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R. W. MONCRIEFF ET AL

2,142,909

TREATMENT OF ARTIFICIAL MATERIALS

Original Filed April 19, 1935 2 Sheets-Sheet 1

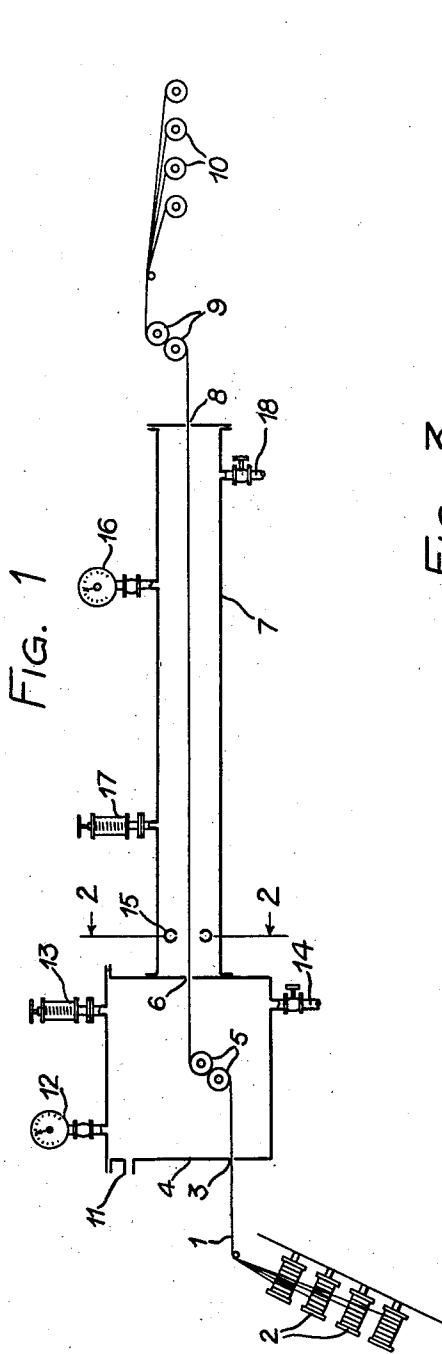


FIG. 3

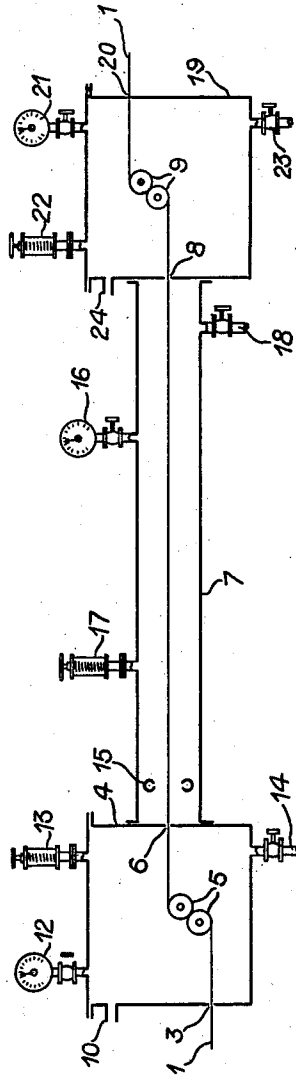
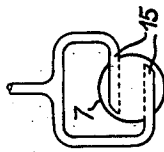


