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(54) **ROLLER SURFACE USED IN LYOCCELL FILAMENT PRODUCTION**

(57) The present invention relates to the surface adaptation of a roller to be employed in the production of lyocell filament yarns.

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Description

[0001] The present invention relates to the surface adaptation of a roller to be employed in the production of cellulose filament yarns.

Background

[0002] Continuous filament yarns are widely used in the textile industry to produce fabrics with a distinct character compared to fabrics produced from yarns made using staple fibers. A continuous filament yarn is one in which all of the fibers are continuous throughout any length of the yarn. A continuous filament yarn will commonly consist of 10 to 300 or more individual filaments which are all parallel to each other and the axis of the yarn when produced. The yarn is produced by extruding a solution or melt of a polymer or a polymer derivative and then winding the yarn produced onto a bobbin or reel or by forming a cake by centrifugal winding.

[0003] Synthetic polymer continuous filament yarns are common. For example, nylon, polyester and polypropylene continuous filament yarns are used in a wide variety of fabrics. They are produced by melt spinning a molten polymer through a spinneret with a number of holes corresponding to the number of filaments required in the yarn produced. After the molten polymer has started to solidify, the yarn may be drawn to orient the polymer molecules and improve the properties of the yarn. Continuous filament yarns can also be spun from cellulose derivatives such as cellulose diacetate and cellulose triacetate by dry spinning. The polymer is dissolved in a suitable solvent and then extruded through a spinneret. The solvent evaporates quickly after extrusion causing the polymer to precipitate in the form of filaments forming a yarn. The newly produced yarn may be drawn to orient the polymer molecules.

[0004] One way to produce Cellulose based filaments and fibers is the so called lyocell process. Lyocell technology is a technology based on the direct dissolution of cellulose wood pulp or other cellulose-based feedstock in a polar solvent (for example n-methyl morpholine n-oxide, hereinafter referred to as 'amine oxide') to produce a viscous highly shear-thinning solution which can be formed into a range of useful cellulose-based materials. Commercially, the technology is used to produce a family of cellulose staple fibers (commercially available from Lenzing AG, Lenzing, Austria under the trademark TEN-CEL®) which are widely used in the textile and nonwovens industries. Other cellulose products from lyocell technology such as filaments, films, casings, beads & nonwoven webs have also been disclosed.

[0005] EP 823945 B1 discloses a process for the manufacture of cellulose fibers, which comprises the extrusion and coagulation of a cellulose spinning solution in accordance with the lyocell process, mandatorily comprising a step of drawing the filaments and cutting the filaments into cellulose fibers, which may be used in var-

ious fields of application. Process step of drawing the coagulated cellulose filaments is essential according to the teaching of this prior art technology in order to obtain in particular staple fibers with a desired balance of properties.

EP 0 853 146 A2 discloses a process for the preparation of cellulose based fibers. According to the teaching of this document two different raw materials having widely differing molecular weights are mixed in order to obtain fibers. WO 98/06754 discloses a similar method, which require that the two different raw materials are first dissolved separately, before admixing the prepared solution to obtain a spinning solution. DE 199 54 152 A1 discloses a method of preparing fibers, wherein spinning solutions having a relatively low temperature are employed.

The benefits of cellulose filament yarns produced from lyocell spinning solution have been described (Krüger, Lenzinger Berichte 9/94, S. 49 ff.). However, due to increasing demands regarding spinning efficiency, attempts have been made to increase spinning speeds in the lyocell process to values of several hundred meters per second. However, at such high spinning velocities various problems may occur, including unsatisfactory high proportion of defects in the individual filaments produced, which may result in a high proportion of products which are not suitable for further use and/or result in stoppage of production.

[0006] During the lyocell process the filaments and/or filament bundles, after the initial filament coagulation in a coagulation bath, are transported by means of various rollers to the further stages of the process, such as washing etc.. Initially the filaments or filament bundles are still susceptible to damage and/or may undesirably adhere to each other when they come into contact, so that care has to be taken that the passage of the filaments or filament bundles around the rollers does not lead to undesired impact on the filaments produced. Example of such negative impacts are slippage due to unsatisfactory adhesion to the roller surface, axial movement of the filaments or filament bundles on the roller surface (i.e. sideways movement on the roller surface, so that there may be undesired contact between neighboring filaments or filament bundles) as well as impact due to too high adhesion to the roller surface so that mechanical forces are exerted on the filament or filament bundles when these detach from the roller surface.

