Feb. 26, 1935. H. DREYFUS 1,992,609

MANUFACTURE OF ARTIFICIAL FILAMENTS AND THE LIKE

Filed Aug. 26, 1932

FIG. 1.

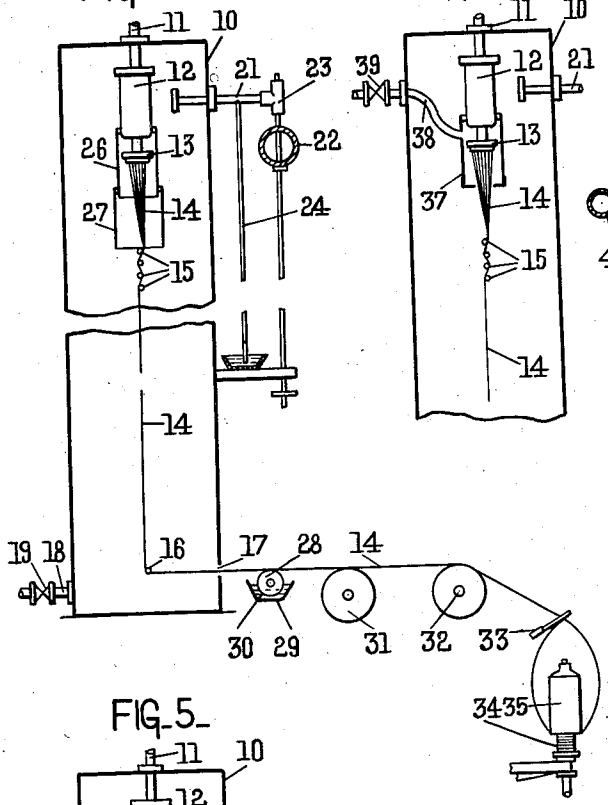


FIG. 2.

FIG. 3.

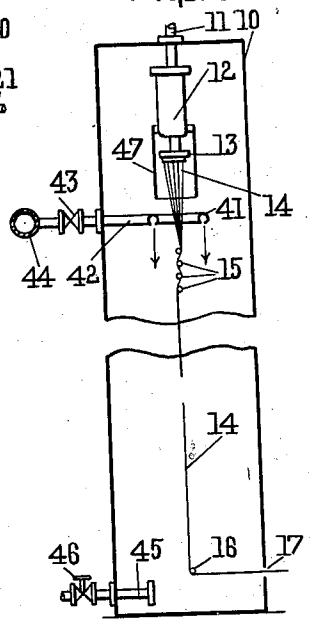


FIG. 5.

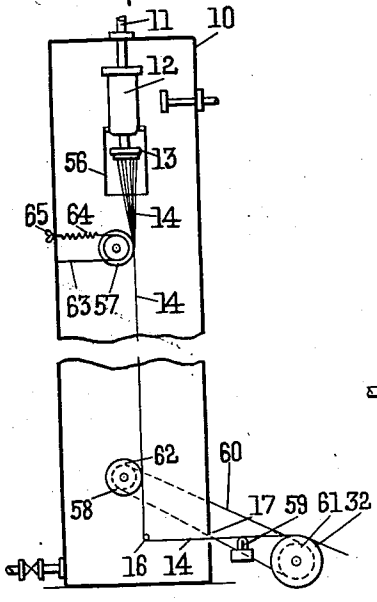


FIG. 4.

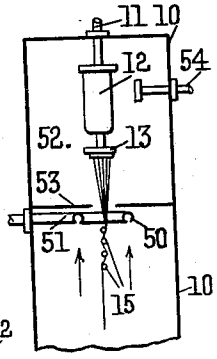
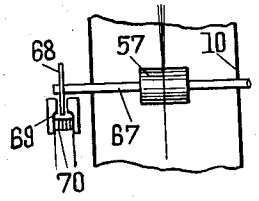


FIG. 6.



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UNITED STATES PATENT OFFICE

1,992,609

MANUFACTURE OF ARTIFICIAL FILAMENTS AND THE LIKE

Henry Dreyfus, London, England

Application August 26, 1932, Serial No. 630,491
In Great Britain October 1, 1931

21 Claims. (Cl. 18—8)

This invention relates to improvements in the manufacture of artificial silk and like filaments, threads, yarns, ribbons and the like from solutions of cellulose acetate or other cellulose esters or ethers by the dry spinning method.

The principal object of the invention is to obtain filaments or other products of very high tenacity.