FIG. 2



ROBERT W. MONCRIEFF  
FRANK B. HILL

INVENTORS

*J. Selby & Co. Lewis & Co.*  
ATTORNEYS

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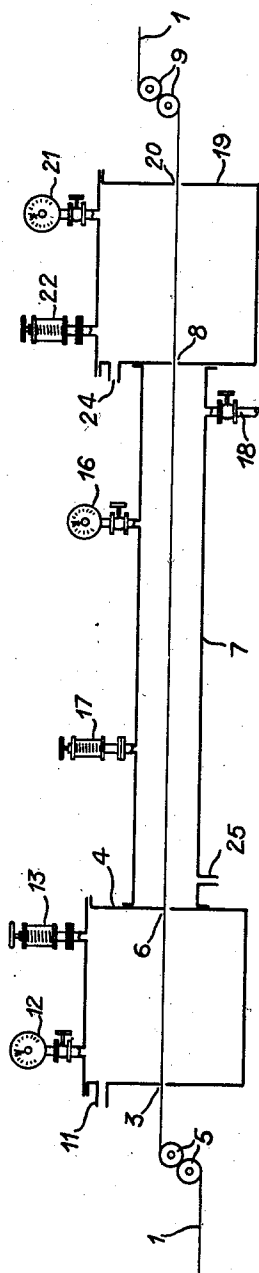
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FIG. 4



ROBERT W. MONCRIEFF  
FRANK B. HILL  
INVENTORS

*P. H. Hill & W. B. Hill*  
ATTORNEYS

# UNITED STATES PATENT OFFICE

2,142,909

## TREATMENT OF ARTIFICIAL MATERIALS

Robert Wighton Moncrieff and Frank Brentnall  
Hill, Spondon, near Derby, England, assignors  
to Celanese Corporation of America, a corpo-  
ration of Delaware

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newed October 29, 1938. In Great Britain May  
30, 1934

13 Claims. (Cl. 8—132)

This invention relates to improved methods and apparatus for the treatment of filaments, threads, foils and similar materials with fluid media under pressure.

5 In U. S. application S. No. 4,510 filed February 1, 1935, processes are described wherein filaments, threads, foils and similar materials having a basis of cellulose acetate or other cellulose derivative are passed through a chamber containing steam under pressure and are stretched while in the chamber. In this manner, materials having increased tensile strength may be obtained in a very simple manner.

10 As indicated in the said specification, the orifices for the passage of the materials into and out of the pressure stretching chamber are preferably as small as possible so as to minimize escape of steam. Across these orifices, there is a pressure drop and the steam escaping exerts a frictional effect on the materials passing through the orifices, thereby setting up in the materials a tension additional to the tension exerted by any mechanical stretching devices.

15 As indicated in the said specification, it is preferable that the condition of the steam be such as to enable the stretching to be carried out with as low a stretching tension as possible. Not only does the stretching tension affect to some extent the properties of the stretched product such as tensile strength and extension, but, in addition, with too high a stretching tension, breakage of the filaments, threads or the like is apt to occur. We have found that for the best results, especially when high degrees of stretch are required, for example degrees of stretch exceeding 500% of the original length of the thread, the tension exerted by the escaping steam on the threads entering the steam chamber is too high, and the present invention has for its object to reduce this tension on the incoming threads, apart from other objects to be referred to hereinafter.

20 We have found that by providing a compressed air chamber in front of the steam stretching chamber and by locating nip rollers or other feed device in the compressed air chamber, the tension exerted on the threads in the steam chamber by the steam escaping from the thread inlets is reduced. The flow of steam across the inlet orifices is dependent on the drop of pressure across these orifices and by maintaining the air in the compressed air chamber at substantially the same pressure as the steam in the stretching chamber, flow is practically avoided, and tension upon the threads during the stretching treat-

ment, due to escape of steam through the inlet orifices, is substantially eliminated.

Thus, in the new process, cellulose acetate threads may be passed through suitable orifices in one side of a compressed air chamber into substantially non-slipping contact with nip rollers or other feed device located in the compressed air chamber, thence through further orifices into the stretching chamber containing saturated or wet steam under pressure, out of orifices at the far end of the steam chamber and thence into substantially non-slipping contact with nip rollers or other stretching device located beyond the stretching chamber. By maintaining the air in the compressed air chamber at the same pressure as the steam in the stretching chamber, tension on the threads due to the escape of steam from the thread inlets to the stretching chamber is entirely eliminated. It is desirable, however, to avoid air entering the stretching chamber from the compressed air chamber, and accordingly it is preferable to maintain the air in the compressed air chamber at a slightly lower pressure than the steam in the stretching chamber so that a small escape of steam into the compressed air chamber takes place but insufficient to give rise to any substantial tension in the threads.

