

## A New Synthetic Fiber Made of Nylon 3\*

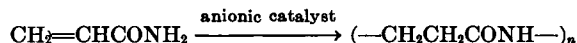
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### Synopsis

Nylon 3 fibers were produced by wet spinning, and properties and structures of the fibers were investigated. Fiber- and film-forming abilities of nylon 3 dopes in various coagulants, using various solvents, were investigated and it was found that only the nylon 3 spinning dope dissolved in formic acid showed excellent fiber- and film-forming abilities in coagulation baths of ethers, esters, or alcohols. As for mass transportation in these coagulants, solvent (formic acid) was diffused into coagulants, with scarce penetration of the coagulants into protofibers. This was due to the fact that these coagulants had moderate solubility parameter values and moderate proton-accepting power, which means the existence of some interaction between solvent and coagulants and no interaction between coagulants and polymer. Protofibers thus obtained were already crystallized and stretching them at room temperature was impossible. However, in such media as silicone oil, hot air, and Wood's metal, which were thought to have no interaction with polyamides, nylon 3 fibers were stretchable above their glass transition temperature (170°–180°C), while in such media as hot water, hot glycerin, and hot steam, which were thought to have some interaction with polyamides, nylon 3 fibers could not be stretched. Mechanical properties of nylon 3 fibers were as follow: strength, 2–3 g/d; elongation, 10%–20%; Young's modulus,  $11 \times 10^5$ – $17 \times 10^5$  psi. High Young's modulus, high transition temperature, and high moisture regain are pointed out as characteristic features of nylon 3 fibers, compared with other synthetic fibers.

### INTRODUCTION

Breslow et al.<sup>1,2</sup> reported that nylon 3 is obtained by the transition polymerization of acrylamide in the presence of an anionic catalyst:



Nylon 3,  $(-\text{CH}_2\text{CH}_2\text{CONH}-)_n$ , is a highly crystalline polymer with a high density of amide group and is considered useful as a material for textiles. The polymer melts at about 340°C with considerable decomposition. Therefore, melt spinning cannot be used, but wet spinning or dry spinning

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may be chosen for the production of the fibers. Nylon 3 is insoluble in most organic solvents and in water, as well as in aqueous basic solutions and dilute acid solutions, but is soluble in hot water above 140°–170°C, strong inorganic acids, organic acids, some aqueous inorganic salts, hot aqueous phenol, and aqueous chloral hydrate.

Some properties of nylon 3 are thought to be different from those of other polyamides. Its glass transition temperature is supposed to be much higher than that of ordinary polyamides. Considering the high density of amide groups leading to high absorption of water, nylon 3 may be expected to be similar to silk or cotton. Considering chemical structure, Young's modulus of nylon 3 should be comparable to that of silk.

Masamoto<sup>3</sup> reported the synthesis of high molecular weight nylon 3 by transition polymerization of acrylamide. In this report, the authors report the wet spinning of nylon 3 whose properties and structures have not yet been reported.

### EXPERIMENTAL

Nylon 3 samples variously reduced viscosities were obtained by the transition polymerization of acrylamide in the presence of an inorganic salt, according to the method previously reported.<sup>4–8</sup> The transition polymerization fraction<sup>9</sup> was attained in a yield of 98% in most cases. In the course of the study polymers having reduced viscosities of 1.4 and 0.9 (polymer concentration 1% in formic acid, at 35°C) were used. The spinning solutions were prepared by dissolving the polymer in 99% formic acid at 33 wt-% polymer concentration and adjusting the polymer concentration by evaporating the excess formic acid at reduced pressure at 40°C.

Fiber-forming abilities of various spinning dopes in various coagulants were investigated by extrusion of a concentrated nylon 3 solution through an orifice into a coagulant. Film-forming abilities of dopes in various coagulants were also investigated. A drop of a nylon 3 solution was smeared on the glass plate and immersed into the coagulant for about 30 min.

The spinning apparatus used in this report is shown schematically in Figure 1. Spinning conditions are given in Table I.

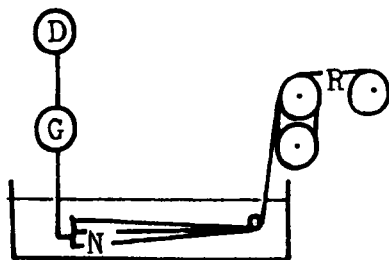


Fig. 1. Apparatus for spinning of nylon 3: D, dope; G, gear pump; N, nozzle; R, roller.