According to the present invention in the dry spinning of filaments, ribbons and the like the spinning conditions are arranged so as to retard evaporation in the initial stages of spinning, and the filaments or other products are subjected to a stretching operation in such a manner that the whole of the stretch does not extend to the spinning nozzle itself. By this means I have found that products of unusually high tenacity may be obtained.

The retardation of the evaporation in the initial stages of the spinning operation may be achieved by any suitable method. The methods described in U. S. application S. No. 561,442 filed 5th September, 1931, are particularly important. This specification describes inter alia the provision around the spinning nozzle of a pipe, tube or other device adapted to shield the filaments in the initial stages of their formation from the evaporative medium in the main body of the spinning cell. By this means the space enclosed by the said pipe, tube or other device becomes saturated with the vapour of the solvent of the spinning solution or it may, if desired, be saturated by suitable injection of such solvent and the evaporation of the solvent from the filaments in the initial stages of their formation is considerably retarded or even prevented altogether. As described in the said specification a comparatively slow current of air may, if desired, be passed through such a funnel, pipe, tube or other device which secludes the spinning nozzles from the main spinning cell. Further if desired instead of providing such tube, pipe or the like surrounding the spinning nozzle a spinning chamber or cell may be divided into two or more chambers, that adjacent the spinning nozzle being traversed at the most by only a slow current of evaporative medium or being provided from an outside source with vapours of the solvent of the spinning solution.

A further means of retarding the evaporation of the solvent in the initial stages of spinning is that described in British application No. 27,076/31 filed 29th September, 1931, namely by drawing off the evaporative medium from the spinning cell before said medium reaches the spinning nozzle, or, in the case of a co-current of evaporative medium, introducing said medium at some distance from the nozzle so that it does not contact therewith.

A still further means of retarding the evaporation consists in incorporating in the spinning solution one or more high boiling solvents or diluents, and in this connection reference is made to U. S. Patents Nos. 1,616,787 and 1,566,384, and U. S. applications S. Nos. 328,305 filed 24th December, 1928, 444,621 filed 15th April, 1930, 522,530 filed 13th March, 1931, and 535,284 filed 5th May, 1931. Such high boiling solvent or diluent may be incorporated in the spinning solution in any desired proportion suitable for retarding the evaporation of the volatile solvent in the initial stages or suitable for maintaining the filaments or other products soft. Preferably the proportion of high boiling solvent does not exceed 100% of the weight of the cellulose acetate or other cellulose derivative. In connection with the employment of such high boiling solvents, it may be pointed out that the solvents which are most valuable are those which yield solutions of the cellulose acetate or other base of the spinning solution which are of substantially higher viscosity than are the corresponding solutions in the volatile solvent itself. Thus, for instance, in spinning a solution of cellulose acetate in acetone highly valuable results are achieved by employing solvents which yield a solution of cellulose acetate of at least 4, and preferably 6 to 10, times or more the viscosity of the corresponding solution of the cellulose acetate in acetone. The employment of such high boiling solvents in the spinning solution is claimed broadly in British application No. 27324/31 filed 1st October, 1931. Examples of such high boiling solvents are mono-acetin, diacetin and glycol mono-acetate, methyl glycol acetate, methyl glycol dioxane, and dimethylene dioxide. The first three of these solvents are especially valuable and yield solutions of cellulose acetate of exceedingly high viscosity. In some cases the solvent need not yield a true solution of the cellulose acetate but it should preferably yield a true solution on addition of a small amount of another agent, and particularly another solvent, such as acetone.

The present invention furthermore comprises the employment in conjunction with one another of any two or more of the above means for retarding the evaporation.

As previously indicated it is characteristic of the present invention that in addition to retarding the evaporation of the solvent in the initial

stages of spinning, the filaments, threads or the like are stretched in such a manner that the whole of the stretch does not run back to the spinning nozzle. This result may be brought about by any suitable means. For example, in carrying out the invention the filaments may encounter means such as a roller, guide or tension device which offers a resistance to their passage, the amount of the resistance determining whether tension applied to the softened portion of the filaments is in part allowed to extend to the nozzle or is wholly confined to a later stage of the drying.

