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(54) **Composite filtering material**

Kompositfiltermaterialien

Matière filtrante composite

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(56) References cited:
US-A- 4 127 706 **US-A- 5 554 722**

EP 1 443 136 B1

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Description

[0001] The present invention concerns a non-woven composite fiber structure obtainable by a method of claim 1.

[0002] Any device and process of manufacturing composite fiber structure mentioned in the present invention are only presented as background art.

[0003] The present invention is related to filtering means, in particular to composite polymeric fiber filters, and to the technology for their manufacture.

[0004] The creation of filtering materials capable of trapping particles of 0.1-10 microns in size and their increasing use is related to increasingly stringent requirements for quality and reliability of manufactured commodities as well as to the rapid development of modern technology and production processes (electronics, aviation, automobile industry, electrochemical industry, biotechnology, medicine). The main industrial manufacturing methods for such materials include production from polymer solutions (V. P. Dubyaga et al., *Polymer Membranes*, "Chemistry" Publishing House, Moscow, 1981 (in Russian); V. E. Gul and V. P. Dyakonova, *Physical and Chemical Principles of Polymer Films Manufacture*, "Higher School Publishing House, Moscow, 1978 (in Russian); German patent DE 3,023,788, "Cationic absorbent for removing acid dyes etc. From waste water - prepared from aminoplast precondensate and aminamide compound"), from powders and powder polymer composites (P. B. Zhivotinskiy, *Porous Partitions and Membranes in Electrochemical Equipment* "Chemistry" Publishing House, Leningrad, 1978 (in Russian); *Encyclopedia of Polymer Science and Engineering*, Wiley, New York, 1987, Vol. 8 p. 533), from macromonolithic films (I. Cabasso and A. F. Turbak, "Synthetic membranes", Vol. 1. *ACS Symposium, Ser. 154*, Washington DC, 1981, p. 267), and from fibers and dispersions of fibrous polymers (T. Miura, "Totally dry nonwoven system combines air-laid and thermobonding technology", *Nonwoven World* Vol. 73 (March 1988) p.46). The latter method is the most widespread, since it facilitates the manufacture of materials with the optimal cost-quality ratio.

[0005] Great interest is also being expressed in the extension of the traditional uses of filtering materials, especially to combination functions of trapping micro-particles in gaseous and liquid media with the adsorption of molecular admixtures, for example, in the removal of mercaptans, as substrate for catalytic reactions, in the enhancement of the bactericidal effect of the filtering material, etc. Fulfillment of these additional functions is possible due to the introduction into the fiber matrix of fillers of some sort or functional groups giving the formation of additional solid phase, i.e., as a result of manufacturing of composite filtering materials.

[0006] At present, polymeric filtering materials are manufactured from synthetic fibers by means of a technology that is similar in many aspects to the traditional

technology applied in the pulp and paper industry. A long fiber thread is cut into pieces of a given length, which are then subjected to some basic and supplementary operations out of more than 50 possibilities, which may include chemical processing for modification of surface properties, mixing with binding and stabilizing compositions, calendaring, drying process, etc. (O. I. Nachinkin, *Polymer Microfilters*, "Chemistry" Publishing House, Moscow, 1985 (in Russian), pp. 157-158). The complexity of such a technological process hampers the manufacture of materials with stable characteristics for subsequent exploitation; results in the high cost of manufactured filtering materials; and practically excludes the manufacture of composites with fillers sensitive to moist, thermal processing.

[0007] There is, however, a method for the manufacture of ultra-thin synthetic fibers (and devices for their production), which facilitates the combination of the process of fiber manufacture with the formation of a microporous filtering material, and thus reduces the number of technological operations, precludes the necessity for aqueous reaction media, and increases the stability of properties of the product being manufactured (see, for example, US-A-2,349,950). According to this method, known as "electrocapillary spinning", fibers of a given length are formed during the process of polymer solution flow from capillary apertures under electric forces and fall on a receptor to form an unwoven polymer material, the basic properties of which may be effectively changed. With this method, fiber formation takes place in the gaps between each capillary, being under negative potential, and a grounded anti-electrode in the form of a thin wire, i.e., in the presence of a heterogeneous field, being accompanied by corona discharge. However, the process of solvent evaporation takes place very rapidly, and as a result the fiber is subjected to varying electric and aerodynamic forces, which leads to anisotropy along the fiber width and formation of short fibers. Manufacture of high-quality filtering materials from such fibers is thus impossible.

[0008] Exploitation of a device for executing the method described above is complicated by a number of technological difficulties:

1. Capillary apertures become blocked by polymer films that form under any deviation from the technological process conditions - concentration and temperature of solution, atmospheric humidity, intensity of electric field, etc.
2. The presence of a large number of such formations leads to a complete halt of the technological process or drops form as a consequence of the rupture of the aforementioned films.

[0009] Therefore, the manufacture of synthetic fibers by this method is possible from only a very limited number of polymers, for example, cellulose acetate and low molecular weight polycarbonate, which are not prone to the

defects described above.

[0010] It is necessary to take into account the fact that such an important parameter of filtering materials as monodispersity of the pores (and the resultant separation efficiency of the product) has, in this case, a weak dependency on fiber characteristics and is largely determined by the purely probabilistic process of fiber stacking.

[0011] Modern filtering materials are subject to strict, frequently contradictory, requirements. In addition to high efficiency of separation of heterogeneous liquid and gas systems, they are required to provide low hydro- (or aero-) dynamic resistance of the filter, good mechanical strength, chemical stability, good dirt absorption capacity, and universality of application, together with low cost.

[0012] The manufacture of such products is conditional on the use of high-quality long and thin fibers with an isometric cross-section, containing monodispersed pores and exhibiting high porosity. The practical value of this product may be greatly increased as possible applications are expanded due to the formation of additional phases, i.e., in the manufacture of the above-mentioned composite filtering materials.

[0013] Therefore, the main objective of the proposed technical solution is removal of the above-listed defects of known solutions for filtering applications (primarily directed at the manufacture of microfilters from polymer fibers) and other purposes, including application as micro-filtering means, i.e., the creation of means and the meeting of the above-listed requirements for technical means for the manufacture of micro-filtering materials with new consumer properties.

