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# (54) FIBER WITH NETWORK STRUCTURE, NONWOVEN FABRIC CONSTITUTED THEREOF, AND PROCESS FOR PRODUCING THE FIBER AND THE FABRIC

FASER MIT NETZWERKSTRUKTUR, DARAUS GEBILDETER VLIESSTOFF UND VERFAHREN ZUR HERSTELLUNG DER FASER UND DES VLIESSTOFFES

FIBRE A STRUCTURE RETICULEE, NON-TISSE CONSTITUE DE TELLES FIBRES, ET PROCEDE DE PRODUCTION DE LA FIBRE ET DU NON-TISSE

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#### Description

(Technical Field)

<sup>5</sup> **[0001]** The present invention relates to fine denier filament having a plexifilamentary structure composed of a mixture of polymers which are not miscible each other, to nonwoven fabric comprising said filament and having a fine fibrillated structure and also to their production, known, for instance, from US-A-5 147 586.

(Background of the Invention)

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**[0002]** With respect to a method of manufacturing fine denier filaments and nonwoven fabric using said filaments, it has been known that multi-component filaments of a sea-island type are subjected to a spinning followed by removing a part of the polymer components therefrom using a solvent and the resulting filaments are subjected to a bonding to afford nonwoven fabric. It has been also known to use the so-called melt-blowing method in which a molten polymer

- <sup>15</sup> is extruded from spinning nozzles followed by drafting with a heated fluid to fibrillate. However, in a method in which a part of the polymer components of the multi-component filaments is removed using a solvent, various steps are necessary. According to a melt-blowing method, it is true to give very fine filaments but, since said melt-blowing method conducts the fibrillation at the stage of molten polymer, there are disadvantages of little drawing orientation and crystallization whereby the resulting filaments are mechanically very weak.
- [0003] On the other hand, the so-called flash-spinning method has been known for preparing very fine filaments from a solution of polymers. As disclosed in U. S. Patent 3,081,519, said method of flash-spinning is a method in which a solution of a polymer in a low-boiling solvent is extruded from spinning nozzles to evaporate the solvent instantly.
   [0004] However, the filaments prepared by such a formulation are composed of one polymer component for any of
- the materials and, therefore, there is a problem that they have a disadvantage inherent to the polymer resulting in a restriction in the development of the use of the product. The art which is disclosed in the Japanese Examined Patent Publication 41/006,215-B and the Japanese Laid-Open Patent Publication 01/097,256-A has the same problem too. To be more precise, polyolefins are excellent in terms of their light weights but are with low modulus whereby they lack the good feel on use and putting on and have specific smoothness while polyesters are the polymers which are inherently suitable for preparing filaments of high tenacity and are with high modulus but they do not give filaments with high tenacity by a method of flash-spinning whereupon they have not been actually used
- <sup>30</sup> high tenacity by a method of flash-spinning whereupon they have not been actually used.
   [0005] EP-A-0.093.021 discloses two-component fibrillated fibres, prepared by flash spinning, comprising an inner core consisting of an olefinic polymer and an outer sheath consisting of a suited amount of hydrophilic polymer, and filaments consisting of aggregates of such individual fibres.

[0006] US-A-5.147.586 discloses a flash spun filament having a plexifilamentary structure and comprising a plexi-<sup>35</sup> filamentary structured polyolefin component.

(Dislosure of the Invention)

[0007] The present invention is to offer fine denier filament having a plexifilamentary structure in which the abovementioned problems have been solved and, particularly, the disadvantages of polyolefins and of polyesters are offset each other while advantages of them are well utilized and also to offer nonwoven fabric comprising said filament as well as methods of preparing them.

**[0008]** The present inventors have carried out extensive studies for solving the above-mentioned problems and achieved the present invention according to independent claims 1, 5, 8, 14 and 16.

- [0009] The filament in accordance with the present invention is composed of a mixture which at least comprises polyolefin and polyester which are not miscible each other and, further, said filament has a plexifilamentary structure. [0010] The method of manufacturing a filament having a plexifilamentary structure according to the present invention comprises that a mixture of polyolefin and polyester having no miscibility each other is dissolved in a solvent under high temperature and high pressure and the resulting one solution phase is spun out from nozzles under the state that the phases of the polymers and the solvent are separated.
- [0011] The filament of the present invention is composed of polyolefin and polyester, has a plexifilamentary structure comprising fibrillated filament of very highly fine denier which has not been available yet and is with high tenacity and modulus. Therefore, it is applicable as materials for textile fabrics, knitted fabrics, nonwoven fabric obtained by a dry method, non-woven fabric obtained by a wet method, etc. and stuffed staple fibres and, in addition, is suitable for
- <sup>55</sup> various purposes such as clothing materials, thermal insulation materials, artificial leather-, absorbing materials used as sanitary materials, protective clothings, curtains, bed sheets, wipers, filters, house wrappings, synthetic paper and the like. Since the filament contains polyester, it is not only coloured by pigments but also suitably dyed in the aftertreatment.

**[0012]** The nonwoven fabric having a fine fibrillated structure in accordance with the present invention contains filaments with a plexifilamentary structure, said filaments contain a mixture of polyolefin and polyester which are not miscible each other in a ratio of within a range of from 5/95 to 95/5 by weight and, further, said filaments having a plexifilamentary structure are bonded each other. The filaments having a plexifilamentary structure are bonded each other in the entire areas of the nonwoven fabric or in such a manner that many heat bonded areas are formed keeping

other in the entire areas of the nonwoven fabric or in such a manner that many heat bonded areas are for a certain distance one another. **100121** The method of manufacturing the manual fabric basis.

**[0013]** The method of manufacturing the nonwoven fabric having a fine fibrillated structure in accordance with the present invention comprises that the filaments having a plexifilamentary structure in which polyolefin and polyester which are immiscible each other are mixed within a range from 5/95 to 95/5 by weight are prepared, then the filaments are made into webs and the webs are bonded by applying heat and pressure thereto so that entire mixed plexifilaments

10 are made into webs and the w of the filaments are adhered.

**[0014]** Another method of manufacturing the nonwoven fabric in accordance with the present invention is that the filaments having a plexifilamentary structure in which polyolefin and polyester which are not miscible each other are mixed within a range from 5/95 to 95/5 by weight are prepared, then the filaments are made into webs and the webs

<sup>15</sup> are partially bonded by application of heat and pressure using an embossing apparatus so that the filaments having a mixed plexifilamentary structure are partially adhered.

**[0015]** As such, nonwoven fabric of a fine fibrillated structure comprising a highly fibrillated fine denier filaments can be prepared in accordance with the present invention. Among them, the nonwoven fabric in which the filaments having a plexifilamentary structure are bonded each other in the entire area are suitable for various purposes including enve-

- 20 lopes, packing materials, floppy sleeves, waterproof materials, labels, thermal insulation materials, synthetic paper, protective clothings as protective materials for sanitary materials, curtains, bed sheets, wipers, filters, house wrappings, etc. In addition, the nonwoven fabric in which the filaments of the plexifilamentary structure are partially bonded is suitable for various uses such as clothings, thermal insulation materials, protective clothings as medical and sanitary materials, curtains, bed sheets, wipers, filters, house wrappings, tents, artificial leather, etc.
- <sup>25</sup> **[0016]** The filament having a plexifilamentary structure in accordance with the present invention will be further illustrated in detail as hereunder.

**[0017]** The plexifilamentary structure of the present invention means the fibril filaments corresponding to  $0.01-10 \,\mu m$  in diameter are formed in a three-dimensional state and in an endless manner to the longitudinal direction.

- [0018] It is necessary that the components constituting the filament of the present invention are the polymer components which are not miscible each other. As a result of the fact that they are not miscible each other, the mixed polymer components are present independently whereby the resulting filament exhibits the fibrous characteristics inherent to each of the polymer components. It has been known that, usually, a mixed fibre of the polymers which are not miscible each other is apt to be divided into each component when a physical force is applied. In the filament of the present invention, the polymers are- not miscible each other and, therefore, fine denier fibril filament of the each polymer
- <sup>35</sup> are dominantly constructed. The specific combination of the polymer components therefor is a combination of polyolefin and polyester.

**[0019]** Examples of the polyolefin which composes the polymer components constituting the filaments of the present invention are polyethylene, polypropylene, copolymer mainly comprising ethylene, copolymer mainly comprising propylene, etc.

- 40 [0020] It is preferred that the viscosity of the ethylenic polymers among the above-given ones is 0.3-30 g/10 min in terms of a melt index value as measured by a method of ASTM-D-1238E. When the melt index value is less than 3 g/ 10 min, the viscosity of the mixed solution becomes too high whereby a fine denier fibril filaments are hardly obtained while, when it is more than 30 g/10 min, the tenacity of the filament per se lowers and, at the same time, smoothness and stickiness of the filament increase whereby there is a tendency of giving the filaments with no good hadling.
- <sup>45</sup> **[0021]** It is preferred that the viscosity of the propylenic polymers is 1-40 g/10 min in terms of a melt flow rate value as measured by a method of ASTM-D-1238L. When the melt flow rate value is less than 1 g/10 min, the viscosity of the mixed solution becomes too high whereby a fine denier fibrillated filament is hardly obtained while, when the melt flow rate value is more than 40 g/10 min, the tenacity of the filament per se lowers and smoothness and stickiness of the filament increase whereby there is a tendency of giving the filaments of no good handling.
- <sup>50</sup> **[0022]** Examples of the polyester which is another polymer component constituting the filament of the present invention are polyethylene terephthalate, polybutylene terephthalate, etc. Further examples which are applicable are the copolymers in which the above-mentioned one is a main components containing up to 40 molar % of the copolymerizing monomers such as isophthalic acid, phthalic acid, glutaric acid, adipic acid, sulfoisophthalic acid, diethylene glycol, propylene glycol, 1,4-butanediol, 2,2-bis(4-hydroxyethoxyphenyl) propane, bisphenol A, polyalkylene glycol, etc. The
- <sup>55</sup> applicable viscosity of the polymer in terms of a relative viscosity as measured by a 0.5% solution in a 1:1 (by weight) mixture of tetrachloroethane and phenol at 20°C is from around 1.3-1.6 (filament grade) to 1.7 (a highly-viscous resin prepared by a solid phase polymerization). The higher the viscosity of the polymer is, the more the filament tenacity is and, therefore, the better.

**[0023]** It is necessary that the components which constitute the filament of the present invention comprise a mixture of the polymers which are at least composed of the components being immiscible each other and having a melting point of not lower than 100°C each. The reason why they should be immiscible each other is as mentioned already while the reason why the polymer components with melting point of not lower than 100°C are necessary is a regulation

- <sup>5</sup> from a practical viewpoint. Thus, when the melting point is lower than 100°C, the polymer is melted even by a boiling water and is extremely restricted from the development of the use in practical side. Accordingly, it is more preferred that if the melting point is 120°C or higher. The phrase reading "at least composed of" means that the above-mentioned mixed polymer components occupy at least 50% by weight in the filaments. If it is less than 50% by weight, the characterisitics of the polyolefin and of the polyester are lost and that is not preferred.
- <sup>10</sup> **[0024]** In the filament of the present invention, it is preferred that the mixing ratio (by weight) of the polyolefin to the polyester is from 5/95 to 95/5. When the mixing ratio of the polyolefin or the polyester is less than the above-mentioned range, the characteristic property of such a polymer is lost and, therefore, that is not preferred. To be more precise, when the mixing ratio of the polyolefin is less than 5% by weight, both lightness and tenacity of the filament lower while, when that of the polyester is less than 5% by weight, modulus of the filament lowers, the stiffness and the touch on
- <sup>15</sup> use and wearing are not achieved and, in addition, the smooth feeling which is characteristic to polyolefin is not resulted. Accordingly, the more preferred range of the mixing ratio is from 15/85 to 85/15 and the most preferred one is from 25/75 to 75/25.