25 The tension on the threads due to escape of steam from the thread outlet orifices from the stretching chamber may be much smaller than the tension exerted on the threads entering the steam chamber by the steam escaping from the thread inlets in the absence of the compressed air chamber, since at the outlet end of the steam chamber the threads are travelling in the same direction as the steam and may, since they have been stretched, be travelling at quite a high speed, whereas at the thread inlet the threads are travelling in a direction opposite to that of the escaping steam. However, if desired, the tension exerted by the escaping steam on the threads passing through the outlets from the stretching chamber may be reduced or eliminated by the provision of a second compressed air chamber containing a feed device, which may in this case be the stretching device, at the outlet end of the stretching chamber.

30 The chamber located at one or both ends of the travel of the threads through the steam chamber may contain, instead of compressed air, any other compressed gas or fluid inert to the materials under treatment.

35 The invention is applicable to any treatment of filaments, threads or the like or of foils or other web materials with fluid media under pres-

sure where it is desired to run the materials continuously through a pressure treatment chamber and to reduce or eliminate tension upon the materials across the inlets or outlets.

5 For convenience the chambers located at one or both ends of the travel of the threads or other materials through the treatment chamber will be referred to as "end pressure chambers", though this term is not to be understood as connoting  
10 that the treatment chamber is necessarily extended in the general direction of travel of the threads or other materials therethrough.

The new apparatus comprises a pressure treatment chamber with an inlet for the treatment fluid in combination with an end pressure chamber with an inlet for inert fluid, and containing a feed device for the materials, and communicating with the treatment chamber, the apparatus having inlet and outlet orifices opening to the  
15 outer air to permit passage of the materials through the chambers in succession but being substantially closed to permit the development of fluid pressure therein.

As previously indicated, the apparatus may  
20 comprise an end pressure chamber containing a feed device for the materials disposed at each end of the travel of the materials through the treatment chamber.

The treatment chamber may be provided with  
25 a pressure gauge, a pressure relief valve and, where steam is the fluid treatment medium, a steam trap or other device for withdrawing condensate from the steam chamber. In addition, the end pressure chamber may be provided with  
30 a pressure gauge and pressure relief valve and, if there is escape of steam into the chamber, with a steam trap or similar device.

In addition to reducing the tension exerted on the threads at the inlet and outlet orifices of the  
35 treatment chamber, the provision of the pressure chamber at both ends of the treatment chamber serves to prevent escape of the fluid treatment medium. This is of considerable advantage where the fluid medium is a gas or liquid, the  
40 escape of which is objectionable, or where it is desirable for economic reasons to prevent loss of the medium.

Other applications of the apparatus of the present invention include saponification of cellulose ester threads, foils or similar materials under  
45 pressure, for example by means of caustic soda, ammonia, methylamine or ethylene diamine, as described in U. S. application S. No. 11,080 filed March 14, 1935, or stretching operations carried out by means of hot water under pressure as  
50 described in U. S. application S. No. 4,511 filed February 1, 1935, or by means of organic softening agents under pressure as described in U. S. application S. No. 11,079 filed March 14, 1935, now Patent No. 2,072,251, dated March 2, 1937.

Where the object is purely to prevent loss of the fluid treatment medium, and the tension on the thread or other material is immaterial, it is unnecessary to provide feed devices in the end  
55 pressure chambers. Thus, in a pressure saponification treatment where escape of saponifying medium into the outer atmosphere is not desirable and at the same time the tension exerted by the fluid medium at the inlets and outlets for the  
60 threads or other materials is of no moment, the apparatus may consist of a pressure treatment chamber, and two end pressure chambers communicating respectively with the inlet and outlet orifices of the treatment chamber and without  
65 feed devices therein.

