A method and device for production of nanofibres through electrostatic spinning of solutions or melts of polymers

Technical field

The invention relates to the method of production of nanofibres through electrostatic spinning of solutions or melts of polymers, at which the produced nanofibres are deposited on the base in the spinning chamber.

The invention also relates to the device for production of nanofibres through electrostatic spinning of solutions or melts of polymers containing the spinning chamber in which the produced nanofibres are deposited on the base.

Background art

The nanofibres are understood as the fibres whose diameter reaches the place values of nanometers (10^{-9} m), while the diameters of nanofibres produced through common methods of spinning vary in the range from about 20 nm to 800 nm.

The layers of nanofibres are in practice used mostly in connection with other layer of textile as the filtration layers for fine filtration, for manufacturing of protective clothes, in medicine as special selective porous bandages and in many other areas and applications.

The most important method for production of nanofibres is electrostatic spinning of solution or of melt of polymer. At the electrostatic spinning the suitable solution of polymer or the melt of polymer is brought in a suitable manner into the static electrical field, which is generated through the difference of electrical potentials of at least one spinning and at least one collecting electrode. Such created electrostatic, in the time not variable, field after then acts on the solution/melt of polymer, which is brought into the electrostatic field for example by rotation of spinning electrode of so called Coulomb forces of a certain value, which through a suitable choice of electrostatic potentials of the spinning and collecting electrode are acting in the direction from the spinning

electrode to the collecting electrode. The required values of these forces, thus also the intensity of electric field are then given by the fact that upon the process of electrostatic spinning these forces must overcome not only the surface tension of the solution/melt of polymer, but also the forces arising out of viscosity of solution/melt. Through acting of forces of a suitable value onto the layer of solution/melt of polymer this layer is being deformed and the equilibrium status of all participating forces then results in creating of the so called Taylor cones. From these Taylor cones, upon further acting of Coulomb force, the elongation of primary nucleus of fibre occurs, which after then through its consequent splitting creates the final nanofibres. The nanofibres are then „drawn out" from the surface of solution/melt of polymer, this approximately in the direction of lines of forces of electrostatic field created by both electrodes. In an ideal case, when only the Coulomb forces of the collecting electrode are acting on the nanofibres, the created nanofibres would copy the direction of lines of forces.

It is necessary to catch in a suitable manner the nanofibres created in this manner, preferably still before their contact with the collecting electrode, and to transport them from the spinning area in which the spinning process is taking place. Catching of nanofibres created according to the above mentioned procedure is mostly performed in such a way that into the space between the spinning and collecting electrode there is inserted a suitable base formed in most cases by the textile configuration on whose surface the nanofibres are being deposited and by its motion are being transported out of spinning area.

Upon depositing of nanofibres on surface of the textile configuration in practice there nevertheless occur several problems which result especially from the fact, that the mentioned textile configuration is not conductive in many cases and it has properties of electrical insulator - dielectric. Upon electrostatic spinning of solution/melt of polymer, to the spinning electrode possibly to the solution/melt of polymer, there is brought a certain electric potential, which is by that the spinning electrode is partially dipped into the solution/melt of polymer, brought also to the solution/melt of polymer, respectively to the spinning electrode. The solution/melt of polymer and out of it produced nanofibres bear

the same charge as the spinning electrode. After the electrically charged nanofibres are brought to the electrically non-conducting textile configuration, thanks to the insulating properties of the textile configuration the electrical charge is not being carried out, and the textile configuration is being charged with the same charge as the incoming nanofibres have. Charging of the textile configuration occurs according to its type and properties, either immediately or in a short time interval after its contact with the charged nanofibres. This fact has an adverse negative impact to the process of electric spinning, this especially because of the two reasons. The first is a total weakening and the fact that the original electrostatic field becomes non homogenous, another then also the mere repulsion of approaching nanofibres by the base due to the known reality of mutual repulsion of identical charges. This results in a considerably worsened creation of nanofibres, which becomes evident by a marked reduction in homogeneity of nanofibrous layer – if only this layer is being produced at all, by a increased diameter of nanofibres, which at the same time are transported and deposited in the whole spinning space in an undesirable manner. Consequently, the whole process of creation of nanofibres may be totally stopped after then.

The objective of the invention is to eliminate or at least to minimise the problems which occur during production of nanofibres through electrostatic spinning of solutions or melts of polymers at which the produced nanofibres are being deposited on the base in the spinning chamber.

The principle of invention

The objective of the invention has been reached through the method of production of nanofibres through electrostatic spinning according to the invention whose principle consists in the fact that before coming into the spinning chamber the electrical conductivity of the base is increased. That means the base with an increased electrical conductivity comes into the electrostatic field in the spinning chamber, e.g. instead of the originally electrically non-conductive base the electrically conductive base, so that the

above described disturbance of electrostatic field does not occur, and the process of electrostatic spinning is running in its principle in an undisturbed manner as in a case of electrically sufficient conductive base materials.