**[0025]** Now, one of the methods for the manufacture of the filament having a plexifilamentry structure in accordance with the present invention will be illustrated as hereunder. In manufacturing the filament of the present invention, the

- 20 commonly known method of flash spinning may be applied. As hereunder, it will be explained in more specific manner. [0026] First, a mixture of polyolefin and polyester is dissolved, at high temperature and high pressure, in a solvent in which both polymers are insoluble at low tempeature but are soluble at high temperature and high pressure. After making it as a single bath phase, it is spun out from nozzles under a state of a phase separation whereby the abovementioned filaments are prepared.
- 25 [0027] Examples of the solvent therefor are commonly known aromatic hydrocarbons such as benzene and toluene; aliphatic hydrocarbons such as butane, pentane and isomers as well as homologues thereof; alicyclic hydrocarbons such as cyclohexane; and unsaturated hydrocarbons. Other examples are halogenated hydrocarbons such as trichlo-romethane, methylene chloride, carbon tetrachloride, chloroform, 1,1-dichloro-2,2-difluoroethane, 1,2-dichloro-1,1-di-fluoroethane, methyl chloride, ethyl chloride, etc. Further examples are alcohols, ethers, ketones, nitriles, amides,
- <sup>30</sup> fluorocarbons, etc. Still further examples are mixtures of the above-mentioned solvents. Taking the recent circumstances where the global environment is a big problem into consideration, the use of the solvents which might destruct an ozone layer is to be particularly avoided. Considering said environmental matter as well, we shall list up methylene chloride, 1,1-dichloro-2,2-difluoroethane, 1,2-dichloro-1,1-difluoroethane, etc. as the solvents which are preferably used in conducting the present invention.
- <sup>35</sup> **[0028]** The range of the concentrations of the polymers will not be so definitely limited since it depends upon the degree of polymerization of the polymer, the type of the solvent, the pressurizing state, etc. but it is recommended to adjust to such an extent that the concentrations of the polymers and of the solvent in the spinning dope are 5-30% by weight and 95-70% by weight, respectively. When the concentration of the polymers is less than 5% by weight, it is difficult to obtain continuous filaments while, when it is more than 30% by weight, no fibrillation takes place resulting
- <sup>40</sup> in cylindrical filaments containing foams therein whereby it is difficult to obtain the fine denier fibrillated filaments of high tenacity. When the concentration of the solvent is less than 70% by weight, the viscosity of the spinning dope becomes too high to dissolve the polymers homogeneously whereby there is a tendency that fine denier fibrillated filaments are not resulted giving the filaments with cavities while, when it is more than 95% by weight, the filaments having a plexifilamentary structure composed of the fibrillated filaments in a continuous manner are not resulted and,
- <sup>45</sup> accordingly, that is not preferred. [0029] It is greatly preferred for increasing the spinning pressure that, before or after preparing the spinning dope, inert gas represented by nitrogen gas or the like is added thereto and injected thereinto. It is particularly preferred to add the inert gas before raising the temperature because degradation of the polymers can be prevented and an ability of rising the temperature is improved whereby the solubility of the polymers in the solvent is accelerated to give a very fine denier filaments with a fibrillated plexifilamentary structure.
- <sup>50</sup> fine denier filaments with a fibrillated plexifilamentary structure.
  [0030] In the manufacture of the filament having a plexifilamentary structure according to the present invention, the elogation and the orientation of the filament are carried out by means of an explosive power accompanied by evaporation of the solvent and the tenacity of the. filament are often decided by the fact whether said filament is well elongated and oriented. The explosive power at that time is a vapourizing power as a result of the speed at the moment. In terms
- <sup>55</sup> of the speed, the solvent is vapourized. at a time within a period of not longer than 0.1 second and, during said process, an increase in the concentration of the polymers is resulted within a short period and, at last, only the mixed polymers are precipitated. The mixed polymers which are precipitated as a result of vapourization of the solvent are cooled. For the tenacity, this cooling step is most important and, in obtaining the filaments with high tenacity, the cooling by means

of a flash flow and the elongation and the orientation depending upon the cooling speed should be well carried out. In a manufacturing method according to the present invention, the polymers which are not miscible each other are used and, therefore, the fibrillation is well promoted by said flash flow whereby very fine denier fibrillated filaments are resulted.

- <sup>5</sup> **[0031]** Since the polymers which are not miscible each other are used in conducting the method of the present invention, the polymers are easily separated even when the polymers are dissolved in the solvent and, therefore, it is preferred to add a surface-active agent thereto. Addition of a surface-active agent is effective in stably keeping the spinnnig dope in an emulsified state. Usually, a surface-active agent of a nonionic type is applied. Examples of the surface-active agent are adducts of polyoxyethylene with lauryl alcohol, stearyl alcohol, oleyl alcohol, etc. and mo-
- noesters of lauric, stearic or oleic acid. The more homogeneous the mixed solution or the dope is, the more fine denier fibrillated plexifilamentary structured filaments are.
   [0032] In carrying out the manufacturing method of the present invention, it is preferred that the dissolving and spinning temperature of the spinning dope is within a range of 170 to 240°C. A lowering in viscosity in the presence of a
- solvent is particularly significant in the case of the polyesters and, therefore, when the temperature is higher than
   240°C, the filaments may be coloured or the degradation is accelerated whereby the filaments with high tenacity may
   not be obtained and, therefore, that is not preferred. When the temperature is lower than 170°C, very fine denier
   fibrillated filaments are not resulted but cylindrical filaments having cavities are formed and, therefore, that is not pre-
- [0033] The retention time of the dissolved state of the spinning dope in carrying out the manufacturing method of the present invention will not be definitely given since it depends upon the above-mentioned dissolving and spinning temperatures. Thus, when the temperature is high, it is necessary that the retention time of dissolution is kept as short as possible while, when the temperature is relatively low, the retention time for dissolution may be long. If mentioned daringly, it is preferred to be from 5 minutes to 90 minutes. When the retention time for dissolution of the mixture for spinning is longer than 90 minutes, colouring or heat degradation of the polyester may take place whereby the tenacity
- <sup>25</sup> of the filaments may lower while, when that time is shorter than 5 minutes, dissolution of the polymers is insufficient giving a problem of choking of the filter or a problem on manufacturing the uniform filaments and, therefore, that is not recommended.

**[0034]** The pressure for spinning the dope in which the polymers are dissolved is not generally regulated because it depends upon the amount of the solvent, the concentration of the polymers and the amount of the inert gas added

<sup>30</sup> but, usually, it is preferred to be not lower than 5884 kPa (60 kg/cm<sup>2</sup>). When the pressure is lower than 5884 kPa (60 kg/cm<sup>2</sup>), the explosive power at the flash spinning is low whereby the orientation of the filaments is low and it is not possible to afford the filaments with high tenacity. In addition, the extrusion becomes uneven and there is a problem that the spinning of filaments with stable and a highly fibrillated state is not prepared. There is no particular restriction for the upper limit of the pressure but, in view of prevention of a decrease in viscosity of the polymer, it is preferably 17652 kPa (180 kg/cm<sup>2</sup>).

<sup>35</sup> 17652 kPa (180 kg/cm<sup>2</sup>).

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**[0035]** In conducting a flash spinning, the spinning dope is spun under a self-generated pressure or via a pressure letdown chamber. With respect to the nozzles for the spinning, commonly known ones may be used.

**[0036]** In the polymers or spinning dope, the agents which are commonly used for filaments such as anti-glassy agents, light-stable agents, heat-stable agents, pigments, filament opening agents, weather-stable agents, ultraviolet absorbent agents, heat-saving agents, stabilizers, etc. may be added within such an extent that they do not deteriorate the effect of the present invention.

**[0037]** Then the nonwoven fabric having a fine fibrillated structure in which the filaments in the plexifilamentary structure are bonded in the entire area and a method of manufacturing it will be illustrated in detail as hereunder.

[0038] Said nonwoven fabric is prepared using the filaments having the above-mentioned plexifilamentary structure.
 [0039] The phrase reading nonwoven fabric having a dense structure used here means the nonwoven fabric in which the apparent density is not less than 0.2 g/cm<sup>3</sup> and the above-mentioned very fine denier plexifilamentary structure is densely bonded.

**[0040]** The filaments which constitute the nonwoven fabric of the present invention are not miscible each other and, therefore, the fibrillated filaments of very fine denier single polymer are mainly formed. The finer the filaments, the

<sup>50</sup> more densely constructed nonwoven fabric is resulted. Thus, as compared with the conventional nonwoven fabric, the product of the present invention exhibits a higher ability as a bacteria barrier which does not pass the bacteria of smaller sizes.

**[0041]** It is necessary that the nonwoven fabric of the present invention is in such a state that the filaments having a plexifilamentary structure are bonded each- other in the entire area. This means that the filaments in a very fine design fibrillated playifilamentary structure are desplay banded at the banded encoded each other in the entire area.

<sup>55</sup> denier fibrillated plexifilamentary structure are densly bonded at the bonded areas and the bonding is not in spots. In other words, the bonding takes place at the bonded areas of very fine denier fibrillated filaments and, therefore, the nonwoven fabric with very dense structure is resulted and the resulting nonwoven fabric is with excellent strength, ability as a bacteria barrier, waterproof pressure and moisture permeability.

**[0042]** Accordingly, it is preferred that the nonwoven fabric of the present invention exhibits a strength of not less than 20 kg/5 cm calculated in terms of the weight of 100 g/m<sup>2</sup>. When the strength is less than 20 kg/5 cm, it is not possible to use for various areas and the use of the nonwoven fabric will be extremely restricted.

- **[0043]** It is preferred that the moisture permeability which shows the property of dispersing the humidity is not less than 100 g/m<sup>2</sup>/hr. When the moisture permeability is less than 100 g/m<sup>2</sup>/hr, the humidity is hardly dispersed and, accordingly, when it is used as a clothing or a house wrapping, humidity is filled in it whereupon droplets of water are formed, uncomfortable feel is resulted or fungi are produced to afford a nonhygienic state. Accordingly, the higher the moisture permeability, the better.
- [0044] Preferred waterproof pressure is 50 cm or higher. The waterproof pressure is a parameter of the degree how hardly the liquid such as water is permeated. When it is less than 50 cm, liquid such as water permeates through said fabric together with bacteria whereupon the bacteria barrier ability is deteriorated. Accordingly, the product cannot be used as a clothing for protecting sanitary materials. Therefore, the higher the value of the water proof pressure, the better.
  - [0045] A manufacturing method of the above-mentioned nonwoven fabric will be given as hereunder.
- <sup>15</sup> **[0046]** In the manufacture of the nonwoven fabric according to the present invention, the filaments having a plexifilamentary structure which constitute the nonwoven fabric should be manufactured at first and, in manufacturing said filaments having a plexifilamentary structure, the method of flash spinning as mentioned above is applied.

**[0047]** When the mixed solution or the dope for spinning is spun out from the nozzles, the flash fluid and the sedimented filaments collide with the rotating plate and, after the filaments in a network structure are subjected to a vibration

20 like twill, a filament opening takes place. The process of the formation of the filaments with a plexifilamentary structure is as mentioned already. With respect to a method for the filament opening, there are two means. One is a frictional electrification by the rotating plate and another is a corona discharge thereafter and any of or both of those means may be applied.

