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Apparatus for Making Artificial Silk

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This invention is an improvement in apparatus for use in dry spinning cellulose esters in the manufacture of artificial silk, under which term are included the cellulose acetate and cellulose nitrate classes or types of artificial silk, artificial horsehair, and similar products, and it further includes a novel method of operating such apparatus in treating the filaments.

A commonly used method of spinning filaments for artificial silk is to dissolve the cellulose acetate, cellulose nitrate or similar ester in a solvent largely or entirely volatile (which I will hereinafter designate as "solution") and then force such solution under considerable pressure through a spinning-nozzle or spinneret provided with a plurality of orifices within a spinning cell. The filaments issuing from the orifices of the spinneret become progressively solidified as they traverse the cell by the evaporation of the solvent in the filaments so that after they emerge from the cell they can be twisted together into a thread.

In such methods it is usual to pass fresh air through the cell in a direction opposite the direction of movement of the filaments, and the volatile solvents have been removed from such air by adsorption in a liquid solvent, or in a solid absorbent such as absorbent carbon; and from these absorbents the volatile solvent may be subsequently recovered for reuse.

In some cases instead of passing a current of air through the entire cell, the latter is divided into zones in which the air is stagnant; such a cell and process are described in my application Serial No. 271,911, filed April 21, 1928, in which there is a stagnant air zone about the spinneret, another stagnant air zone at the exit end, and an intermediate zone traversed by a rapidly moving air current.

Some qualities of artificial silk depend on the temperature to which the filament is subjected during solidification and the rate of evaporation of the solvent, which latter is determined by the temperature and the amount of solvent vapor in the air in the cell, the rate at which the air is renewed, and the amount of stretching to which the filament is subjected before it is hardened. The commercial production of high quality artificial silk depends on maintaining the above conditions at the optimum, continuously and simultaneously, in each of a large number of cells over a long period of time.

Some of the objects of my present invention are to pass the artificial silk filaments through an air current whose major movement is longitudinally along but counter to the movement of the filaments, and without any substantial lateral deflection of air in the cell at right angles to the direction of movement of the filaments before they harden; to control temperatures; to produce a natural circulation of air in the cell by producing differences in density of the columns of air in the condenser and the cell; to remove the solvent from the air so completely that the air is free of ether laden air escaping from the cell with the filament will be practically obliterated; to reunite the same air in a closed circuit thereby preventing dust particles entering the cell and adhering to the filaments and injuring the quality of the thread; and to recover a very high percentage of solvent for reuse.

Further objects are to construct the cell so that it can be economically maintained in effective condition and operation, its interior may easily be kept clean and contain nothing on which broken filaments or drops of the solution may lodge; and the operating conditions therein can be readily controlled.

Another object of the invention is to provide removable pierced diaphragms inserted in the ducts connecting the spinning chamber and condenser; and by inserting diaphragms having the proper orifices the speed of the circulation of the air can be controlled without changing the temperature of the spinning chamber or the condenser. Also by this device it is possible to change the concentration of solvent vapor in the air near the spinneret without changing the temperature.

Other objects and advantages of the invention will be hereinafter set forth.

The accompanying drawing diagrammatically illustrates an apparatus embodying the invention and I will explain the construction of said apparatus and the method of operating same to enable others to adapt and use the invention. I refer to the claims for summaries of the essentials of the invention, and of the novel features of construction and novel combination of parts, for which protection is desired.

As shown in said drawing the cell or spinning chamber comprises an elongated body 1 the central and major portion of which is surrounded by a jacket 2, the space between the jacket 2 and the body 1 forming a chamber B which can be heated by hot water or any other suitable means. Within the cell adjacent the upper end thereof is a spinneret 3 which is connected to a supply tube 3a that extends out of the upper end of the body and is connected to suitable means for forc-
2 ing a cellulose nitrate solution or cellulose acetate solution through said tube to the spinneret. At the lower end of the body is an outlet tube 4 through which the filaments pass out of chamber A. The lower end of the body is preferably contracted and surrounded by a jacket 5, and the space between the end of the body and the jacket forms a heating chamber C, which chamber may be heated by steam or any other suitable means. The chambers B and C can be independently heated as desired, or chamber B alone may be used.

