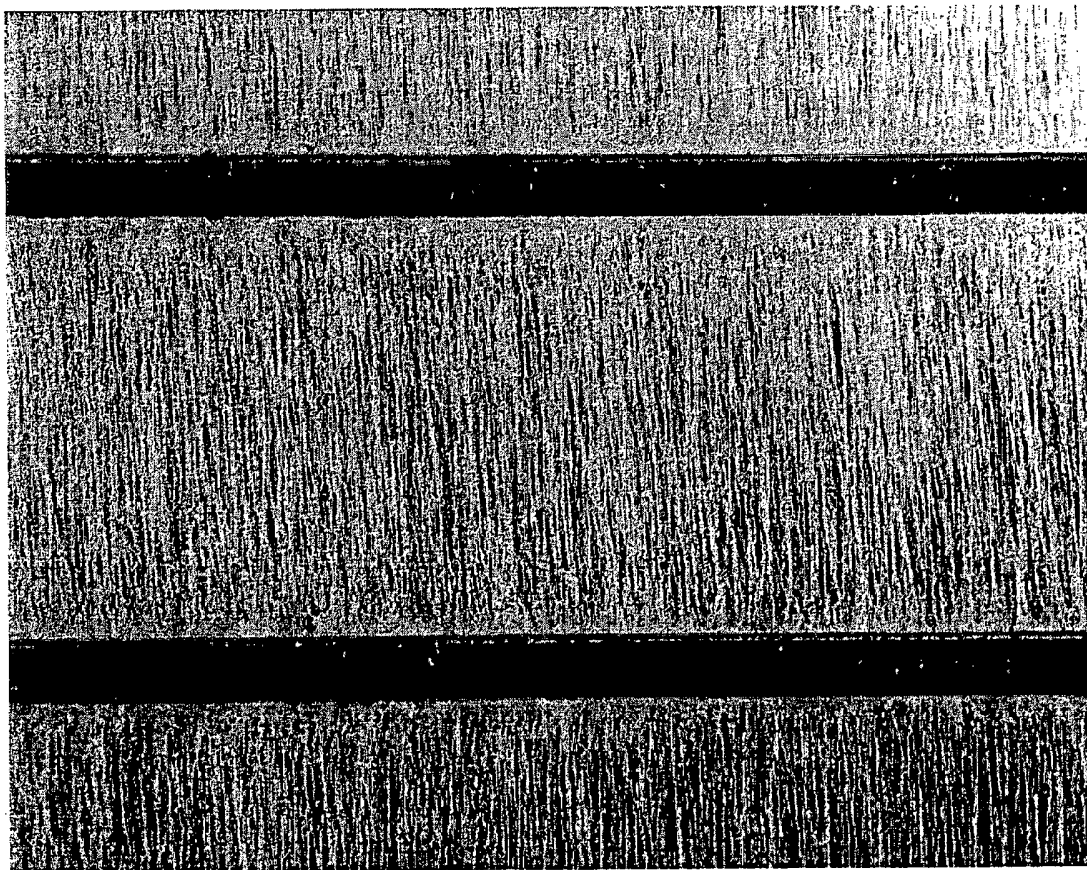


Fig. 6

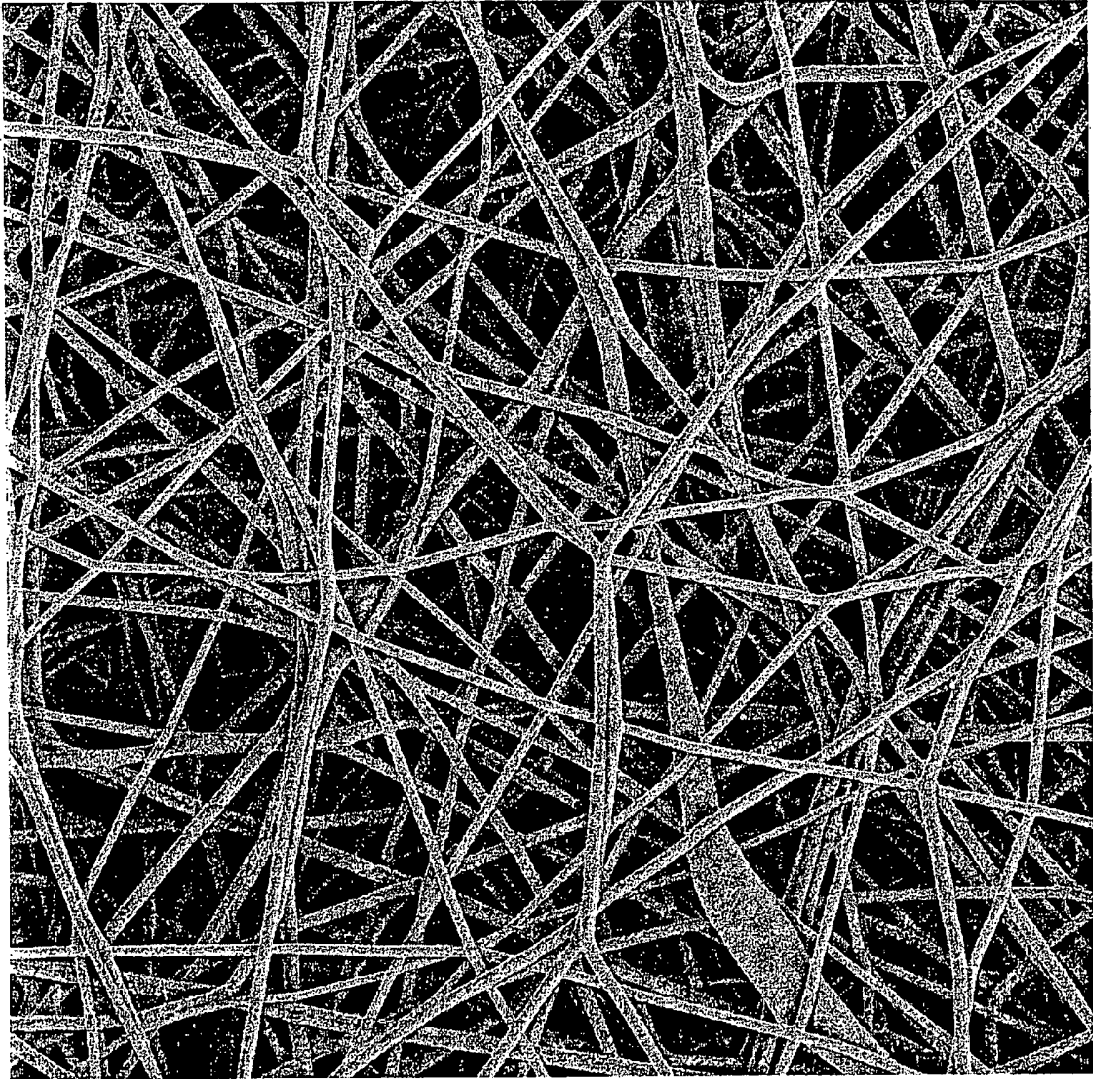