[0048] The filaments with a plexifilamentary structure which are opened as such are accumulated on a conveyer and

- the resulting filament web is subjected to a bonding by application of heat and pressure by means of a group of rolls. The conditions for the bonding are as follows. Thus, the temperature is made not lower than "(melting point of the polymer having the lowest melting point among the polymers comprising the filaments) - (40°C)" and not higher than said melting point while the nip pressure of roll axis direction is made from 0.5 kg/cm to 20 kg/cm. The definite conditions are suitably chosen at last. The bonding with pressure by means of a group of rolls at room temperature and further
- <sup>30</sup> bonding with pressure under the above-mentioned condition are more preferred since a strong bonding is resulted at the bonded area between the filament and another.
   [0049] When the temperature of lower than "(melting point of the polymer) (40°C)" is applied in the final stage, the

adhesion or the bonding throughout the filaments decreases whereby the tenacity of the nonwoven fabric is lowered while, when the bonding with heat and pressure is applied at the temperature of higher than the melting point, the filaments are melted and the effect of preparing a fine denier fibrillated filaments is deteriorated. Accordingly, those

## are not preferred.

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**[0050]** With respect to the nip pressure of roll axis direction, an adhesion throughout the filaments is deteriorated and the tenacity of the nonwoven fabric lowers when it is less than 0.5 kg/cm while, when it is more than 20 kg/cm, there is a tendency that the filament layer becomes filmy and, therefore, those are not preferred.

- <sup>40</sup> **[0051]** Steel rolls, rubber rolls and resin rolls are jointly used as to the above-mentioned group of rolls. When steel rolls are applied, coating with fluoroethylene resin, rubber, etc. is preferred because a formation of unusual lustre on the resulting nonwoven fabric can be prevented. Two to thirty rolls may be used. The use of a device called a calender roll apparatus as a common device for such a group of rolls is most preferred.
- [0052] In the nonwoven fabric of the present invention, adjustment of the weight can be carried out by changing the above-mentioned vibration-like twill and conveyer speed. A big change in the weight is possible by laminating the formed web or nonwoven fabric.

**[0053]** Then a nonwoven fabric having a fine fibrillated structure in which the filaments having a plexifilamentary structure are bonded each other to form many heat bonded areas with a certain distance kept one another and its manufacturing method will be illustrated in detail.

<sup>50</sup> **[0054]** Such a nonwoven fabric can be manufactured using the above-mentioned filaments having a plexifilamentary structure as well.

**[0055]** It is necessary that the nonwoven fabric in that case is in such a state that the filaments having a plexifilamentary structure is partially bonded. This means that the very fine denier filaments having a fibrillated network structure are partially bonded in certain areas in a state of a mixed spinning and also by a polymer which is a low melting.

<sup>55</sup> component. Thus they are not bonded in its entirety. In other words, very fine denier fibrillated filaments are partially bonded by the polymer which is a low-melting component whereby the shape of the nonwoven fabric is maintained. The filaments where the partial bonding does not take place are constructed by a very fine denier filaments having a fibrillated plexifilamentary structure. Accordingly, in spite of a dense structure, the resulting nonwoven fabric is soft and

is with excellent tenacity and moisture permeability. In addition, because of such a dense structure, it naturally exhibits a property as a bacteria barrier.

[0056] Such a partially bonded state can be expressed by the ratio of the bonded area to the entire area of the obtained nonwoven fabric. Said ratio is measured as follows. Thus, a picture of the small piece of the nonwoven fabric

- <sup>5</sup> is taken by a scanning electron microscope and the average value of the ten measurements of the ratio of the sum of the bonded area to the area of the minimum repeating unit. The preferred ratio of the bonded area to the entire area is not more than 50%. When the ratio is more than 50%, the bonded area becomes too much whereby the freedom of the nonfixed filaments in the nonwoven fabric is restricted and there is a tendency that the nonwoven fabric becomes hard and is without softness. On the other hand, when said ratio is too small, the shape of the nonwoven fabric is
- deteriorated. Accordingly, it is preferred to be not less than 4% from a practical viewpoint. Taking the above into consideration, the most preferred range is from 5% to 30%.
  [0057] The state of such a partial bonding can be expressed by the density of the bonded area too. The density of the bonded area is calculated as follows. Thus, a picture of a small piece of the nonwoven fabric is taken by a scanning
- electron microscope, the ratio of the sum of the numbers of the bonded area to the area of the minimum repeating unit
   is calculated in terms of per cm<sup>2</sup>, such a measurement is conducted for ten times independently and an average value
   is calculated therefrom. The preferred density of the bonded area is from 15/cm<sup>2</sup> to 120/cm<sup>2</sup>. When it is less than 15/cm<sup>2</sup>, the shape of the nonwoven fabric is no good and, in addition, there is a decrease in the abrasion resistance of the nonwoven fabric resulting in a problem of fluffiness. Accordingly, the higher the density of the bonded area, the less the practical problems and, therefore, the better. However, when said density is too high, handling of the nonwoven
- fabric may be hard and without softness. Accordingly, the preferred ratio is not more than 120/cm<sup>2</sup>. In view of the above, the more preferred range is from 20 to 100/cm<sup>2</sup> and the most preferred one is 30 to 90/cm<sup>2</sup>.
   [0058] It is far preferred that both of the ratio of the bonded area to the entire area and the density of the bonded area are within the above-mentioned ranges.
- [0059] The shape of such bonded areas may be any of circular, ellipsoidal, diamond-shaped, triangular, T-shaped, <sup>25</sup> I-shaped, parallel-crossed and lattice-shaped.

**[0060]** The preferred property of the nonwoven fabric is that the strength calculated per  $100 \text{ g/m}^2$  is not less than 5 kg/5 cm. This is because, when the strength is less than 5 kg/5 cm, there may be a problem on actual use and the use of the nonwoven fabric is quite limited.

- [0061] Preferred softness at compression is not more than 200 g. The softness at compression stands for the softness of the nonwoven fabric and when its value is smaller, the product is softer. Said softness at compression is measured as follows. Thus, five test pieces of the nonwoven fabric having a width of 50 mm to the direction of the apparatus (longitudinal direction) and a length of 100 mm to the direction which is rectangular thereto are prepared, each of them is laterally curved to form a hollow cylinder, its both ends are adhered, the resulting sample is compressed to its axial direction at the speed of 50 mm/minute using a Tensilon UTM-4-1-100 (Toyo Baldwin), the stress at its maximum load
- <sup>35</sup> is measured, the average value thereof is divided by a weight of the nonwoven fabric and the value calulated per 100 g/m<sup>2</sup> is defined as a softness at compression. The softness at compression of the nonwoven fabric with more than 200 g is not preferred because of a decrease in the softness and an appearance of a hardness. Accordingly, it is more preferably not more than 160 g or, most preferably, not more than 120 g.
- **[0062]** The preferred moisture permeability is not less than 100 g/m<sup>2</sup>/hr. The moisture permeability shows an ability of dispersing the humidity and, when the value is higher, the property is better. When the moisture permeability is less than 100 g/m<sup>2</sup>/hr, the humidity is hardly dispersed and, therefore, when it is used as a clothing or a house wrapping, humidity is filled therein resulting in a condensation of water droplets whereby unpleasant feel is accompanied or the nonhygienic state is resulted because of the generation of fungi. Accordingly, the higher the moisture permeability, the better.
- <sup>45</sup> **[0063]** The weight of the nonwoven fabric may be as thick as 500 g/m<sup>2</sup> and, usually, it is from 20 to 200 g/m<sup>2</sup>.

**[0064]** A method of manufacturig the nonwoven fabric will be given as hereunder.

**[0065]** For the manufacture of the nonwoven fabric, the filaments having a plexifilamentary structure which constitutes the nonwoven fabric is to be manufactured at first. In the manufacture of such filaments having a network structure, the above-mentioned method of flash spinning is applied.

- <sup>50</sup> **[0066]** When the dope for spinning is spun out from the nozzles, the flash flow and the sedimented filaments collide with the rotating plate, the filaments having a plexifilamentary structure is vibrated like twill and the filament opening takes place. The process of formation of the filaments having a plexifilamentary structure is the same as given above. With respect to a method of the filament opening, there are two means a frictional electrification using a rotating plate and a corona discharge thereafter and any of them or both may be applied.
- <sup>55</sup> **[0067]** The filaments having a plexifilamentary structure which are open as such are accumulated on a conveyer and the resulting filament web is partially subjected to a bonding with heat and pressure using an embossing roll apparatus. Examples of the embossing roll apparatus are a thermal embossing apparatus and a pin-sonic processing apparatus by means of ultrasonic wave.

**[0068]** The conditions for bonding with heat and pressure using a thermal embossing apparatus are as follows. Thus, the temperature is not lower than "(melting point) -  $(40^{\circ}C)$ " and is not higher than the melting point of the polymer having the lowest melting point among the polymers constituting the filaments. The nip pressure of roll axis direction is chosen from a range of 0.5 to 50 kg/cm. Incidentally, the processing may be carried out under the nip pressure

- <sup>5</sup> condition within the above range with a clearance of 0.02-0.2 mm. The clearance is taken for preventing a filmation due to a complete fusion of the bonding area and that may be suitably conducted depending upon the use. Further, it is preferred to conduct an embossing under the above-mentioned conditions after a preliminary bonding with heat and pressure using rolls at room temperature because a disorder in the fibrillated filament web is not generated. When the bonding with heat and pressure is carried out at the temperature of higher than the melting point of the polymer at the
- 10 embossing step, the filaments are sometimes melted whereby the web is wound round the roller whereby no sheet is produced. Further, there is an unpreferred tendency that the very fine denier filaments with a plexifilamentary structure are fused whereby the effect of the very fine denier fibrillated filament is deteriorated. On the other hand, the application of the temperature of lower than "(melting point of the polymer) - (40°C)" at the final stage is not preferred since the bonding among the filaments lowers whereby the strength of the nonwoven fabric lowers.
- <sup>15</sup> **[0069]** With respect to the nip pressure of roll axis direction, the pressure of lower than 0.5 kg/cm is not preferred because the bonding at the bonding areas among the filaments lowers whereby the strength of the nonwoven fabric lowers. On the other hand, the pressure of higher than 50 kg/cm is not preferred as well because the bonding area tends to become filmy and, if things go on further, the nonwoven fabric with holes is resulted.
- [0070] The conditions for bonding with heat and pressure when a pin-sonic processing apparatus is used are, for example, that the filaments are partially bonded by means of an oscillation using an ultrasonic wave of about 20 kHz so that the state of the nonwoven fabric is maintained. Degree of the heat bonding can be suitably selected by changing the amplitude of the ultrasonic wave. When said method of heat bonding by ultrasonic waves is applied, the areas other than the bonding area are rarely affected by heat at the bonding step of the web with high heat bonding ability and, accordingly, the nonwoven fabric as a whole can be held in such a state that the thermal shrinkage properties
- 25 can be still maintained. Accordingly, when the nonwoven fabric with a high thermal shrinkage property is manufactured, the effect can be more effectively achieved.

**[0071]** The embossing shape of those embossing apparatuses are usually formed of an engraving roll having a projected pattern and a flat roll. The projected pattern can be constructed by regulating the above-mentioned ratio of the bonding area and the density of the bonding area. The terminal shape of projected area of the engraved roll may

- <sup>30</sup> be any of circular, ellipsoidal, diamond-shaped, triangular, T-shaped, I-shaped, parallel-crossed or lattice-shaped. [0072] Incidentally, other embossings may be conducted under the above-mentioned or different conditions before or after the above-mentioned embossing processing or a calender processing may be conducted together provided that they are not out of the coverage of the present invention.
- 35 (Explanations of the Examples)

**[0073]** The present invention will now be further illustrated by way of the following examples. Measurements and evaluations of various properties in the following examples are carried out by the following methods.