TABLE I  
Main Spinning Conditions Adopted in This Study

|                         |               |  |
|-------------------------|---------------|--|
| Dope                    | Polymer       | $\eta_{sp}/c = 0.9$                                |
|                         | Solvent       | HCOOH  |
|                         | Concentration | 37 wt-%  |
| Coagulation             | Nozzle        | $0.15 \times 10 \text{ mm}\phi \times \text{hole}$ |
|                         | Output        | 0.47 cc/min  |
|                         | Coagulant     | AcOEt  |
|                         | Length        | 2m   |
|                         | Temperature   | 20°C   |
| 2nd Bath                | Winding speed | 4.4 m/min  |
|                         | Medium        | AcOEt  |
|                         | Length        | 3.5m   |
|                         | Temperature   | 20°C   |
| Drawing                 | Medium        | Metal Plate  |
|                         | Temperature   | 280°C  |
|                         | Length        | 24 cm  |
|                         | Feed speed    | 3.6 m/min  |
|                         | HCOOH content | 6-10 wt-%  |
| Boiling water treatment | Medium        | Water  |
|                         | Temperature   | 100°C  |
|                         | Time          | 30 min   |

## RESULTS AND DISCUSSION

### Effects of Coagulant on Fiber-Forming Abilities of Nylon 3 Solution in Formic Acid

Fiber- and film-forming abilities of nylon 3 dope, using formic acid as the solvent, were investigated in various types of coagulants. Results were shown in Table II. With water as the coagulant, no fiber formation of nylon 3 was attained. This might have been attributable to the weak coagulation power of water. In this paper, coagulation power was defined as  $10/K$ , where  $K$  is that volume (in ml) of a coagulant which clouds 10 ml of 5% polymer solution.

Using aqueous salts such as potassium formate and sodium formate as coagulants, fiber and film formation of nylon 3 was observed to some extent. However protofibers thus obtained in aqueous coagulants were microscopically quite nonhomogeneous and porous, probably because of the penetration of the coagulant into the protofibers. Stretching of these protofibers by the methods mentioned later was impossible. The protofibers obtained in such coagulants as esters, ethers, and some alcohols were quite homogeneous and deswelled.

Relations between fiber-forming abilities of the dope in various coagulants and chemical structures of coagulants were studied. The coagulants in which nylon 3 solution showed superior fiber-forming abilities had moderate strength of proton-accepting power and moderate solubility parameters. Cohesive energy density of formic acid and nylon 3 was considered quite large due to the high concentration of hydrogen bonds.

TABLE II  
Fiber- and Film-forming Abilities of Nylon 3 Dope  
in Various Coagulants, Using Formic Acid as Solvent<sup>a</sup>