I find it desirable to employ in the spinning cell at least two rollers, preferably small, between which stretch or additional stretch is applied. If desired three, four or more rollers or similar devices may be employed so as to obtain a number of stretching stages. Such rollers may be mounted on ball bearings or other anti-friction bearings so as to facilitate rotation. Two rollers between which stretch or additional stretch is applied to the filaments may differ in diameter. The rollers may be constructed of very light material, as for example of aluminium or other light metal, or of insoluble phenol-aldehyde synthetic resin or other resistant plastic material. Furthermore the rollers may be made relatively short and may if desired be grooved so as to receive the filaments. Preferably such rollers are provided with means adapted to resist rotation thereof, such resistance differing from roller to roller. Thus, for instance, the rollers may carry vanes or like devices adapted to provide an air or liquid resistance to the rotation of the roller by rotating in a compartment outside the spinning cell or inside the spinning cell and separated from the main body of the cell. Any other means may be adopted so as to set up in the rollers a differential resistance to rotation, or the rollers may be positively driven at different speeds, the roller nearest the jet being rotated at a speed calculated to impart little or no stretch to the filaments between it and the jet.

The filaments may be further stretched if desired after they have left the spinning cell, such stretch being facilitated, if desired or requisite, by suitable application of a solvent or softener.

Though the invention is especially of value in the spinning of filaments or other products of cellulose acetate, it may also be applied to the spinning of solutions of other cellulose esters, as for example cellulose formate, cellulose propionate, cellulose butyrate, cellulose nitrate or nitro-acetate, and of cellulose ethers, for instance methyl cellulose, ethyl cellulose or benzyl cellulose.

The invention will now be described in greater detail with reference to the accompanying drawing, but it is to be understood that this description is given by way of illustration only, and is in no respect limitative.

Figure 1 is a side elevation in section of a spinning cell for carrying out the present invention,

Figure 2 shows a variation in detail of Figure 1,

Figures 3, 4 and 5 show three further forms of apparatus for carrying out the invention, and

Figure 6 shows a variation in detail of Figure 5.

In Figure 1 a spinning cell 10 is provided with pipe 11 for the supply of spinning solution, pipe 11 leading the solution through a filter candle 12 to a spinning jet 13. The filaments 14 issuing from the jet pass in and out of a number of tension rods 15 and down the spinning cell 10, turning at the bottom of the cell around a guide rod 16 and leaving the cell through an aperture 17. A current of evaporative medium enters the cell 10 at the bottom by an inlet 18 controlled by a cock 19 and leaves by a draw-off pipe 21 conveying it to a header 22, valve means 23 being provided in order that the rate of flow of evaporative medium may readily be adjusted. A flow-gauge 24 may, if desired, be connected in the pipe 21.

In order that evaporation of the filaments 14 in the neighbourhood of the pipe 13 may be retarded, the jet 13 and the filaments 14 as far down as the rods 15 are surrounded by a cowl which is made in two parts 26, 27 which may be telescoped and slid up the candle 12, by which they are supported, when it is desired to reach the jet 13. In this manner the zone is kept more or less stagnant within the hood, and the evaporation of solvent from the filaments between the jet 13 and rods 15 is retarded.

The filaments 14 leaving the cell 10 by the aperture 17 pass over a small roller 28 freely rotating, preferably in ball bearings, in a trough 29 containing a liquid 30 which is a solvent, softener, or gelling agent for the substance of the filaments. The filaments 14 then pass successively over two rollers 31, 32, the roller 31 rotating at such a speed as to stretch the filaments within the cell 10, while the roller 32 rotates at a higher speed in order to stretch the filaments which, after setting in the cell 10, have been softened in passing over the roller 28, between the rollers 31, 32. The stretch imparted by the roller 31 extends as far as the tension rods 15, but is prevented by the rods from extending to any large extent to the part of the filaments lying between rods 15 and the spinning jet 13. On leaving the roller 32 the filaments 14 pass to the balloon guide 33 of a cap spinning device 34 by means of which they are twisted, and wound on to a bobbin 35.

Figure 2 shows a variation in detail of the upper part of the cell shown in Figure 1. In this figure a hood is provided around the jet 13 in the form of a single piece 37 which is fitted with a flexible tubing 38 controlled by a valve 39 lying outside the cell. By means of the tube 38 a proportion of air, preferably small, may be drawn off from the hood 37, or alternatively a small quantity of air may be passed into the cell by the same means. Or again, the stagnant nature of the atmosphere within the hood 37 may be augmented by supplying through the tube 38 vapours of material which is a solvent for the substance of the filaments, e. g. the solvent which is used in the spinning solution from which the filaments are produced.