<sup>40</sup> Melting Point of the Polymer:

**[0074]** A differential scanning calorimeter (type DSC-2, Parkin Elmer) was used and the temperature giving a polar value of the melting absortion curve measured at the rising rate of 20°C/minute was established as a melting point.

<sup>45</sup> Fineness of the Filament:

[0075] Fineness based upon a corrected weight was measured in accordance with JIS L-1090.

Tenacity and Elongation of the Filament:

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**[0076]** Tensilon (type UTM-4-1-100; manufactured by Toyo Baldwin) was used. A sample of 10 cm length was twisted to an extent of 20 times/5 cm at the gauge length of 5 cm and the strength and the elongation were measured at a tensile speed of 5 cm/minute. The measured strength was divided by the above-mentioned fineness to give a strength of the filament. In those operations, the measurements were conducted twenty times and the average value thereof was given.

#### Modulus of the Filament:

**[0077]** The initial tensile resistance (g/dtex (g/d)) was measured in accordance with JIS L-1013 7.10 (Measurement of Initial Tensile Resistance) in the measurements of the above strength and elongation of the filament and was established as the modulus value.

Specific Surface Area:

[0078] The specific surface area (m<sup>2</sup>/g) of the filament was measured by a BET nitrogen absorption method using <sup>10</sup> Belsorp 28 of Nippon Bel KK.

Dyeing Ability of the Filament:

[0079] The filament was subjected to the following dispersed dyeing or the cationic dyeing, then subjected to the reduced dyeing, washed with water, dried and the dyeing ability of the filament was evaluated in accordance with the following standards:

(i) : very good;  $\bigcirc$ : good;  $\triangle$ : relatively good; x: no good

- (Dispersed Dyeing) Blue E-FBL (a disperse dye manufactured by Sumitomo Chemical) (1% owf), 1 g/liter of a dispersing agent (Disper-TL; manufactured by Meisei Kagaku) and 0.1 g/liter of formic acid (as a complement agent) were prepared and the filament was subjected to a boil dyeing for 60 minutes with a volume ratio of 1:50. (Cationic Dyeing) Astrazon Blue FFR (a cationic dye manufactured by Bayer) (1% owf), 0.5 g/liter of a mixed dyeing agent (Migregal WA-10; manufactured by Senka) and 10% owf of sodium sulfate (as a complement agent) were prepared and the filament was subjected to a boil dyeing for 60 minutes with a volume ratio of 1:50.
- 25 (Reduced Dyeing) Sanmole RL-100 (a refinement agent manufactured by Nikka Kagaku KK) (1 g/liter), 2 g/liter of hydrosulfite and 1 g/liter of sodium hydroxide were prepared and the filament was treated therewith at 80°C for 20 minutes with a volume ratio of 1:50.

KS Strength and Tensile Elongation of the Nonwoven Fabric:

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**[0080]** Tensilon (Type UTM-4-1-100; manufactured by Toyo Baldwin) was used. In accordance with a strip method mentioned in JIS L-1096, ten test. pieces with a width of 5 cm and a length of 20 cm each were prepared and the measurement was conducted at a gauge length of 10 cm and tensile speed of 10 cm/minute. The maximum tensile strength for each test piece at that time was averaged and calculated in terms of 100 g/m<sup>2</sup> to establish the KS strength.

<sup>35</sup> Then the maximum elongations were averaged and the average value was established as the tensile elongation of the nonwoven fabric.

Apparent Density of the Nonwoven Fabric:

<sup>40</sup> **[0081]** Five test pieces with a width of 10 cm and a length of 10 cm each were prepared, weight of each test piece was measured, then a load of 4.5 g/cm<sup>2</sup> was applied using a thickness measuring device (manufactured by Daiei Kagaku Seiki Seisakusho), the thickness after allowing to stand for ten seconds was measured, the apparent density was calculated by the following formula and the average value was established as the apparent density of the nonwoven fabric.

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Apparent Density  $(g/cm^3)$  = Weight  $(g/m^2)$ /Thickness (mm)/1000

Moisture Permeability of the Nonwoven Fabric:

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**[0082]** In accordance with the regulation of JIS-L 1099-A-1, the moisture permeability (g/m<sup>2</sup>/hr) was measured under the conditions of 40°C temperature and 90% humidity.

Dyeing Ability of the Nonwoven Fabric:

[0083] The same operation and also evaluation were made by the same manner as those in the case of the filament.[0084] Hereunder, examples for the filaments will be given.

Example 1.

[0085] An autoclave of 500 cc was filled up with 20 g of polyethylene of high density with a melting point of 132°C, a density of 0.96 g/cm3 and a melt index of 0.8 g/10 min, 30 g of polyethylene terephthalate with a melting point of 256°C and a relative viscosity of 1.6 and methylene chloride (a solvent). Further, each 0.2% by weight (to the above mixed polymer) of polyoxyethylene (POE) (3 moles) lauryl ether and isotridecyl stearate were added thereto as the surface-active agents. Then the autoclave was closed, nitrogen was introduced into the autoclave until the pressure became 40 kg/cm<sup>2</sup> and the stirring was initiated at a suitable speed together with heating up. Concentrations of each of the components in the solution were 20% by weight and 80% by weight for the polymers and the solvent, respectively.

10 [0086] The time required for the temperature rise from 100°C to 220°C was 25 minutes. When the temperature became 220°C, the solution was stirred for ten minutes to give a homogeneous solution. The gauge pressure at that time was 10787 kPa (110 kg/cm<sup>2</sup>). Then the valve was immediately released in such a state that a continuously introducing device of compressed nitrogen gas was applied so that the pressure in the autoclave was kept at 10787 kPa (110 kg/cm<sup>2</sup>) whereby the solution was spun out into air from the nozzles (hole diameter: 0.75 mm; L (length)/D (di-

ameter): 1) equipped with a pressure letdown chamber. The pressure in the pressure letdown chamber was 9022 kPa (92 kg/cm<sup>2</sup>).

**[0087]** The resulting filaments were with very good state of fibrillation. No colouration of the filaments was noted and both strength and modulus were high. The filaments were dyed with a disperse dye whereupon it was confirmed that they were clearly dyed. Properties of the filaments were as follows:

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Tensile Strength: 4.36 g/dtex (4.85 g/d) Elongation: 64% Modulus: 15 g/dtex (16.7 g/d) Specific surface area: 31 m<sup>2</sup>/g

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Example 2.

**[0088]** The same autoclave as in Example 1 was used and 20 g of polypropylene with a melting point of  $162^{\circ}$ C, density of 0.910 g/cm<sup>3</sup> and a melt flow rate of 4 g/10 min, 30 g of polyethylene terephthalate with a melting point of  $256^{\circ}$ C and a relative viscosity of 1.7 and methylene chloride (a solvent) were filled in this autoclave.

[0089] Then each 0.2% by weight (to the mixed polymer) of POE (3 moles) lauryl ether and isooctyl laurate were added thereto as the surface-active agents. The autoclave was closed, nitrogen was introduced thereinto until the pressure became 4903 kPa (50 kg/cm<sup>2</sup>) and a stirring with a suitable speed was initiated together with a heating. The concentrations of the mixed polymer and the solvent in the solution were 20% by weight and 80% by weight, respectively.

**[0090]** The time required from the temperature rose from 100 to  $200^{\circ}$ C was 30 minutes. When the temperature became  $200^{\circ}$ C, stirring was conducted for ten minutes to give a homogeneous solution. The gauge pressure at that time was 10297 kPa (105 kg/cm<sup>2</sup>). Then the valve was released immediately in such a state that nitrogen gas was introduced from a continuous introducing device of pressurized nitrogen to keep the inner pressure of the autoclave

40 at 10297 kPa (105 kg/cm<sup>2</sup>) and the solution was spun into air from the nozzles of 0.75 mm diameter with a L/D ratio of 1 equipped with a pressure letdown chamber. The pressure in the pressure letdown chamber was 8336 kPa (85 kg/ cm<sup>2</sup>).

**[0091]** The resulting filaments were with a good fibrillated state without colouring and both filament strength and modulus were high. It was also confirmed that the filament was clearly dyed when dyed with a disperse dye. Properties of the filament were as follows:

Tensile Strength: 3.12 g/dtex (3.47 g/d) Elongation: 50% Modulus: 16.74 g/dtex (18.6 g/d) Specific surface area: 29 m<sup>2</sup>/g.

Example 3.

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[0092] The same operations as in Example 2 were conducted with an exception that polyethylene terephthalate copolymerized with 5 molar % of sulfoisophthalic acid (melting point: 247°C; relative viscosity: 1.3) was used and that the dissolving and the spinning temperatures were made 200°C to manufacture the filaments. The pressure during the dissolution was 10297 kPa (105 kg/cm<sup>2</sup>) and the pressure of the pressure letdown chamber was 8434 kPa (86 kg/cm<sup>2</sup>).
[0093] The resulting filaments were with a good fibrillated state without colouring and both filament strength and

modulus were high. It was also confirmed that, when the filaments were dyed with a cationic dye, only the polyester component was clearly dyed. Properties of the filaments were as follows:

Tensile Strength: 4.01 g/dtex (4.53 g/d) Elongation: 69% Modulus: 12.7 g/dtex (14.1 g/d) Specific surface area: 38 m<sup>2</sup>/g.

Examples 4-8 and Comparative Examples 1-2.

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**[0094]** Polyethylene of high density (melting point: 132°C; density: 0.96 g/cm<sup>3</sup>; melt index value: 0.6 g/10 min) and polyethylene terephthalate (melting point: 256°C; relative viscosity: 1.4) in various mixing ratios and 200 g of methylene chloride (a solvent) were filled in the same autoclave as in Example 1. Each 0.2% by weight (to the above mixed polymer) of POE (3 moles) lauryl ether and isooctyl laurate were'added as the surface-active agents thereto. The autoclave was closed, nitrogen was introduced thereinto until 4903 kPa (50 kg/cm<sup>2</sup>) and the stirring with a suitable

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speed was initiated together with a heating. **[0095]** Concentrations of each of the components are as given in Table 1. The time required for rising the temperature from 100°C to 200°C was 35 minutes. When the temperature became 200°C, stirring was continued for ten minutes to give a homogeneous solution. The gauge pressure at that time was about 10787 kPa (110 kg/cm<sup>2</sup>). Then the valve

20 was immediately released in such a state that nitrogen was introduced thereinto from a continuous introducing apparatus of pressurized nitrogen to keep the inner pressure of the autoclave at 10787 kPa (110 kg/cm<sup>2</sup>) and the solution was spun into air from the nozzles (diameter: 0.75 mm; L/D = 1) equipped with a pressure letdown chamber. [0096] The results are given in Table 1.

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5		Dyeing Ability of Filament	×	<u>م</u> 0	000	0	
10 15		Specific Surface Area (m²/g)	17	28 36	43 34 24	18	
20		Modulus (g/dtex (g/d))	91 (10.1)	11.3 (12.5) 12.2 (13.5)	13.1 (14.6) 11.9 (13.2) 7.5 (8.3)	4.3 (4.8)	
		Elon- gation (%)	60	59	51	46	
25	Table 1	Tensile Strength (g/d))	ц.08	4.45 4.68	5.28 3.56 2.26	0.85	te sity
30		Pressure at Spinning Stage (kPa(kg/cm <sup>2</sup> ))	8826 (90)	8924 (91) 8924 (91)	9022 (92) 8924 (91) 9022 (92)	8924 (91)	PET: polyethylene terephthalate PE : polyethylene of high density
35		Mixing Ratio of Polymers (PET/PE)	0/100	5/95 15/85	50/50 80/15 95/ 5	100/0	thylene te thylene of
40			tive 1	а Ю	8-76	tive 2	polye
45			Comparative Example 1	Example Example	Example Example Example	Comparative Example 2	PET: PE :

50 [0097] It is clear from Table 1 that, in the Examples of the present invention, there was a tendency that the more the mixing ratio of the polyester is, the better the dyeing ability by a disperse dye is and the resulting filaments were with very good fibrillated state, no colouring and both filament strength and modulus were high.