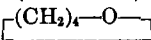
|                                       | Coagulant   | Film forming | Fiber forming | Coagulation power, |
|---------------------------------------|---|--------------|---------------|--------------------|
| Alcohols                              | CH <sub>3</sub> OH  | ×            | ×             | 0.67               |
|                                       | C <sub>2</sub> H <sub>5</sub> OH  | ×            | ×             | 0.66               |
|                                       | n—C <sub>3</sub> H <sub>7</sub> OH  | ⊙            | ⊙             | 0.56               |
|                                       | iso—C <sub>3</sub> H <sub>7</sub> OH  | ⊙            | ⊙             | 0.64               |
|                                       | n—CH <sub>2</sub> (CH <sub>2</sub> ) <sub>3</sub> OH  | ⊙            | ⊙             | 0.50               |
|                                       | iso—(CH <sub>2</sub> ) <sub>2</sub> CHCH <sub>2</sub> OH  | ⊙            | ⊙             | —                  |
|                                       | sec—(C <sub>2</sub> H <sub>5</sub> )CH <sub>2</sub> CHOH  | ⊙            | ⊙             | —                  |
|                                       | n—C <sub>8</sub> H <sub>11</sub> OH   | ⊙            | ⊙             | 0.68               |
|                                       | n—C <sub>8</sub> H <sub>13</sub> OH   | ⊙            | ⊙             | 1.0                |
|                                       | n—C <sub>8</sub> H <sub>17</sub> OH   | ⊙            | ⊙             | —                  |
|                                       | HO—(CH <sub>2</sub> ) <sub>2</sub> OH   | ×            | ×             | 0.87               |
|                                       | HO—CH <sub>2</sub> CH(CH <sub>3</sub> )(CH <sub>2</sub> ) <sub>3</sub> OH                       | ×            | ×             | 0.64               |
|                                       | HO—CH <sub>2</sub> CHOHCH <sub>2</sub> OH   | ×            | ×             | 0.08               |
|                                       |  OH            | ⊙            | ⊙             | 0.56               |
|                                       | C <sub>6</sub> H <sub>5</sub> CH <sub>2</sub> OH  | ×            | ×             | —                  |
| Ketones                               | CH <sub>3</sub> CO·CH <sub>3</sub>  | ×            | ×             | 0.71               |
|                                       | CH <sub>3</sub> CO·C <sub>2</sub> H <sub>5</sub>  | ○            | ○             | 0.66               |
|                                       |  =O            | ○            | ○             | 0.47               |
|                                       | C <sub>6</sub> H <sub>5</sub> COCH <sub>3</sub>   | ○            | ○             | 0.32               |
| Ethers                                | C <sub>2</sub> H <sub>5</sub> OC <sub>2</sub> H <sub>5</sub>                                    | ⊙            | ⊙             | 0.95               |
|                                       | (CH <sub>3</sub> ) <sub>2</sub> ·CH·OCH(CH <sub>3</sub> ) <sub>2</sub>                          | ⊙            | ⊙             | 1.79               |
|                                       | n—(C <sub>4</sub> H <sub>9</sub> ) <sub>2</sub> O   | ⊙            | ×             | n.m                |
|                                       | (—C <sub>2</sub> H <sub>4</sub> O—) <sub>2</sub>  | ⊙            | ⊙             | 0.83               |
|                                       |              | ⊙            | ⊙             | 0.67               |
| Ester                                 | HCOOCH <sub>3</sub>   | ○            | ○             | 0.29               |
|                                       | HCOOC <sub>2</sub> H <sub>5</sub>   | ⊙            | ⊙             | 0.35               |
|                                       | CH <sub>3</sub> COOCH <sub>3</sub>  | ⊙            | ⊙             | 0.57               |
|                                       | CH <sub>3</sub> COOC <sub>2</sub> H <sub>5</sub>  | ⊙            | ⊙             | 0.73               |
|                                       | CH <sub>3</sub> COOCH(CH <sub>3</sub> ) <sub>2</sub>  | ⊙            | ⊙             | 1.16               |
|                                       | CH <sub>3</sub> COO(CH <sub>2</sub> ) <sub>3</sub> CH <sub>3</sub>                              | ⊙            | ⊙             | 1.28               |
|                                       | CH <sub>3</sub> COO(CH <sub>2</sub> ) <sub>5</sub> CH <sub>3</sub>                              | ⊙            | ⊙             | 2.28               |
|                                       | C <sub>2</sub> H <sub>5</sub> O <sub>2</sub> CCH <sub>2</sub> CO <sub>2</sub> H <sub>5</sub>    | ⊙            | ⊙             | —                  |
|                                       | CH <sub>2</sub> O <sub>2</sub> C(CH <sub>2</sub> ) <sub>4</sub> CO <sub>2</sub> CH <sub>3</sub> | ⊙            | ⊙             | —                  |
| Hydro-carbon & halogenated derivative | C <sub>6</sub> H <sub>6</sub>   | ⊙            | ×             | n.m                |
|                                       | n—CH <sub>2</sub> (CH <sub>2</sub> ) <sub>4</sub> CH <sub>3</sub>                               | ⊙            | ×             | n.m                |
|                                       | CCl <sub>4</sub>  | ⊙            | ×             | n.m                |
| Amide                                 | HCON(CH <sub>3</sub> ) <sub>2</sub>   | △            | ×             | 1.23               |
|                                       | CH <sub>3</sub> CON(CH <sub>3</sub> ) <sub>2</sub>  | △            | ×             | —                  |
| Amide                                 | HCONH <sub>2</sub>  | ×            | ×             | —                  |

TABLE II (continued)

|               | Coagulant                                       | Film forming | Fiber forming | Caagulation power, |
|---------------|---|--------------|---------------|--------------------|
| Nitrile       | CH <sub>3</sub> CN                              | △            | ×             | 0.30               |
|               | CH <sub>3</sub> CH <sub>2</sub> CN              | △            | ×             | —                  |
|               | CN—CH <sub>2</sub> —CN                          | △            | ×             | —                  |
| Water and     | H <sub>2</sub> O                                | ×            | ×             | 0.0—               |
|               | NH <sub>4</sub> Cl                              | ×            | ×             | 0.21               |
| saturated     | NaNO <sub>3</sub>                               | ×            | ×             | 0.0—               |
|               | NH <sub>4</sub> NO <sub>3</sub>                 | ×            | ×             | 0.0—               |
| aqueous salts | Na <sub>2</sub> SO <sub>4</sub>                 | ×            | ×             | 0.0—               |
|               | (NH <sub>4</sub> ) <sub>2</sub> SO <sub>4</sub> | ×            | ×             | 0.55               |
|               | HCOONa  | ○            | △             | 0.40               |
|               | HCOOK   | ○            | ○             | 1.54               |
|               | HCOONH  | ×            | ×             | 0.59               |
|               | CH <sub>3</sub> COOK                            | △            | △             | 0.71               |
|               | aq. 20% KOH                                     | ×            | ×             | 0.26               |
|               | aq. 40% KOH                                     | △            | △             | 0.50               |
|               | 10% KCl—  | ○            | ○             | 0.71               |
|               | aq. 40% KOH                                     |              |               |                    |