In Figure 3 a current of evaporative medium enters the spinning cell 10 through an annular pipe 41, fed by a pipe 42 and valve 43 from a pressure header 44 or other supply. The current of evaporative medium flows in the same direction as the filaments and leaves by a draw-off device 45 at the bottom of the cell 10, the draw-off device being provided with control means indicated diagrammatically at 46. Since the annular pipe 41 through which evaporative medium is introduced into the cell lies below the level of the nozzle 13, that part of the cell lying above the ring 41, including the nozzle 13, is not swept by the current of evaporative medium and, in consequence, evaporation therein is retarded. In order to protect the filaments 14 as they issue from the nozzle 15 from small irregular currents of evaporative medium, a hood 47 is provided

about the nozzle 13. Stretch is applied to the filaments 14 lying within the cell in a manner similar to that described with reference to Figure 1, the stretch being prevented from running back to the filaments 14 in the neighbourhood of the nozzle 13 by means of tension rods 15 in and out of which the filaments pass.

Figure 4 shows the evaporative medium entering at the bottom of the cell, as described with reference to Figure 1, and leaving by an annular tube 50 lying below the level of the spinning nozzle 13. A draw-off pipe 51 leads from the annular pipe 50 out of the cell and is provided with control means similar to those described with reference to Figure 1. The nozzle 13 and the filaments immediately issuing therefrom are contained in a separate chamber 52 divided from the rest of the spinning cell 10 by means of a partition 53. Since the major part of the evaporative medium is drawn off before entering the chamber 52, evaporation of the filaments therein is retarded. An additional draw-off device 54 may be provided in the chamber 52 whereby a slow current of evaporative medium may be caused to pass through the chamber 52, so that the stagnant nature of the atmosphere in the chamber may be modified at will. The proportion of evaporative medium leaving the cell in this manner may be controlled by means similar to those described in connection with the draw-off device 21 in Figure 1. As described with reference to Figures 1 to 3 stretch applied to the filaments 14 outside the cell 10 is prevented from running back to the nozzle 13 by means of tension bars 15 in and out of which the filaments pass.

In Figure 5 the restriction of evaporation is effected in the manner described with reference to Figure 1, a hood 56 being provided round the spinning jet 13 for this purpose. The filaments 14 issuing from the hood 56 pass successively round two rollers 57, 58 inside the cell before turning round the guide 16 and leaving the cell by the orifice 17. The filaments then pass over a feed roller 32 driven at a constant peripheral speed, the roller 58 being driven from the roller 32 by means of a chain 60 and gears 61, 62. The peripheral speed of the roller 58 is lower than that of the roller 32 so that the filaments 14 are stretched between them, the filaments being softened by passage over a wick 59 supplied with a solvent or the like. The roller 57 is provided with a band brake 63 adjustable by means of a spring 64 and a wing nut 65, the effect of the braked roller being to allow stretch to be imparted to the filaments 14 between the rollers 57, 58 and to prevent such stretch from being communicated beyond the roller 57 to the filaments 14 lying between it and the jet 13. The filaments 14 leaving the roller 32 pass to a cap spinning device as described with reference to Figure 1, or to any other convenient collecting means (not shown).

Figure 6 shows an alternative form of braking device for the roller 57, the shaft 67 on which the roller is mounted extending outside the cell 10 and being provided with a metal disc 68 rotating between the poles of a magnet 69 excited by means of a coil 70. In this manner a resistance to rotation is imparted to the shaft 67 and the roller 57, similar to that imparted by the brake 63 shown in Figure 5. The disc 68 and the magnet 69 may be of such size as to serve a series of rollers 57 rotating in a series of cells and mounted on a common shaft 67 running along the length of the series, so that uniformity in the manner of applying stretch is ensured throughout the series of cells.

What I claim and desire to secure by Letters Patent is:—

1. Process for the production of artificial filaments, threads, yarns, ribbons and the like by the dry or evaporative method, which process comprises extruding a spinning solution into a current of evaporative medium, secluding the extruded product in the initial stages of spinning from the effect of the main body of such current so as to retard its initial evaporation, submitting the product to a stretching operation, and restraining the stretch so imparted so that the whole of it does not extend to the part of the product so secluded.

2. Process for the production of artificial filaments, threads, yarns, ribbons and the like by the dry or evaporative method, which process comprises extruding a spinning solution into a current of evaporative medium flowing past the point of extrusion, secluding the extruded product in the initial stages of spinning from the effect of the main body of such current so as to retard its initial evaporation, submitting the product to a stretching operation, and restraining the stretch so imparted so that the whole of it does not extend to the part of the product so secluded.