[0098] In Comparative Example 1, the resulting filaments were with good fibrillated state and both filament strength and modulus were relatively high although, since no polyester was contained at all, the dyeing by a disperse dye was no good. In Comparative Example 2, the dyeing with a disperse dye was good because it contained no polyethylene at all although the fibrillated state was not so good and, in addition, both filament strength and modulus were low.

Example 9.

**[0099]** Filaments were manufactured under the same conditions as in Example 6 with an exception that polybutylene terephthalate (melting point: 228°C; relative viscosity: 1.7) was used and the dissolving and spinning temperatures were made 200°C. The pressure at the dissolving stage was 10983 kPa (112 kg/cm<sup>2</sup>) and the pressure in the pressure letdown chamber was 9120 kPa (93 kg/cm<sup>2</sup>).

**[0100]** The resulting filaments were with a very good fibrillated state without colouring and both filament strength and modulus were high. It was also confirmed that the filaments were well dyed with a disperse dye. Properties of the filaments were as follows:

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Tensile Strength: 4.16 g/dtex (4.62 g/d) Elongation: 63% Modulus: 12.2 g/dtex (13.6 g/d) Specific surface area: 38 m<sup>2</sup>/g.

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Example 10.

**[0101]** A ten-liter autoclave was used and 600 g of polyethylene of high density (melting point:  $132^{\circ}$ C; density: 0.96 g/cm<sup>3</sup>; melt index value: 0.8 g/10 min), 900 g of polyethylene terephthalate (melting point:  $256^{\circ}$ C; relative viscosity:

- 1.7) and methylene chloride (a solvent) were filled therein. Each 0.2% by weight (to the above-mentioned mixed polymers) of POE (3 moles) lauryl ether and isotridecyl stearate were added. The autoclave was closed, nitrogen was introduced thereinto until the pressure became 1961 kPa (20 kg/cm<sup>2</sup>) and a stirring with a suitable speed was initiated together with a heating. Concentrations of the polymers and the solvent in the solution were 20% by weight and 80% by weight, respectively.
- [0102] The time required for rising the temperature from 100°C to 220°C was 40 minutes. Stirring was continued for ten minutes when the temperature became 220°C to give a homogeneous solution. The gauge pressure at that time was 10689 kPa (109 kg/cm<sup>2</sup>). Then three valves were released in such a state that a continuously introducing device of compressed nitrogen gas was applied so that the pressure in the autocalve was kept at 10787 kPa (110 kg/cm<sup>2</sup>) whereupon the solution was spun out from three nozzles (diameter: 0.75 mm; L/D = 1) equipped with a pressure
- letdown chamber. This was collided with a rotating plate, then opened and accumulated on a moving conveyer net to give webs. The pressure in the pressure letdown chamber was 9022 kPa (92 kg/cm<sup>2</sup>).
   [0103] Then the webs were layered and passed through a clearance calender machine of an oil pressure type having three pairs of rollers to give nonwoven fabric of 50 g/m<sup>2</sup> weight. All of the surfaces of the upper rollers for this calender machine were made of urethane rubber while the lower rollers were heating rollers wherein the steel surface was
- coated with fluorine resin. The temperatures and the nip pressures of roll axis direction from the first to the third rollers were made 60, 120 and 125°C and 0.3, 0.8 and 1.5 kg/cm, respectively.
   [0104] The resulting nonwoven fabric was with a very good state of fibrillation of the filaments without colouring. In
- addition, the filaments were bonded throughout the entire surfaces and, therefore, the strength of the nonwoven fabric was high and both moisture permeability and waterproof pressure were high as well. It was also confirmed that the
   nonwoven fabric was able to be dyed clearly when dyed with a disperse dye. Properties of this nonwoven fabric were as follows.

Specific surface area: 31 m<sup>2</sup>/g

KS Strength of the nonwoven fabric (MD/CD): 38.4/39.8 kg/5 cm (MD: machine direction; CD: cross direction) Elongation of the nonwoven fabric (MD/CD): 18/26%

Apparent density: 0.40 g/cm<sup>3</sup> Moisture permeability: 280 g/m<sup>2</sup>/hr Waterproof pressure: 180 cmH2O Dyeing ability: (0)

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Example 11.

**[0105]** The same apparatus as in Example 10 was used and 400 g of polypropylene (melting point: 162°C; density: 0.910 g/cm<sup>3</sup>; melt flow rate value: 4 g/10 min), 1,100 g of polyethylene terephthalate (melting point: 256°C; relative viscosity: 1.6) and methylene chloride (a solvent) were filled in the autoclave. Each 0.2% by weight (to the mixed polymers) of POE (3 moles) lauryl ether and isooctyl laurate were added to the above mixed polymers as the surface-active agents. The autoclave was closed, then nitrogen was introduced into the autoclave until the pressure became 3923 kPa (40 kg/cm<sup>2</sup>) and a stirring at a suitable speed was initiated together with a heating. The concentrations of

the mixed polymers and the solvent in the solution were 20% by weight and 80% by weight, respectively.

[0106] The time required for rising the temperature from 100°C to 200°C was 30 minutes and, when the temperature became 200°C, a stirring was conducted for ten minutes to give a homogeneous solution. The gauge pressure at that time was 11572 kPa (118 kg/cm<sup>2</sup>). Then three valves were immediately released keeping the pressure of the autoclave

5 at 11768 kPa (120 kg/cm<sup>2</sup>) by means of a continuous introducing apparatus of compressed nitrogen gas so that the solution was spun out from three nozzles (hole diameter: 0.75 mm; L/D = 1) equipped with a pressure letdown chamber. This was collided with a rotating plate, opened and accumulated on a moving conveyer net to prepare webs. The pressure of the pressure letdown chamber was 9709 kPa (99 kg/cm<sup>2</sup>). Then the webs were laminated and the same operations as in Example 10 were carried out with an exception that the temperatures of the first to the third rollers 10 were made 60, 150 and 155°C, respectively to manufacture nonwoven fabric of a weight of 100 g/m<sup>2</sup>.

[0107] The resulting nonwoven fabric was with a very good state of fibrillation of the filaments without colouring and, in addition, the filaments were bonded throughout the entire surfaces. Accordingly, the strength of the nonwoven fabric was high and both moisture permeability and waterproof pressure were high as well. It was also confirmed that the nonwoven fabric was able to be dyed clearly when dyed with a disperse dye. The properties of the resulting nonwoven

15 fabric were as follows.

> Specific surface area: 29 m<sup>2</sup>/g KS Strength of the nonwoven fabric (MD/CD): 32.3/33.6kg/5cm Elongation of the nonwoven fabric (MD/CD): 22/29% Apparent density: 0.47 g/cm<sup>3</sup>

20 Moisture permeability: 263 g/m<sup>2</sup>/hr Waterproof pressure: 159 cmH2O Dyeing ability: (0)

25 Example 12.

> [0108] Nonwoven fabric was manufactured by the same conditions as in Example 11 with an exception that polyethylene terephthalate which was copolymerized with 5 molar % of sulfoisophthalic acid (melting point: 247°C; relative viscosity: 1.3) was used and the dissolving and the spinning temperatures were made 200°C. The pressure at the dissolving stage was 11670 kPa (119 kg/cm<sup>2</sup>) and the pressure of the pressure letdown chamber was 9807 kPa (100

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kg/cm<sup>2</sup>). [0109] The resulting nonwoven fabric was with a very good state of fibrillation of the filaments without colouring. In addition, the filaments were bonded throughout the entire surfaces. Accordingly, the strength of the nonwoven fabric was high and both moisture permeability and waterproof pressure were high as well. It was also confirmed that the

35 nonwoven fabric was able to be clearly dyed with a cationic dye. The properties of the nonwoven fabric were as follows.

Specific surface area: 28 m<sup>2</sup>/g KS Strength of the nonwoven fabric (MD/CD): 29.2/30.1kg/5cm Elongation of the nonwoven fabric (MD/CD): 17/25 % Apparent density: 0.39 g/cm<sup>3</sup> Moisture permeability: 278 g/m<sup>2</sup>/hr Waterproof pressure: 172 cm H<sub>2</sub>O Dyeing ability: (0)

45 Examples 13-17 and Comparative Examples 3-4.

> [0110] The apparatus of Example 10 was used and polyethylene with high density (melting point: 132°C; density: 0.96 g/cm<sup>3</sup>; melt index value: 0.6 g/10 min) and polyethylene terephthalate (melting point: 256°C; relative viscosity: 1.4) in varying ratio were filled in the autoclave together with 6,200 g (kept constant) of methylene chloride as a solvent.

50 Each 0.2 % by weight (to the above-mentioned mixed polymers) of POE (3 moles) lauryl ether and isooctyl laurate were added and the autoclave was closed. Then nitrogen was introduced into the autoclave to make the inner pressure 3923 kPa (40 kg/cm<sup>2</sup>) and a stirring at a suitable speed was initiated together with a heating.

[0111] Concentrations of each of the components in the solution were as given in Table 2. The time required for rising the temperature from 100°C to 200°C was 35 minutes and, when the temperature became 200°C, a stirring was 55 continued for ten minutes to give a homogeneous solution. The gauge pressure at that time was about 10787 kPa (110 kg/cm<sup>2</sup>). Then three valves were immediately released keeping the pressure in the autoclave at 10787 kPa (110 kg/ cm<sup>2</sup>) by an introduction of nitrogen gas to conduct a spinning and, by the same manner as in Example 10, webs were formed and nonwoven fabric was obtained.

5		Dyeing Ability	×	40000	Ø	
10		Water- proof Pressure (cm)	110	130 183 235 165 72	35	
15		ic Moisture Permea- bility (**)	230	240 280 315 250 100	60	
20		Properties of Nonwoven Fabric KS Tensil Apparent M Strength Elon- Density P (MD/CD) gation b (kg/5cm) (MD/CD) (*) (	0.43	0.42 0.41 0.39 0.38 0.36	0.35	
25		s of Nonu Tensil Elon- gation (MD/CD)	29/30	28/29 22/25 21/23 19/21 14/16	12/12	
30	Table 2	Properties KS Strength (MD/CD) (kg/5cm)	46.2/47.1	ц6.9/ц7.7 ц8.3/ц9.8 53.6/55.3 ц3.2/ц5.3 22.3/25.0	11.7/13.0	
35		Specific Surface Area (m²/g)	17	18 34 24 24	18	e sity m²/hr)
40		Pressure Specifor of Surfac Letdown Area Chamber (kPa(kg/cm <sup>2</sup> )) (m <sup>2</sup> /g	8826 (90)	8924 (91) 8924 (91) 9022 (92) 8924 (91) 9022 (92)	8924 (91)	polyethylene terephthalate polyethylene of high density apparent density $(g/m^3)$ moisture permeability $(g/m^2/hr)$
45 50		Mixing Ratio of Polymers (%)	0/100	5/95 15/85 50/50 80/15 95/ 5	100/0	polyethylene tereph polyethylene of hig apparent density (g moisture permeabili
55			Comparative Example 3	Example 13 Example 14 Example 15 Example 16 Example 17	Comparative Example 4	<pre> PET: polye     PE : polye     * : appar     ** : moist</pre>

**[0113]** It is clear from Table 2 that, in Examples 13-17, there was a tendency that the more the mixing ratio of the polyester, the better the dyeing ability by a disperse dye and the resulting nonwoven fabric was with a very good state of fibrillated filaments without colouration. Moreover, the filaments were bonded throughout the entire surfaces and, therefore, the strength of the nonwoven fabric was high and both moisture permeability and waterproof pressure were high as well.

