Production and Characterization of Carbon Fiber from Iodinated *Bombyx mori* Silk Fibroin

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INTRODUCTION:

In the development of new materials from silk fibroin (SF), the researches concerning the carbonization of silk are involved. Recently, it has been reported that by carbonization at 800 °C of SF, non-graphitizing silk based carbon could be obtained with a carbon yield of 30%, which was much higher than the value measured for the cellulose based carbon. Iodine is a good stabilizing agent for producing higher yield pitch carbon. In addition, carbon yield for some kinds of polymers such as polyacrylonitrile and poly(vinyl alcohol) have been considerably enhanced by carbonization with iodine treatment. To investigate the effects of iodine treatment on the carbonization behavior of SF, silk fibers were treated with iodine vapor and evaluated the carbon yield, fiber morphology, fine structure and the mechanical properties.

MATERIALS AND METHODS:

Bombyx mori degummed SF fiber used in this experiment was provided by North Eastern Industrial Research Center, Shiga prefecture, Japan.

A single or multi step carbonization process was used for the preparation of silk based carbon fiber. In the single step process, SF fibers were heated from 25 to 800 °C with a heating rate of 5 °C min-1 under Ar atmosphere. However, the carbon fiber obtained was partially melted and was too fragile to handle. For better performance, SF fibers were treated with iodine vapor at 100 °C for 12 h and untreated and iodinated SF fibers were heated from 25 to 800 °C by a multi step carbonization process as shown in Fig. 1. The multi step process was defined based on the optimum thermal degradation rate of silk, which was determined by dynamic TGA measurement using untreated SF.

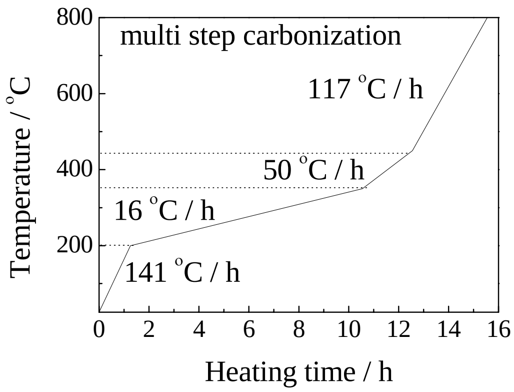

Fig. 1. Heating schedules for multi step carbonization

RESULTS AND DISCUSSION:

Fig. 2 shows the carbon yield of SF as a function of different iodine treatment times. In case of untreated specimen, the carbon yield was ca. 29wt%. The carbon yields considerably increased and reached up to ca. 36wt% as a function of iodine treatment for 12 h. After 12 h, the yield gradually decreased though iodine content increased. So we used only 12 h treated specimen for experiments. SEM observation showed that obtained carbon fibers from iodinated SF were structurally intact and stable in appearance (Fig. 3). The strength of carbon fibers prepared from iodinated SF using the multi step carbonization was considerably increased compared to that of untreated SF. To evaluate the structural features, untreated and iodinated SF were heated to 800, 1200 and 1400 ^{o}C, respectively. Fig. 4 shows the WAXD profiles of SF based carbons obtained at different heating schedules. Untreated SF exhibited amorphous pattern in different temperature. On the other hand, at 1400 ^{o}C iodinated SF showed the peak at $2\theta=26.0^{o}$ (d= 3.42 Å), which corresponds to the scattering from the (002) plane of graphite.

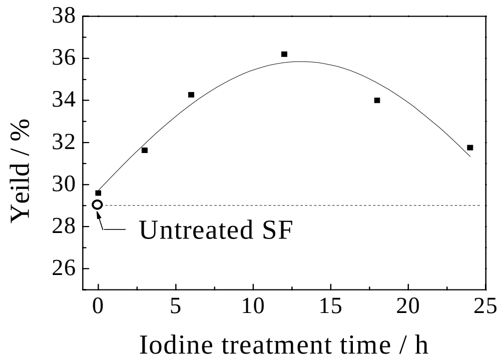

Fig. 2. The carbon yield of SF fiber according to multi step carbonization

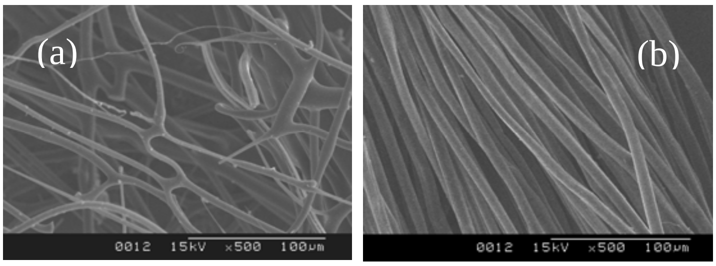

Fig. 3. SEM images of silk based carbon fibers: (a) untreated SF, single step carbonization; (b) iodinated SF, multi step carbonization.

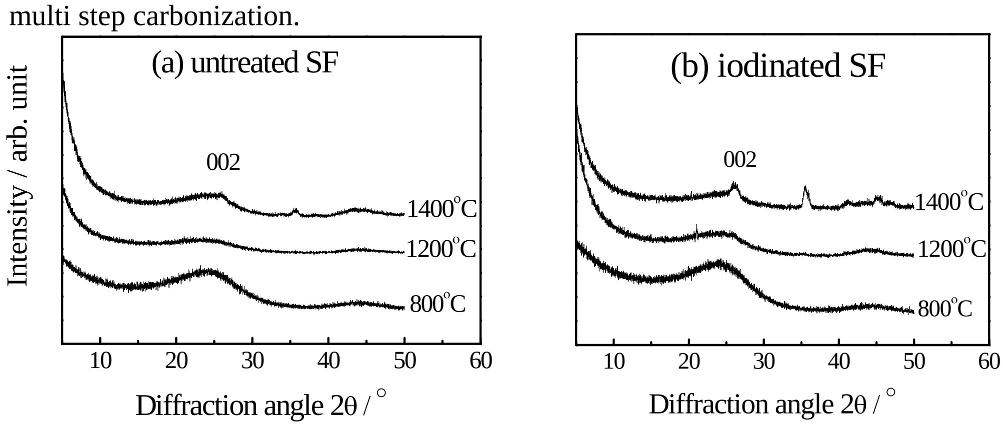

Fig. 4. Equatorial WAXD profiles of silk carbon fibers prepared by multi step carbonization at different temperature: (a) untreated SF, (b) iodinated SF.