<sup>a</sup> Polymer  $\eta_{sp}/c = 1.4$ ; polymer conc. = 36 vol-%; coagulation power =  $10/K$ , where  $K$  is volume (ml) of coagulant which clouds 10 ml 5% polymer solution; n.m = nonmixing; (⊙) homogeneous, flexible fibers; (○) flexible, nonhomogeneous fibers; (△) nonhomogeneous, inflexible fibers; (×) non-fiber forming.

Thus when the solubility parameters of the coagulants were large (such as methanol) the coagulants might have interacted with formic acid and the polymer and penetration of coagulants into protofibers might have occurred easily. Nonhomogeneous protofibers with many microscopic voids might be obtained because of the penetration of the coagulant.

When the solubility parameters of the coagulants were small (such as silicone oil), the coagulants had little interaction with formic acid and the polymer. The solvent did not diffuse into the coagulants and the coagulant did not penetrate into the polymer solution, and no coagulation occurred.

When the solubility parameters of the coagulants were moderate, there might have been some interaction of the coagulant with formic acid and little interaction with the polymer. Thus desolvation might have occurred with little penetration of the coagulant into the protofibers and homogeneously deswelled protofibers might be obtained. Considering the nature of the solvent and the polymer, proton-accepting power of the coagulants might be important. As shown in Table II, esters, ethers, and some alcohols, in which nylon 3 dope showed superior fiber formation, had moderate proton-accepting power. Thus in such coagulants, diffusion of the formic acid into coagulants might be easy due to the proton-donating nature of the formic acid, while penetration of the coagulants into proto-

fibers might be difficult due to the weak proton-accepting nature of the polymer.

The coagulants in which nylon 3 dope showed only film-forming abilities were xylene, toluene, carbon tetrachloride, etc. These coagulants had moderate values of solubility parameter but poor proton-accepting or proton-donating power. Thus there was little interaction between nylon 3 and the solvent, and the diffusion of the solvent into the coagulant was too slow for fiber formation. However, because diffusion slow enough for film formation might be possible, homogeneously deswelled films were obtained.

In the wet spinning process, in addition to diffusion of the solvent and penetration of the coagulant into the protofibers, gelation of the spinning dope flow through an orifice, crystallization of the protofibers, and solvent power of the solvent must be considered. But in the wet spinning process involving nylon 3, diffusion of solvent, desolvation, and no penetration of coagulants into the protofibers, followed by gelation and crystallization, were believed to be the most important factors.

### Nylon 3 Solutions in Other Solvents

Fiber-forming abilities of nylon 3 dope dissolved in dichloroacetic acid, trifluoroacetic acid, sulfuric acid, nitric acid, aqueous phenol, aqueous calcium thiocyanate, zinc chloride, and aqueous chloral hydrate were investigated in the various coagulants. With these solvents only poor fiber- and film-forming abilities were observed, as shown in Table III. This could be attributed to the difference of solvent power of the solvents used and the differences of the nature of the solvent.

### Fiber-Forming Process

In this section, the fiber-forming process using formic acid as solvent was investigated. In Figure 2, mass transfer in the wet spinning process of

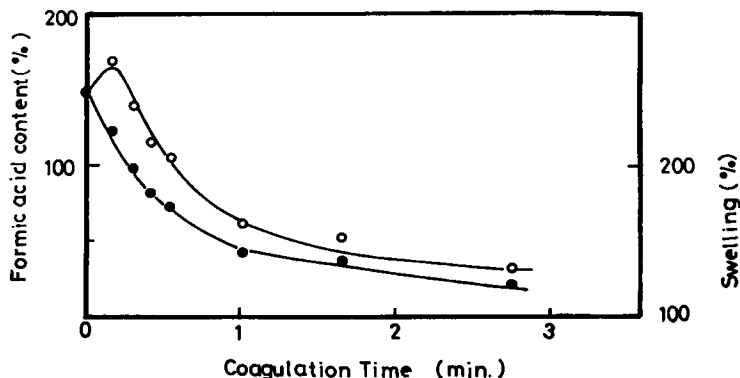


Fig. 2. Fiber-forming process of nylon 3, using ethyl acetate as the coagulant: swelling % [(weight of protofibers)/(weight of polymer in protofibers)]  $\times$  100.

