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Estimation of miniaturized carbon fibers obtained through exfoliation process

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Abstract
Exfoliated PAN based carbon fibers were prepared through electrochemical processing and then rapid heat treatment. Carbon material electrode to obtain the high capacity was examined through estimation of oxygen functional groups by using Boehm titration and TPD analysis. In addition, amount of the edges which become a field of the reaction in the enhancement of capacity was also examined by TPO. The edges area having ExCFs were 110 m²/g.

Introduction
Electric double layer capacitor (ELDC) is used as a peak assist power supply for a main power supply. Porous carbon materials such as activated carbon or activated carbon fibers with large surface area can utilize it for physical adsorption is used as its electrode materials. In recently, it is examined that the nano carbon materials such as CNTs (Carbon nanotube) is applied as a carbon material electrode for obtaining the high capacity. Exfoliated carbon fibers (ExCFs) having unique morphology were obtained through electrochemical treatment and following rapid heat treatment¹-³. It was found that large capacity was recognized, when the ExCFs was applied to the electrode of EDLC⁴-⁵. It is related that ExCFs have suitable pores absorbing ions in electrolyte in spite of small surface area. However, the estimation of the edges of ExCFs which become a field of the reaction has not been examined. In this study, amount of oxygen functional groups which contribute capacity, and amount of the edges will be discussed.

Experimental
Sizing agent on surface of PAN-based carbon fibers was rinsed by acetone through dipping. Its carbon fibers were electrolyzed in 13 mol/dm³ nitric acid electrolyte as an anode. Electrolyzed condition was done to 3600 °C at the 10 A/g in current density. Obtained samples were rinsed by distillated water and then dried for 24 h at room temperature. Their samples were rapid heat treated at 1000 °C for 12 s. Part of ExCFs obtained carried out by re-heat treatment at 2500 °C for 30 min for the improvement of the graphitization. Estimation of oxygen functional groups on the ExCFs surface was
carried out by using Boehm titration\(^6\), and TPD (Temperature Programmed Desorption) connected with gas chromatography and Karl-Fisher aquameter. In addition, amount of edges were estimated by calculating amount of the hydrogen obtained from TPO (Temperature Programmed Oxidation).

**Results and Discussions**

Carbon fibers changed from submicro meter to nano meter size through electrolysis and following rapid heat treatment. Its morphology change is shown in Fig. 1. Oxygen functional groups of the ExCFs obtained by Boehm titration is summarized at Table 1. A lot of oxygen functional groups such as hydroxyl, carboxyl and lactone groups were introduced into ExCFs by heat treatment under air atmosphere, and its amount of hydroxyl and carboxyl groups were 0.39 and 0.45 m mol/g, respectively. In addition, existence of quinone and carbonyl groups were confirmed by CO and CO\(_2\) spectrum analysis of TPD. Amount of edges also were estimated by TPO. Hydrogen atom which existed in the edges of graphite layers assumed at the proportion of 1:1 for carbon atom. From their assumption, their edges were decided at 0.0832 nm\(^2\). Therefore, their edges area having ExCFs and re-heat treated one was 110 m\(^2\)/g and 5 m\(^2\)/g, respectively.

**Conclusions**

There were many oxygen functional groups such as carboxyl, hydroxyl, quinone and carbonyl groups on surface of ExCFs. The edges area having ExCFs were 110 m\(^2\)/g. Therefore, the field of reaction to obtain the high capacity was so large.

**References**