Fig. 7

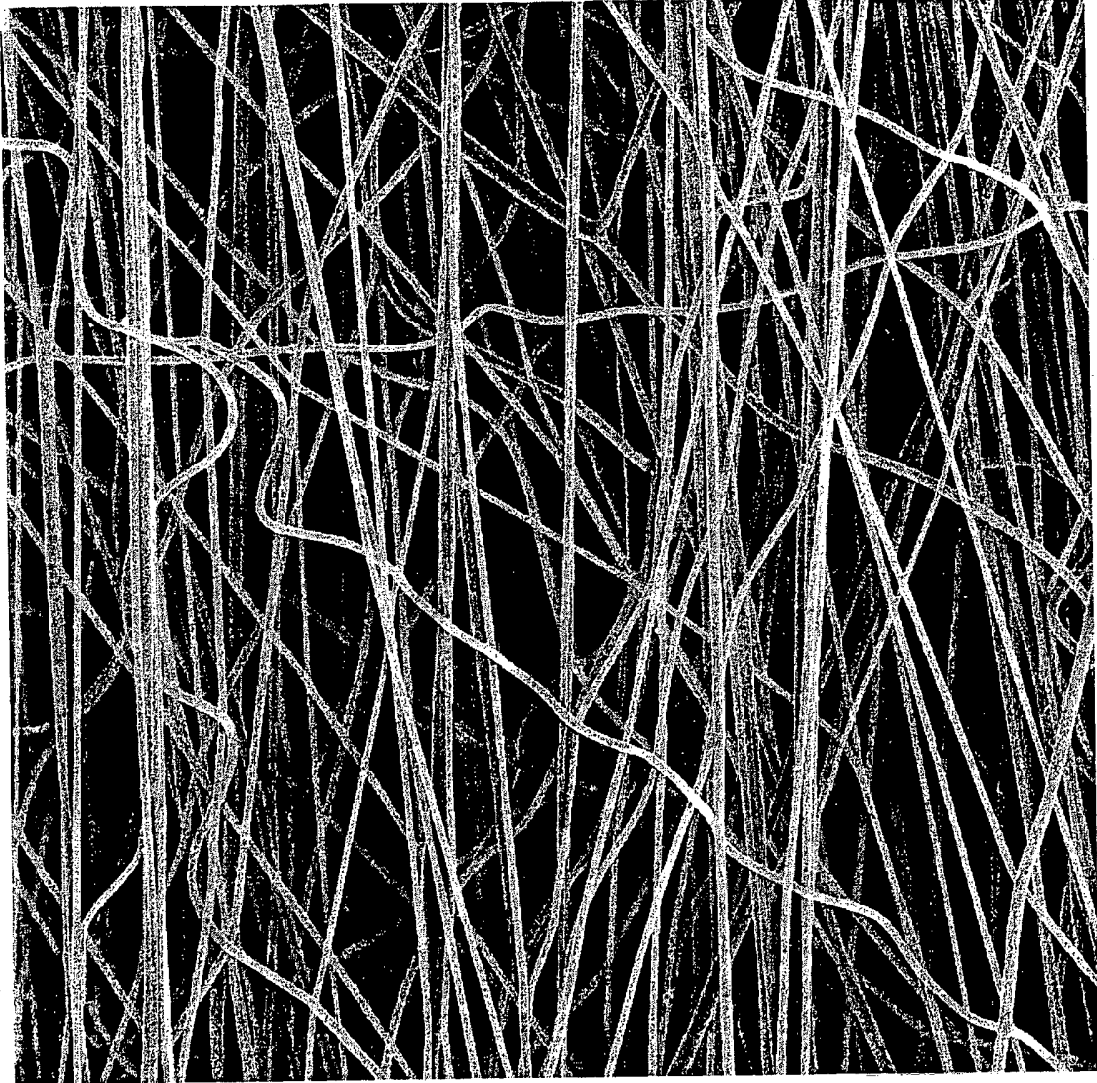


Fig. 8