3. Process for the production of artificial filaments, threads, yarns, ribbons and the like by the dry or evaporative method, which process comprises extruding a spinning solution into a current of evaporative medium flowing in a direction opposite to the direction of extrusion, withdrawing said current of evaporative medium before it reaches the point of extrusion so as to seclude the extruded product in the initial stages of spinning from the effect of the main body of such current and to retard its initial evaporation, submitting the product to a stretching operation, and restraining the stretch so imparted so that the whole of it does not extend to the part of the product so secluded.

4. Process for the production of artificial filaments, threads, yarns, ribbons and the like by the dry or evaporative method, which process comprises extruding a spinning solution into a current or evaporative medium flowing in the direction of extrusion but originating at a point on the extrusion side of the point of extrusion so as to seclude the extruded product in the initial stages of spinning from the effect of the main body of such current and to retard its initial evaporation, submitting the product to a stretching operation, and restraining the stretch so imparted so that the whole of its does not extend to the part of the product so secluded.

5. Process for the production of artificial filaments, threads, yarns, ribbons and the like by the dry or evaporative method, which process comprises extruding a spinning solution into a current of evaporative medium, secluding the extruded product in the initial stages of spinning from the effect of the main body of such current so as to retard its initial evaporation but allowing a small quantity of evaporative medium to flow past the point of extrusion, submitting the product to a stretching operation, and restraining the stretch so imparted so that the whole of it does not extend to the part of the product so secluded.

6. Process for the production of artificial filaments, threads, yarns, ribbons and the like by the dry or evaporative method, which process comprises extruding a spinning solution into a current of evaporative medium flowing in a direc-

tion opposite to the direction of extrusion, withdrawing all but a small proportion of evaporative medium before it reaches the point of extrusion so as to seclude the extruded product in the initial stages of spinning from the effect of the main body of such current and to retard its initial evaporation, submitting the product to a stretching operation, and restraining the stretch so imparted so that the whole of its does not extend to the part of the product so secluded.

7. Process for the production of artificial filaments, threads, yarns, ribbons and the like by the dry or evaporative method, which process comprises extruding a spinning solution into a current of evaporative medium flowing in the direction of extrusion but originating mainly at a point on the extrusion side of the point of extrusion so as to seclude the extruded product in the initial stages of spinning from the effect of the main body of such current and to retard its initial evaporation, submitting the product to a stretching operation, and restraining the stretch so imparted so that the whole of it does not extend to the part of the product so secluded.

8. Process for the production of artificial filaments, threads, yarns, ribbons and the like by the dry or evaporative method, which process comprises extruding a spinning solution into a current of evaporative medium, secluding the extruded product in the initial stages of spinning from the effect of the main body of such current so as to retard its initial evaporation, submitting the product to a stretching operation, restraining the stretch so imparted so that the whole of it does not extend to the part of the product so secluded, and introducing in the neighborhood of the product in the initial stages of spinning vapors of a solvent for the substance of the product.

9. Process for the production of artificial filaments, threads, yarns, ribbons and the like by the dry or evaporative method, which process comprises extruding a spinning solution containing a high boiling solvent into a current of evaporative medium, secluding the extruded product in the initial stages of spinning from the effect of the main body of such current so as to retard its initial evaporation, submitting the product to a stretching operation, and restraining the stretch so imparted so that the whole of it does not extend to the part of the product so secluded.

10. Process for the production of artificial filaments, threads, yarns, ribbons and the like from solutions of cellulose derivatives by the dry or evaporative method, which process comprises extruding a spinning solution of a cellulose derivative into a current of evaporative medium, secluding the extruded product in the initial stages of spinning from the effect of the main body of such current so as to retard its initial evaporation, submitting the product to a stretching operation, and restraining the stretch so imparted so that the whole of it does not extend to the part of the product so secluded.

11. Process for the production of artificial filaments, threads, yarns, ribbons and the like from solutions of cellulose acetate by the dry or evaporative method, which process comprises extruding a spinning solution of cellulose acetate into a current of evaporative medium, secluding the extruded product in the initial stages of spinning from the effect of the main body of such current so as to retard its initial evaporation, submitting the product to a stretching operation, and restraining the stretch so imparted so that the whole of it does not extend to the part of the product so secluded.

12. Apparatus for the production of artificial filaments, threads, yarns, ribbons and the like by the dry or evaporative method, which comprises a spinning cell, a spinning jet therein for the extrusion of material, means for passing a current of evaporative medium through said cell, means for secluding the extruded product from the main effect of said current in the neighborhood of said jet, means for stretching said product and means for preventing the applied stretch from reaching the secluded part of said product.