TABLE III  
Fiber- and Film-Forming Abilities of Nylon 3 Dopes in Various Coagulants, Using Various Solvents<sup>a</sup>

| Coagulants  | Fiber forming |    |    |     | Film forming |    |    |    |                 |                 | W <sup>d</sup> |                 |
|---|---------------|----|----|-----|--------------|----|----|----|-----------------|-----------------|----------------|-----------------|
|   | DCA           | CS | ZC | DCA | CS           | ZC | SA | NA | PW <sup>b</sup> | FW <sup>b</sup> |                | CH <sup>c</sup> |
| Water   | ×             | ×  | ×  | ×   | ×            | ×  | ×  | ×  | ×               | ×               | ×              | ×               |
| aq. Na <sub>2</sub> SO <sub>4</sub>                 | ×             | ×  | ×  | ×   | ×            | ×  | ×  | ×  | ×               | ×               | ×              | ×               |
| aq. (NH <sub>4</sub> ) <sub>2</sub> SO <sub>4</sub> | ×             | ×  | ×  | ×   | ×            | ×  | ×  | ×  | ×               | ×               | ×              | ×               |
| aq. HCOOK   | ×             | △  | △  | ×   | ○            | △  | ×  | ×  | ×               | ×               | ×              | ×               |
| MeOH  | ×             | ×  | ×  | ×   | ×            | ×  | ×  | ×  | ×               | ×               | ×              | ×               |
| iPrOH   | ×             | ×  | ×  | △   | ×            | ×  | ×  | ×  | ×               | ×               | ×              | ×               |
| AcOEt   | △             | △  | △  | △   | △            | △  | △  | △  | △               | △               | △              | △               |
| AcOBu   | △             | △  | △  | △   | △            | △  | △  | △  | △               | △               | △              | △               |
| (iPr) <sub>2</sub> O                                | ×             | ×  | ×  | △   | ×            | ×  | ×  | ×  | ×               | ×               | ×              | ×               |
| Dioxane   | △             | △  | △  | △   | △            | △  | △  | △  | △               | △               | △              | △               |
| MEK   | ×             | ×  | ×  | ×   | ×            | ×  | ×  | ×  | ×               | ×               | ×              | ×               |







<sup>a</sup> DCA = dichloroacetic acid; CS = 38% aqueous calcium thiocyanate; ZC = 60% aqueous zinc chloride; SA = sulfuric acid; NA = nitric acid; PW = 70% aqueous phenol; FW = 37% aqueous formaline, CH = 73% aqueous chloral hydrate; W = water. Polymer conc. (vol-%): DCA, 33.3; SA, 15.8; NA, 37.5; CS, 33.3; ZC, 41.9; PW, 27.3; FW, 20; CH, 23.0; W, 27.3. Polymer  $\eta_{sp}/c = 1.4$ . (△) nonhomogeneous, inflexible fibers; (×) non-fiber forming.

<sup>b</sup> Dissolved at 150°C.

<sup>c</sup> Dissolved at 60°C.

<sup>d</sup> Dissolved at 180°C.

TABLE IV  
Effects of Coagulants

| Coagulant            | Section of fibers   | Coagulation power | Solubility parameter | Maximum draft ratio |
|----------------------|---|-------------------|----------------------|---------------------|
| AcOMe                |  | 0.57              | 9.6                  | 3.8                 |
| AcOEt                |  | 0.73              | 9.1                  | 3.8                 |
| AcOiPr               |  | 1.16              | 8.4                  | 3.0                 |
| AcOnBu               |  | 1.28              | 8.5                  | 3.0                 |
| Dioxane              |  | 0.83              | 9.9                  | 3.3                 |
| (iPr) <sub>2</sub> O |  | 1.79              | —                    | 2.6                 |

nylon 3, using ethyl acetate as the coagulant, is shown. As mentioned previously, little penetration of the coagulants into protofibers was observed. These phenomena support the assumption mentioned in a previous section, and might account for the superior fiber formation of nylon 3 in the wet spinning process. In other coagulants, such as methyl acetate, isopropyl acetate, and dioxane diisopropyl ether, mass transfer was also investigated and similar phenomena were observed.

