PHYSICAL PROPERTIES OF ACTIVATED CARBON AEROGELS

Y. Hanzawa and K. Kaneko
Physical Chemistry, Material Science, Graduate School of Science and Technology, Chiba University, 1-33 Yayoi-cho, Inage-ku, Chiba 263, Japan

Introduction

Carbon aerogels prepared by Pekala et al [1] have the monolith form and their physical properties have been studied by Dresselhaus et al [2]. This carbon aerogel has a network structure of primary carbon particles, providing predominant mesopores. These carbon particles have slight micropores. The network structure and the size of the primary particle can be controlled by the sol-gel chemistry. If we can donate a uniform microporosity to the mesoporous carbon aerogel without change of the skeletal carbon gel structure, a promising new carbon having a wide variety of functions can be obtained. However, there are few examples of activated carbon having a bimodal distribution in the micropore and mesopore ranges [3]. This report describes the pore structure and physical properties of activated carbon aerogels.

Experimental

Carbon aerogel was prepared by the Pekala method. Resorcinol-formaldehyde (RF) gels were derived from the sol-gel polymerization of resorcinol and formaldehyde with a slight amount of sodium carbonate as a basic catalyst. The molar ratio of resorcinol (R) to catalyst (C) was held at R/C=200. The RF aerogels were dried under the supercritical condition with CO_2, followed by the carbonization under N_2 flow at 1323 K. The resultant vitreous black monoliths are carbon aerogels, which are denoted by CA in this work. The activation of carbon aerogels under CO_2 flow was carried out at 1173 K. The activated carbon aerogel is designated a-CA-x. Here, x is the activation time at 1173 K.

The adsorption isotherm of nitrogen was measured at 77K. The dc electrical conductivity of the carbon aerogel sample was measured using evaporated Aluminum electrodes. The magnetic susceptibility was measured by use of SQUID system in the temperature range of 2-300K.

Results and Discussion

The N_2 adsorption isotherms are shown in Figure 1 for various values of the activation time at 1173 K in a stream of CO_2. The longer the activation time, the greater the amount of adsorption. In particular, the uptake at relatively low pressure (P/P_0) noticeably increases with increasing activation time. The adsorption isotherms have a characteristic adsorption hysteresis. The hysteresis loops of non-activated carbon aerogel (CA) and a-CA-5 are of type H1, coinciding with the network structure of agglomerates of uniform spherical particles. Activation for longer times tends to change the loop shape from type H1 to H2, suggesting the decrease of the primary particle size and a partial opening of the network structure. However, even such an activation treatment does not change the basic network structure and the activated carbon aerogel in a monolith from has both micropores and mesopores. Both of microporosity and mesoporosity were separately evaluated using the subtracting pore effect (SPE) method for the high resolution α_S-plots [4, 5]. As the activation process progresses, the total surface area remarkably increases up to 2600 $m^2 \cdot g^{-1}$. Both the

micropore surface area and micropore volume increase with the activation time until 5 h and then become almost saturated over 5 h. Figure 2 shows the temperature dependence of the dc electrical conductivity of CA and a-CA-5. The electrical conductivity of a-CA-5 is lower than that of CA in the whole range. Although the log σ vs. T^{-1} relation of CA is almost linear, that of a-CA-5 is not simple. The activation energies of both samples are in the order of meV, which is smaller than that of activated carbon fiber (ACF). The electrical conduction should be carried by thermal hopping conduction in carbon aerogels. The micrographitic units of CA should be smaller than those of ACF and activated CA should have smaller micrographitic units than CA. The Mott plot of log σ vs. $T^{-1/3}$ was constructed for both samples. However, the plots were not good linear. The electrical conductivity behaviors will be discussed. The preliminary SQUID measurement of a-CA-5 suggested the random magnetism.

Acknowledgment

This work was partially supported by a Grant-in-Aid for Scientific Research on Priority Areas by the Ministry of Education, Science and Culture, Japanese Government.

References

1. Pekala, R.W., Alviso, C.T., Kong, F.M. and Hulsey, S.S., *J. Non-Cryst. Solids*, **1992**, *145*, 90.
2. Fung, A.W.P., Wang, Z.H., Dresselhaus, M.S., Dresselhaus, G., Pekala, R.W. and Endo, M., *Phys. Rev. B*, **1994**, *49*, 17325.
3. Ghosal, R., Kaul, D., Boes, U., Sanders, D., Smith, D.M. and Maskara, A., *Mater. Res. Soc. Symp. Proc.*, **1995**, *371*, 413.
4. Kaneko, K., Ishii, C., Ruike, M. and Kuwabara, H., *Carbon*, **1992**, *30*, 1075.
5. Hanzawa, Y., Kaneko, K., Pekala, R.W. and Dresselhaus, M.S., *Langmuir*, **1996**, *12*, 6167.

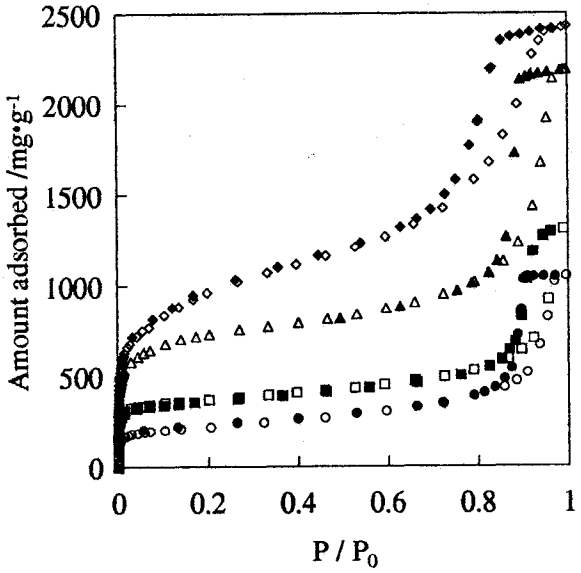

Figure 1. Adsorption isotherms of nitrogen at 77K on activated carbon aerogels. Solid symbols denote desorption. (circle), CA; (square), a-CA-1; (triangle), a-CA-5; (diamond), a-CA-7.

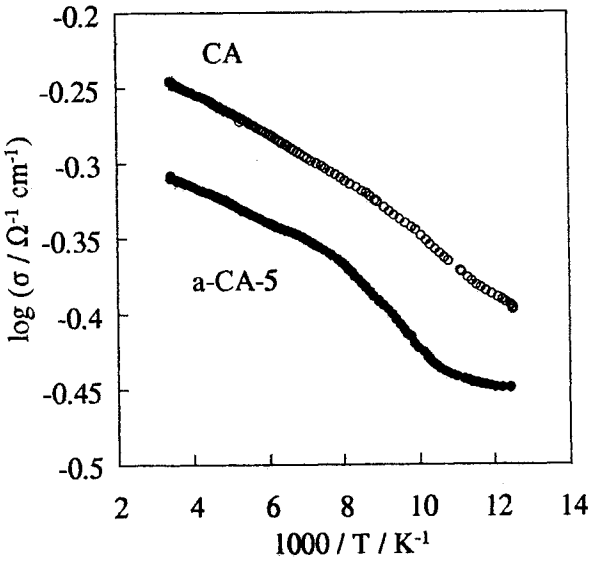

Figure 2. The $T^{-1/3}$ dependence of dc electrical conductivity of carbon aerogels. (open circle), CA; (closed circle), a-CA-5.