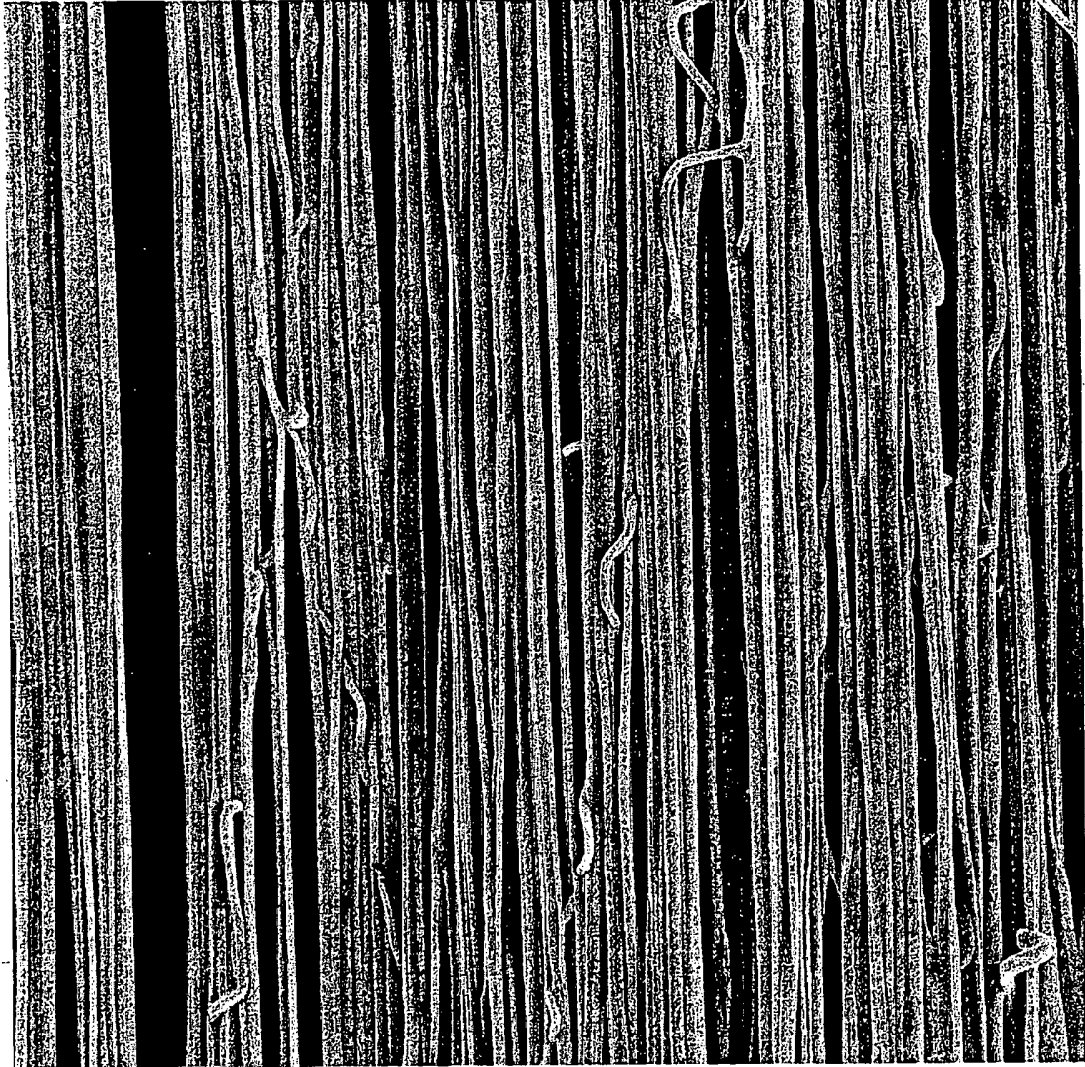


Fig. 9

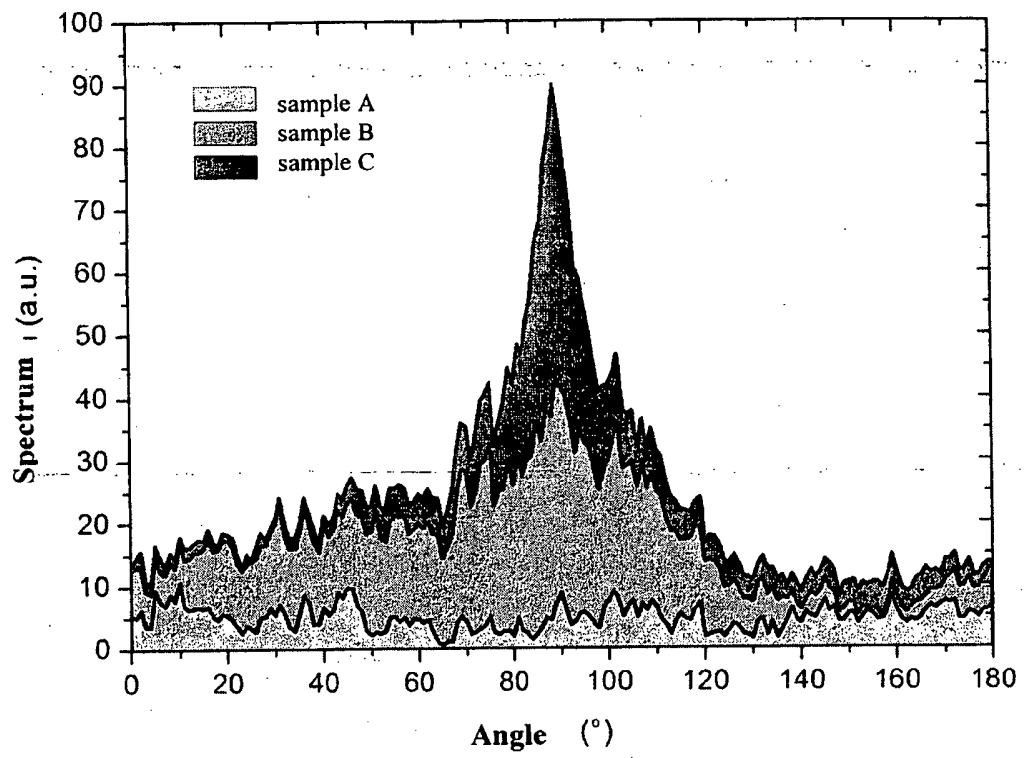


