DISCUSSION OF INTERCALATION BEHAVIOR FOR THE PITCH-BASED CARBON FIBERS

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Introduction

It has been reported that the intercalation to the carbon fiber (CF) is difficult to be synthesized further than the intercalation to the natural graphite. Therefore typical large exfoliation such as showing on graphite flakes could not recognized. From the examination of the decomposition behavior of Li-ICCF (intercalation compounds of carbon fiber), it has been reported that intercalation of Li is generated with coexistence of high and low stage of Li-intercalation compounds [1]. However, there is some other factors existence to form the intercalation compounds in the case of CF, because inner orientation and cross sectional morphology having CF are different It was chosen the Pitch -based CF having the corrugate radial texture with different graphitization degree as host materials for the synthesis of Li-ICCF. This Li-ICCF prepared from pitch-based carbon fibers were synthesized through short-circuit of lithium metal and carbon fibers to clarify behavior of intercalation to the carbon fibers. Behavior of its intercalation were discussed the crystallinity of carbon fibers. It was suggested that behavior of intercalation of the carbon fibers were related with crystallinity of its internal texture.

Experimental

3 kinds of pitch-based carbon fiber showing different graphitization degree were used for the host carbon fiber (CF). Synthesis of Li-ICCF were synthesized through short-circuit of lithium metal and carbon fibers. This short circuit was carried out by external short circuit of Li metal compressed on Ni-mesh and carbon fibers in EC/DEC(1/1 vol. %) in which 1 mol/dm^3 $LiBF_4$ dissolved. The accuracy measurement of *002* and *004* diffraction line using X ray diffraction (XRD) were carried out by mixed grounded carbon fiber and 10 wt.% silicon powder (National Institute of Standards and Technology, 640 c) as an internal standard by the agate mortar. Condition of XRD measurement is 40 kV in applied voltage, 40 mA in current and 1/4 ^{o}/min in scan rate. Scanning range was done 20 to 30 ^{o} and 50 to 60 ^{o}, respectively. Crystallinity [graphitization degree: (P1)] of the CFs was calculated by referring to the paper by Noda et al. [2]. The separation of peak obtained (*002* and *004* diffraction line) was carried out according to the Gaussian function, and then *d*-spacing, crystallite size and graphitization degree were calculated by usingt*002* and *004* diffraction line.

Results and Discussions

The decomposition behavior with time course of Li-ICCF prepared from CF having high crystallinity is shown in Fig. 1. Diffraction lines of 2θ = 24.9 ^{o} and 26.5 ^{o} recognized after short circuit are corresponded stage-2 of *002* diffraction line and Li-ICCF showing the high-stage, respectively. Each peak shifted to the high angle with the decomposition of Li-ICCF, and it seemed to be decomposing to the high-stage in comparison with after short-circuit. On the other hands, in the case of CF showing low graphitization degree, the peak which corresponded to the random stage was recognized, however, the shift to the high angle with decomposition could not be recognized.

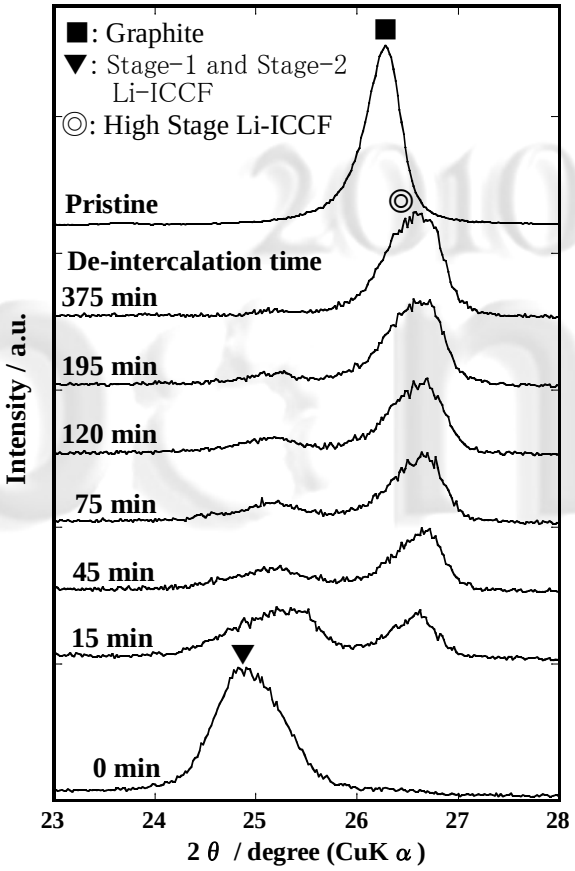

Fig. 1 Behavior of de-intercalation.

Result of peak separation in *002* diffraction lines for the pristine carbon fiber showing high crystallinity is shown in Fig. 2. From this peak separation in *002* diffraction line, carbon fiber with the high crystallinity consist 43.8, 30.1 and 26.9 nm in comparatively large crystallite size (Lc), and 8.8 nm comparatively small crystallite size (Lc). The internal texture of carbon fiber means being the coexistence of high

and low crystallinity. It seemed to connect it with the behavior in which the existence of the part of the high crystallinity shifted to the high angle side. In the case of carbon fiber with the low crystallinity, it was guessed that peak did not shift to the high angle, since the part with the low crystallinity is mainly included. Therefore, it might be the storage of Li^+ to the part with the low crystallinity.

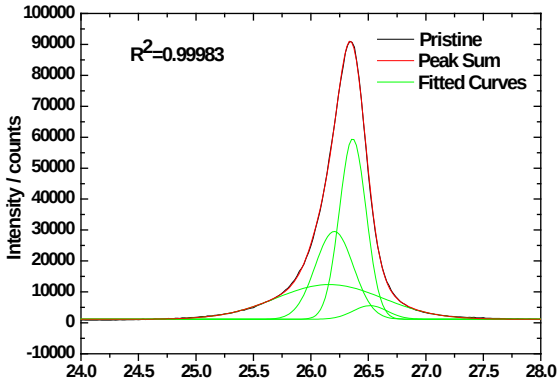

Fig. 2 Fitting curves of 002 diffraction lines

Reference

[1] Takenaka A., Tsumura T., Toyoda M., Synthesis of ternary intercalation compounds from pitch based carbon fibers and its application. TANSO, 2008:**233**: 131-135.

[2] Noda T., Inagaki M., et. al. TANSO. 1962:47:14-22.