

Microscopic Examination of Rayon

By G. HEINK, BERLIN, GER.

In consideration of the great importance which rayon has gained it is of utmost interest when analyzing the product to do this not only in a chemical way for the determination of its quality but also to subject the rayon to an optical examination to obtain a true picture of its chemical nature as well as its quality.

The chemical reactions alone often do not give us any indications as to the chemical origin of the product when comparing the different types of rayon; the optical analysis, however, will answer all these questions clearly.

In the following a selection of the more frequent methods of optical analysis shall be discussed which may serve as a contribution to emphasize to the reader the great importance of microscopy as an aid in the examination of rayon.

The simplest microscopical examination is represented by the so-called side view or longitudinal view of a single thread in regard to the width of the thread respectively the single fiber, its structure, impurities, torn ends, etc.

This examination will already disclose to the experienced eye the quality of the rayon in question. The thread is first examined in surrounding air, and in this investigation the most important step is the gauging of the fiber-width that is measuring the thickness of the single fiber. This criterion, however, varies greatly with the different classes of rayon; in case of viscose for instance the fiber thickness is about $30\text{-}35\mu$, and is furthermore dependent upon the titer in each case. The measurement is taken with the aid of an ocular micrometer by taking the middle value of

about 10 measured fibers to obtain a figure as exact as possible.

Furthermore, the swelling value is of great importance for the estimation of the quality of rayon. By moistening the rayon fiber previously measured in air and by measuring the increase in width or the "swelling" the swelling value is obtained in per cent. As an average 30% is taken. The smaller the swelling value of a rayon is the better the quality and vice versa.

As far as impurities are concerned we find mostly air bubbles or blisterlike formations besides solid particles. These latter may be of a most diversified nature. In dyed rayon for instance these particles may have been present in the dyestuff, or as is often the case with viscose they may be traced to an insufficient removal of sulphur. See Figures 1, 2 and 3.

The preparation of the cross-section is by far more difficult than preparing the fiber for a side-view, but it is just the cross-section picture which discloses such important points regarding the quality of the product.

As far as the preparation of the cross-section is concerned we can only touch upon this procedure briefly. Every one experienced in microscopy knows the secrets and intricacies of a perfect cross-section.

Three to four threads are imbedded into paraffine with the aid of a small frame especially designed for the purpose. They are tiny rectangular frames made of aluminum or brass over which the threads to be examined are wound, preferably using lengths of about 10 cms.

The interior of the frame is now filled with melted paraffine. It is not necessary to

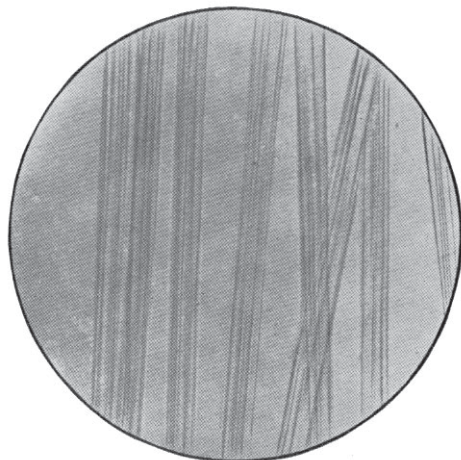


Figure 1

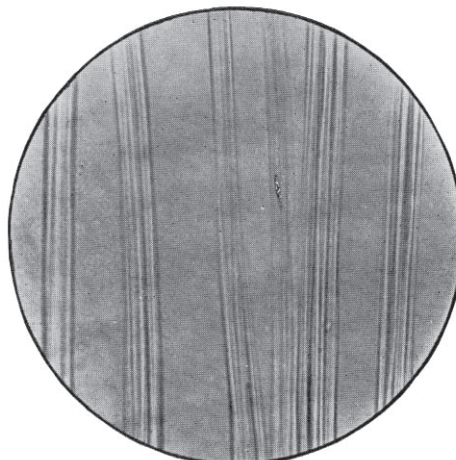


Figure 2

Figure 1 shows a non-desulphurized rayon fiber in the longitudinal or side view; the numerous small specks in the interior are sulphur particles. Figure 2 is the same filament after desulphurization. The small sulphur particles appear in the cross-section (Figure 3) as dot-like enclosures. Figure 4 gives us again the same rayon fiber after desulphurization; the dots in the interior are no longer visible. These impurities can only be seen in the side view, a critical examination and identification, however, is only possible after a cross-section picture has been prepared.

dyed these threads; to the contrary sometimes disadvantageous, as impurities may appear in the cross-section picture which may be due to the dyestuff and may lead to erroneous conclusions. After the hardening of the paraffine the little block is removed from the frame. It should be cut pointed at the upper end in order to avoid getting too much paraffine on the glass slab when the cutting with the microtome is performed. It is also advisable to have the piece well cooled. Only a sufficiently cold piece refrigerated with small pieces of ice will furnish a clean cut on the microtome.