In some cases it is advantageous if the electrical conductivity on the surface of the base is increased, on its impacting side. In other cases it is necessary because of the technological reasons to increase the electric conductivity in the whole volume of the base.

As described in the claim 4 it is advantageous, if after depositing of the nanofibres on the base the electric conductivity of the base is decreased.

At the same time it is advantageous, if the electric conductivity of the base after the base with the deposited nanofibres leaves the spinning chamber, is decreased. The electrically non-conductive base for a certain period of time then changes to the electrically conductive base, while the length of this period of time to a certain extend depends on the speed of motion of the base material in the device for electrostatic spinning of the solution/melt of polymer, in some case on the efficiency with which the electrically conductive liquid is removed from the electrically conductive base, while it can be said that the base is electrically conductive only after it enters the equipment for increasing of the electrical conductivity of the base, in the spinning chamber and before leaving the device for decreasing the electric conductivity of the base, which are the part of the device for electrostatic spinning according to the invention. Electric conductivity of the base unless it is a technological requirement, does not intervene into possible further production operations which are performed outside the spinning device before or after depositing of nanofibrous layer.

Increasing of electric conductivity of the base is reached by bringing the conductivity increasing liquid to the base, which through interaction with the base causes increase in electric conductivity of the base to a required level.

According to an advantageous execution mentioned in the claim 7 the conductivity increasing liquid is brought to the base in a gaseous status. Through interaction with the base either the condensate in the form of droplets or a liquid film covering the fibres of the base is created or the molecules of the

liquid through the physical forces bind to the fibres and simultaneously to a various extent penetrate into the volume of fibres of the base. In the real practice, the common participation of all these actions could almost always be expected.

At the execution according to the claim 8 the liquid is brought to the base in a liquid state, while according to the claim 9 it is advantageous if the liquid is brought in the form of aerosol.

The suitable conductivity increasing liquids are the polar solvents. If there is a requirement for preserving of conductivity of the base after applying the layer of nanofibres, the conductivity increasing liquid are the solutions of salts, which after drying create on the base the conductive layer at least from the impacting side of the base. With advantage decreasing of electric conductivity of the base could be achieved through removing the conductivity increasing liquid from the base or at least by reducing the quantity of the conductivity increasing liquid in the base.

The principle of the equipment for production of nanofibres through electrostatic spinning of solutions or melts of polymers according to the invention lies in the fact that in the direction of motion of the base before the spinning chamber there is arranged a device for increasing the electrical conductivity of the base.

At the same time it is advantageous if in the direction of motion of the base after the spinning chamber there is arranged the device for decreasing the electrical conductivity of the base.

The device for increasing of electrical conductivity of the base contains in the advantageous execution the means for bringing the conductivity increasing liquid to the base, this either to both sides of the base or to the impacting side of the base.

Description of the drawing

The example of embodiment of the device according to the invention is schematically shown in the drawing.

Examples of embodiment

The method of production of nanofibres through electrostatic spinning and the device for production of nanofibres through electrostatic spinning of solutions or melts of polymer will be explained on the example of embodiment of the device for production of nanofibres through electrostatic spinning of solution of polymer schematically shown in the drawing, which contains the spinning chamber **1**, in which there is positioned the spinning electrode **11**, the collecting electrode **12** and the reservoir **13** of the solution **14** of polymer. The spinning electrode **11** is in a known manner in a pivoted way mounted in the reservoir **13** of solution of polymer and with a section of its surface intervenes into the solution **14** of polymer in the reservoir **13**. Upon rotation of the spinning electrode **11** around its longitudinal axis the spinning electrode **11** carries out with its surface a certain quantity of solution **14** of polymer into the spinning space **15** between the spinning electrode **11** and the collecting electrode **12**.

The collecting electrode **12** is arranged above the spinning electrode **11**, while the longitudinal axis of the spinning electrode **11** is parallel with the plane, which is tangential to the surface of the collecting electrode **12**, while the collecting electrode **12** and the spinning electrode **11** are in a known not illustrated manner connected to the opposite poles of the source of high voltage or the one is grounded and to the second there is brought one pole of a high voltage. In this way there is between them created the electrical field, which through its action of force to the volume of solution of polymer, carried out by the rotation motion of the spinning electrode **11** on the surface of spinning electrode **11** into the electrostatic field, ensures origination of Taylor cones and after then also origination of nanofibres. Voltage to the spinning electrode **11** can be brought also through bringing voltage into the spinning solution **14** of polymer through some of the known methods.