[0014] US-A-4 127 706 describes a method of preparing a porous sheet product which comprises the step of introducing a spinning liquid comprising an organic fiber forming polymeric material into an electric field whereby fibers are drawn from the liquid to an electrode and collecting the fibers so produced upon the electrode. PTFE and other fluorinated polymer mats produced by the electrostatic process are useful as electrolytic cell diaphragms, battery separators etc.

[0015] US-A-5 554 722 discloses aromatic polyamide compositions with improved electrostatic properties, formed structures produced therefrom, and use and production thereof.

[0016] Described are compositions comprising an aromatic polyamide which is soluble in organic solvents and from 0.01 to 30% by weight, based on the weight of the composition, of an organic or organometallic charge control agent.

[0017] The compositions can be used for producing formed structures, in particular fibers; these fibers are preferably used for producing webs which can be used for producing dust filters.

[0018] The non-woven composite fiber structure obtainable by a method of forming a polymer is defined in claim 1.

[0019] Any device and process of manufacturing com-

posite fiber structure mentioned in the present invention are only presented as background art.

SUMMARY OF THE INVENTION

[0020] According to the present invention there is provided a device for transforming a liquefied polymer into a fiber structure, including: (a) a substantially planar precipitation electrode; (b) a mechanism for charging the liquefied polymer to a first electrical potential relative to the precipitation electrode; (c) a mechanism for forming a surface on the liquefied polymer of sufficiently high curvature to cause at least one jet of the liquefied polymer to be drawn by the first electrical potential to the precipitation electrode of high surface curvature are formed by forcing the polymer solution through a bank of nozzles. The nozzles of the electrode-collector are inserted lengthwise in cylindrical holes sited at intervals in a negatively charged cover plate of the electrode-collector. The source of solvent vapors is connected to the holes. In an alternative configuration, the nozzles are connected by a system of open channels to the solvent vessel.

[0021] In one of the implementations, the device is provided with an additional grounded electrode, which is placed in parallel to the surface of the nozzles of the electrode-collector and which is able to move in the direction normal to the plane of the electrode-collector's nozzles.

[0022] In order to improve the manufacturing process, the additional electrode may take the form of a single wire stretched over the inter-electrode space.

[0023] The additional electrode may also take the form of a perforated plate with flange, in which case the surface of the additional electrode, the flange, and the electrode-collector form a closed cavity, and the apertures of the perforated plate are co-axial to the apertures of electrode-collector.

[0024] Preferably, a device of the present invention also includes an aerosol generator, made in the form of a hollow apparatus (fluidized bed layer) divided into two parts by a porous electro-conducting partition, which is connected to a mainly positive high-voltage source. The lower part of the cavity forms a pressure chamber, which is connected to a compressor, and the upper part of the cavity is filled with the dispersible filler, for example, polymer powder.

[0025] Alternatively, the aerosol generator may be made in the form of a slot sprayer, connected to a positive high-voltage source and a dry fluid feeder, provided with an ejector for supplying powder to the sprayer.

[0026] Secondly, the objective put forward in the current invention is obtained by the suggested method of manufacturing of a composite filtering material, stipulating the following operations (stages):

- preparation of a polymer solution from a polymer, an organic solvent and solubilizing additives, for example, by mixing at elevated temperatures;

- pouring the polymer solution into the electrode-collector and introducing the dispersible filler, for example, from a polymer of the same chemical composition as that in the solution, into the cavity of electrified aerosol generator;
- supply of negative high voltage to the electrode-collector, and creation of hydrostatic pressure to facilitate ejection of the polymer solution through the electrode-collector nozzles to produce polymer fibers with a negative electric charge;
- transfer of the aforementioned fibers under the action of electric and, inertial forces to the precipitation electrode and chaotic stacking of the fibers on its surface to transform the fibers into an unwoven polymer material;
- displacement of above-described polymer material with the help of the precipitation electrode, followed by interaction of the polymer material with the electrified aerosol cloud formed from the dispersible filler in the aerosol generator under positive high voltage and air pressure, accompanied by penetration of the aerosol cloud into the structure of the negatively charged unwoven polymer material to form a homogeneous composite filtering material.

[0027] The filtering material prepared by the current method can be subjected to pressing.

[0028] The invention is herein described, by way of example only, with reference to the accompanying drawings, wherein:

FIG. 1 is a schematic diagram of a device of the present invention, including two alternative electrified aerosol generators;

FIG. 2A is a top view of the electrode-collector of the device of FIG. 1;

FIG. 2 is a lateral cross section of the electrode-collector of FIG. 2A;

FIGs. 3 and 4 are lateral cross sections of alternative nozzle-based electrode-collectors;

FIG. 5 is a lateral cross section of an electrode-collector based on a rotating wheel;

FIG. 6 is a lateral cross section of an electrode collector based on reciprocating needles.

[0029] The present invention is of a device and process for the electrostatic precipitation of a polymer fiber composite structure. Specifically, the present invention can be used to make a composite nonwoven filter.

[0030] The principles and operation of the electrostatic precipitation of a nonwoven polymer fiber structure according to the present invention may be better understood with reference to the drawings and the accompanying description.

[0031] The technological process of preparation of the composite filtering material includes two basic stages, which take place simultaneously. The first consists of the formation and precipitation on a constantly moving sur-

face (base) of ultra-thin fibers from the polymer solution that flows out of the capillary apertures under the action of an electric field.

[0032] The second operation is the introduction of micro-dispersed particles of filler of a particular composition into the fiber structure (matrix) formed previously in the first stage of production.

[0033] The suggested method of manufacture of the composite filtering material is based on the realization of these two basic operations and also includes the operations described above.

[0034] A basic variant of the device of the present invention (Figure 1) includes a high-voltage electrode-collector 1, manufactured as bath, filled with the polymer solution and provided with a base 2 and a cover 2'. The electrode-collector is connected to a feeder 3 (Figure 2B) by a flexible pipe, installed so as to allow vertical movement, and a source 4 of high voltage of negative polarity.