On the other hand where it is desired to prevent escape of the fluid treatment medium and the tension exerted by the flow of treatment medium across the orifices for the passage of  
5 the threads or other materials into and out of the treatment chamber is only of moment at the inlet, the apparatus may comprise a pressure treatment chamber provided at one end with an end pressure chamber containing a feed device for the materials and at the other end with a  
10 second end pressure chamber without a feed device therein.

The accompanying drawings illustrate diagrammatically some forms of apparatus in accordance with the present invention,  
15

Fig. 1 being a longitudinal cross-section of an apparatus suitable for stretching cellulose acetate threads in steam,

Fig. 2 a cross-section (to a larger scale) on the line 2—2 of Fig. 1,  
20

Fig. 3 a longitudinal cross-section of another form of apparatus suitable for stretching cellulose acetate threads in steam, and

Fig. 4 a longitudinal cross-section of an apparatus suitable for the treatment of threads of cellulose acetate under pressure with a fluid such as an organic softening agent for stretching or an organic amine solution for saponification wherein loss of the active agent is to be avoided.  
25

Referring to Figs. 1 and 2, the threads 1 from a creel of bobbins 2 pass through small orifices 3 into a compressed air chamber 4 in non-slipping contact with nip rollers 5 in the compressed air chamber through small orifices 6 into a steam chamber 7, out of the steam chamber through  
30 orifices 8, in non-slipping contact with nip rollers 9 for stretching, and are finally wound on a creel of bobbins 10. The compressed air is introduced through the inlet 11 into the chamber 4 which is provided with a pressure gauge 12, a pressure relief valve 13, and a steam trap 14. Steam enters through perforated pipes 15 so positioned that wet or saturated steam is directed on to the threads immediately on their entry into the steam chamber 7. The chamber 7 is also provided with a pressure gauge 16, a pressure relief valve 17, and a drain 18 for condensate.  
35

In Fig. 3, the stretching rollers 9 are located in a second compressed air chamber 19 out of which the threads pass by orifices 20 to the take-up device. The compressed air chamber 19 is provided with a pressure gauge 21, a pressure relief valve 22, and a drain 23 for condensate, and is filled with compressed air through the inlet 24.  
40

For the general conditions of the steam stretching operation, reference is made to U. S. application S. No. 4,510. By means of the present invention very much higher degrees of stretch are obtainable than without the use of the end pressure chamber. By treating cellulose acetate threads with wet or saturated steam at the appropriate temperature and pressure in accordance with the present invention, stretches of over 2,000% of the original length of the threads are obtainable and, moreover, the qualities of the thread such as tensile strength and extensibility appear to be beneficially affected by the use of the end pressure chamber.  
45

The apparatus illustrated in Figs. 1 and 3 may also be used for stretching cellulose acetate threads in hot water by providing instead of the steam inlets 15, an inlet and outlet for the water and a heater in the chamber 7, as illustrated in the drawings accompanying U. S. application S. No. 4,511. For the conditions for hot water  
50

stretching, reference is made to this specification.

Referring to Fig. 4, which is an apparatus suitable for example for stretching in an organic solvent or for saponification of threads of cellulose acetate, the threads 1 pass through the orifices 3 into a compressed air chamber 4 after passing in non-slipping contact with the nip rollers 5. From the compressed air chamber 4, they pass through orifices 6 into the pressure treatment chamber 7, and thence through orifices 8 into a second compressed air chamber 19 and through orifices 20 into substantially non-slipping contact with nip rollers 9. The compressed air chamber 19 is provided with a pressure gauge 21, a pressure relief valve 22 and an inlet 24 for compressed air. The treatment fluid enters the pressure treatment chamber 7 by the inlet 25.

In a modification of the invention the end pressure chamber, instead of being provided with nip rollers or other feed device for the materials, may contain a bobbin, cheese, roll or other package adapted to supply the material to the pressure treatment chamber. The terms "device for delivering the materials" and "delivery device" in the appended claims are used generically to include such packages as well as feed devices, and the term "feed device for the materials" excludes the packages and includes only nip rollers or similar devices for forwarding the running materials. The use of packages in the end pressure chambers permits of batches of material being subjected to treatment with fluid media under pressure. It will be apparent however that the invention is most advantageously applied when feed devices are located in the end pressure chambers so that the materials may be passed from the outer air through the pressure treatment chamber and finally again into the outer air.