The upper end of chamber A is connected by a duct 6 to a recovery device 7 and at a point adjacent its lower end chamber A is connected by a duct 8 to the other end of the condenser 7. In the example shown the condenser comprises a chamber D within which is a tube 8 through which should be circulated a suitable cooling fluid. The solvents condensed in the condenser 7 accumulate in the lower end thereof below the duct 8 and form a pool of condensate, indicated at E, excess condensate passing off through a pipe 10, which may be provided with a glass section 10a through which the flow of condensate may be observed. In the ducts 6 and 8 are inserted removable diaphragms 6a and 8a, which have perforations of such size as will allow an exact control of the amount of air circulated through and between the cell and condenser. The diaphragms 6a and 8a are preferably made of non-heat-conducting material to prevent direct interchange of heat between the heated cell and the cold condenser, but they may be made of metal and insulated with non-heat-conducting material. The gaskets employed to render the diaphragm tight should be of non-heat-conducting material and the bolts used in connection therewith so arranged as not to conduct heat between the parts.

The cell is preferably covered with non-heat-conducting material: and condenser 7 is also preferably covered with non-heat-conducting material to prevent gain of heat or sweating.

The cell is preferably provided with transparent openable ports, as shown at 11, 11a and 12, 12a, these being hermetically closable, but affording means for inspection of access to the top and bottom portions of the cell.

The air is circulated in a closed circuit through the chamber A to and through the condenser chamber D back to the chamber A. In passing through the condenser chamber D the vapor in the air condenses on the surface of the tube 8 and this condensate collects in a pool E at the bottom of chamber D. Through the transparent section 10a of pipe 10 the rate of condensate flow can be observed, thus enabling a check to be maintained on the rate of condensation.

The outlet tube 4 when of suitable length and diameter will very definitely limit the escape of solvent from the chamber with the filaments, and this action is enhanced by a nozzle 4a on the lower extremity of said tube. The upper end of the orifice in this nozzle is restricted to a minimum area, just sufficient to allow ready passage of the filaments. Said orifice enlarges downwardly and outwardly, and its interior surfaces are smooth and have no sharp edges that might injure the delicate filaments.

It is a well known physical fact that if two chambers are connected by means of a restricted passage and the atmosphere in one of the chambers is different from the atmosphere in the other, the rate of diffusion of the different atmospheres through the said passage is proportionate to the minimum area of cross section of the said passage. In accordance with this fact the length of outlet tube 4 is such as to lower the rate of diffusion of air, or other gaseous atmosphere, between its two extremities to a desired minimum, while providing a uniform gradient along its whole internal length. The restricted area of the bore in nozzle 4 is the final restricting factor in the reduction of diffusion through the filament outlet. By employing a tube ¾” in diameter and ten inches in length I have found the loss of solvent to be entirely negligible.

A high temperature in the lower end of the chamber A (produced by the heating chamber C) will cause convection currents of air, in this part of the chamber, which will move upwardly opposite to the direction of movement of the filaments, and such currents will take from the filaments any solvent vapors that may still adhere to or surround them at this point.

The solvent content of the atmosphere returned to the spinning chamber A through the duct 8 is of utmost importance in determining the physical properties of the final filaments produced and should be observed. In the example described the plastic state of the solvents depends upon:

1. the velocity of evaporation of the solvent from the filaments during solidification;
2. the solvent content of the atmosphere in chamber A;
3. the velocity of circulation of the atmosphere through the said chamber;
4. the temperature of said chamber;
5. the temperature of the atmosphere returned from the condenser into the chamber; and
6. the amount of stretching to which the filaments are subjected while still in the plastic state in which they remain for quite a distance downward from the spinnereet.