[0035] Spinnerets 5 with nozzles 6 having capillary apertures are screwed into threaded openings in cover 2 of the electrode-collector as on a chess board (Figure 2). Because the height of the spinnerets is slightly less than the width of cover 2' and the length of each nozzle 6 exceeds the width of cover 2', the nozzle section is placed

above cover 2' on the axis of cylindrical depressions 7, connected to each other by a system of open channels 8 (Figure 2A). The solvent is fed into this system of channels from a vessel 9. A precipitation electrode 10 is situated at a certain distance above cover 2'. Precipitation

electrode 10 is manufactured in the form of a constantly moving surface (when in the operating mode), for example, a bell made of electrical conducting material. Precipitation electrode 10 is grounded. Shafts 11 and 12, connected to an electrical motor (not depicted on the pictures), are responsible for driving precipitation electrode 10, keeping precipitation electrode 10 under tension, and preliminary compression of the material on precipitation

electrode 10. Part of precipitation electrode 10 is wound around shaft 13, which has a large diameter, and is thus immersed in the rectangular cavity of the electrified aerosol generator. The cavity of the electrified aerosol generator is divided into two sections by a porous conducting partition 15. The latter is connected, to a high-voltage source 16 of positive polarity. The lower part 14 of the

electrified aerosol generator, forming pressure chamber 17, is connected to a compressor (not shown on drawings). A micro-dispersible filler is poured onto the surface of the porous partition 15 in the upper part of the generator. The entire device depicted in Figure 1 is contained

in a hermetically sealed container, provided with a suction unit and a settling chamber for trapping and recirculation of the solvent vapors (not shown on drawings).

[0036] The electrified aerosol generator may also be implemented in the form of a slot sprayer 18, connected by a pipe to a dry powder ejection feeder 19 and a source of positive high voltage 16. The use of the slot sprayer with a charging of aerosol in the field of the corona discharge is preferred in the case of metallic powders (in-

cluding graphite powder) and powders that are not easily fluidized.

[0037] The direction of fiber feeding on the vertical surface may be reversed, and the dimensions of the electrode-collector and the number of capillaries may be minimized with the help of the device depicted in Figure 3. The device consists of an electrode-collector frame 20, manufactured from a dielectric material and having a central channel 21, for example, of cylindrical shape. This channel is connected by a pipe to a feeder (not shown on the drawing) and is provided with aperture 22 to facilitate exchange of gases with the atmosphere. A busbar 23 with spinnerets 5 and nozzles having capillary apertures is installed in the lower part of frame 20. The nozzles are connected to a source of high voltage (not shown on the drawing). Cover 24 with apertures 25 is placed before the busbar. Nozzles 6 are placed in these apertures with coaxial clearance. The internal surface of the cover and busbar form a cavity 26, which is connected to a saturator (not shown on drawing) by a pipe.

[0038] In a number of cases, the process of manufacturing the composite filtering material may be improved by implementation of the device shown in Figure 4. Here, a dielectric flange 28 serves as a base for a perforated grounded plate 27, which is installed, with a certain clearance C, parallel to the surfaces of the electrode-collector 20 and the busbar 23. Plate 27 rests on the flange in such a way as to provide for vertical movement for regulation of the size of the clearance C. Apertures 29 of the perforated plate are co-axial to the apertures of electrode-collector's nozzles. The internal surface of perforated plate 27 and busbar 23 form a cavity 26, which is connected by a pipe to a saturator.

[0039] The proposed device in its basic form functions as follows: From feeder 3 (Figure 2B), the polymer solution runs into electrode-collector bath 1, and under the action of hydrostatic pressure the polymer solution begins to be extruded through the capillary apertures of nozzles 6. As soon as a meniscus forms in the polymer solution, the process of solvent evaporation starts. This process is accompanied by the creation of capsules with a semi-rigid envelope, the dimensions of which are determined, on the one hand, by hydrostatic pressure, the concentration of the original solution and the value of the surface tension, and, on the other hand, by the concentration of the solvent vapor in the area of the capillary apertures. The latter parameter is optimized by choice of the area of free evaporation from cover 2' and of the solvent temperature.

[0040] An electric field, accompanied by a unipolar corona discharge in the area of nozzle 6, is generated between cover 2' and precipitation electrode 10 by switching on high-voltage source 4. Because the polymer solution possesses a certain electric conductivity, the above-described capsules become charged. Coulombic forces of repulsion within the capsules lead to a drastic increase in hydrostatic pressure. The semi-rigid envelopes are stretched, and a number of point microruptures

(from 2 to 10) are formed on the surface of each envelope. Ultra-thin jets of polymer solution start to spray out through these apertures. Moving with high velocity in the inter-electrode interval, these jets start to lose solvent and form fibers that are chaotically precipitated on the surface of the moving precipitation electrode 10, forming a sheet-like fiber matrix. Since the polymer fiber possesses high surface electric resistance and the volume of material in physical contact with precipitation electrode surface is small, the fiber matrix preserves the negative electric charge for a long time.

[0041] When compressed air is fed into pressure chamber 17 of electrified aerosol generator 14 and high-voltage source 16 is switched on, the micro-dispersible filler becomes fluidized and acquires a positive electric charge. Under the action of electric and aerodynamic forces, the filler particles move to the surface of precipitation electrode 10, which holds the fiber matrix. As a result of the action of Coulombic forces, the filler particles interact with the fiber matrix, penetrate its structure, and form a composite material.

[0042] When the belt of precipitation electrode 10 passes between shafts 11, preliminary material compression takes place, accompanied by re-distribution of the filler particles in the matrix volume. Spherical particles, attached to the fiber material solely by electrical forces, move along paths of least resistance into micro-zones having a minimum volume density of matrix material, filling large pores, and thus improving the homogeneity of the composite and the degree of micro-dispersity of the pores.

[0043] The micro-dispersible powders from the following materials may be used as fillers: a polymer of the same chemical composition as that in the matrix, polymer latexes, glass, or Teflon, as well as active fillers that lead to the production of composite microfiltering materials with new consumer properties. These new materials may find application as adsorbents, indicators, catalysts, ion-exchange resins, pigments bactericides, etc.