Thus fiber formation of nylon 3 is characterized by desolvation and deswelling, contrary to that of other polymers in the wet spinning process.

In Table IV, the effects of coagulation power of the coagulants on fiber formation and fiber properties are shown. The maximum draft ratio, a

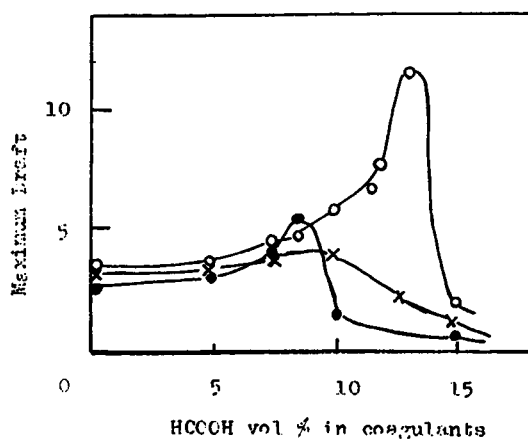


Fig. 3. Effects of addition of formic acid into the coagulants on the maximum draft ratio: (○) ethyl acetate; (●) isopropyl ether; (×) dioxane.



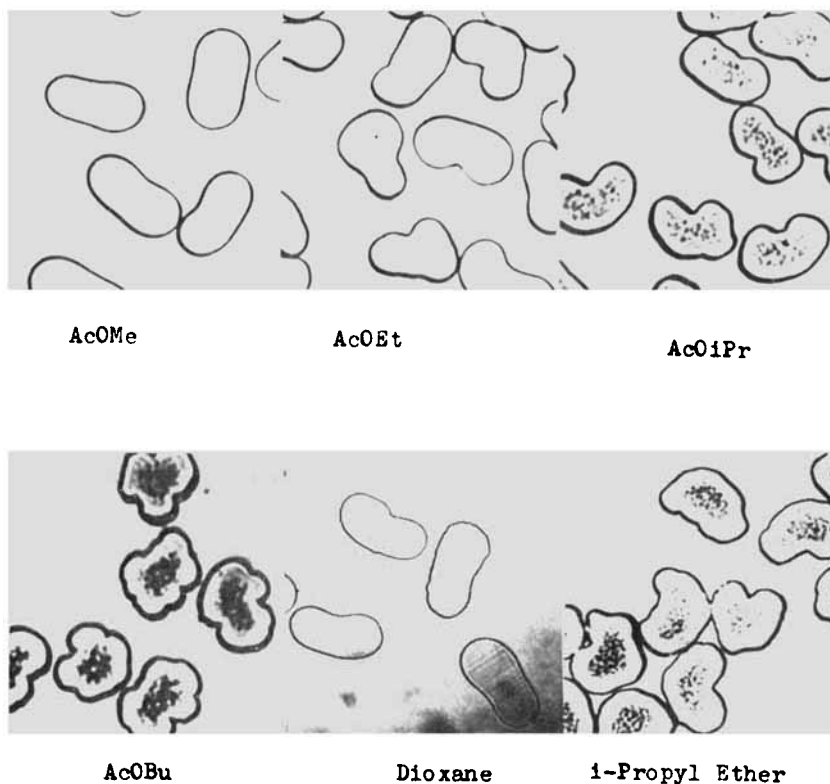


Fig. 4. Sectional shape of the fibers obtained in the various coagulants.

measure of the spinnability in the wet spinning process, was increased with decreasing coagulation power of the coagulants, which might be related to the solubility parameter of the coagulants. This was also confirmed by the fact that formic acid added to the coagulant to lower the coagulation power increased the maximum draft ratio, as shown in Figure 3.

In Figure 4, the sectional shape of the fibers obtained in various coagulants is shown. When the coagulation power of the solvents was low such as methyl acetate, ethyl acetate, and dioxane, the fibers were dog bone shaped. The inner structure of the fibers was microscopically homogeneous, probably because of the weak coagulation of the surface of spinning dope flow. The fibers obtained in such coagulants as isopropyl acetate, *n*-butyl acetate, and diisopropyl ether, which had strong coagulation power, were uneven in shape. The inner structure of the fibers was microscopically nonhomogeneous with much porosity, probably because of the strong coagulation to the surface of spinning dope flow. X-Ray diffraction patterns of protofibers showed that the fibers were already crystallized. Thus molecular orientation of nylon 3 was thought to be quite difficult, considering the high density of amide groups.