[0007] The preparation of continuous filament lyocell yarns at high velocities therefore presents new process challenges. It would be beneficial if slippage or other problems during the process could be minimized as much as possible in a reliable manner. In particular it would be beneficial if these problems could be minimized or avoided altogether not only during the initial stages of the process (immediately after spinning as well as the initial washing steps) but also during later stages of the process, such as application of additional substances, such as finishing (avivage) processes or the application of oils, moisturizing compositions etc.

Object of the invention

[0008] Accordingly it is the object of the present invention to provide a means for preventing defects to lyocell filaments or filament bundles as well as a means for preventing manufacturing problems, for example due to slippage etc. during lyocell filament production, in particular at high speed.

Brief description of the Invention

[0009] Accordingly the present invention provides the roller as defined in claim 1 as well as the method according to claim 2. Preferred embodiments are given in claims 3 to 11 and the specification, as well as in use claims 12 to 15.

Detailed description of the Invention

[0010] As is well known in the art, the lyocell process comprises, after preparation of the spinning solution, the extrusion of filaments through nozzles, followed in most cases by an initial cooling in an air gap, followed by coagulation in a bath. Thereafter the filaments or filament bundles are taken up, usually by a guidance roller to guide the product to the further process steps, such as liquid removal and washing steps. Also during these stages the filaments or filament bundles are moved to the next process steps by means of rollers. Additional stages of filament production may be stages to apply further substances to the filament or the filament yarn, such as application of finishing agents etc.

[0011] Concerning these rollers, in particular the initial guidance roller taking up the filaments after exiting the coagulation bath, but as well for the next roller and any further roller, it is important to carry out these and the further processing steps with all due care in order to avoid that the filaments are subjected to forces detrimental to the filaments while also avoiding in particular again during contact with the first couple of rollers, movement of the filaments and/or filament bundles in order to avoid that same come into contact with each other. During these initial stages after coagulation, the filaments are still susceptible for mechanical impact as well as for generating undesired contact points between individual filaments and/or filament bundles. As this may lead to problems further downstream during the filament processing it is relevant to control these process stages carefully. However, no compromise can be made in relation with production speeds as this is one of the most important factors concerning commercial considerations. Similar considerations however also apply to the later stages of the process, as again the filaments or filament yarns have to be transported around the rollers of the respective process steps with all due care, in particular avoiding slippage, as this may also during later process steps (when the filaments already have been consolidated) lead to detrimental effects, such as filament breakage or

production problems.

[0012] In particular, it is a requirement that the filaments and/or filament bundles are transported via the rollers without slippage, as this in particular may lead to various problems that may require highly undesired stoppages of the overall process.

[0013] As outlined above, these requirements for the roller surface in this respect are not easily fulfilled, as the different requirements due to the fiber nature are conflicting. In addition, these requirements may be different at the different production stages identified above. In order to allow a quick transport without slippage as well as without axial movement of the filaments and/or filament bundles, the adhesion to the surface should be as high as possible. At the same time, at a given point the filaments or filament bundles have to detach themselves from the roller surface (i.e. release of the filaments and/or filament bundles from the roller surface without exerting undesired high mechanical forces on the filaments or filament bundles), which requires that adhesion is as low as possible. Accordingly, it is required to find a solution in order to comply with these conflicting demands.

[0014] In accordance with the present invention, it has been found that these conflicting requirements can be fulfilled if the surface of the roller to be in contact with the lyocell filament or filament bundle is selected so that the roller surface comprises first sections displaying a surface tension so that, depending on the liquid adhering to the filament or yarn, one of the following conditions is fulfilled:

a) surface tension of the first sections is in the range of from 0.25 to 3.5 times, preferably 0.35 to 2.5 times, more preferably 0.4 to 0.8 times the surface tension of the liquid, provided that the surface tension of the liquid is 45 mN/m or more; or

b) surface tension of the first sections is in the range of from 0.5 to 10 times, preferably 0.8 to 2.5, more preferably 1 to 2 times the surface tension of the liquid, provided that the surface tension of the liquid is less than 45 mN/m.

