

## Structure and Re-orientation in the First Stage for Polyamide/Iodine Complex. [II] Change of Structures on Iodine Doping and Termination.

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### Introduction

Polyamide-6 (PA6, Nylon<sup>®</sup>-6) is a popular hydrophilic polymer with high crystallinity, and it also shows ordered structures even in coordinated structures with guest molecules or ions. "Polyamide-6/iodine complex" can be prepared by easy and rapid operation using an aqueous solution containing polyiodide ions at room temperature.[1-3]

As one of interesting behaviors for the complex structure, rapid formation of the complex is reported; the complex structures are formed rapidly in a few seconds.[4] However, the initial structure observed in the first stage shows different spacing of intercalated structures from essential one shown in ordinary stable structure.[5] In this work, *in-situ* observation for the doubly oriented film of PA6 was achieved using an enclosing housing.[6]

### Experiments

*Sample preparation : Doubly-oriented PA-6 film:*

PA6 used was a commercial polymer ( $M_w=4.2 \times 10^5$ ) and a melt-pressed film was quenched in ice water. The film was drawn three times to be a "doubly-oriented" film. ( $\chi \sim 25\%$ ) This film (0.15 mm in thickness) was cut to be a strip which could be set in a quartz capillary of 2.0 mm in diameter.

*Pouring system : housing, dispenser pump, solution:*

For the process of "iodine-doping", an aqueous solution of I<sub>2</sub>-KI (0.4N) was used.

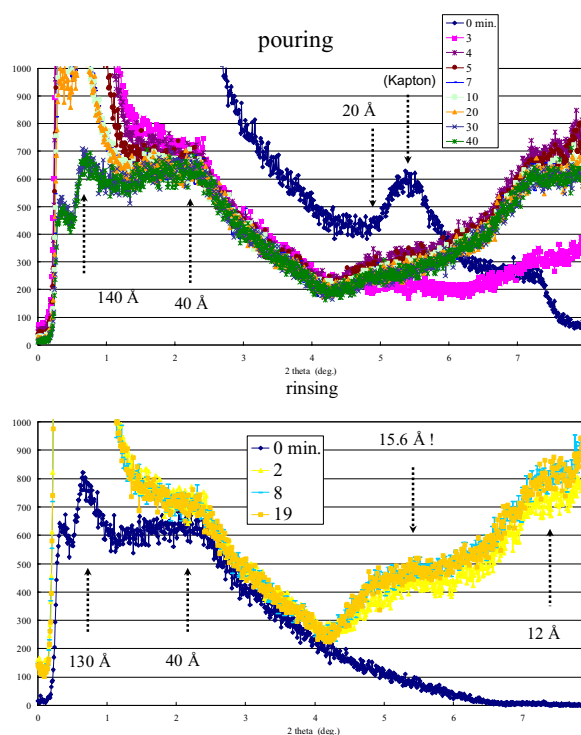
Scheme of the housing vessel has been reported.[5] Remote-controlled operation of a dispenser pump introduced the 0.4N I<sub>2</sub>-KI solution and rinsing water into a capillary holding the filmy sample; regular rotation of the pump introduced the solution from the capillary bottom as a suction pump and reverse rotation led distilled water which was reserved in a tube connected at the capillary head. Each flow was synchronized to start of time-sliced measurement.

*Beam, detector, layout:*

As an incident, a monochromatized beam ( $\lambda = 0.15$  nm) on BL-15A was used and diffraction profile was measured with an 1-dim PSPC.[7] The detector was set to detect diffraction along the meridian; the line detector was arranged normal to the chain axis in this experiment. Camera length was 510mm and exposure time was set as 1min. for each shot. For each sequence of observation, 20-45 shots were taken continuously.

### Results

The spacing of 1.56nm which is ordinarily and stably observed for the complex did not appear in the first stage of complex but was observed after rinsing by water. At the same time, another structure rose or vanished through operation. Iodine-doping and rinsing suggest complication in preparation for the complex and more investigation has been required.



Figures: Diffraction profiles of *in-situ* observation on pouring solution (upper) and on rinsing with water (lower).

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### References

- [1] H. Arimoto, et al., J. Polym. Sci., A3, 317 (1965).
- [2] A. Kawaguchi. Polymer, 33, 3981(1992).
- [3] A. Kawaguchi., Sens. Act. B. 73, 174 (2001).
- [4] A. Kawaguchi, et al., SPring-8 User Exp. Rep. 5, 354 (2000).
- [5] A. Kawaguchi, et al., Polym. Prep. Jpn., 50, 2000 (2001).
- [6] A. Kawaguchi, et al., PF Activity Rep. 2003, 172 (2004).
- [7] [http://pfwww.kek.jp/users\\_info/station\\_spec/bl15/bl15a.html](http://pfwww.kek.jp/users_info/station_spec/bl15/bl15a.html)

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