[0044] The use of an electrified aerosol generator, as described above with the fluidized layer, facilitates high productivity of the process and product homogeneity. However, several powders have difficulty in forming a fluidized layer: metallic powders, particularly catalytic metals, can be subjected to electric precipitation only in the field of a unipolar corona discharge. Therefore, in these cases, as well as in the case in which it is necessary to measure out exact amounts of filler, it is worthwhile to use a slot sprayer 18 as the electrified aerosol generator (Figure 1). When compressed air from a compressor is fed into the dry powder feeder and the high voltage source is switched on, the powdered filler is ejected into slot sprayer 18. The aerosol cloud coming out of the sprayer apertures becomes charged in the unipolar corona discharge field, and under the action of electric and aerodynamic forces is transferred to the precipitation electrode, where it interacts with the fiber matrix as described above.

[0045] The functioning of the device described in Figure 3 corresponds, in the main aspects, with the operation of the basic device. The main difference is as follows: solvent vapor from the saturator under slight excess pressure is fed into cavity 26 and exits via aperture 25, flowing over the edges of the apertures of nozzles 6. The advantage of this configuration lies in the facts that it provides the possibility of easy spatial re-orientation and fiber feeding in any direction and that it can be manufactured in compact form with a small number of capillaries. A device of this type is not efficient in installations aimed at high throughput due to difficulties in obtaining homogeneous distribution of the vapor-air mixture through a large number of apertures and to the possibility of vapor condensation in pipes and subsequent falling of drops.

[0046] Intensification of the fiber matrix manufacturing process and a reduction of fiber width in order to produce filtering materials with a minimum pore size assumes, on the one hand that the intensity of the electric field should be increased to values close to the level at which electrical discharges would begin to form between the emerging fibers and precipitation electrode 10 and, on the other hand, that the concentration of solvent vapors in the inter-electrode interval be increased in order to maintain the capability of consolidating fiber formation. The optimal electric field strength, both between electrode-collector I and precipitation electrode 10, and between the electrified aerosol generator and precipitation electrode 10, is between about 2.5 KV/cm and about 4 KV/cm.

[0047] An increase in the average intensity and heterogeneity of the electric field, leading to corona discharge, may be realized by installing, in the inter-electrode interval, one or more grounded electrodes manufactured, for instance, in the form of wires. This solution facilitates an increase in the productivity of the process by 1.5-2 times, but it does not lead to formation of short fibers with, varying strength and size parameters. The negative effect of using a linear grounded electrode instead of a planar grounded electrode, thereby producing an inhomogeneous electrical field, may be reduced by increasing the solvent vapor concentration in the fiber-formation area, which is difficult in open devices and increases solvent consumption and danger of fire.

[0048] This deficiency may be overcome by application of the device described above and depicted in Figure 4.

[0049] Switching on the high-voltage source 4 in the C clearance produces an homogeneous electric field, the intensity of which may be easily increased to 10-15 KV/cm. Under these conditions, the impact of the electric field upon the jet of polymer solution increases significantly. The fiber comes out thinner and more homogeneous along its length. The initial fiber velocity also increases, and thereafter it comes through apertures 29 of perforated plate 27 and is stacked on precipitation electrode surface as described above. A change of the size of clearance C facilitates regulation of fiber thickness and device productivity, as well as the degree of material po-

rosity.

[0050] The present invention may be used to produce the polymer fiber structure from a much wider range of polymers than is possible using the prior art of US 2,349,950. Polymers amenable to the present invention include polysulfone, polyphenyl sulfone, polyether sulfone, polycarbonate in general, ABS; polystyrene, polyvinylidene fluoride, postchlorinated polyvinyl chloride and polyacrylonitrile. Suitable solvents include, *inter alia*, chloroform, benzene, acetone and dimethylformamide. The optimal concentration of the solution depends on the specific polymer and solvent used. Generally, the higher the concentration of polymer in the solution, the higher the process yield and the lower the product porosity. Concentrations of between about 10% and about 12% have been found optimal for the polymer solution used in electrode-collector 1. It has been found advantageous to add certain additives to the solutions of these polymers. Amine salts such as tetraethyl ammonium bromide and benzyltriethylammonium bromide, arc used to regulate the conductivity of the polymer solution. Small amounts of high molecular weight (order of 500,000) polyoxyalkylene additives such as polyethylene glycol and polyvinyl pyrrolidone promote the formation of the polymer solution jets by reducing intermolecular friction. Surfactants such as dimethylimidazole and ethoxytrimethylsilane enhance fiber thickness and uniformity.

[0051] More generally, the scope of the present invention includes the manufacture of the polymer fiber structure from a liquefied polymer, and not just from a polymer solution. By a liquefied polymer is meant a polymer put into a liquid state by any means, including dissolving the polymer in a solvent, as described above, and melting the polymer.

[0052] Also more generally, the scope of the present invention includes the formation of a surface on the liquefied polymer, of sufficient curvature to initiate the process discussed above of the charged capsules, leading to the formation of the jets of liquefied polymer that turn into fibers and precipitate onto precipitation electrode 10. As discussed above, if the liquefied polymer is a polymer solution, the fibers are formed by evaporation of the solvent. If the liquefied polymer is a melt, the fibers are formed by solidification of the jets.

[0053] In the process of the present invention as described above, the highly curved surfaces are the menisci of polymer solution emerging from nozzles 6. Other mechanisms for forming these highly curved surfaces are illustrated in Figures 5 and 6. Figure 5 illustrates a variant of electrode-collector I in which the polymer solution, stored in a tank 33, is pumped by a pump 32 through a feed pipe 31 to a delivery chamber 36. Rotatably mounted in delivery chamber 36 is a circular wheel 30 made of an electrically conductive material. Mounted on rim 38 of wheel 30 are triangular protrusions 40 made of a material that is wetted by the polymer solution. Tips 42 of protrusions 40 point radially outward from wheel 38. Wheel 38 is charged negatively by source 4. As the

polymer solution is delivered to chamber 36, wheel 30 rotates and each of protrusions 40 is successively coated with a layer of the polymer solution, which in turn acquires a negative charge. The surface of the portion of this polymer solution layer that surrounds tip 42 constitutes the highly curved surface whence the charged jets emerge. Polymer solution not consumed in the course of precipitating fibers onto precipitation electrode 10 is returned to tank 33 via an outlet pipe 35 by a pump 34. The optimal concentration of polymer solution used in this variant of electrode-collector 1 generally has been between about 14% and about 17%.