**[0114]** In Comparative Example 3, the resulting nonwoven fabric was with a good state of fibrillation and the strength of the nonwoven fabric was relatively high but the dyeing ability was no good since no polyester was contained therein at all.

[0115] In Comparative Example 4, the resulting nonwoven fabric exhibited good dyeing ability with a disperse dye
 since it contained no polyethylene at all but the fibrillated state was not so good and, in addition, all of the strength of
 the nonwoven fabric, moisture permeability and waterproof pressure were low.

Example 18.

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- <sup>15</sup> **[0116]** The same conditions as in Example 15 were applied for the manufacture of the nonwoven fabric with an exception that polybutylene terephthalate (melting point: 228°C; relative viscosity: 1.7) was used and the dissolving and the spinning temperatures were made 200°C. The pressure at the dissolving stage was 10983 kPa (112 kg/cm<sup>2</sup>) and the pressure in the pressure letdown chamber was 9120 kPa (93 kg/cm<sup>2</sup>).
- [0117] The resulting nonwoven fabric was with a very good state of fibrillation of the filements without colouring. Moreover, the filaments were bonded throughout the entire surfaces and, therefore, the strength of the nonwoven fabric was high and both moisture permeability and waterproof pressure were high as well. It was also confirmed that the nonwoven fabric was able to be clearly dyed with a disperse dye. Properties of the nonwoven fabric were as follows.

Specific surface area: 31 m<sup>2</sup>/g

 KS Strength of the nonwoven fabric (MD/CD): 36.3/37.4kg/5cm Elongation of the nonwoven fabric (MD/CD): 21/29% Apparent density: 0.37 g/cm<sup>3</sup> Moisture permeability: 292 g/m<sup>2</sup>/hr Waterproof pressure: 191 cm H<sub>2</sub>O
 Dueing ability: (0)

Example 19.

[0118] A 10-liter autoclave was used and 600 g of polyethylene of high density (melting point: 132°C; density: 0.96 g/m<sup>3</sup>; melt index value: 0.8 g/10 min), 900 g of polyethylene terephthalate (melting point: 256°C; relative viscosity: 1.70) and methylene chloride (a solvent) were filled in this autoclave. Each 0.2% by weight (to the above-mentioned mixed polymers) of isooctyl stearate and isostearyl ester were added as the surface-active agents. The autoclave was closed, then nitrogen was introduced thereinto to make the pressure 1961 kPa (20 kg/cm<sup>2</sup>) and a stirring at a suitable speed was initiated together with a heating. The concentrations of the components were 20% by weight of the polymers and 80% by weight of the solvent.

**[0119]** The time required for rising the temperature from 100°C to 220°C was 40 minutes and, when the temperature became 220°C, a stirring was continued for ten minutes to give a homogeneous solution. The gauge pressure at that time was 10689 kPa (109 kg/cm<sup>2</sup>). Then three valves were immediately released by keeping the pressure of the autoclave at 10787 kPa (110 kg/cm<sup>2</sup>) by means of a continuous introducing apparatus of compressed nitrogen gas so that

45 the solution was spun out from three nozzles (hole diameter: 0.75 mm; L/D = 1) equipped with a pressure letdown chamber. This was collided with a rotating plate, opened and accumulated on a moving conveyer net to give webs. The pressure of the pressure letdown chamber was 9022 kPa (92 kg/cm<sup>2</sup>).

**[0120]** Then the webs were laminated and passed through a clearance embossing apparatus of an oil pressure type to manufacture nonwoven fabric of a weight of 50 g/m<sup>2</sup>. The upper roll of the embossing apparatus was an engraved roll while the lawer one was a flat roll and both were beating rolls. An embossing process was carried out at the pin

- <sup>50</sup> roll while the lower one was a flat roll and both were heating rolls. An embossing process was carried out at the nip pressure of roll axis direction of 20 kg/cm, the temperature of 125°C and the speed of 10 m/min without taking a clearance between the upper and the lower rolls. The rate of the bonded area of the engraved roll was 25% and the desity of the bonded area was 60/cm<sup>2</sup>.
- **[0121]** The resulting nonwoven fabric was with a very good state of fibrillated filaments without colouring. In addition, the filaments were in such a structure that there were many small bonded areas with pressure and, accordingly, the nonwoven fabric was with excellent softness and moisture permeability keeping a practically sufficient strength as the nonwoven fabric. It was also confirmed that the nonwoven fabric was able to be clearly dyed with a disperse dye. The properties of the nonwoven fabric were as follows.

<sup>30</sup> Dyeing ability: (0)

Specific surface area:  $31 \text{ m}^2/\text{g}$ KS Strength of the nonwoven fabric (MD/CD): 17.3/18.6kg/5cm Tensile elongation of the nonwoven fabric (MD/CD); 28/31%Apparent density:  $0.28 \text{ g/cm}^3$ Softness at compression: 125 gMoisture permeability:  $250 \text{ g/m}^2/\text{hr}$ Dyeing ability: ( $\overline{0}$ )

Example 20.

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**[0122]** The same apparatus as in Example 19 was used and 400 g of polypropylene (melting point:  $162^{\circ}$ C; density: 0.910 g/cm<sup>3</sup>; melt flow rate value: 4 g/10 min), 1,100 g of polyethylene terephthalate (melting point:  $256^{\circ}$ C; relative viscosity: 1.6) and methylene chloride (a solvent) were filled in the autoclave. Each 0.2% by weight (to the above-mentioned mixed polymers) of isooctyl stearate and isostearyl ester were added as the surface-active agents. The autoclave was closed, then nitrogen was introduced into the autoclave until the pressure became 3923 kPa (40 kg/ cm<sup>2</sup>) and a stirring at a suitable speed was initiated together with a heating. The concentrations of the mixed polymers

- and the solvent in the solution were 20% by weight and 80% by weight, respectively. **[0123]** The time required for rising the temperature from 100°C to 200°C was 30 minutes and, when the temperature became 200°C, a stirring was carried out for ten minutes to give a homogeneous solution. The gauge pressure at that
- time was 11572 kPa (118 kg/cm<sup>2</sup>). Then three valves were released by keeping the pressure of the autoclave at 11768 kPa (120 kg/cm<sup>2</sup>) by means of a continuous introducing apparatus of compressed nitrogen gas and the solution was spun out from three nozzles (hole diameter: 0.75 mm; L/D = 1) equipped with a pressure letdown chamber. This was collided with a rotating plate, opened and accumulated on a moving conveyer net to give webs. The pressure in the pressure letdown chamber was 9709 kPa (99 kg/cm<sup>2</sup>). Then the webs were laminated and treated by the same manner
- <sup>25</sup> as in Example 19 with an exception that the temperature of the embossing process was made 150°C to give nonwoven fabric with a weight of 100 g/m<sup>2</sup>.

**[0124]** The resulting nonwoven fabric was with a very good state of fibrillation of the filaments without colouring. Moreover, the filaments were in such a structure that there were many small bonded areas with pressure and, accordingly, the nonwoven fabric maintained a practically sufficient strength and also excellent softness and moisture permaphility. It was also excellent softness and moisture per-

<sup>30</sup> meability. It was also confirmed that the nonwoven fabric was able to be clearly dyed with a disperse dye. Properties of the nonwoven fabric were as follows.

Specific surface area: 29 m<sup>2</sup>/g

KS Strength of the nonwoven fabric (MD/CD): 16.2/17.7kg/5cm Tensile elongation of the nonwoven fabric (MD/CD): 30/33% Apparent density: 0.29 g/cm<sup>3</sup> Softness at compression: 120 g Moisture permeability: 231 g/m<sup>2</sup>/hr Dyeing ability: (i)

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Example 21.

[0125] Nonwoven fabric was manufactured by the same conditions as in Example 20 with an exception that polyethylene terephthalate copolymerized with 5 molar % of sulfoisophthalic acid (melting point: 247°C; relative viscosity: 1.3) was used and that the dissolving and the spinning temperatures were made 200°C. The pressure at the dissolving stage was 11670 kPa (119 kg/cm<sup>2</sup>) and that of the pressure letdown chamber was 9807 kPa (100 kg/cm<sup>2</sup>).

[0126] The resulting nonwoven fabric was with a very good state of fibrillation of the filaments without colouring. Moreover, the filaments were in such a structure that there were many small bonding areas with pressure and, therefore, it maintained a practically sufficient strength as the nonwoven fabric and exhibited excellent softness and moisture permeability. It was also confirmed that the nonwoven fabric was able to be clearly dyed with a cationic dye. The presenting of the permeability areas of the permeability were as follows.

properties of the nonwoven fabric were as follows.

Specific surface area: 28 m<sup>2</sup>/g
 KS Strength of the nonwoven fabric (MD/CD): 14.5/15.7kg/5cm
 Tensile elongation of the nonwoven fabric (MD/CD): 37/39 %
 Apparent density: 0.30 g/cm<sup>3</sup>
 Softness at compression: 118 g
 Moisture permeability: 247 g/m<sup>2</sup>/hr

Dyeing ability: (0)

Examples 22-26 and Comparative Examples 5-6.

- <sup>5</sup> [0127] The apparatus of Example 19 was used and polyethylene with high desity (melting point: 132°C; density: 0.96 g/cm<sup>3</sup>; melt index value: 0.6 g/10 min) and polyethylene terephthalate (melting point: 256°C; relative viscosity: 1.4) with varied ratio and 6,200 g (kept constant) of methylene chloride (a solvent) were filled in the autoclave. Each 0.2% by weight (to the above-mentioned mixed polymers) of isooctyl stearate and isostearyl ester were added as the surface-active agents and the autoclave was closed. Then nitrogen was introduced into the autoclave to make the pressure 3923 kPa (40 kg/cm<sup>2</sup>) and a stirring at a suitable speed was initiated together with a heating.
- **[0128]** The concentrations of the components were as given in Table 3. The time required for rising the temperature from 100°C to 200°C was 35 mintues and, when the temperature became 200°C, a stirring was continued for ten minutes to give a homogeneous solution. The gauge pressure at that time was about 10787 kPa (110 kg/cm<sup>2</sup>). Then three valves were immediately released keeping the pressure in the autoclave at 10787 kPa (110 kg/cm<sup>2</sup>) by means
- of a continuous introducing apparatus of compressed nitrogen gas to conduct a spinning and, by the same manner as in Example 19, webs were formed and nonwoven fabric was prepared therefrom.
   [0129] The results are given in Table 3.