Otherwise the principle design of the roller in accordance with the present invention is, except for the surface structure explained herein, in accordance with usual roller designs (such as diameter etc.) so that these details are not further explained here.

It has been found that by adjusting the surface tension of the first sections of the roller surface as identified above it is possible that the filament and/or yarn is guided around the roller at the respective process step without detrimental effect on the filament or yarn. Taking the washing steps as an example, the principle as outlined above means that (taking water as the washing liquid, having a surface tension of about 72.8 mN/m) the first sections may have a surface tension of from 254.8 mN/m to 18.2 mN/m. In case of a low surface tension liquid,

such as an avivage composition, which may for example have a surface tension of about 29 mN/m, the range of surface tension for the first sections may range from 14,5 mN/m to 290 mN/m. Surface tensions in the range as identified above provide the required adhesion for the filament or filament bundle, which as outlined above prevents slippage as well as axial movement of the filament and/or filament bundle on the roller surface. At the same time, it is, however, required that the roller also enables an easy removal of the filament or filament bundle from the surface of the roller at the required position. This requirement, as indicated above is likewise met with a roller selected in accordance with the principles outlined above.

However, the roller surface may also comprise second sections having a surface tension lower than the surface tension of the first sections. This may in particular aid with the proper detachment of the filament or filament bundles from the roller surface, which further avoids undesired effects with regard to the filament or filament bundle, such as breaking of filaments etc.. Accordingly, the roller in accordance with the present invention may comprise second surface sections, which allow an even better removal or detachment of the filament or filament bundle from the roller surface without detrimental effect on the filament or filament bundle. In accordance with the present invention, this is ensured by providing appropriate detachment sections over the surface of the roller, preferably in regular intervals, wherein these sections are characterized in that these display a surface tension of less than the surface tension of the first sections. Preferably, these second sections display a surface tension, which is at least 5%, more preferable at least 10%, even more preferably at least 25% lower than the surface tension of the first sections. In embodiments, the difference is at least 10 mN/m, preferably at least 25 mN/m. As is readily apparent from the above, the surface tension of the second sections may be in a range in principle covered by the ranges defined for the first sections. However, as long as the surface tension of the second sections is lower than the surface tension of the first sections, both types of sections can be clearly defined and it is possible to distinguish between the two types of surface sections. In case of the second sections having another structure (surface structure, such as roughness or a different three dimensional structure, such as recesses or creases) or being made from a different material this is more readily apparent. However, as long as the required lower surface tension is given, the second sections are able to serve the desired purpose (aiding with the detachment of the filament/yarn from the roller), as during use these differences are sufficient (even if the absolute values would still be within the surface tension range defined for the first sections).

[0015] In accordance with the present invention, various means are available in order to provide such detachment sections. One possibility is the provision of creases or grooves, preferably extending parallel to the axis of

the roller, on the roller surface. By providing such creases or grooves, the (theoretical) surface of the roller changes from the principle high surface tension material employed for the roller (fulfilling the requirements identified above required for good adhesion) by means of simply providing an absent surface (i.e. air). Such an "absent" surface is considered in the context of the present invention as a material having a low surface tension. During filament processing it may also be that these creases or grooves are filled with water, which likewise provides a surface tension within the required range.

[0016] A further option is the reduction of the surface roughness, as also this will lead to surface tension values for the detachment sections in the range as identified above.

[0017] Furthermore, it is also possible to fill creases or grooves provided on the roller surface with materials, preferably synthetic polymers, which provide a surface tension within the range identified above. In a similar manner, i.e. by employing low surface tension materials, preferably synthetic polymers, it is also possible to provide coated sections on the surface of the roller, which provide a surface tension in the range as required. One way of ensuring that such a coating works in the desired manner is the filling of micro-cracks or micro-fissures within the surface of the roller material. However, the present invention likewise envisages to provide such coatings also over macroscopic surface areas of the roller surface.