At the illustrated example of embodiment there is in the space between the collecting electrode **12** and the spinning electrode **11** created in the spinning chamber **1** in the vicinity of the collecting electrode **12** the track for guiding the base **2**, which is usually represented by a strip mostly of a textile material.

In the direction of motion of the base 2 before the spinning chamber 1 there is arranged an equipment 3 for increasing the electric conductivity of the base. In the direction of movement of the base after the spinning chamber 1 there is arranged a device 4 for decreasing of electrical conductivity of the base 2.

In the illustrated example of embodiment the base 2 is in a known manner unwound from the carrier roll 5 and before entering the device 3 for increasing the electrical conductivity of the base it passes through a pair of feeding rollers 6. After leaving the device 4 for decreasing the electrical conductivity of the base the base 2 with the deposited layer of nanofibres passes through a pair of take-up rollers 7 and it is in a known manner wound onto the fabric roll 8.

In the device 3 for increasing the electrical conductivity of the base 2 there are against the base 2 arranged the means 31 and 32 for bringing the conductivity increasing liquid 311 to the base 2, while the first means 31 for bringing the conductivity increasing liquid 311 to the base 2 are oriented to the impacting side 21 of the base 2 and the second means 32 for bringing the conductivity increasing liquid 311 to the base 2 are oriented to the opposite side of the base 2. The impacting side 21 is the side of the base 2 facing the spinning electrode 11. At the same time the conductivity increasing liquid 311 may be in a liquid or gaseous state.

In case of a liquid state to the base 2 there is brought aerosol of a liquid which on the surface of the base creates from one or from both sides a liquid film and/or the molecules of the liquid through the physical forces bind to the base and simultaneously to a various extent penetrate into the volume of the base 2. At the same time for a quality spinning it is essential that the conductivity increasing liquid 311 is applied at least to the impacting side 21 of the base 2.

If there is a requirement to preserve conductivity of the base 2 after applying a layer of nanofibres, the conductivity increasing liquid 311 are the

solutions of salts, which after drying create on the base 2 the conductive layer at least from the impacting side 21 of the base.

In case of a gaseous state of the conductivity increasing liquid 311 to the base there are brought the vapours of a liquid, which through interaction with the base 2 create a condensate in the form of droplets and/or a liquid film and/or the molecules of the liquid through the physical forces bind to the base and simultaneously to a various extent penetrate into the volume of the base 2.

The conductivity increasing liquid is the polar solvent, e.g. alcohol or an acid. The polar solvents are understood as the solvents which contain a dipole in its molecule.

In this manner the electrically non-conductive base becomes the electrically conductive base and at the electrically conductive base the electrical conductivity is being increased. The treatment of electrically non-conductive base 2 to the electrically conductive base 2 is running in the device 3 for increasing of the electrical conductivity of the base, which is positioned before the spinning chamber 1. After entering the device 3 for increasing of the electrical conductivity of the base to the base 2 there is applied the conductivity increasing liquid 311, which renders to the electrically non-conductive base 2 a certain electrical conductivity, as from the point of view of fluency of the process of electrostatic spinning it is advantageous that the electrically non-conductive base 2 after being brought into the electrostatic field between the spinning electrode 11 and collecting electrode 12 was acting to a certain level as an electric conductor. Applying of the conductivity increasing liquid 311 to the base 2 is being realised through some of the known methods and the liquid is brought to the surface of the base 2 from its one and/or second side, where it is captured. From the above mentioned it is then obvious that into the device 3 for increasing the electrical conductivity of the base there is entering the electrically non-conductive base 2 and from it leaves the electrically conductive base whose electrical conductivity is caused by presence of the conductivity increasing liquid 311 on the impacting side 21 of the base 2 or in the whole volume of the base 2. From the device 3 for increasing the

electrical conductivity of the base the electrically conductive base 2 enters the spinning chamber 1.

In the spinning chamber 1 through the force acting of electric field between the spinning electrode 11 and collecting electrode 12 the nanofibres are produced and they are deposited on the electrically conductive base 2. After depositing of a layer of nanofibres of required parameters the electrically conductive base 2 with deposited layer of nanofibres leaves the spinning chamber 1 and it is led to the device 4 for reducing the electrical conductivity of the base 2, which, in the illustrated embodiment, consists of drying chamber in which the electrically conductive liquid is removed from the electrically conductive base 2 with deposited layer of nanofibres, through which the electrically conductive base 2 with deposited layer of nanofibres becomes an electrically non-conductive base with deposited layer of nanofibres. One of possible ways for removing the electrically conductive liquid is for example drying when the electrically conductive base 2 with deposited layer of nanofibres is subject to stream of a certain quantity of air having a certain temperature and speed. The resulting product leaving the device 3 for increasing the electrical conductivity of the base is after then electrically non-conductive base 2 with deposited layer of nanofibres. The decreasing of electrical conductivity of the base 2 takes place gradually also in the spinning chamber, nevertheless to the greatest extent the conductivity is reduced only after the nanofibres are deposited. The above mentioned solution illustrated in the Fig. 1 seams to be an optimum one.