The microtome is a mechanical cutting device which alone permits to cut a great number of fine slices of equal thickness in the easiest and quickest manner. Of course,

It is extremely rare that good or well checking results are obtained, especially in the case of viscose, its forms being dependent entirely on the manufacturing proc-

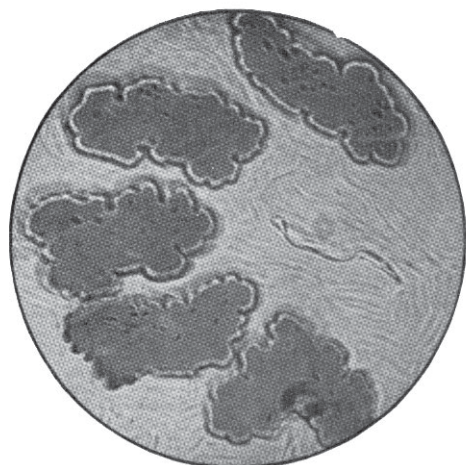


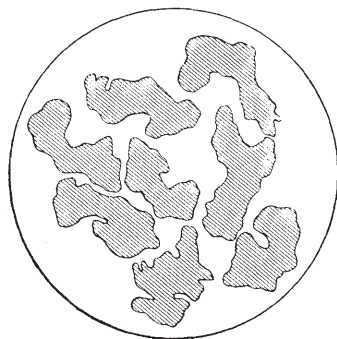
Figure 3



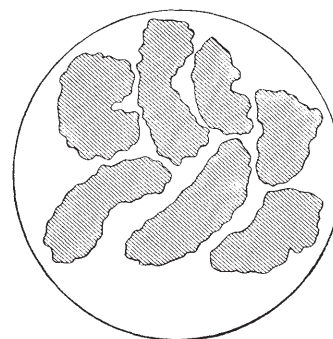
Figure 4

the number of sharp cuts depends on the sharpness of the cutting knife, because only with the sharpest knife is it possible to obtain a series of such exact cuts. The thickness of the cut is 2.5-7 μ . (1 μ = $\frac{1}{1000}$ mm.) The cuts are carefully placed on the object-

ess, and more especially on the composition of the precipitation bath. If the salt contents of the precipitation bath are increased, the original straight-edged form of the cross-section will become irregular, the more or less round form disappears, and the



Glanzfäden A. G.



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carrier, covered with the protection glass slab, and the paraffine is dissolved and washed off with xylol. The prepared fibers are examined at an enlargement of 400 times (see the eight cross-section pictures).

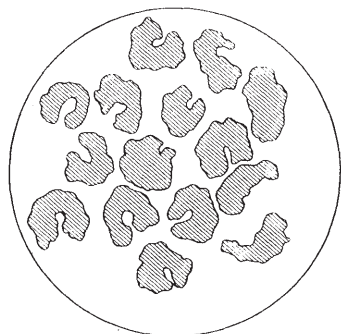
The great variety of pictures to be observed can only be understood by the expert, who has to prepare and examine daily a great number of such cross-sections.

oddest formations become perceivable, as may be seen from the accompanying figures. Also the nature and amounts of the different salt additions contained in the precipitation bath are expressed characteristically in the cross-section picture, and it is possible for an experienced eye to calculate, and find out the composition of the precipitation bath down to the finest details.

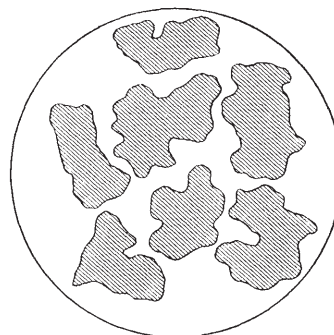
The rayon industry is an industry surrounded with secrets, and yet often a single tiny cross-section picture may reveal a great secret.

Especially the viscose rayon shows great

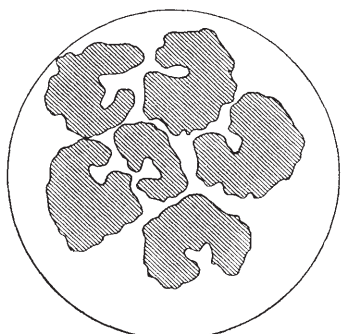
The cross-section picture of a rayon fiber is, therefore, a far more reliable criterion, than the less exact and questionable chemical reactions, which are also helpful in determining the type of the product.



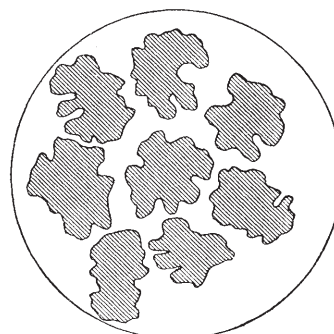
Glanzstoff



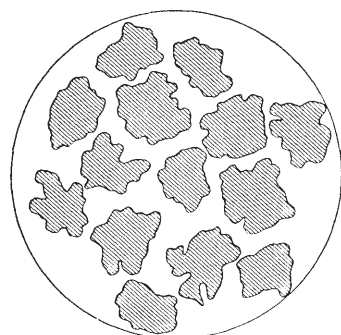
Küttner



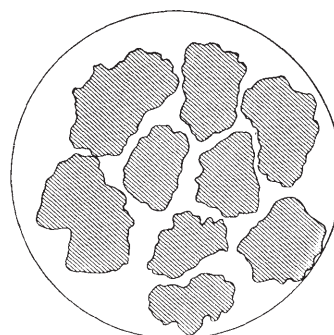
Agfa



Tubize



Elsterberg



Glauchau

variations in the cross-section pictures, while cuprammonium rayon always has the same, smooth round forms, and for that reason can be easily distinguished.

To distinguish cellulose acetate from nitro-cellulose, the microscopical examination is the only means, as the chemical tests are but little reliable.