Fig. 10



Fig. 11a

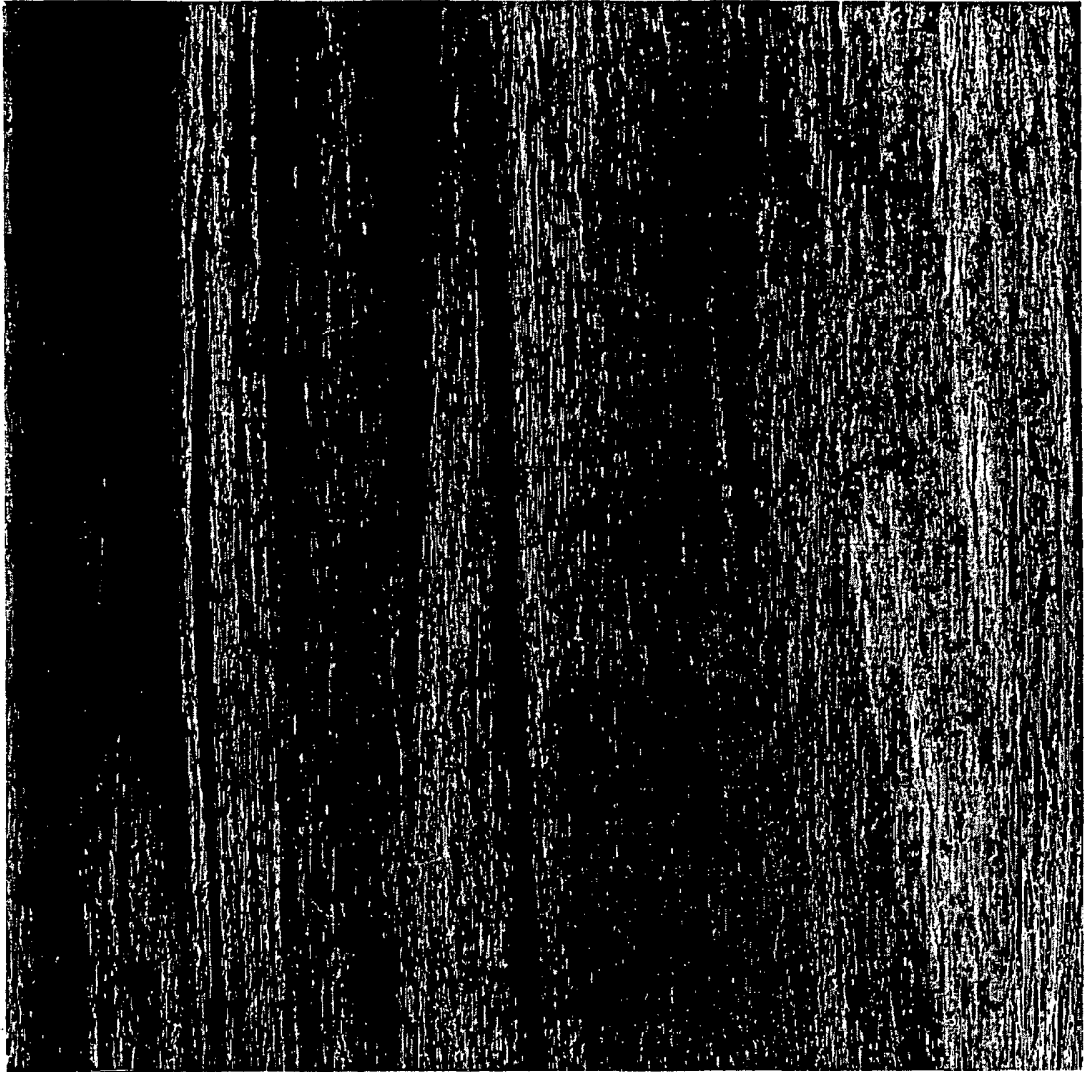


Fig. 11b