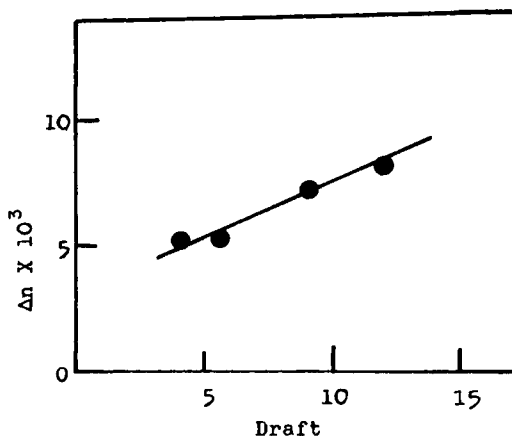


Fig. 5. Effect of draft ratio on molecular orientation of the fibers.

In Figure 5, the effects of spinning draft ratio on molecular orientation of the fibers are shown. With increasing draft ratio, molecular orientation was increased slightly and birefringence reached a value of  $1 \times 10^{-2}$ , which is insufficient for textile.

Considering the strong inter- and intramolecular interaction of nylon 3, stretching of the fibers in a state of lowered transition points of the polymer was thought plausible. From this view point, stretching of the fibers containing much formic acid or stretching of the fibers in such media as hot water and glycerin, which are thought to have some interaction with polyamides, was thought plausible. By varying the coagulation time, nylon 3 fibers containing 20–30 wt-% formic acid were obtained, but stretching these fibers at room temperature was impossible. The stretching of the undrawn fibers in such media as hot water, hot steam, and hot glycerin was also investigated. However, as shown in Table V, nylon 3 fibers could not be stretched in these media. In such medias as silicone, Wood's metal, and hot air, which were thought to have no interaction with polyamides,

TABLE V  
Effects of Stretching Media on Maximum Draw Ratio

| Medium*      | Temperature, °C | Max. draw ratio |
|--------------|-----------------|-----------------|
| Silicone     | 240°C           | 5.0             |
| Wood's metal |                 | 4.5             |
| PEG 400      | 220°C           | 4.0             |
| EG           | 150°C           | 1.2             |
| Glycerin     | 150°C           | 1.0             |
| Water        | 100°C           | —               |
| Hot air      | 240°C           | 4.0             |
| Vapor        | 200°C           | —               |
| aq HCOOK     | 120°C           | 1.2             |

\* PEG, polyethylene glycol; EG, ethylene glycol.

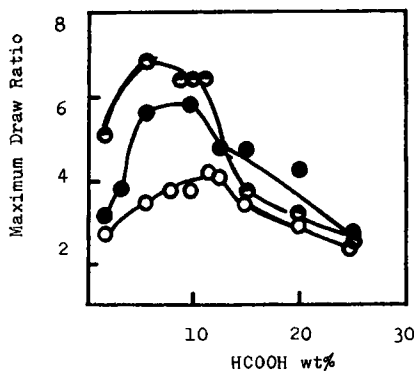


Fig. 6. Effect of formic acid content fibers on maximum draft ratio. Draw temperature: (○) 200°C.; (●) 240°C.; (◐) 280°C.

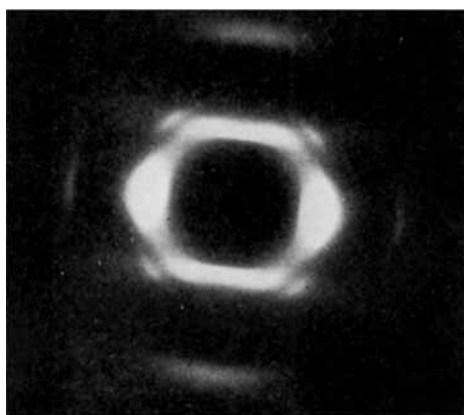


Fig. 7. X-Ray diffraction pattern of drawn nylon 3 fibers.

nylon 3 fibers could be stretched above their glass transition temperature (170°–180°C).

In Figure 6, the effects of drawing temperature and formic acid content in fibers on the maximum draw ratio are shown. The most favorable drawing temperature with maximum draw ratio of the undrawn fibers became lower with increase of the formic acid contents in undrawn fibers.

In Figure 7, x-ray diffraction pattern of drawn nylon 3 fibers is shown, which reveals good molecular orientation. Birefringence of the most oriented fibers was increased to a value of  $8 \times 10^{-2}$ , a higher value, considering the fact that ordinary polyamides showed a value of  $6 \times 10^{-2}$ . This was due to the high amide concentration. From the x-ray diffraction pattern, the crystal structure of nylon 3 was determined, which will be reported elsewhere.<sup>10</sup> The unit cell of the nylon 3 crystal was monoclinic, though that of other odd-numbered nylons was triclinic or pseudohexagonal.