[0018] In case of the provision of creases or grooves, preferably these are provided with a breadth (i.e. the dimension perpendicular to the axis of the roller) in the range of from 0.5 to 5 mm, preferably 1.5 to 2.5 mm. The distance between two such grooves or creases may be in the range of from 2 to 25 mm, preferably 2.5 to 10 mm, more preferably 3 to 4.5 mm. This dimension may also be employed with respect to the coating of the roller surface in order to create detachment points, for example by using synthetic polymers.

[0019] Since the creation of the detachment sections, i.e. second sections in accordance with the present invention requires additional manufacturing steps, such as surface modification processes and/or coating processes, it is preferred if the majority of the roller surface is made from the first sections. As these sections typically are made from the principle material used for roller surface manufacture, it often is commercially beneficial if these surface sections make up the majority of the roller surface. Typical first section proportions in relation with the overall roller surface are from 40 to 95%, preferably from 50 to 80%, including from 60 to 70%. In embodiments, the roller surface consists of the first sections only.

[0020] The creases and grooves as well as the micro-cracks or micro-fissures to be provided on the roller surface may be prepared in accordance with methods known to the skilled person, by appropriate surface treatment methods, such as etching processes. Likewise, the skilled person is in a position to coat the roller surface,

for example, with synthetic polymers in order to create coatings providing the detachments sections. Likewise, the provision of sections with reduced surface roughness is likewise well within the usual practice of the skilled person.

[0021] Suitable materials for the high surface tension section of the roller surface are in particular metals, in particular iron based materials, stainless steel, chromium, aluminum etc.. However, it has been found that as long as the surface tension requirement is fulfilled, that also other materials may be employed, such as ceramics. The materials suitable for providing the detachment sections typically, in case that not simply creases or grooves are provided, are synthetic polymers, such as PVC, PA, polyolefins, PTFE, PET, PMMA, etc. In order to facilitate production of a roller surface as provided by the present invention, the use of thermoplastic polymers is preferred.

[0022] Surprisingly, it has been found that by following the above-outlined guidance, namely by ensuring that the surface tension of the principal material employed for the roller surface is within the range identified above, while providing optional detachment sections fulfilling the surface tension requirements as again outlined above, it is easily possible to secure a proper transport of filaments or filament bundles around rollers during lyocell filament production, without giving rise to the undesired effects, such as slippage, contact between filaments or filament bundles and filament breakage.

[0023] Accordingly, the present invention proposes to use a roller as defined and described herein during lyocell filament production, in particular as the guidance roller taking up the filaments or filament bundles after coagulation, but also for any subsequent roller employed for example during liquid removal, washing or drying operations.

[0024] Using chromium plated steel rollers lyocell filaments were produced. The surface tension of the roller was determined according to the methodology described below. Using rollers having surfaces with surface tensions of about 37 mN/m (surface tension of roller about 0.5 times the surface tension of the washing liquid) high quality lyocell filament yarns could be produced without any problems concerning filament breakage etc. even during high speed filament production. Similar results were obtained using roller surfaces with surface tensions of about 44, 56, 93 and 185 mN/m, i.e. with factors 0.6, 0.75, 1.3 and 2.4.

Surface tension values as referred to herein have been determined according to the methodology of Owens-Wendt-Rabel-Kaelble using a surface energy measuring device from Kruss (DSA10HS) employing the corresponding software (DSA10HSAJ1H "Drop shape Analysis System Serial Number 20012303 (Q161F800 INR.59028). Measurements (60 seconds after droplet deposition) of at least 1 droplet each (30 measurements per droplet) were employed to calculate surface tension values, employing the following three standard liquids:

- HPLC water
- Ethylene glycol for analysis (purity > 99.5%, available for example from Merck: K42538921 133 1.09621.1000)
- Diiodomethane (purity > 99%, stabilized, available for example from Acros organics: 169830250 A0390451)