These six essential features are all controllable in my cell by reason of:

1. the control of temperature in chamber B;
2. the control of flow through ducts 6 and 8 by diaphragms 6a and 8a; and
3. the control of temperature of the fluid circulating through tube 9.

My apparatus is inexpensive and easily operated and, due to the temperatures used, is peculiarly adapted for spinning artificial silk from cellulose ester solutions and particularly from cellulose acetate dissolved in acetic acid for the making of a cellulose acetate thread.

When spinning at a constant speed (i.e., constant length of filament per second) the spinning chamber adjustments and methods of operation will vary with every varying denier of filament produced. For example in the spinning of 75 denier filaments the condenser 7 is cooled by a suitable brine solution to a desired temperature and at the point where the solvent is led off the solvent chamber 135 has a temperature of within 5° C. of the cooling fluid circulating through tube 9. The spinning chamber A is heated by hot water, or hot glycerine-water mixture, circulated through the chamber B; the temperature of the heating fluid being maintained to allow ready passage of the filaments. At the spinnereet 3 an atmospheric temperature of about 80° C. is maintained. The atmosphere at that point carries from about 500 to 400 grams of acetic acid per cubic meter. In the upper part of chamber A, in the vicinity of duct 8, a temperature of about 0° C. is maintained. The temperature at this point not only controls the solvent content in the atmosphere returning to the spinning chamber, but also...
fects the physical characteristics of the filament by reducing or increasing its plasticity, and therefore its reaction toward stretch. The solvent content of the returning atmosphere should be not more than 150 grams per cubic meter, and the temperature of the said atmosphere returned should be kept constant within plus or minus 2°C. For a more specific example, the condenser is cooled by brine at about −14°C. The temperature of the acetone at the point at which it is drawn off is about −10°C. The spinning cell is heated by hot water at about 90°C. The temperature of the atmosphere at the spinneret is about 89°C. The humidity conditions in the exterior insure a thread of very high uniformity, which will not be easily affected in subsequent textile operations by varying atmospheric conditions.

The recovered solvent is free from impurities and can be re-used without further concentration.

The final minimum amount of solvent retained by the filaments can be easily controlled by varying the temperature of the heater B, by varying the length of the spinning chamber A or by varying the temperature of the outlet heater C. The latter especially controls the amount of solvent removed from the filaments in the lower end of the spinning chamber A, and thereby the solvent content in the finished thread.

By the same means as those described in the preceding paragraph, any desired or determined amount of solvent may be allowed to be retained by the filaments so as to facilitate the subsequent operation of twisting and fix the thread. This is done by evaporating the desired small amount of residual solvent after the filaments have been twisted, thereby fixing the twist in the colloidal texture of the filaments.

Direct heat transfer between the chambers A and D is prevented by the distance between these chambers, by the heat insulating gaskets of the diaphragms, and also by the non-conductive coverings of these chambers which is an important factor in the commercial operation of my apparatus and facilitates exact temperature control of both chambers.

The atmosphere is withdrawn from the spinning chamber at such a distance from the spinneret that the atmosphere immediately around the circumference of the spinneret remains homogeneous and prevents the filaments at one side of the spinneret from being in a richer or poorer solvent atmosphere than those on the other side of said spinneret.

The condenser D, consisting of an inner cooling tube surrounded by atmosphere containing solvent, is very efficient because the said tube takes heat directly from the solvent laden atmosphere; and it not only acts as a condenser but provides for the formation of a constant pool of condensed liquid solvent. The surface of such pool being at a very low temperature, it also will tend to lower the vapor tension of the portion of atmosphere passing over it in returning to the spinning chamber, so that such portion is practically destitute of its solvent content.

The spinning chamber being heated by an external heater will immediately raise the cold and denuded atmosphere reentering from the condenser to a proper temperature. The heating chamber B preferably terminates a short distance below the spinneret.