In Figure 8, the effects of draw ratio on the mechanical properties of nylon 3 fibers are shown. With draw ratio, tensile strength was increased

linearly and tensile elongation was decreased. Undrawn fibers had no tensile strength and no elongation in the wet condition, while drawn fibers showed superior retention of mechanical properties in wet condition. This

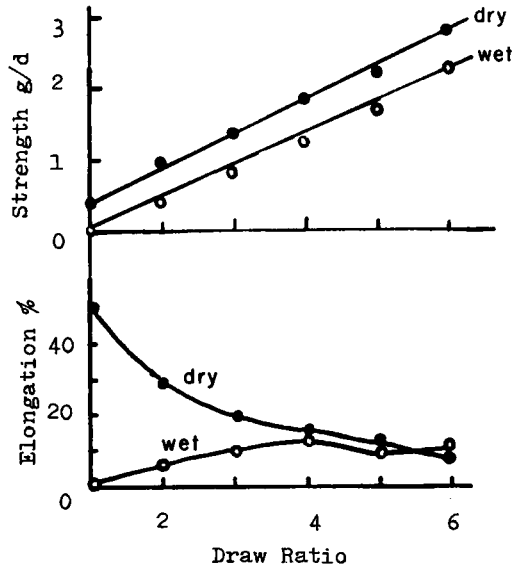


Fig. 8. Effects of draft ratio on mechanical properties of fibers.

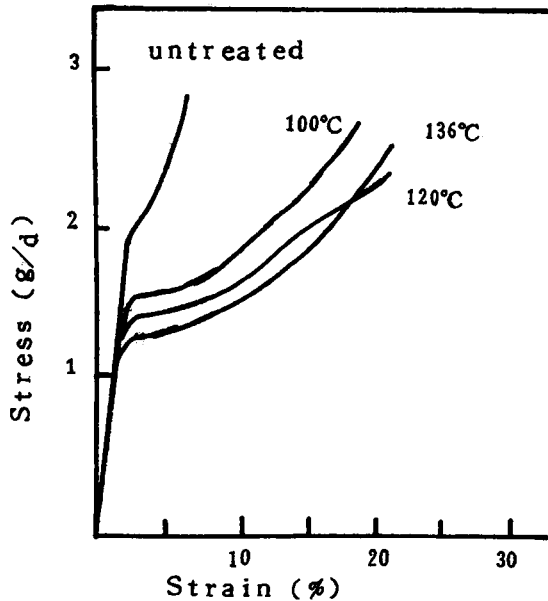


Fig. 9. Stress-strain curve of wet heat-treated nylon 3 fibers.

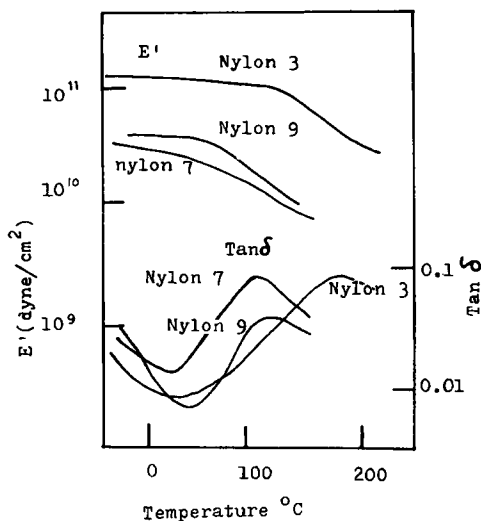


Fig. 10. Viscoelastic properties of nylon 3 fibers.

might be partly due to the molecular orientation and partly due to the transformation of the crystal structure in the drawing process.<sup>10</sup>

In Figure 9, strain-stress curves of the wet heat-treated drawn nylon 3 fibers are shown. Tensile elongational properties were very much improved. Mechanical properties were as follows: tensile elongation strength, 2-3 g/d; tensile elongation, 10%-20%; Young's modulus,  $11 \times 10^5$ - $17 \times 10^5$  psi. Young's modulus was quite high, comparable to that of silk.

In Figure 10, the viscoelastic properties of nylon 3 fibers are shown. The temperature showing the maximum  $\tan \delta$ , which might be attributed to the temperature of the scission of hydrogen bonds in the amorphous region,<sup>11</sup> was higher by about 70°C than ordinary polyamides. This might be due to the high concentration of amide groups.

High moisture regain of nylon 3 fibers, a value of 7%-9% (20°C, 65% RH), was observed, which was comparable to that of silk.

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