

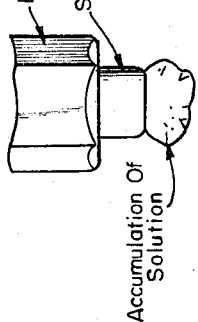
May 21, 1968

R. J. LOCKHART

3,384,690

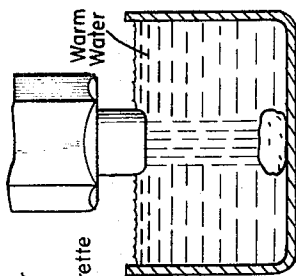
METHOD OF SPINNERETTE CLEANING DURING THE PREPARATION
OF INORGANIC OXIDE MONOFILAMENTS
Filed Jan. 21, 1965

FIG. 1.



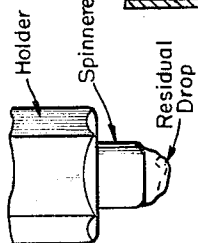
Before Treatment
To Remove
Accumulation

FIG. 2.



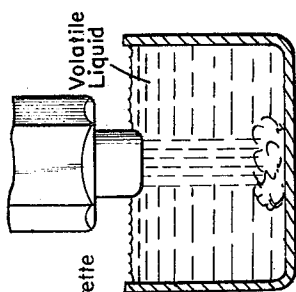
After Spinnerette Face Has
Been Immersed In Warm Water.
Accumulation Has Dropped
Off And Spinning Solution Is
Now Being Extruded Through
Orifices

FIG. 3.



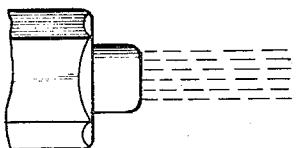
Immediately After
Warm Water Has
Been Removed

FIG. 4.



After Spinnerette Face
Has Been Immersed In
Volatile Liquid

FIG. 5.



Spinnerette After
Residual Volatile
Liquid Has Evaporated.
Filaments Being
Extruded Uniformly
Into Air.

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3,384,690

METHOD OF SPINNERETTE CLEANING DURING THE PREPARATION OF INORGANIC OXIDE MONOFILAMENTS

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Filed Jan. 21, 1965, Ser. No. 426,875
5 Claims. (Cl. 264—39)

ABSTRACT OF THE DISCLOSURE

A procedure for removing the accumulations which form in the vicinity of the extrusion orifices of a spinnerette through which inorganic oxide filaments are extruded from a viscous concentrate, in which the face portion of the spinnerette is cleared of any accumulations by immersing the face portion of said spinnerette in a succession of liquids while extrusion takes place to assist in physically detaching the accumulations from the spinnerette face.

This invention relates to the spinning of monofilaments of inorganic polymers of the type described in French Patent 1,316,535 which corresponds to United States Patent 3,180,741 issued Apr. 27, 1965, and in United States patent application 252,083 filed Jan. 17, 1963 which issued on Mar. 28, 1967, as United States Patent 3,311,689 and the disclosures of which are incorporated herein by reference.

Each of the patents describes the preparation of a liquid concentrate from which monofilaments of inorganic oxides may be dry spun, into air or other gaseous atmospheres, the preparation of said concentrate forming no part of the present invention which is directed to a procedure for treating the spinnerette orifices in order to improve the continuity of the extrusion process.

When producing filaments of inorganic oxides by the dry spinning processes described in the above noted patents, it has been found difficult to produce continuous filaments for any extended intervals of time, due principally to the accumulation of spinning solution on parts of the face of the spinnerette until the accumulated material falls under the influence of gravity. When these accumulations break loose they often whip around and snap the filaments forming from adjacent unobstructed spinnerette holes. In addition, accumulations attached to the spinnerette hang down and catch or tangle emerging filaments. A similar phenomenon is experienced in melt spinning of synthetic polymer filaments as described, for example in United States Patent 3,129,272 which also describes procedures for minimizing the formation of such accumulations in dry spinning, such as blanketing the face of the spinnerettes uniformly and completely with an "inert" gas. Such a procedure adds considerably to the expense and requires extremely careful control when applied to dry spinning of inorganic oxide filaments.

Various methods of overcoming the formation of such accumulations which have been attempted in dry spinning of inorganic filaments include application of surface coatings to the spinnerettes to retard the spreading of liquids on the exit side of the spinnerettes and mechanical polishing of the spinnerette face to remove any scratches and to eliminate any surface discontinuities which might favor the utilization and propagation of such accumulations.

When extruding inorganic oxide monofilaments, coating the spinnerette with silicone oils, silanes, waxes, Teflon, or other lubricants did not eliminate the formation of the undesired accumulations, which appeared on the spinnerettes just as if no coatings had been applied.

Examination of new "unused" spinnerettes revealed an overall pattern of parallel scratches apparently from the finishing operations incidental to the manufacture of the spinnerettes. Examination of the same spinnerettes after use revealed that they had become much more scratched, owing to the softness of the platinum rhodium alloy of which the spinnerettes are fabricated. Polishing the spinnerettes to a mirror finish noticeably improved the process, but the effect was only temporary, since the scratches formed again during normal handling and cleaning operations.

