CHARACTERIZATIONS OF CARBON FIBER SURFACES BY DIRECT FLUORINATION

Y. S. Lee, J. H. Kim, H. J. Son[*], B. K. Lee[*], J. S. Rho[*], and W. C. Oh[**]
Dept. of Chemical Eng., Sunchon National Univ., Sunchon 540-742, Korea
[*]Dept. of Fine Chemical Eng. & Chemistry, Chungnam National Univ., Taejon 305-764, Korea
[**]Dept. Of Chemistry, Hanseo Univ., Seosan 356-820, Korea

Introduction

Carbon fibers are widely used as reinforcements in composites, especially in composites with epoxide matrices. They combine a high stress factor and strength with a low density, which has led to their increasing use in high performance construction materials. To improve the fiber matrix adhesion, it is necessary to increase the surface polarity, create more sites for hydrogen bonding and improve the possibility for covalent linking between the fiber material and the surrounding polymer matrix in order to achieve good stress transfer from the matrix material to the filling fiber material[1,2]. Normally, Various methods used for the modification of such non-polar carbon fiber surfaces include oxidation in various plasmas, oxidation in air, electrochemical oxidation of carbon fibers used as anodes in various electrolytes or wet chemical methods, such as boiling in nitric acid[3]. Recently, as an alternative technique, fluorine gas has been used as a surface treatment agent for many kinds of organic and inorganic plastic materials, because of its very high reactivity[4].
In this study, carbon fiber was fluorinated as a function of partial pressure of O_2. and than, physical properties, such as wettability, surface polarity and solid surface tension of fluorinated carbon fiber were estimated.

Experimental

2.1 Starting Materials
Pitch-based carbon fibers were used for the studies without any additional surface sizing ; HT polyacrylonitrile (PAN)-based Carbon fibers from Taekwang (TZ 307)
2.2 Fluorination procedure
Fluorine gas was supplied Messer at 99.8% purity. The impurity was almost only nitrogen, and the amount of hydrogen fluoride was under 0.01%. Trace amount of fluoride in the fluorine gas were removed by sodium fluoride pellets heated at 100□.
2.3 Contact angle measurement
For investigation of the wettability of carbon fiber, we measured the contact angle. since the contact angle θ of a test liquid on carbon fibers is not directly measurable because of the small fiber diameter, a gravimetric method was used to estimate the contact angle to water and to methylene iodide using the DCA analyzer (CAHN DCA 315). Because of the very small mass change during the measurement of a monofilament, we used bundle type for measurement.
2.4 measurement of surface tension
For calculation of the surface tension of carbon fiber, we used the Owens-Wendt-geometric mean method[5] by water and methylene iodide.

$$\gamma_{LV}(1+\cos\theta) = 2(\gamma_S{}^d \cdot \gamma_{LV}{}^d)^{1/2} + 2(\gamma_S{}^P \cdot \gamma_{LV}{}^P)^{1/2} \qquad (1)$$

Include Young Eq.

$$\gamma_{SL} = \gamma_S + \gamma_{LV} - 2(\gamma_S{}^d \cdot \gamma_{LV}{}^d)^{1/2} - 2(\gamma_S{}^P \cdot \gamma_{LV}{}^P)^{1/2} \qquad (2)$$

By use of the equation in (1),(2), the surface tension can be evaluated.

Results and Discussion

We are particulary interested in the influence of surface fluorination on the physical properties, such as wettability, surface polarity and solid surface tension. The effect of fluorination pressure(F_2/O_2=1) on the contact angle of carbon fibers is shown in Fig. 1. As shown in this Fig., contact angle measured against water shows minimum value in the condition of fluorination at 0.05bar and room temperature for 30 min . And then, the contact angles measured against water was increased remarkably again at higher fluorine pressures. The contact angles measured against methylene iodide show a similar tendency accoding to the fluorination pressure. Fig. 2 shows the surface tension change of the fluorinated carbon fibers accoding to the fluorination pressure. The surface tension of carbon fiber fluorinated by the same conditions has maximum value at 0.05bar. This can be explained by the chemical modification of surface due to fluorination at fluorination pressure with 0.05bar. Methylene iodide has a very small polar component of surface tension, so that the polar interaction is small compared with that of water and the major contribution should stem from dispersive interaction. The effect of O_2 Partial pressure on the contact angles and the surface tension of carbon fiber is shown in Fig. 3, 4 respectively. As shown in these Figs., the contact angle shows minimum value at O_2 Partial pressure 0.5 ratios, and the surface tension shows maximum value at O_2 Partial pressure 0.5 ratios, also. These changes of surface property are due to increase surface polarity of carbon fiber by fluorination.

Conclusion

[1]. Wettability of fluorinated carbon fiber is dependent upon partial pressure of O_2.
[2]. Fluorination of carbon fiber is optimized in mixture gas ratio 5:5 and gas pressure 0.05bar.
[3]. Surface property of carbon fiber was improved by slight fluorination.
[4]. Change of wettability due to increased polarity of fluorinated carbon fiber.