The conductivity increasing liquid is brought to the base in the form of vapours and/or aerosol, while aerosol may be replaced by a stream of liquid from the nozzles arranged along the width of the base 2. The means for bringing the conductivity increasing liquid 311 in the gaseous state are with advantage created by nozzles arranged along the width of the base 2.

The rate of increasing of electrical conductivity of the base 2 is being regulated either by changes of forward motion of the base 2 or by a quantity of the conductivity increasing liquid 311 being brought.

The conductivity increasing liquid **311** is from the base **2** removed in various manners in the washing and/or drying device, for example it is at least partially removed by washing in a suitable solvent, after which the solvent and the possible rests of the conductivity increasing liquid **311** are from the base **2** removed by heating and/or flushing and/or rinsing by air and/or other gas. In other embodiment the conductive increasing liquid **311** is from the base removed by heating and/or flushing and/or rinsing by air and/or by other gas. Heating is performed by means of microwave and/or infrared radiation and/or using a warm air and/or other warm gas.

List of referential markings

1	spinning chamber
11	spinning electrode
12	collecting electrode
13	reservoir for solution of polymer
14	solution of polymer
15	spinning space
2	base
21	impacting side of the base
3	device for increasing the electrical conductivity of the base
31,32	means for bringing the conductivity increasing liquid to the base
311	conductivity increasing liquid
4	device for decreasing the electrical conductivity of the base
5	carrier roll
6	pair of feeding rollers
7	pair of take-up rollers
8	fabric roll

CLAIMS

1. The method of production of nanofibres through electrostatic spinning of solutions or melts of polymers, at which the produced nanofibres are deposited on the moving base in the spinning chamber **characterised by that the** electrical conductivity of the base (2) is increased before entering into the spinning chamber (1).

2. The method according to the claim 1, **characterised by that the** electrical conductivity of the base is increased on surface of the base (2) on its impacting side (21).

3. The method according to the claim 1 **characterised by that the** electrical conductivity of the base is increased in the whole volume of the base.

4. The method as claimed in any of the claims 1 to 3 **characterised by that the** electrical conductivity of the base (2) is decreased after depositing of nanofibres.

5. The method according to the claim 4 **characterised by that the** electrical conductivity of the base (2) is being decreased after the base (2) with deposited nanofibres leaves the spinning chamber (1).

6. The method according to any of the previous claims **characterised by that the** increasing of electrical conductivity of the base (2) is reached through bringing the conductivity increasing liquid (311) to the base (2).

7. The method according to the claim 6 **characterised by that the** conductivity increasing liquid (311) is brought to the base (2) in a gaseous state.

8. The method according to the claim 6 or 7 **characterised by that the** conductivity increasing liquid (311) is brought to the base (2) in a liquid state.

9. The method according to the claim 8, **characterised by that the** liquid is brought in a form of aerosol.

10. The method according to any of the previous claims **characterised by that the** conductivity increasing liquid (311) is a polar solvent.

11. The method as claimed in any of the claims 1 to 10, **characterised by that the** conductivity increasing liquid (311) is a solution of salt.

12. The method as claimed in any of the claims 4 to 11, **characterised by that the** decreasing of electrical conductivity of the base (2) is reached by removing the conductivity increasing liquid (311) from the base (2) or at least by reducing the quantity of the conductivity increasing liquid (311) in the base (2).

13. The device for production of nanofibres through electrostatic spinning of solutions or melts of polymers containing the spinning chamber, in which the created nanofibres are deposited to the base, **characterised by that** the device (3) for increasing of the electrical conductivity of the base (2) is arranged in the direction of motion of the base (2) before the spinning chamber (1).

14. The device according to the claim 13, **characterised by that the** device (4) for decreasing the electrical conductivity of the base (2) is arranged after the spinning chamber (1).

15. The device according to the claim 13 or 14, **characterised by that the** device (3) for increasing the electrical conductivity of the base (2) contains the means (31, 32) for bringing the conductivity increasing liquid (311) to the base (2).

16. The device according to the claim 15, **characterised by that the** means (31, 32) for bringing the conductivity increasing liquid (311) are arranged along both sides of the base (2).

17. The device according to the claim 15, **characterised by that the** means (32) for bringing the conductivity increasing liquid (311) to the base (2) are arranged on the impacting side of the base (2).