[0054] Figure 6 is a partial illustration, in cross-section, similar to the cross-section of Figure 2B, of a variant of electrode-collector 1 in which nozzles 6 are replaced by reciprocating needles 40, made of an electrically conductive material that is wetted by the polymer solution. Each needle 40 is provided with a mechanism 42 for raising and lowering needle 40. When a needle 40 is lowered, the sharpened tip 44 thereof is wetted and coated by the polymer solution. The surface of the polymer solution is highly curved at tip 44. When a needle 40 is raised towards precipitation electrode 10, the high voltage difference between needle 40 and precipitation electrode 10 causes jets of the polymer solution to emerge from the polymer solution surrounding tip 44 and to stream towards precipitation electrode 10. It should be noted that in this variant of electrode-collector 1, only needles 40, and hence the polymer solution thereon, are negatively charged by source 4.

[0055] Also shown in Figure 6 is a speaker 50 of a system for producing acoustical vibrations in the air above electrode-collector 1. Speaker 50 emits a tone of a single frequency, preferably in the range between about 5000 Hz and about 30,000 Hz, towards needles 40. The vibrations thus induced in the highly curved surfaces of the polymer solution on tips 44 have been found to stimulate the emission of jets of polymer solution towards precipitation collector 10.

[0056] While the invention has been described with respect to a limited number of embodiments, it will be appreciated that many variations, modifications and other applications of the invention may be made.

Claims

1. A nonwoven composite fiber structure obtainable by a method of forming a polymer into said nonwoven composite fiber structure, the method comprising the steps of:

- (a) liquefying the polymer, thereby producing a liquefied polymer;
- (b) providing a substantially planar precipitation electrode (10);
- (c) charging said liquefied polymer to a first electrical potential relative to said precipitation elec-

trode (10);

(d) forming a surface on said liquefied polymer of sufficiently high curvature to cause at least one charged jet of said liquefied polymer to be drawn to said precipitation electrode (10) by said first electrical potential difference, thereby forming a charged matrix of polymer fibers on said precipitation electrode (10); and

characterized in that

(e) supplying an aerosol of charged filler powder to said precipitation electrode (10), in a manner that electrical forces between said charged filler powder and said charged matrix uniformly distribute said charged filler powder within micro-zones having a minimum volume density of said charged matrix, thereby forming a composite material therewith.

2. The nonwoven composite fiber structure of claim 1, wherein said liquefying is effected by dissolving the polymer in a solvent, thereby creating a polymer solution and the method further comprises the step of:

(f) adding to said polymer solution an additive selected from the group consisting of amine salts, polyoxyalkylenes and surfactants.

Patentansprüche

1. Eine Vlies-Verbundfaserstruktur, erhaltbar durch ein Verfahren des Formens eines Polymers zu besagter Vlies-Verbundfaserstruktur, wobei das Verfahren die Schritte umfasst des:

- (a) Verflüssens des Polymers, **dadurch** Herstellens eines verflüssigten Polymers;
- (b) Vorsehens einer im Wesentlichen planaren Niederschlagselektrode (10);
- (c) Aufladens besagten verflüssigten Polymers auf ein erstes elektrisches Potential in Bezug auf besagte Niederschlagselektrode (10);
- (d) Bildens einer Oberfläche auf besagtem verflüssigtem Polymer von ausreichend hoher Krümmung, um das Anziehen wenigstens eines geladenen Strahls besagten verflüssigten Polymers zu besagter Niederschlagselektrode (10) durch besagte erste elektrische Potentialdifferenz zu verursachen, wodurch eine geladene Matrix von Polymerfasern an besagter Niederschlagselektrode (10) gebildet wird; und

gekennzeichnet durch

(e) Zuführen eines Aerosols von geladenem Füllstoffpulver zu besagter Niederschlagselektrode (10), auf eine Weise, dass elektrische Kräfte zwischen besagtem geladenem Füllstoffpulver und besagter geladener Matrix besagtes geladenes Füllstoffpulver gleichförmig inner-

halb von eine minimale Volumendichte aufweisenden Mikrozonen besagter geladener Matrix verteilen, wodurch sie ein Verbundmaterial damit bilden.

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2. Die Vlies-Verbundfaserstruktur von Anspruch 1, wobei besagte Verflüssigung durch Auflösen des Polymers in einem Lösungsmittel bewirkt wird, wodurch eine Polymerlösung erzeugt wird, und das Verfahren weiter den Schritt umfasst des:

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(f) besagter Polymerlösung Zusetzens eines Additivs, gewählt aus der aus Aminsalzen, Polyoxyalkylenen und grenzflächenaktiven Stoffen bestehenden Gruppe.

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(f) ajouter à ladite solution polymère un additif choisi parmi le groupe constitué par des sels d'amines, des polyoxyalkylènes et des agents tensioactifs.

Revendications

1. Structure de fibre composite non-tissée que l'on obtient via un procédé de transformation d'un polymère pour obtenir ladite structure de fibre composite non-tissée, le procédé comprenant les étapes consistant à :

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(a) liquéfier le polymère pour ainsi obtenir un polymère liquéfié ;

(b) procurer une électrode de précipitation (10) essentiellement plane ;

(c) charger ledit polymère liquéfié à un premier potentiel électrique par rapport à ladite électrode de précipitation (10) ;

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(d) former une surface sur ledit polymère liquéfié possédant une courbure suffisamment importante pour obtenir une aspiration d'au moins un jet chargé dudit polymère liquéfié en direction de ladite électrode de précipitation (10) via ladite différence procurée par le premier potentiel électrique, pour ainsi obtenir une matrice chargée de fibres polymères sur ladite électrode de précipitation (10) ;

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caractérisée en ce que

(e) on alimente avec un aérosol de poudre de matière de remplissage chargée ladite électrode de précipitation (10), de telle sorte que les forces électriques entre ladite poudre de matière de remplissage chargée et ladite matrice chargée distribuent de manière uniforme ladite poudre de matière de remplissage chargée au sein de zones microscopiques de ladite matrice chargée, possédant une densité volumique minimale, pour ainsi former un matériau composite.