The principal objects of this invention are to provide a simple and effective way to delay or prevent the formation of accumulations on the spinnerettes during the dry spinning of inorganic oxide monofilaments and to provide a simple and effective way to remove such accumulations, if and when formed, without serious interruption of the spinning process.

Briefly, in accordance with the present invention, these objects were achieved by placing a container of a suitable liquid under the spinnerette orifices, and then moving the container or spinnerettes relative to one another so as to immerse the entire outer face of the spinnerette cup in the liquid, and keeping the two in contact for a time sufficient to clear the faces of the spinnerettes of any accumulated fresh or partially dried spinning solution.

One liquid found to be suitable was warm water, but it was also found that after the water had cleared the spinnerette holes and face, it would remain on the face of the spinnerette after the container was moved away, and would be in the form of a large drop, which remained long enough to permit the emerging inorganic oxide dope to recoat the surface. The disadvantage was overcome by removing the container of water and replacing it as promptly as possible with a container of a volatile liquid in which the spinning solution is insoluble. Acetone is one such liquid and when the container of acetone was removed, the acetone evaporated and the face of the spinnerette remained clear permitting the continuous spinning of inorganic filaments for hours as compared with fractions of an hour prior to this invention.

The procedure just described is shown schematically in the drawings accompanying this application in which FIGURE 1 shows a spinnerette bearing an accumulation of spinning solution sufficient to impair satisfactory operation of the process;

FIGURE 2 shows the spinnerette of FIGURE 1 shortly after it was immersed in warm water, and just after the accumulation has dropped off of the spinnerette permitting the resumption of extrusion through the spinnerette orifices;

FIGURE 3 shows the spinnerette immediately after the body of warm water has been removed from the spinnerette leaving one residual drop of water suspended from the spinnerette face;

FIGURE 4 shows the spinnerette after it has been immersed in a volatile liquid; and

FIGURE 5 shows the spinnerette after the volatile liquid has evaporated and shows filaments of inorganic oxide as they are extruded into air.

Other liquids which may be used in place of warm water (125° F.) to clear the spinnerettes of accumulated spinning solution included acetic acid, either as glacial acetic acid or as aqueous solutions of the acid and it appears that the accumulation-clearing liquid could be any liquid in which the spinning solution is at least partly soluble and which did not react chemically or corrosively upon the spinnerette.

Other volatile liquids which were used in place of acetone were chloroform, carbon tetrachloride, ethyl ether and cyclohexane each of which evaporates quite rapidly

from the face of the spinnerette once the bath is removed from the spinnerette.

It has been found that the best results are obtained by using warm water for the spinnerette clearing liquid and a water miscible liquid such as acetone for the water removing, spinnerette drying liquid.

Having now described preferred embodiments of the invention it is not intended that it be limited except as may be required by the appended claims.

I claim:

1. In a process in which a liquid concentrate suitable for the production of an inorganic oxide monofilament is extruded into air through a spinnerette plate having a plurality of extrusion orifices therein, the improvement which comprises: clearing away any accumulation from the face of said spinnerette plate by immersing the face portion of said spinnerette in a first liquid selected from the group consisting of warm water, aqueous and glacial acetic acid in which said accumulations are at least partly soluble; withdrawing said spinnerette from said first liquid while extruding said inorganic oxide monofilament producing concentrate through said spinnerette face;

promptly after withdrawing said spinnerette from said first liquid, immersing said face portion of said spinnerette in a volatile second liquid in which said first liquid is soluble to remove any residue of said first liquid from said spinnerette face; and thereafter withdrawing said spinnerette from said volatile second liquid while extruding said concentrate through said spinnerette face and continuing said extrusion while any residual volatile second liquid evaporates into the surrounding atmosphere.

2. The process of claim 1 in which the first liquid is warm water and the second liquid is acetone.

3. The process of claim 1 in which the second liquid is a volatile organic liquid.

4. The process of claim 1 wherein the inorganic oxide is alumina.

5. The process of claim 1 wherein the inorganic oxide is zirconia.

No references cited.

JAMES A. SEIDLECK, *Primary Examiner*.

H. MINTZ, *Examiner*.

UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 3,384,690

May 21, 1968

Robert J. Lockhart

It is certified that error appears in the above identified patent and that said Letters Patent are hereby corrected as shown below:

Column 1, line 42, "spining" should read -- spinning --.
Column 4, line 19, cancel "No references cited." and insert

References Cited

UNITED STATES PATENTS

1,901,658	3/1933	Ladrette	-----264-39
3,180,741	4/1965	Wainer et al.	-----106-39
3,311,689	3/1967	Kelsey	-----264-210

Signed and sealed this 7th day of October 1969.

(SEAL)

Attest:

Edward M. Fletcher, Jr.

Attesting Officer

WILLIAM E. SCHUYLER, JR.

Commissioner of Patents