Claims

1. Roller, suitable to be used during lyocell filament production, wherein the roller surface comprises first sections displaying a surface tension so that, depending on the liquid adhering to the filament or yarn, one of the following conditions is fulfilled:

- a) surface tension of the first sections is in the range of from 0.25 to 3.5 times, preferably 0.35 to 2.5, more preferably 0.4 to 0.8 times the surface tension of the liquid, provided that the surface tension of the liquid is 45 mN/m or more; or
- b) surface tension of the first sections is in the range of from 0.5 to 10 times, preferably 0.8 to 2.5, more preferably 1 to 2 times the surface tension of the liquid, provided that the surface tension of the liquid is less than 45 mN/m.

2. Method of selecting a roller for a process step during lyocell filament production, **characterized in that** the roller is selected so that the roller surface comprises first sections displaying a surface tension so that, depending on the liquid adhering to the filament or yarn, one of the following conditions is fulfilled:

- a) surface tension of the first sections is in the range of from 0.25 to 3.5 times, preferably 0.35 to 2.5, more preferably 0.4 to 0.8 times the surface tension of the liquid, provided that the surface tension of the liquid is 45 mN/M or more; or
- b) surface tension of the first sections is in the range of from 0.5 to 10 times, preferably 0.8 to 2.5, more preferably 1 to 2 times the surface tension of the liquid, provided that the surface tension of the liquid is less than 45 mN/m.

3. Roller according to claim 1 or method according to claim 2, wherein the surface tension of the first sections is so that the following condition is fulfilled: surface tension of the first sections of condition a) is in the range of from 1 to 1.75 times; the surface tension of the first sections of condition b) is in the range of from 0.9 to 5 times the surface tension of the liquid.
4. Roller according to claim 1 or 3 or method according to claim 2 or 3, wherein the roller surface comprises

second sections having a surface tension lower than the first sections.

5. Roller or method according to any of the preceding claims, wherein the first sections are made from metal, preferably chromium plated steel. 5
6. Roller or method according to any of claims 4 or 5, wherein the second sections are made from synthetic polymers. 10
7. Roller or method according to any of claims 4 or 5, wherein the second sections are creases or grooves.
8. Roller or method according to any of the preceding claims, wherein the roller surface comprises only first sections or wherein the first and second sections substantially extend parallel along the roller axis. 15
9. Roller or method according to any of claims 4 to 8, wherein the second sections show a breadth of from 0.5 to 5 mm, preferably of from 1.5 to 2.5 mm. 20
10. Roller or method according to any of claims 4 to 9, wherein the second sections are spaced apart by 2 to 25 mm, preferably 3 to 4.5 mm. 25
11. Roller or method according to any of the preceding claims, wherein the first sections amount to from 40 to 95% of the surface area of the roller surface. 30
12. Use of a roller according to any of the preceding claims, wherein the roller is employed during lyocell filament production. 35
13. The use according to claim 12, wherein the roller is used as guidance roller taking up the lyocell filaments or filament bundles after these exit the coagulation bath. 40
14. The use according to claim 12, wherein the roller is employed during liquid removal and/or washing steps.
15. The use according to claim 12, wherein the roller is used during application of additional substances to the filament 45

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EUROPEAN SEARCH REPORT

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EP 19 20 4806

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A	OLGA SANTOS ET AL: "Modified stainless steel surfaces targeted to reduce fouling--surface characterization", JOURNAL OF FOOD ENGINEERING, vol. 64, no. 1, 1 September 2004 (2004-09-01), pages 63-79, XP055678925, GB ISSN: 0260-8774, DOI: 10.1016/j.jfoodeng.2003.09.013 * Parts 2.1 and 2.7; tables 11,12 *	1-15	
			TECHNICAL FIELDS SEARCHED (IPC)
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The present search report has been drawn up for all claims			
Place of search The Hague		Date of completion of the search 23 March 2020	Examiner Malik, Jan
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**ANNEX TO THE EUROPEAN SEARCH REPORT
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This annex lists the patent family members relating to the patent documents cited in the above-mentioned European search report.
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