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5	Dyeing Ability	400©©	× ©	
10	Moisture Permea- bility (**)	210 250 285 220 80	200	
15	ic Softness at Compres- sion (g)	195 130 85 100 110	210 120	
20	Properties of Nonwoven Fabric KS Tensil Apparent S Strength Elon- Density a (MD/CD) gation C (kg/5cm) (MD/CD) (*) S ((()) (*) (1)	0.32 0.30 0.26 0.29 0.33	0.33 0.35	
25	5 of Nonw Tensil Elon- gation (MD/CD) (%)	38/39 32/35 31/33 29/31 24/26	39/40 22/22	
30	Properties ( KS Tr Strength E (MD/CD) gr (kg/5cm) ()	16.9/17.7 18.3/19.8 23.6/25.3 13.2/15.3 12.3/15.0	16.2/17.1 8.7/ 8.0	
35	Specific Surface Area (m²/g)	24 24 24 24 26 26 26	17 18	late ensity ³) (g/m²/hr)
40	Pressure Specif of Surfac Letdown Area Chamber (kPa(kg/cm <sup>2</sup> )) (m <sup>2</sup> /g)	8924 (91) 8924 (91) 9022 (92) 8924 (91) 9022 (92)	8826 (90) 8924 (91)	polyethylene terephthalate polyethylene of high density apparent density (g/cm <sup>3</sup> ) moisture permeability (g/m²/hr)
45	Mixing Ratio of PET/PE (%) ()	5/95 15/85 50/50 85/15 95/ 5	0/100	ethylene rethylene rent dens sture perm
50		Example 22 Example 23 Example 24 Example 25 Example 25	Comparative Example 5 Comparative Example 6	PET: pol, PE : pol, * : appe ** : mois

<sup>55</sup> **[0130]** It is clear from Table 3 that, in Examples 22-26, there was a tendency that the more the mixing ratio of polyester, the better the dyeing ability with a disperse dye and the resulting nonwoven fabric was with a very good state of fibrillated filaments without colouring. Moreover, the filaments were in such a structure that there were many small bonded areas with pressure and, therfore, the nonwoven fabric kept a practically sufficient strength and exhibited excellent softness

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and moisture permeability. It was also confirmed that the nonwoven fabric was able to be clearly dyed with a disperse dye.

**[0131]** In Comparative Example 5, the resulting nonwoven fabric was with a good fibrillated state and the strength was relatively high but, since it contained no polyester at all, the dyeing ability was no good.

**[0132]** In Comparative Example 6, no polyethylene was contained at all and, therefore, the resulting nonwoven fabric was with good dyeing ability with a disperse dye but the fibrillated state was not so good and, in addition, both strength and moisture permeability were low.

Example 27.

**[0133]** Nonwoven fabric was manufactured under the same conditions as in Example 24 with an exception that polybutylene terephthalate (melting point: 228°C; relative viscosity: 1.7) was used and the dissolving and the spinning temperatures were made 200°C. The pressure at the dissolving stage was 10983 kPa (112 kg/cm<sup>2</sup>) and the pressure of the pressure letdown chamber was 9120 kPa (93 kg/cm<sup>2</sup>).

<sup>15</sup> **[0134]** The resulting nonwoven fabric was with a very good state of fibrillation of the filaments without colouring. Moreover, the filaments were in such a structure that there were many small bonded areas with pressure and, therefore, the nonwoven fabric kept a practically sufficient strength and exhibited excellent softness and moisture permeability. It was also confirmed that the nonwoven fabric was able to be clearly dyed with a disperse dye. The properties of the nonwoven fabric were as follows.

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Specific surface area:  $31 \text{ m}^2/\text{g}$ KS Strength of the nonwoven fabric (MD/CD): 15.3/16.5kg/5cm Tensile elongation of the nonwoven fabric (MD/CD): 37/39 %Apparent density:  $0.27 \text{ g/cm}^3$ 

Softess at compression: 109 g
 Moisture permeability: 262 g/m<sup>2</sup>/hr
 Dyeing ability: (0)

Examples 28-34.

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[0135] In the manufacture of nonwoven fabric according to Example 25, the type of the embossing rolls was changed so that the ratio of the bonded area and the density of the bonded areas were changed. The results are given in Table 4.
[0136] It is clear from Table 4 that the nonwoven fabric of Examples 28-33 maintained a practically sufficient strength and exhibited excellent softness and moisture permeability with a tendency that the more the ratio of bonded area and the density of the bonded area, the more the strength and the less the softness.

**[0137]** In Example 34, the ratio of the bonded areas became 100% due to a bonding with pressure only by a flat roll and, accordingly, the softness of the resulting nonwoven fabric was somewhat inferior though it still exhibited practically sufficient strength and moisture permeability.

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	Dyeing Ability	00	00	) ()	00	
	Moisture Permea- bility (**)	280 290	295 300	335	310 250	
Ŀ.	Softness at Compres- sion (g)	190 180	145 100	20	68 715	
Table 4 Properties of Nonwoven Fabric	Apparent Density (*)	0.36 0.34	0.32 0.29	0.23	U.26 0.38	
ut of Non	Tensil Elon- gation (MD/CD) (%)	22/23 26/27	28/30 29/31	34/36	32/33 19/21	
Table 4 Properties	KS Strength (MD/CD) (kg/5cm)	26.9/27.7 20.8/22.9	18.4/19.8	12.3/24.0	6.2/ 7.1 43.2/45.3	
Specific	Surface Area (m²/g)	34 34	34 34	34	34 34	alate density m <sup>3</sup> ) (g/m²/hr)
Density	of Bonded Area (/cm²)	80 60	60 60	17	40 -	<pre>PET; polyethylene terephthalate PE : polyethylene of high densi * : apparent density (g/cm<sup>3</sup>) ** : moisture permeability (g/m</pre>
Ratio	of Bonded Area (%)	40	33 17	17	د 100	ethylene ethylene rent den ture per
		Example 28 Example 29	Example 30 Example 31		Example 33 Example 34	PET; poly PE : poly * : appa ** : mois

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#### Claims

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- 1. A filament having a fine fibrillated structure, said filament being a flash spun filament having a plexifilamentary structure and comprising a plexifilamentary structured polyolefin component, **characterized in that** it further comprises a plexifilamentary structured polyester component which is immiscible with the plexifilamentary structured polyolefin component, said polyolefin and said polyester being present in a ratio within a range of from 5/95 to 95/5 by weight.
- **2.** A filament according to claim 1, in which the polyolefin is any one of polyethylene, polypropylene, a copolymer mainly comprising ethylene and a copolymer mainly comprising propylene.
  - 3. A filament according to claim 1 or 2, in which the polyester is any one of polyethylene terephthalate and polybutylene terephthalate.
- <sup>15</sup> **4.** A filament according claim 1, in which the mixing ratio of the polyolefin to the polyester in the polymer mixture is within a range of from 15/85 to 85/15 by weight.
  - **5.** A method for manufacturing a filament having a fine fibrillated structure according to claim 1, **characterized in that** a mixture of polyolefin and polyester is dissolved in a solvent under high temperature and high pressure in a ratio within a range of from 5/95 to 95/5 by weight to give a one solution phase and then is flash spun in such a state that said filament comprises a plexifilamentary structured polyolefin component and a plexifilamentary structured polyolefin component and a plexifilamentary structured polyester component which are immiscible with each other.
    - 6. A method according to claim 5, in which an inert gas is added to and introduced into a dope for the spinning.
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- 7. A method according to claim 5 or 6, in which a surface-active agent is added to the dope for the spinning.
- 8. A nonwoven fabric having a fine fibrillated structure, comprising filaments according to claim 1, **characterized in that** said filaments are bonded to each other.
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- **9.** A nonwoven fabric according to claim 8, in which the polyolefin is any one of polyethylene, polypropylene, a copolymer mainly comprising ethylene and a copolymer mainly comprising propylene.
- **10.** A nonwoven fabric according to claim 8 or 9, in which the polyester is any one of polyethylene terephthalate and polybutylene terephthalate.
  - **11.** A nonwoven fabric according to any one of claims 8 to 10, in which the mixing ratio of the polyolefin to the polyester in the polymer mixture is within a range of from 15/85 to 85/15 by weight.
- 40 12. A nonwoven fabric according to any one of claims 8 to 11, in which the filaments having a fine fibrillated structure are bonded to each other in the entire area of the obtained nonwoven fabric and the properties of said nonwoven fabric are that the strength is not less than 20 kg/5 cm, the waterproof pressure is not less than 50 cm and the moisture permeability is not less than 100 g/m<sup>2</sup>/hr.
- 45 **13.** A nonwoven fabric according to any one of claims 8 to 11, in which the filaments having a fine fibrillated structure are bonded to each other to form heat bonded areas spaced apart from one another and the properties of said nonwoven fabric are that the strength is not less than 5 kg/5 cm, the softness at compression is not more than 200 g and the moisture permeability is not less than 100 g/m<sup>2</sup>/hr.
- 50 14. A method for manufacturing a nonwoven fabric having a fine fibrillated structure, whereby filaments according to claim 1, are prepared, characterized in that the resulting filaments are made into webs and then the webs are subjected to a bonding with heat and pressure using a group of rolls so that the filaments are bonded to each other in the entire area of the nonwoven fabric.
- 55 15. A method for manufacturing a nonwoven fabric according to claim 14, in which the temperature when the webs are subjected to a bonding with heat and pressure using a group of rolls is higher than the melting point of the polymer having the lowest melting point in the polymers which constitute the filaments 40°C and not higher than said melting point and the nip pressure of roll axis direction at the stage of bonding with heat and pressure is within

a range of from 0.5 kg/cm to 20 kg/cm.

- **16.** A method for manufacturing a nonwoven fabric having a fine fibrillated structure whereby filaments according to claim 1, are prepared, **characterized in that** the resulting filaments are made into webs and then the webs are subjected to a bonding with heat and pressure using a group of rolls so that the filaments are bonded to each other to form heat bonded areas spaced apart from one another.
- **17.** A method for manufacturing a nonwoven fabric according to claim 16, in which the temperature for the bonding of the webs with. heat and pressure using an embossing apparatus is higher than the melting point of the polymer having the lowest melting point among the polymers constituting the filaments -40°C and not higher than said melting point and the nip pressure of roll axis direction is within a range of from 0.5 kg/cm to 50 kg/cm.
  - **18.** A method for manufacturing a nonwoven fabric according to claim 16 or 17, in which the ratio of the bonded area to the entire area of the nonwoven fabric is from 4 % to 50 %.
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**19.** A method for manufacturing a nonwoven fabric according to any one of claims 16 to 18, in which the density of the bonded areas is from 15/cm<sup>2</sup> to 120/cm<sup>2</sup>.

#### 20 Patentansprüche

- 1. Filament mit einer feinfibrillierten Struktur, das ein durch Flash-Spinning hergestelltes Filament mit einer geflechtartigen Struktur ist und eine Polyolefinkomponente mit einer geflechtartigen Filamentstruktur aufweist, dadurch gekennzeichnet, dass es außerdem eine Polyesterkomponente mit einer geflechtartigen Filamentstruktur, die mit der Polyolefinkomponente mit der geflechtartigen Filamentstruktur nicht mischbar ist, aufweist, wobei das Polyolefin und der Polyester in einem Massenverhältnis im Bereich von 5 : 95 bis 95 : 5 vorliegen.
- 2. Filament nach Anspruch 1, dadurch gekennzeichnet, dass das Polyolefin ein Polyethylen, Polypropylen, ein hauptsächlich aus Ethylen bestehendes Copolymer oder ein hauptsächlich aus Propylen bestehendes Copolymer ist.
- **3.** Filament nach Anspruch 1 oder 2, **dadurch gekennzeichnet**, **dass** der Polyester Polyethylenterephthalat oder Polybutylenterephthalat ist.
- **4.** Filament nach Anspruch 1, **dadurch gekennzeichnet**, **dass** das Massenmischungsverhältnis des Polyolefins zum Polyester in dem Polymergemisch in dem Bereich von 15 : 85 bis 85 : 15 liegt.
  - 5. Verfahren zur Herstellung eines Filaments mit einer feinfibrillierten Struktur nach Anspruch 1, **dadurch gekenn**zeichnet, dass ein Gemisch aus Polyolefin und Polyester in einem Lösungsmittel bei hoher Temperatur und hohem Druck in einem Massenverhältnis im Bereich von 5 : 95 bis 95 : 5 gelöst wird, um eine Lösungsphase zu erhalten, und dann durch Flash-Spinning so versponnen wird, dass das Filament eine Polyolefinkomponente mit einer geflechtartigen Filamentstruktur und eine Polyesterkomponente mit einer geflechtartigen Filamentstruktur, die nicht miteinander mischbar sind, aufweist.
- 45 6. Verfahren nach Anspruch 5, dadurch gekennzeichnet, dass in die Spinnlösung ein Inertgas eingeleitet wird.
  - 7. Verfahren nach Anspruch 5 oder 6, **dadurch gekennzeichnet**, **dass** ein grenzflächenaktiver Stoff zur Spinnlösung gegeben wird.
- 50 **8.** Vliesstoff mit einer feinfibrillierten Struktur, der Filamente nach Anspruch 1 aufweist, **dadurch gekennzeichnet**, **dass** die Filamente miteinander verklebt sind.
  - 9. Vliesstoff nach Anspruch 8, dadurch gekennzeichnet, dass das Polyolefin ein Polyethylen, Polypropylen, ein hauptsächlich aus Ethylen bestehendes Copolymer oder ein hauptsächlich aus Propylen bestehendes Copolymer ist.
  - **10.** Vliesstoff nach Anspruch 8 oder 9, **dadurch gekennzeichnet**, **dass** der Polyester Polyethylenterephthalat oder Polybutylenterephthalat ist.