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

1. Process for improving the strength of artificial threads, foils and similar materials, which comprises running the materials from the outer air into an end pressure chamber, which contains compressed air or other inert fluid under pressure and which communicates with a pressure treatment chamber, in substantially non-slipping contact with a positively driven feed device in the end pressure chamber, thence through the communication between the end pressure and treatment chambers into a stretch-assisting fluid, which is contained in the treatment chamber and is under a pressure substantially the same as that of the inert fluid, and finally into the outer air, and stretching the materials while in the stretch-assisting fluid.

2. Process for improving the strength of filaments, threads, foils and similar materials of organic derivatives of cellulose, which comprises running the materials from the outer air into an end pressure chamber, which contains compressed air or other inert fluid under pressure and which communicates with a pressure treatment chamber, in substantially non-slipping contact with a positively driven feed device in the end pressure chamber, thence through the communication between the end pressure and treatment chambers into hot water, which is contained in the treatment chamber and is at a temperature above 100° C. and under a pressure higher, but only slightly higher, than that of the inert fluid, and finally into the outer air, the materials being stretched while in the hot water.

3. Process for improving the strength of filaments, threads and similar materials of cellulose acetate, which comprises running the materials from the outer air into an end pressure chamber, which contains compressed air or other inert fluid under pressure and which communicates with a pressure treatment chamber, in substantially non-slipping contact with a positively driven feed device in the end pressure chamber, thence through the communication between the end pressure and treatment chambers into hot water, which is contained in the treatment chamber and is at a temperature above 120° C. and under a pressure higher, but only slightly higher, than that of the inert fluid, and finally into the outer air, the materials being stretched while in the hot water.

4. Process for improving the strength of filaments, threads, foils and similar materials of organic derivatives of cellulose, which comprises running the materials from the outer air into an end pressure chamber, which contains compressed air or other inert gas under pressure and which communicates with a pressure treatment chamber, in substantially non-slipping contact with a positively driven feed device in the end pressure chamber, thence through the communication between the end pressure and treatment chambers into saturated or wet steam, under a pressure at least substantially the same as that of the inert gas and at a temperature above 100° C., contained in the treatment chamber and finally into the outer air, and stretching the material while in the steam.

5. Process for the saponification of threads, foils and similar materials of organic esters of cellulose, which comprises running the materials from the outer air into an end pressure chamber, which contains compressed air or other inert fluid under pressure and which communicates with a pressure treatment chamber, in substantially non-slipping contact with a positively driven feed device in the end pressure chamber, thence through the communication between the end pressure and treatment chambers into a saponifying fluid which is under a pressure substantially the same as that of the inert fluid and is contained in the treatment chamber, wherein the materials are saponified, and finally into the outer air.

6. Process for improving the strength of filaments, threads and similar materials of cellulose acetate, which comprises running the materials from the outer air into an end pressure chamber, which contains compressed air or other inert gas under pressure and which communicates with a pressure treatment chamber, in substantially non-slipping contact with a positively driven feed device in the end pressure chamber, thence through the communication between the end pressure and treatment chambers into a steam stretching chamber wherein saturated or wet steam at a temperature above 100° C. and under a pressure higher, but only slightly higher, than that of the inert gas is directed on to the materials immediately after their entry into the chamber, and finally into the outer air, and stretching the materials while in the steam.

7. Process for improving the strength of filaments, threads and similar materials of cellulose acetate, which comprises running the materials from the outer air into an end pressure chamber which contains compressed air or other inert gas under pressure and which communicates with a pressure treatment chamber, in substantially

non-slipping contact with a positively driven feed device in the end pressure chamber, thence through the communication between the end pressure and treatment chambers into saturated or wet steam, at a temperature above 120° C. and under a pressure higher, but only slightly higher, than that of the inert gas, contained in the pressure treatment chamber, and finally into the outer air, and stretching the materials while in the steam.