Due to the residual amount of acetone in the atmosphere reentering the chamber A friction between the filament and incoming warm dry air is avoided and the tendency of the filaments to become electrified and fly apart, thereby causing breakage and irregular winding of the thread, is prevented.

As the atmosphere is used over and over again in a closed circuit it is freed from water vapor by cooling in the condenser, the condensed water vapor passing off with the solvent, whereas in apparatus and methods where fresh air is continually introduced the air in winter is at a low humidity (say 15%) and in summer at high relative humidity (30–95%).

The percentage of evaporation of the solvent from the filaments is controllable and will be in part determined by the distance traveled by the
filament in the spinning cell, six feet being a good commercial length.

The use of changeable diaphragms to control the speed of the air without changing the temperature of either the jacket or the condenser, is of great importance, because varying the size of the filaments being produced will vary the atmospheric conditions unless movement of the atmosphere in the spinning cell is controlled. While the temperature of the condenser can be changed that is possible only when a single cell or at most a few cells are in use to spin the same size filament; but in a plant where there are thousands of cells spinning filaments of different sizes, it is not practical to use different temperatures for the various condensers without great complication of the installation. With my invention by merely changing the diaphragms helps to simple and easy control of the flow of air over the filaments is obtained.

It will be noted that my apparatus has means for independently controlling the temperature, the concentration of solvent in air, the velocity of circulation, the temperature at which condensation of solvent takes place, and the rate of the diffusion with external air therefrom. Accordingly, the speed of drying of the filament can be closely controlled and filaments of the desired shape and cross section obtained.

In the appended claims where filaments of artificial silk are mentioned, it is to be understood that the term includes filaments of artificial horsehair, artificial straw, and similar products made from cellulose acetate solutions and other cellulose ester solutions as well as filaments of artificial silk in the more limited sense.

I claim:

1. Apparatus for dry spinning filaments of artificial silk comprising: a spinning cell, a spinning nozzle in the upper part of the cell, an outlet tube in the lower part of the cell through which the filaments extruded from the nozzle are withdrawn, the ratio of the diameter of said tube to the length thereof and to the size of the filament to pass therethrough being such as substantially to prevent passage of interior atmosphere therethrough into the atmosphere external to the cell, a condenser separate from the cell and exterior thereto, a duct connecting the cell above the nozzle with the inlet of the condenser, and a duct connecting the discharge of the condenser with the cell adjacent the outlet tube.

2. Apparatus for splitting artificial silk filaments comprising a spinning chamber having at one end an outlet for spun filaments, heating means for heating the chamber at and immediately adjacent to the outlet, and means for heating the main body of said chamber.

3. Apparatus for splitting artificial silk filaments comprising an elongated cylindrical spinning chamber having a tapered end terminating in an outlet for spun filaments, and a heating jacket surrounding said tapered end.

4. Apparatus for splitting filaments of artificial silk comprising a spinning chamber, a condensing system, an outlet connection between the upper part of the chamber and said system, an inlet connection between the lower part of the chamber and said system, means for heating the chamber between said inlet and outlet connections, and means independent of said means for heating the chamber below said inlet connection and adjacent the filament outlet of the chamber.

5. Apparatus for splitting filaments of artificial silk comprising a spinning cell, spinning means therein, means for circulating a solvent-evaporating atmosphere through the cell, and means for preventing ingress of air through the filament outlet, comprising an outlet pipe having a long central bore coaxial with the filaments and having a slight clearance therefrom, the ratio of the diameter of said outlet pipe to the length thereof and the size of the filament passing therethrough being such that the downward movement of a filament through said outlet bore substantially will prevent ingress of air through said outlet by reason of the slight clearance between the filaments and the walls of the bore.

6. Apparatus for splitting artificial silk filaments including a spinning chamber, a condensing chamber, a duct connecting the chambers, and a removable perforated diaphragm including a heat insulating medium extending across said duct.

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