- **11.** Vliesstoff nach einem der Ansprüche 8 bis 10, **dadurch gekennzeichnet**, **dass** das Massenmischungsverhältnis des Polyolefins zum Polyester in dem Polymergemisch in dem Bereich von 15: 85 bis 85 : 15 liegt.
- 12. Vliesstoff nach einem der Ansprüche 8 bis 11, dadurch gekennzeichnet, dass die Filamente mit einer feinfibrillierten Struktur über die gesamte Fläche des erhaltenen Vliesstoffs miteinander verklebt sind und dass der Vliesstoff als Eigenschaften eine Festigkeit von mindestens 20 kp/5 cm, einen Wasserundurchlässigkeitsdruck von mindestens 50 cm und eine Feuchtigkeitsdurchlässigkeit von mindestens 100 g/m<sup>2</sup>/h hat.
- 13. Vliesstoff nach einem der Ansprüche 8 bis 11, dadurch gekennzeichnet, dass die Filamente mit einer feinfibril <sup>10</sup> lierten Struktur so miteinander hitzeverklebt werden, dass räumlich voneinander getrennte Hitzeverklebungsbe reiche entstehen und dass der Vliesstoff als Eigenschaften eine Festigkeit von mindestens 5 kp/5 cm, eine Weichheit beim Zusammendrücken von höchstens 200 g und eine Feuchtigkeitsdurchlässigkeit von mindestens 100 g/
   m<sup>2</sup>/h hat.
- 15 14. Verfahren zur Herstellung eines Vliesstoffs mit einer feinfibrillierten Struktur, mit dem Filamente nach Anspruch 1 hergestellt werden, dadurch gekennzeichnet, dass aus den resultierenden Filamenten Flor hergestellt wird und der Flor dann mit Wärme und Druck unter Verwendung einer Walzengruppe so verfestigt wird, dass die Filamente über die gesamte Fläche des Vliesstoffs miteinander verklebt werden.
- 15. Verfahren zur Herstellung eines Vliesstoffs nach Anspruch 14, dadurch gekennzeichnet, dass die Temperatur beim Verfestigen des Flors mit Wärme und Druck unter Verwendung einer Walzengruppe höher als die Differenz "Schmelzpunkt des Polymers mit dem niedrigsten Schmelzpunkt von den die Filamente bildenden Polymeren minus 40 °C" und nicht höher als dieser Schmelzpunkt ist und dass der Quetschdruck in Achsrichtung der Walzen beim Verfestigen mit Wärme und Druck im Bereich von 0,5 bis 20 kg/cm liegt.
  - 16. Verfahren zur Herstellung eines Vliesstoffs mit einer feinfibrillierten Struktur, mit dem Filamente nach Anspruch 1 hergestellt werden, dadurch gekennzeichnet, dass aus den resultierenden Filamenten Flor hergestellt wird und der Flor dann mit Wärme und Druck unter Verwendung einer Walzengruppe so verfestigt wird, dass Filamente so miteinander verklebt werden, dass räumlich voneinander getrennte Hitzeverklebungsbereiche entstehen
  - 17. Verfahren zur Herstellung eines Vliesstoffe nach Anspruch 16, dadurch gekennzeichnet, dass die Temperatur beim Verfestigen des Flors mit Wärme und Druck unter Verwendung einer Gaufriervorrichtung höher als die Differenz "Schmelzpunkt des Polymers mit dem niedrigsten Schmelzpunkt von den die Filamente bildenden Polymeren minus 40 °C" und nicht höher als dieser Schmelzpunkt ist und dass der Quetschdruck in Achsrichtung der Walzen im Bereich von 0,5 bis 50 kg/cm liegt.
    - **18.** Verfahren zur Herstellung eines Vliesstoffs nach Anspruch 16 oder 17, **dadurch gekennzeichnet**, **dass** der Anteil der Verklebungsbereiche an der Gesamtfläche des Vliesstoffs 4 bis 50 % beträgt.
- 40 19. Verfahren zur Herstellung eines Vliesstoffs nach einem der Ansprüche 16 bis 18, dadurch gekennzeichnet, dass die Dichte der Verklebungsbereiche 15 bis 120/cm<sup>2</sup> beträgt.

#### Revendications

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- Filament présentant une structure fibrillée fine, ledit filament étant un filament obtenu par filage éclair présentant une structure plexifilamenteuse et comprenant un composant de polyoléfine à structure plexifilamenteuse, caractérisé en ce qu'il comprend en outre un composant de polyester à structure plexifilamenteuse qui est non miscible au composant de polyoléfine à structure plexifilamenteuse, ladite polyoléfine et ledit polyester étant présents suivant un rapport dans une plage de 5/95 à 95/5 en poids.
- Filament selon la revendication 1, dans lequel la polyoléfine est l'un quelconque parmi un polyéthylène, un polypropylène, un copolymère comprenant principalement de l'éthylène et un copolymère comprenant principalement du propylène.
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- **3.** Filament selon la revendication 1 ou 2, dans lequel le polyester est l'un quelconque parmi un polytérephtalate d'éthylène et un polytérephtalate de butylène.

- **4.** Filament selon la revendication 1, dans lequel le rapport de mélange de la polyoléfine sur le polyester dans le mélange de polymères se situe dans une plage de 15/85 à 85/15 en poids.
- 5. Procédé de fabrication d'un filament présentant une structure fibrillée fine selon la revendication 1, caractérisé en ce qu'un mélange de polyoléfine et de polyester est dissous dans un solvant à une température élevée et une pression élevée suivant un rapport dans une plage de 5/95 à 95/5 en poids pour donner une phase en une seule solution et subit ensuite un filage éclair dans un état tel que ledit filament comprend un composant de polyoléfine à structure plexifilamenteuse et un composant de polyester à structure plexifilamenteuse qui sont non miscibles l'un à l'autre.

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- 6. Procédé selon la revendication 5, dans lequel un gaz inerte est ajouté à une solution destinée au filage et est introduit dans celle-ci.
- 7. Procédé selon la revendication 5 ou 6, dans lequel un agent tensioactif est ajouté à la solution destinée au filage.
  - 8. Etoffe non tissée présentant une structure fibrillée fine comprenant des filaments selon la revendication 1, caractérisée en ce que lesdits filaments sont liés les uns aux autres.
- 9. Etoffe non tissée selon la revendication 8, dans laquelle la polyoléfine est l'un quelconque parmi un polyéthylène, un polypropylène, un copolymère comprenant principalement de l'éthylène et un copolymère comprenant principalement du propylène.
  - **10.** Etoffe non tissée selon la revendication 8 ou 9, dans laquelle le polyester est l'un quelconque parmi un polytérephtalate de butylène.
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- **11.** Etoffe non tissée selon l'une quelconque des revendications 8 à 10, dans laquelle le rapport de mélange de la polyoléfine sur le polyester dans le mélange de polymères se situe à l'intérieur d'une plage de 15/85 à 85/15 en poids.
- 30 12. Etoffe non tissée selon l'une quelconque des revendications 8 à 11, dans laquelle les filaments présentant la structure fibrillée fine sont liés les uns aux autres dans la surface entière de l'étoffe non tissée obtenue et les propriétés de ladite étoffe non tissée sont que la résistance n'est pas inférieure à 20 kg/5 cm, la pression d'imperméabilité n'est pas inférieure à 50 cm et la perméabilité à l'humidité n'est pas inférieure à 100 g/m²/h.
- 13. Etoffe non tissée selon l'une quelconque des revendications 8 à 11, dans laquelle les filaments présentant une structure fibrillée fine sont liés les uns aux autres pour former les surfaces liées à chaud espacées les unes des autres, et les propriétés de ladite étoffe non tissée sont que la résistance n'est pas inférieure à 5 kg/5 cm, la mollesse à la compression n'est pas supérieure à 200 g et la perméabilité à l'humidité n'est pas inférieure à 100 g/m²/h.
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- 14. Procédé de fabrication d'une étoffe non tissée présentant une structure fibrillée fine grâce à quoi des filaments selon la revendication 1 sont préparés, caractérisé en ce que les filaments résultants sont réalisés en bandes et ensuite les bandes sont soumises à un liage avec de la chaleur et une pression en utilisant un groupe de rouleaux de sorte que les filaments soient liés les uns aux autres dans la surface entière de l'étoffe non tissée.
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- 15. Procédé de fabrication d'une étoffe non tissée selon la revendication 14, dans lequel la température lorsque les bandes sont soumises à un liage avec de la chaleur et une pression en utilisant un groupe de rouleaux est supérieure au point de fusion du polymère présentant le point de fusion le plus bas dans les polymères qui constituent les filaments -40 °C et non supérieure audit point de fusion, et la pression de pincement de la direction de l'axe des rouleaux à l'étape de liage avec de la chaleur et une pression se situe dans une plage de 0,5 kg/cm à 20 kg/cm.
- 16. Procédé de fabrication d'une étoffe non tissée présentant une structure fibrillée fine grâce à quoi des filaments selon la revendication 1 sont préparés, caractérisé en ce que les filaments résultants sont réalisés en bandes et ensuite les bandes sont soumises à un liage avec de la chaleur et une pression en utilisant un groupe de rouleaux de sorte que les filaments soient liés les uns aux autres afin de former des surfaces liées à chaud espacées les unes des autres.
  - 17. Procédé de fabrication d'une étoffe non tissée selon la revendication 16, dans lequel la température pour le liage

des bandes avec de la chaleur et une pression en utilisant un dispositif de gaufrage est supérieure au point de fusion du polymère présentant le point de fusion le plus bas parmi les polymères constituant les filaments -40 °C et non supérieure audit point de fusion, et la pression de pincement de la direction de l'axe des rouleaux se situe dans une plage de 0,5 kg/cm à 50 kg/cm.

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- **18.** Procédé de fabrication d'une étoffe non tissée selon la revendication 16 ou 17, dans lequel le rapport de la surface liée sur la surface entière de l'étoffe non tissée est de 4 % à 50 %.
- 19. Procédé de fabrication d'une étoffe non tissée selon l'une quelconque des revendications 16 à 18, dans lequel la densité des surfaces liées est de 15/cm<sup>2</sup> à 120/cm<sup>2</sup>.

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