8. Process for improving the strength of filaments, threads and similar materials of cellulose acetate, which comprises running the materials from the outer air into an end pressure chamber, which contains compressed air or other inert gas and which communicates with a stretching chamber, in substantially non-slipping contact with a positively driven feed device in the end chamber, thence through the communication between the end and stretching chambers into the stretching chamber, which contains saturated or wet steam under a pressure higher, but only slightly higher, than that of the inert gas and at a temperature above 100° C., and from the stretching chamber directly into the outer air, and stretching the materials while in the steam.

9. Process for improving the strength of filaments, threads and similar materials of cellulose acetate, which comprises running the materials from the outer air into an end pressure chamber, which contains compressed air or other inert gas under pressure and which communicates with a pressure treatment chamber, in substantially non-slipping contact with a positively driven feed device in the end pressure chamber, thence through the communication between the end pressure and treatment chambers into a steam stretching chamber wherein saturated or wet steam at a temperature above 120° C. and under a pressure higher, but only slightly higher, than that of the inert gas is directed on to the materials immediately after their entry into the chamber, and finally into the outer air, and stretching the materials to at least 500% of their original length while in the steam.

10. Process for improving the strength of filaments, threads, foils and similar materials of organic derivatives of cellulose, which comprises running the materials from the outer air into an end pressure chamber, which contains compressed air or other inert gas under pressure and which communicates with a pressure treatment chamber, in substantially non-slipping contact with a positively driven feed device in the end pressure chamber, thence through the communication between the end pressure and treatment chambers into saturated or wet steam, under a pressure at least substantially the same as that of the inert gas and at a temperature above 100° C., contained in the treatment chamber, from said treatment chamber into an end pressure chamber which contains an inert gas under pressure and which communicates with the treatment chamber, through the communication between the said chambers, and finally into the outer air,

the materials being stretched while in the steam.

11. Process for improving the strength of filaments, threads, foils and similar materials of organic derivatives of cellulose, which comprises running the materials from the outer air into an end pressure chamber, which contains compressed air or other inert gas under pressure and which communicates with a pressure treatment chamber, thence through the communication between the end pressure and treatment chambers into saturated or wet steam, under pressure and at a temperature above 100° C. contained in the treatment chamber, from said treatment chamber into an end pressure chamber, which contains an inert gas under pressure and which communicates with the treatment chamber, through the communication between the said chambers, and finally into the outer air, the inert gas in the end pressure chambers being under a pressure at the most substantially the same as that of the steam, the materials being stretched by passing them in substantially non-slipping contact with positively driven feed devices contained in the end pressure chambers.

12. Process for improving the strength of artificial threads, foils and similar materials, which comprises running the materials from the outer air into an end pressure chamber, which contains compressed air or other inert fluid under pressure and which communicates with a pressure treatment chamber, in substantially non-slipping contact with a positively driven feed device in the end pressure chamber, thence through the communication between the end pressure and treatment chambers into a stretch-assisting fluid under pressure contained in the treatment chamber and finally into the outer air, and stretching the materials while in the stretch-assisting fluid, the pressure of the inert fluid being lower than, but only slightly lower than, that of the stretch-assisting fluid so that substantially no tension on the materials in the treatment chamber is exerted by the flow of fluid from chamber to chamber.

13. Process for improving the strength of filaments, threads and similar materials of cellulose acetate, which comprises running the materials from the outer air into an end pressure chamber, which contains compressed air or other inert gas under pressure and which communicates with a pressure treatment chamber, in substantially non-slipping contact with a positively driven feed device in the end pressure chamber, thence through the communication between the end pressure and treatment chambers into saturated or wet steam, at a temperature above 100° C. and under a pressure higher, but only slightly higher, than that of the inert gas so that substantially no tension on the materials in the treatment chamber is exerted by the flow of fluid from chamber to chamber, and finally into the outer air, and stretching the materials while in the steam.

ROBERT WIGHTON MONCRIEFF.  
FRANK BRENTNALL HILL.