Reference

[1]. Ruland W, Adv. Mater., 2(1990) 528.
[2]. Toit F J, and Sanderson., R. D., J. of Fluorine Chem., 98(1999) 107.
[3]. Bismarck A, Tahhan A, et al, J. of Fluorine Chem., 84 (1997) 127.
[4]. Chong YB, and Ohara H, J. of Fluorine Chem., 57 (1992) 169.
[5]. Fujimoto H, Maeda T, and Yoshikawa M, J. of Fluorine Chem., 60(1993) 69.

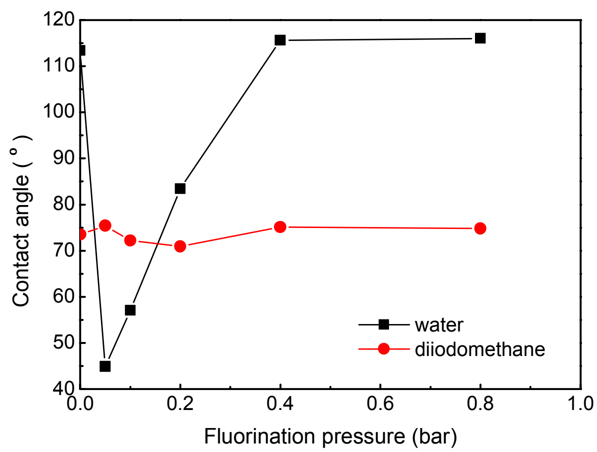

Fig. 1. Contact angle of fluorinated carbon fiber as a function of fluorination pressure (F_2/O_2=1, fluorination time = 30min).

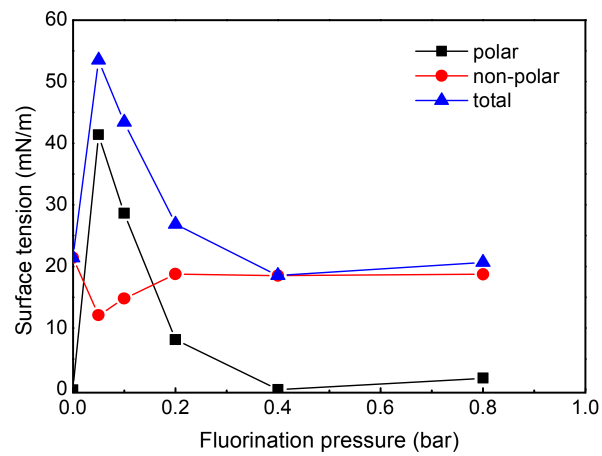

Fig. 2. Surface tension of fluorinated carbon fiber as a function of fluorination pressure (F_2/O_2=1, fluorination time = 30min).

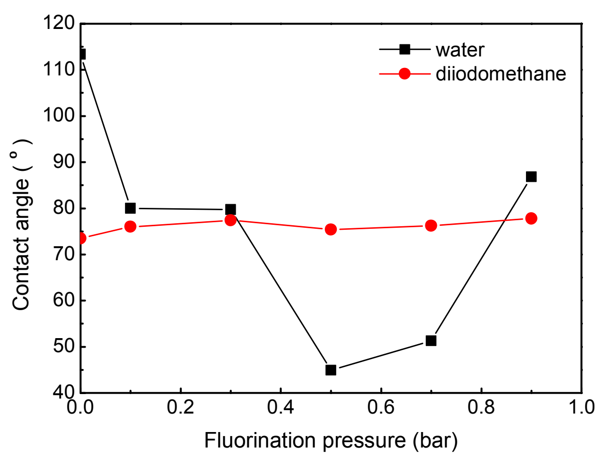

Fig. 3. Contact angle of fluorinated carbon fiber as a function of O_2 partial pressure (fluorination pressure = 0.05bar, fluorination time = 30min).

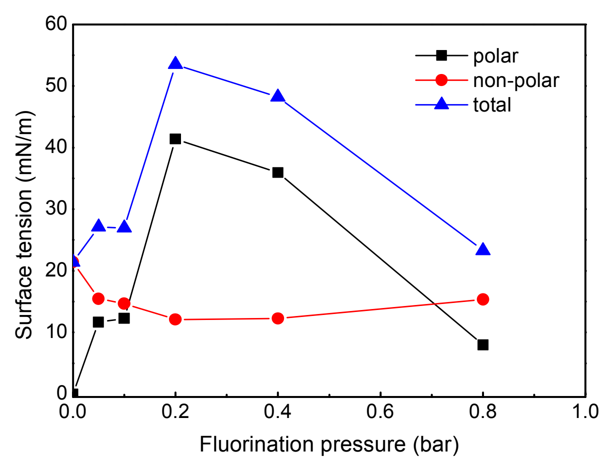

Fig. 4. Surface tension fluorinated carbon fiber as a function of O_2 partial pressure (fluorination pressure = 0.05bar, fluorination time = 30min).