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2. Structure de fibre composite non-tissée selon la revendication 1, dans laquelle ladite liquéfaction est mise en oeuvre par dissolution du polymère dans un solvant, pour ainsi obtenir une solution polymère, le procédé comprenant en outre l'étape consistant à :

55

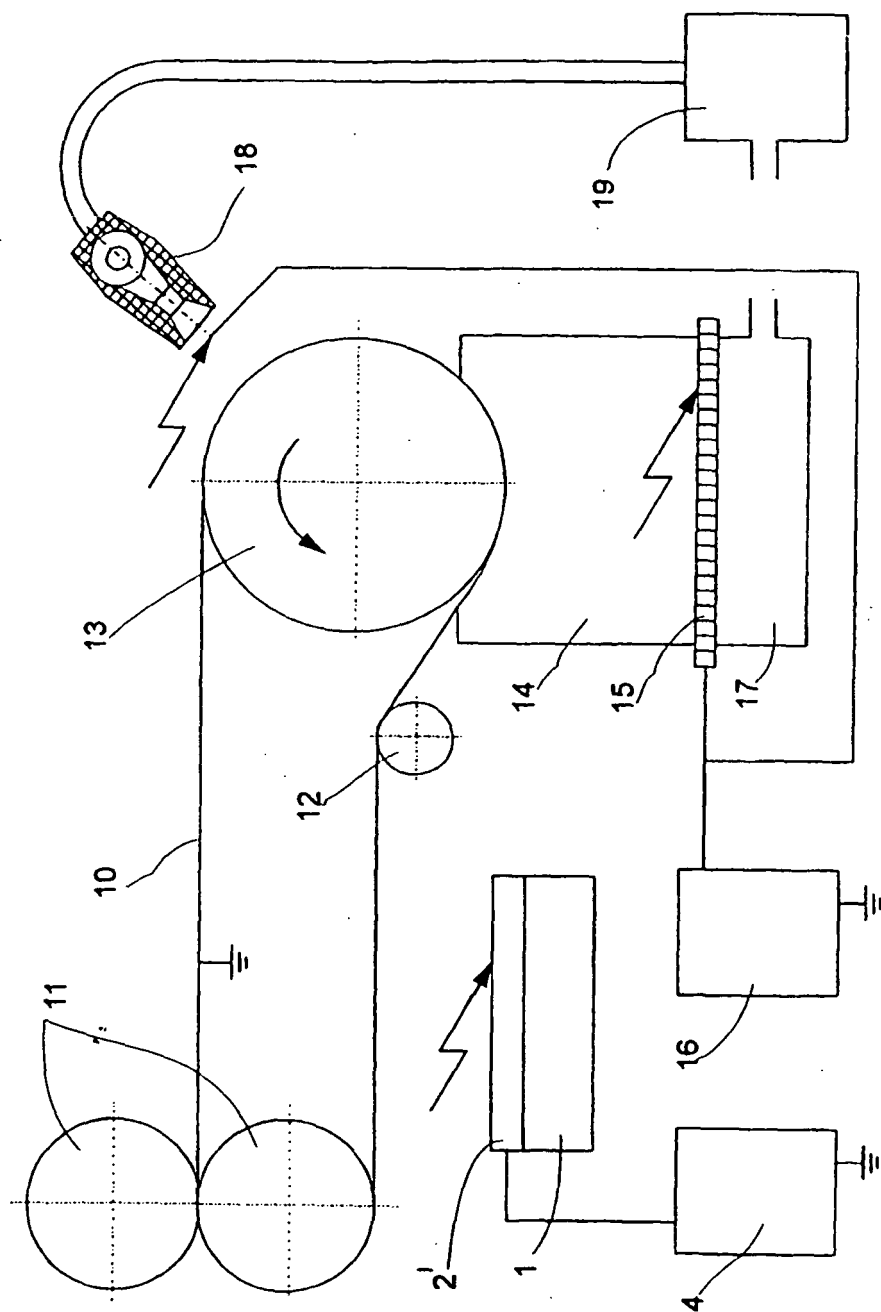


Fig. 1

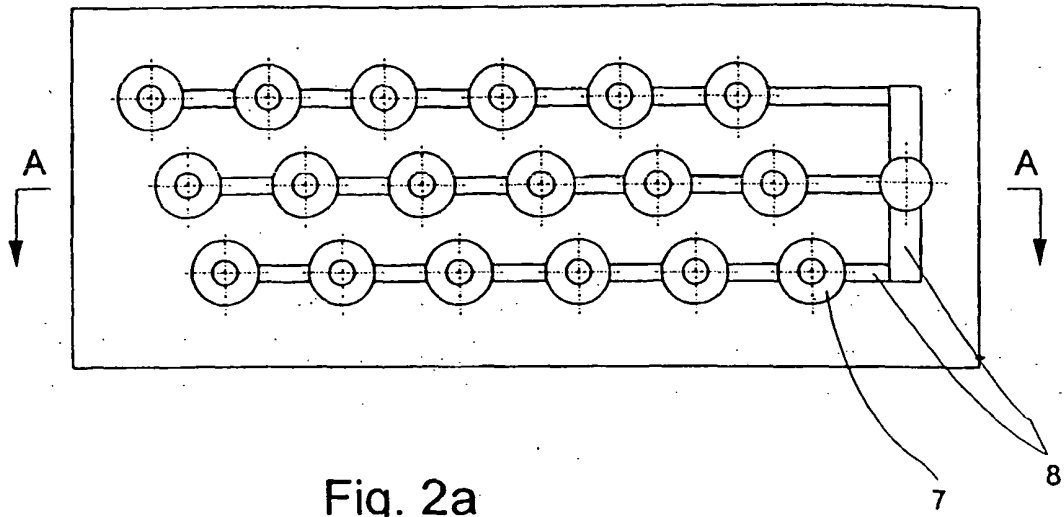


Fig. 2a

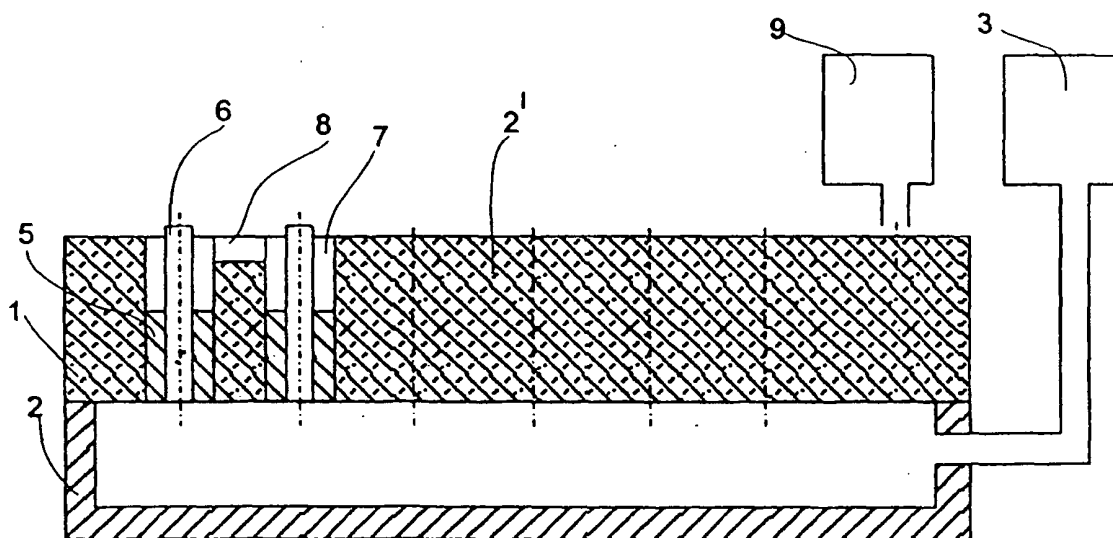