13. Apparatus for the production of artificial filaments, threads, yarns, ribbons and the like by the dry or evaporative method, which comprises a spinning cell, a spinning jet therein for the extrusion of material, means for passing a current of evaporative medium through said cell, a shield adapted to surround the neighborhood of said jet for secluding the extruded product from the main effect of said current in the neighborhood of said jet, means for stretching said product and means for preventing the applied stretch from reaching the secluded part of said product.

14. Apparatus for the production of artificial filaments, threads, yarns, ribbons and the like by the dry or evaporative method, which comprises a spinning cell, a spinning jet therein for the extrusion of material, means for passing a current of evaporative medium through said cell in a direction opposite to the direction of extrusion, means for withdrawing said current at a point short of the spinning jet so as to seclude the extruded product from the main effect of said current in the neighborhood of said jet, means for stretching said product and means for preventing the applied stretch from reaching the secluded part of said product.

15. Apparatus for the production of artificial filaments, threads, yarns, ribbons and the like by the dry or evaporative method, which comprises a spinning cell, a spinning jet therein for the extrusion of material, means for passing a current of evaporative medium through said cell in the direction of extrusion and from a point on the extrusion side of said jet so as to seclude the extruded product from the main effect of said current in the neighborhood of said jet, means for stretching said product and means for preventing the applied stretching from reaching the secluded part of said product.

16. Apparatus for the production of artificial filaments, threads, yarns, ribbons and the like by the dry or evaporative method, which comprises a spinning cell, a spinning jet therein for the extrusion of material, means for passing a current of evaporative medium through said cell, means for secluding the extruded product from the main effect of said current in the neighborhood of said jet, means for stretching said product, means for preventing the applied stretch from reaching the secluded part of said product, and means for introducing a small quantity of evaporative medium to reach the product in the initial stages.

17. Apparatus for the production of artificial filaments, threads, yarns, ribbons and the like by the dry or evaporative method, which comprises a spinning cell, a spinning jet therein for the extrusion of material, means for passing a current of evaporative medium through said cell.

a shield adapted to surround the neighborhood of said jet for secluding the extruded product from the main effect of said current in the neighborhood of said jet, means for stretching said product, means for preventing the applied stretch from reaching the secluded part of said product, and means for introducing a small quantity of evaporative medium within said shield.

18. Apparatus for the production of artificial filaments, threads, yarns, ribbons and the like by the dry or evaporative method, which comprises a spinning cell, a spinning jet therein for the extrusion of material, means for passing a current of evaporative medium through said cell in a direction opposite to the direction of extrusion, means for withdrawing the greater part of said current at a point short of the spinning jet so as to seclude the extruded product from the main effect of said current in the neighborhood of said jet, means for stretching said product, means for preventing the applied stretch from reaching the secluded part of said product and means for withdrawing the remainder of said current at a point behind said spinning jet.

19. Apparatus for the production of artificial filaments, threads, yarns, ribbons and the like by the dry or evaporative method, which comprises a spinning cell, a spinning jet therein for the extrusion of material, means for passing a current of evaporative medium through said cell in the direction of extrusion and for the most part from a point on the extrusion side of the said jet so as to seclude the extruded product from the main effect of said current in the neighborhood of said jet, means for stretching said product, means for preventing the applied stretch from reaching the secluded part of said product, and means for introducing a small current of evaporative medium at a point behind the spinning jet.

20. Apparatus for the production of artificial filaments, threads, yarns, ribbons and the like by the dry or evaporative method, which comprises a spinning cell, a spinning jet therein for the extrusion of material, means for passing a current of evaporative medium through said cell, means for secluding the extruded product from the main effect of said current in the neighborhood of said jet, means for stretching said product, and a plurality of tension rods disposed on the extrusion side of the spinning jet for preventing the applied stretch from reaching the secluded part of said product.

21. Apparatus for the production of artificial filaments, threads, yarns, ribbons and the like by the dry or evaporative method, which comprises a spinning cell, a spinning jet therein for the extrusion of material, means for passing a current of evaporative medium through said cell, means for secluding the extruding product from the main effect of said current in the neighborhood of said jet, a pair of rollers within the spinning cell for stretching said product, means for driving the second of said rollers and means for braking the first of said rollers so as to prevent the applied stretch from reaching the secluded part of said product.

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