

drawing.

Some determination of contraction of filament through wet and dry heat have also been carried out. By wet heat at 90°C filament drawn to nine times of its original length contracts almost completely to its original length. By dry heat at 250°C filament drawn as high as five times, contracts completely.

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### **37. On the Heat Decomposition of Polyvinyl Alcohol Fiber**

*Kiyoshi Hirabayashi and Jun Hiramatsu*

(Sakurada Laboratory)

X-ray investigation of the process of heat decomposition of polyvinyl alcohol (P. V. A.) fiber have been carried out. After the heat treatment (200°C, 7 min.) P. V. A. were dried (110°C, 24 hrs.) and subjected to heat decomposition in an electric oven. The oven was heated from 150°C, and 415°C, and during the heating fibers were not allowed to contract. The process may be divide into four stages. 1), growth of crystallites between 150° and 200°C. 2), growth of crystallites and their disorientation between 200° and 250°C. 3), melting of crystallites and decomposition between 250° and 350°C. 4), carbonization above 350°C. Rate of the weight loss of the sample due to dehydration was the greatest between 250° and 300°C.

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### **38. Determination Carboxylic Groups in Vinylon Fiber**

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Determination of carboxylic groups of Vinylon fiber have been carried out according to the method of reversible methylene-blue absorption. Polyvinyl alcohol (P. V. A.) fiber is subjected to heat-treatment (5 min. at ca. 215°C), then partially (35-40 mol %.) formalized so Vinylon is obtained. As is shown in table 1, various pretreatment gives different results, so we cannot determine the true contents of the carboxylic groups of the Vinylon fibr. But it seems, that this method is useful for the technical characterization of Vinylon fiber.

This table 2 shows M. B. absorption of Vinylon after bleaching.

Table 1. Determination of carboxylic groups under various condition.

| No. Substance and pretreatment.                       | mg. M.B./Subst. | Mol. vinylgroup.<br>/mol. COOH |
|-------------------------------------------------------|-----------------|--------------------------------|
| 1. P. V. A. fiber.                                    | 3.02            | 2420                           |
| 2. No. 1. after the heat-treatment.                   | 1.099           | 6600                           |
| 3. No 2. after formalization (Vinylon)                | 0.250           | 27500                          |
| 4. After immersion of No. 2 in water (40°C, 24 hrs.)  | 1.129           | 5650                           |
| 5. After immersion of No. 3 in water (45°C, 17 hrs.)  | 0.472           | 14600                          |
| 6. After immersion of No. 3 in boiling water (1 min.) | 0.624           | 11200                          |
| 7.                   "                   (5 min.)     | 0.695           | 10000                          |
| 8.                   "                   (10 min.)    | 0.777           | 8990                           |
| 9.                   "                   (30 min.)    | 1.028           | 6690                           |
| 10.                  "                  (60 min.)     | 2.38            | 2940                           |
| 11.                  "                  (120 min.)    | 2.30            | 3010                           |

Table 2. Determination of carboxylic groups after bleaching.

(The original sample is No. 3 of table 1.)

| Bleaching agent.              | Condition of bleaching                                                     | mg. M.B./g. Subt. | Mol. vinylgroup.<br>/Mol. COOH |
|-------------------------------|----------------------------------------------------------------------------|-------------------|--------------------------------|
| Bleaching-powder              | Cl <sub>2</sub> 1g/L, N/10H <sub>2</sub> SO <sub>4</sub> , 10c, 17 hrs.    | 0.851             | 8100                           |
|                               | Cl <sub>2</sub> 1g/L, CH <sub>3</sub> COOH, 1g./L 17 hrs.                  | 1.73              | 3960                           |
|                               | Cl <sub>2</sub> , 3.4g/L, CH <sub>3</sub> COOH, 1g./L, 17 hrs.             | 0.803             | 8580                           |
|                               | Cl <sub>2</sub> , 5g/L, CH <sub>3</sub> COOH, 1g./L, 17 hrs.               | 9.12              | 760                            |
| H <sub>2</sub> O <sub>2</sub> | g./L, 45°C, 17 hrs, PH=8.0                                                 | 0.460             | 15000                          |
| "                             | 50g./L, 45°C 17 hrs, PH=8.0                                                | 1.345             | 5090                           |
| K-permangan.                  | 2g./L, 10°C, afterwards immersed in oxalic acid, washed with water, dried. | 0.510             | 13680                          |
| Sodiumchlorite.               | 5g./L, 45°C, 17 hrs, without CH <sub>3</sub> OOH.                          | 0.530             | 13040                          |
|                               | 1g./L, 45°C, 17 hrs, CHCOOH 1g./L.                                         | 0.952             | 7240                           |
|                               | 5g./L, 10°C, 17 hrs, without CH <sub>3</sub> COOH                          | 0.576             | 11960                          |
|                               | 5g./L, 10°C, 17 hrs, CH <sub>3</sub> COOH 1g./L.                           | 0.438             | 15620                          |
| Sodium hydrosulphite.         | 5g./L, 45°C, 17 hrs,                                                       | 0.741             | 9300                           |
|                               | 50g./L, 45°C, 17 hrs,                                                      | 0.973             | 7070                           |

### 39. On the Low Formalization of Polyvinyl Alcohol Fiber

*Ichiro Sakurada and Naofumi Nakamura*

(Sakurada Laboratory)

By the manufacture of synthetic fiber Vinylon, polyvinyl alcohol fiber, which has been subjected to heat treatment, is formalized ordinary with a bath of the following composition: H<sub>2</sub>SO<sub>4</sub> 250g/L, Na<sub>2</sub>SO<sub>4</sub> 300g/L, HCHO 60g/L.

For the purpose of utilization of the wash liquor, formalization of the fiber with this wash liquor have been undertaken. The liquor have the following composition: H<sub>2</sub>SO<sub>4</sub> 80~150 g/L, Na<sub>2</sub>SO<sub>4</sub> 100g/L, HCHO 10~2 g/L. It have been found that polyvinyl alcohol fibers can be easily formalized with this dilute bath and formaldehyde is almost exhausted. This process may have a practical application.