Fig. 2b

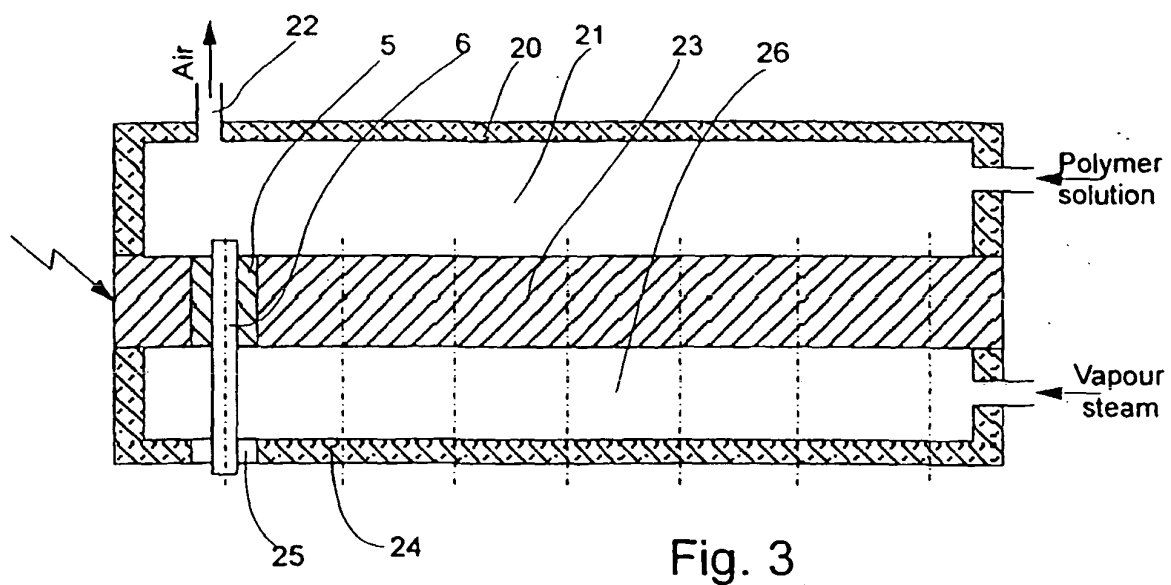


Fig. 3

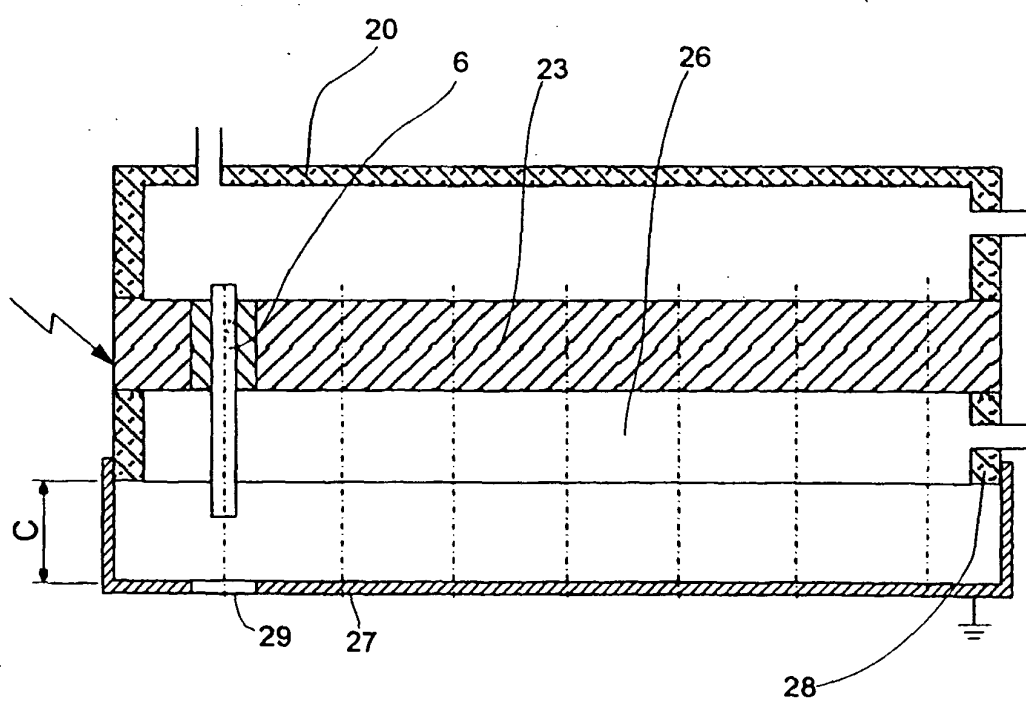


Fig. 4

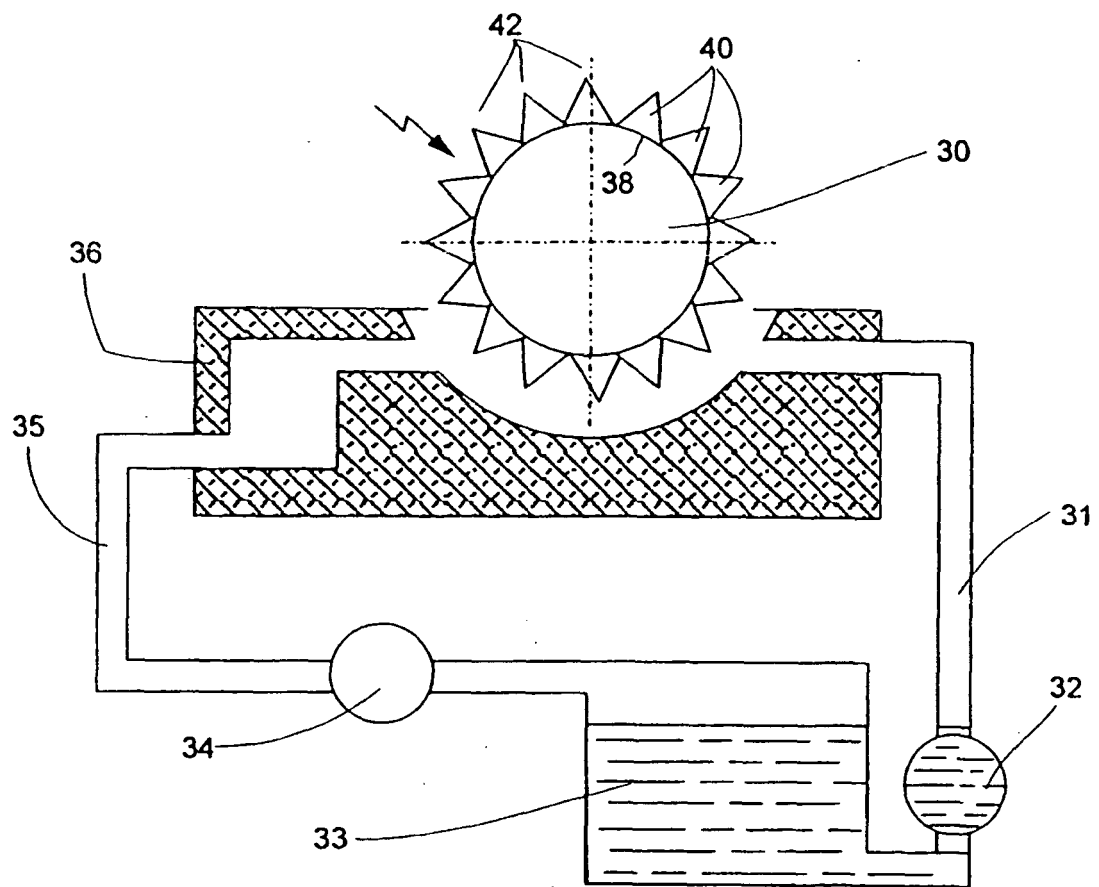


Fig. 5

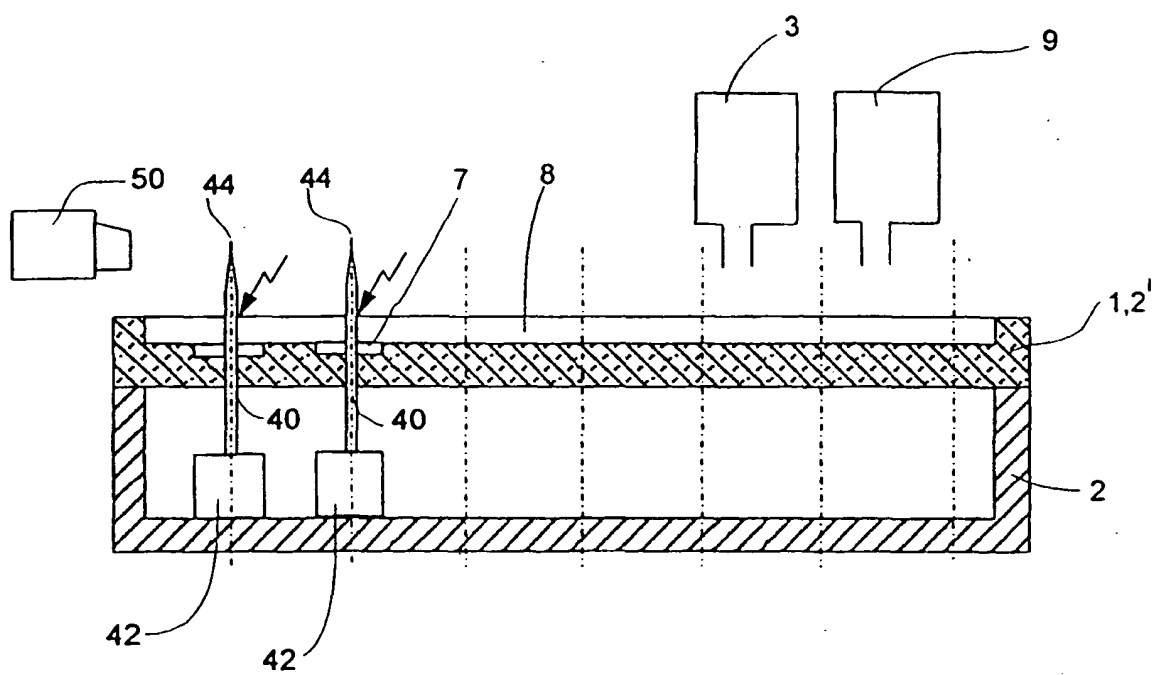


Fig. 6