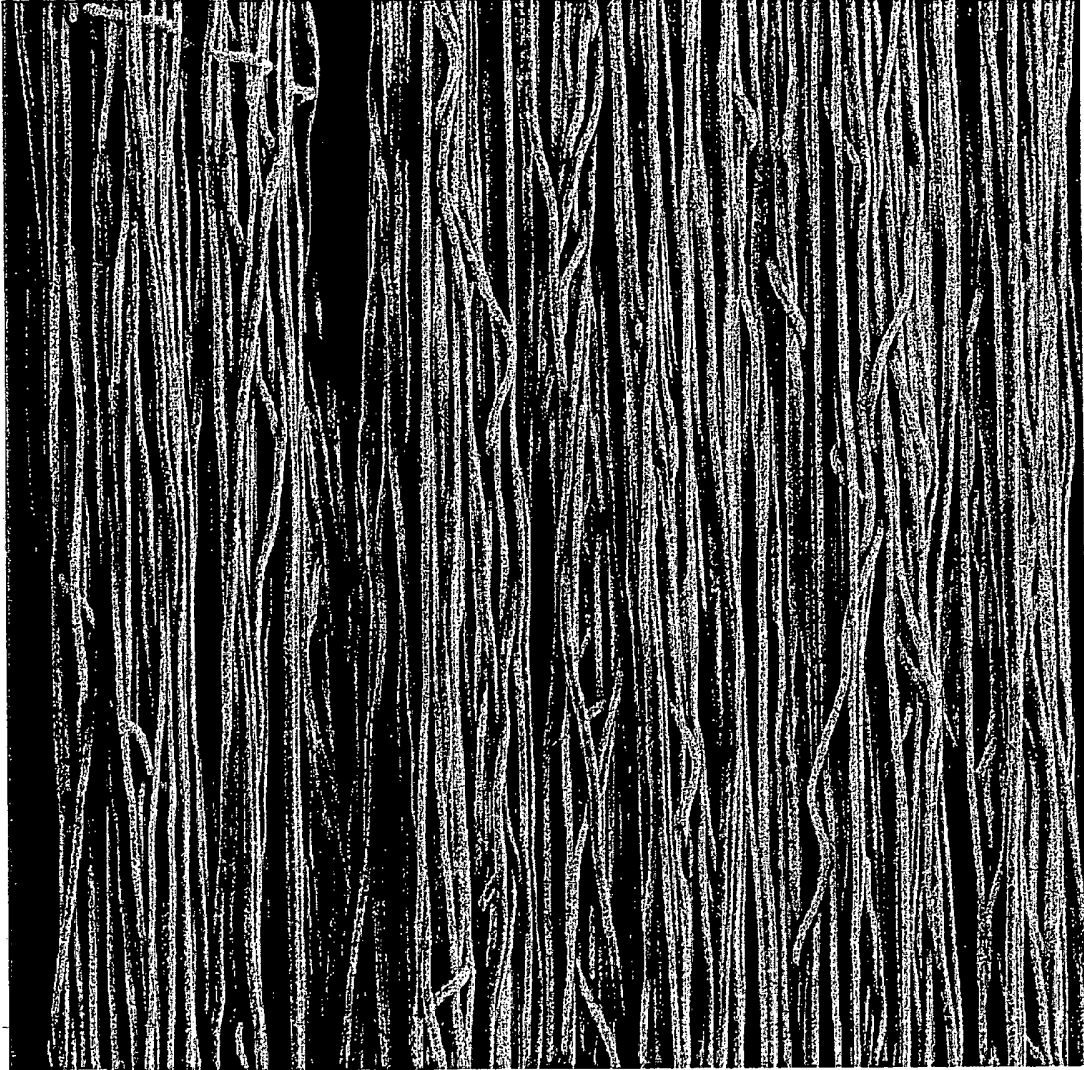


Fig. 11c

REFERENCES CITED IN THE DESCRIPTION

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Szabadalmi igénypontok:

1. Berendezés mikroszálas és nanoszálas kétdimenziós vagy háromdimenziós szálas anyagok előállítására, amely tartalmaz legalább egy fém fonó fúvókát (3), mely egy első potenciálra van csatlakoztatva, egy kollektornak az elektróda (6) készletét, a kollektornak az elektróda (6) készlete tartalmazza a kollektornak legalább két elektródáját (6), amelyek egy síkban vannak elhelyezve, szemben a fúvókával vagy fúvókákkal (3), és úgy vannak elhelyezve, hogy egymáshoz képest állandó távolságra vannak, és egy második potenciálra vannak csatlakoztatva, **azzal jellemezve**, hogy tartalmaz egy gyűjtő lapot (7) vagy egy gyűjtő hengert (14) a mikroszálaknak vagy nanoszáalaknak az összegyűjtésére, amelyek a kollektornak az egymással szomszédos, párt alkotó elektródái (6) között helyezkednek el, és a gyűjtő lap (7) el van látva résekkel, amelyeken keresztül a kollektor elektródái (6) haladnak, míg a gyűjtő lap (7) a metszésvonalában vagy a gyűjtő hengernek (14) egy érintője, amely merőleges a kollektor elektródáinak (6) a síkjában levő kontaktus vonalra, a kollektor elektródáinak (6) a síkjával egy α szöget alkot, amelynek mérete 0° és 90° közötti tartományban van, a gyűjtő lap (7) vagy a gyűjtő henger (14) a kollektor elektródáihoz (6) viszonyítva mozgathatóan van elhelyezve abban az irányban, amely abban a síkban van, mely merőleges a kollektor elektródáinak (6) a síkjára, és amelyben az elektróda (6) tengelye fekszik, míg a gyűjtő lap (7) vagy a gyűjtő henger (14) mozgásának az iránya az elektróda (6) tengellyel egy β szöget alkot, amelynek mérete 0° és 90° közötti tartományban van.
2. Az 1. igénypont szerinti berendezés mikroszálas és nanoszálas kétdimenziós vagy háromdimenziós szálas anyagok előállítására, **azzal jellemezve**, hogy a gyűjtő lap (7) el van látva nyitott párhuzamos résekkel, melyek mindegyike a kollektor elektródáinak (6) egyikével szemben van elhelyezve, míg a gyűjtő lapnak (7) két szomszédos rés közötti kiemelkedései a kollektor két szomszédos elektródái (6) között levő térbe vannak beillesztve.
3. Az 1.-2. igénypontok bármelyike szerinti berendezés mikroszálas és nanoszálas kétdimenziós vagy háromdimenziós szálas anyagok előállítására, **azzal jellemezve**, hogy a kollektornak az elektróda (6) készlete tartalmazza a kollektornak legalább három párhuzamos elektródáját (6), amelyek egymáshoz képest állandó távolságra vannak.

4. Az 1. igénypont szerinti berendezés mikroszálas és nanoszálas kétdimenziós vagy háromdimenziós szálas anyagok előállítására, **azzal jellemezve**, hogy a gyűjtő lap (7) tartalmaz egy felületet, amely a kollektor elektródáitól (6) el van fordítva, mely felület be van vonva egy eltávolítható szubsztrátummal, a mikroszálas vagy nanoszálas réteg szubsztrátum általi beborításának lehetővé tételére.
5. Az 1. igénypont szerinti berendezés mikroszálas és nanoszálas kétdimenziós vagy háromdimenziós szálas anyagok előállítására, **azzal jellemezve**, hogy a gyűjtő lap (7) tartalmaz egy felületet, amely a kollektor elektródáitól (6) el van fordítva, és amely el van látva egy horonnyal a gyűjtő lap (7) által összegyűjtött mikroszálas vagy nanoszálas rétegeknek az elhelyezésére.
6. Az 1. igénypont szerinti berendezés mikroszálas és nanoszálas kétdimenziós vagy háromdimenziós szálas anyagok előállítására, **azzal jellemezve**, hogy a kollektor elektródái (6) keresztmetszetének az alakja négyszög vagy derékszögű négyszög alakzat 0,1 mm – 10 mm közötti szélességi tartománnyal.
7. A 6. igénypont szerinti berendezés mikroszálas és nanoszálas kétdimenziós vagy háromdimenziós szálas anyagok előállítására, **azzal jellemezve**, hogy a kollektor elektródái (6) keresztmetszetének az alakja négyszög vagy derékszögű négyszög alakzat 1 mm – 5 mm közötti szélességi tartománnyal.
8. A 6. igénypont szerinti berendezés mikroszálas és nanoszálas kétdimenziós vagy háromdimenziós szálas anyagok előállítására, **azzal jellemezve**, hogy a kollektor elektródái (6) egymástól egy légréven át vannak elválasztva, ahol az elektródáknak az egymástól mért távolságuk oldalirányban 0,1 mm – 200 mm közötti tartományban van.
9. A 8. igénypont szerinti berendezés mikroszálas és nanoszálas kétdimenziós vagy háromdimenziós szálas anyagok előállítására, **azzal jellemezve**, hogy a kollektor elektródáinak (6) az egymástól mért távolsága oldalirányban 1 mm – 100 mm közötti tartományban van.