The present invention relates to the manufacture of rayon and more particularly to spinnerets used in the production of viscose rayon which are treated to inhibit or minimize the contamination of the orifices of the spinnerets during the spinning operation.

A major problem in the manufacture of viscose rayon is encountered as a result of the contamination of the spinnerets through which the spinning solution is extruded into the spin bath. While this problem has been solved by the use of dissolved cation-active materials in the spin bath as described and claimed in United States Patent No. 2,125,031, issued July 26, 1938, attention has been directed toward solving the problem by covering the spinnerets with very thin films to thereby inhibit their clogging during spinning. U.S. Patent No. 2,100,681 is directed to the provision of a film of wax-like materials such as ozocerite and ceresine on the spinnerets. United States Patent No. 2,284,154 discloses a method of treating spinnerets with a cation active substance. Although somewhat satisfactory results have been had in the following of the teachings of these last referred to United States patents, their utility in commercial operations is limited due to the temporary nature of the films. In both cases the film either dissolves or wears off of the face of the spinnerets in a relatively short time and therefore the spinnerets have to be replaced and the entire treatment repeated before they can be reused.

In order to spin viscose through an acid spin bath of the order of 10% sulphuric acid and maintain optimum spinning conditions over long periods of time such as several weeks, it is necessary to reduce the involuntarily spinneret change to a minimum. Involuntary spinneret change, means the replacement of spinnerets necessary as a result of their becoming fouled and the minute openings therein becoming totally or partially clogged with impurities occurring in the system, including precipitated cellulose, sulphur deposits, secondary reaction products and the like.

It is now proposed to utilize a novel type of substance as a film-forming material on the surface of the spinneret to inhibit the incrustation thereof that is long lasting, but is suitable for this use. These substances are of the class and character of those described in the U.S. Patent No. 2,308,222 in the name of Winton I. Patnode and assigned to General Electric Company.

This invention has for its purpose the application of these substances, namely organo-silicon halides, to the surface of the spinnerets and the use of the treated spinnerets in the production of rayon by the viscose process.

In treating the spinnerets with the organo-silicon halides, very much the same technical procedure should be adopted as that disclosed in the Patnode Patent No. 2,308,222. Initially, the spinnerets are thoroughly cleaned by washing in strong sulphuric acid baths, neutralized in a weak alkaline bath and finally washed in water and dried. The method next involves the contacting of the spinnerets with an organo-silicon halide by exposing them to the vapor of, for example, a mixture of methyl silicon chlorosilane consisting principally of (CH₃)₂SiCl and (CH₃)₂SiCl₃ in accordance with the disclosure of the Patnode patent. As there disclosed, these compounds react with the water adsorbed at the surface of the spinneret and a very thin silicone film is deposited on the spinneret which is water-repellent and is insoluble in the acid spin bath. After a few seconds of the vapor contact with the spinnerets, portions thereof will be coated after which it is preferable to introduce vapors of an alkaline reagent, such as ammonia, into the presence of the spinnerets in order to neutralize any acid traces that may be present as a result of the reaction of the organo-silicon halide.

It is not essential that these organo-silicon halides be utilized in the gaseous state as they may also be applied, for example, by rubbing or wiping the surfaces of the spinnerets.

The thus treated spinnerets are subsequently embodied on spinning machines and a viscose solution is extruded through the spinnerets into an acid spin bath, the treatment being for the purpose of causing the spinneret to remain clean and to avoid the partial or total closing of the orifices even over prolonged periods of spinning such as several weeks. Therefore, the spinnerets do not have to be changed except periodically when voluntary changes are made at the time the spinning machines are shut down for cleaning and other repairs.

Although it is not certain, it is believed that it is the water-repellent characteristic of the organo-silicon halides that will prevent or minimize the contamination of the spinneret orifices.

Among the various organo-silicon halides that are intended for treating spinnerets may be mentioned the alkyl silicon halides such as methyl, ethyl, propyl, and butyl chlorides or bromides, the ary1 silicon halides such as phenyl silicon halides, aralkyl silicon halides such as phenyl-
methyl silicon halides, alkaryl silicon halides such as tolyl silicon halides, and similar alkyl, aryl, etc., halosilanes such as chlorosilanes.

What is claimed is:

1. Incident to the manufacture of viscose rayon wherein a viscose solution is extruded through minute spinneret orifices into an acid spinbath to form threads and the like therefrom, a process of treating spinnerets having moisture adsorbed on the surface thereof, which comprises contacting the spinnerets with an organo-silicon halide to effect the reaction of the halide with the moisture and the resultant formation in situ of a very thin, substantially permanent, acid resistant, water repellent organo-silicone film on the surface of the spinnerets whereby contamination of the spinneret orifices during the spinning operation is inhibited.

2. Incident to the manufacture of viscose rayon wherein a viscose solution is extruded through minute spinneret orifices into an acid spinbath to form threads and the like therefrom, a process of treating spinnerets having moisture adsorbed on the surface thereof, which comprises contacting the spinnerets with a methyl silicon halide to effect the reaction of the halide with the moisture and the resultant formation in situ of a very thin, substantially permanent, acid resistant, water repellent methyl silicone film on the surface of the spinnerets whereby contamination of the spinneret orifices during the spinning operation is inhibited.

3. In the manufacture of synthetic threads wherein a liquid is extruded through minute spinneret orifices to form filamentous threads therefrom, a process of treating spinnerets having moisture adsorbed on the surface thereof, which comprises contacting the spinnerets with an organo-silicon halide to effect the reaction of the halide with the moisture and the resultant formation in situ of a very thin, substantially permanent, acid resistant, water repellent organo-silicone film on the surface of the spinnerets whereby fouling of the spinnerets during the spinning operation is inhibited.

REFERENCES CITED

The following references are of record in the file of this patent:

UNITED STATES PATENTS

<table>
<thead>
<tr>
<th>Number</th>
<th>Name</th>
<th>Date</th>
</tr>
</thead>
<tbody>
<tr>
<td>2,100,581</td>
<td>Weeldenburg</td>
<td>Nov. 30, 1937</td>
</tr>
<tr>
<td>2,273,638</td>
<td>Graves et al.</td>
<td>Feb. 17, 1942</td>
</tr>
<tr>
<td>2,306,222</td>
<td>Fatnode</td>
<td>Dec. 22, 1942</td>
</tr>
</tbody>
</table>

OTHER REFERENCES


Chemistry of the Silicones, by E. G. Rochow, pp. 62-82, John Wiley & Sons, Inc., N. Y., 1946. (Copy in Div. 6.)