Electrothermal properties of regenerable carbon contained porous ceramic fiber media

**Sung Park · Young-Pil Kwon · Hyuck-Chon Kwon ·
Ju-Hyeon Lee · Hae-Weon Lee · Jae Chun Lee**

Abstract Electrically regenerable carbon contained porous
ceramic fiber media have been prepared for adsorption/
regeneration system. The effect of carbon coating methods
on the electrothermal properties of the media was studied
by investigating electrical resistivity, and adsorption and
electrothermal desorption behavior of gaseous adsorbates.
At the similar levels of carbon content, the electrical
resistivity of the media produced from mixtures of ceramic
fiber and phenolic resin powder was almost one order of
magnitude lower than the value of those produced by
infiltration of liquid phenolic resin into porous ceramic
body. In situ thermal desorption and adsorption experiments
show that the adsorption abilities of the media apparently
depended on the Brunauer-Emmett-Teller surface area of
carbon.

Keywords Ceramic fiber · Carbon · Joule heating ·
Adsorption · Electrothermal desorption · BET surface area

1 Introduction

Advanced ceramic materials are often required to accomplish multi-function performance. Carbon-coated porous
ceramic monoliths have a range of potential applications in
the areas of filtration [1], hazardous gas adsorption [2, 3],
active heater and thermal insulation [4, 5]. Electrically
conductive nanoporous carbon coated on ceramic fibers has
an adsorption capability for hazardous gases and the
regenerability by Joule heating. Many of studies regarding
electrically carbon adsorbents and heaters found in the
literature concern the electrical and electrothermal properties of the carbon monoliths and not the carbon-coated
ceramic monoliths [6, 7]. Because of the diversity of
physical and electrical properties of the ceramic media
containing carbon due to the fabrication conditions [3–5], it
is necessary for the design of electrically regenerable
adsorption unit to evaluate the electrothermal desorption
behavior of these materials. The objectives of this study is
to compare and characterize the adsorption and electrothermal desorption behavior of regenerable fiber media containing carbon adsorbents prepared by different process
routes. Thus, in this paper, results of electrical resistivity
and electrothermal desorption measurements are presented
for two different types of carbon contained ceramic media.
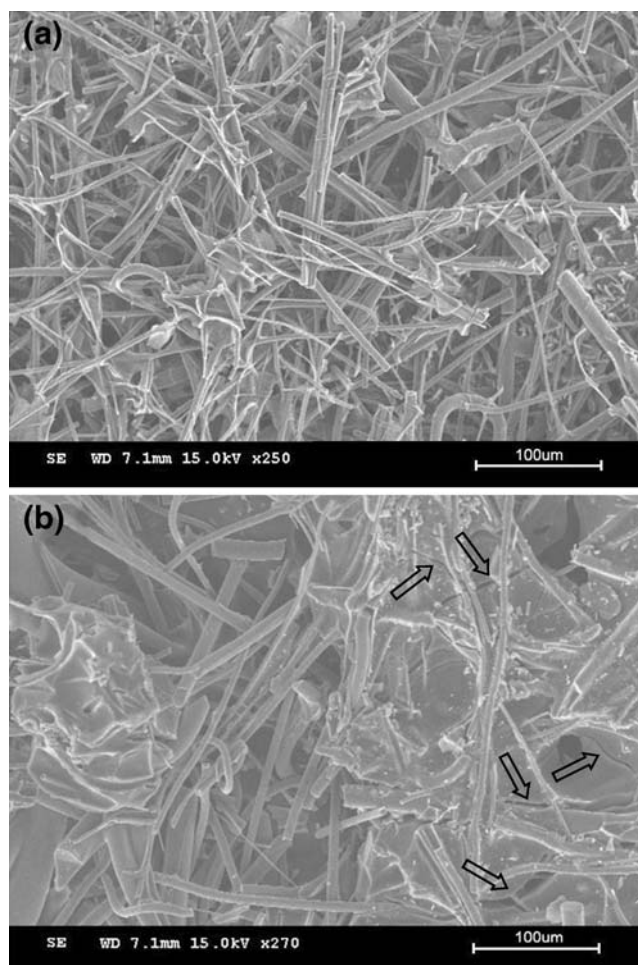
2 Experimental

Non-conductive alumino-silicate ceramic fibers chopped to
a few millimeters in length were used as a support for
carbon. Two sets of carbon contained samples were
prepared, one using slurry mixing method and the other
via resin infiltration process. In samples prepared by the
first method, chopped ceramic fibers were mechanically

S. Park · Y.-P. Kwon · H.-C. Kwon · J. C. Lee (✉)
Department of Materials Science and Engineering,
Myongji University,
Yongin, Kyunggi-do 449-728, South Korea
e-mail: jcl@mju.ac.kr

J.-H. Lee
Department of Electronic Materials Engineering.,
Sunmoon University,
Asan, Choongnam 336-708, South Korea

H.-W. Lee
Nanomaterials Research Center,
Korea Institute of Science and Technology,
Seoul 136-791, South Korea

slurried in water, and phenolic resin powder, organic and inorganic binders were added to the water–fiber mixture. The weight ratio of the phenolic resin to the ceramic fiber was varied from batch to batch. The slurry was molded into disc shape of about 50 mm diameter and 10 mm thickness using a vacuum forming process described elsewhere [3]. To convert the resin to nanoporous carbon the resin coated porous composite media were pyrolysed at 800 °C for 1 h in flowing N_2, followed by steam activation at 850 °C with various hold times [6]. In this way, porous ceramic fiber media containing various amounts of nanoporous carbon with different specific surface area were obtained. In samples obtained using the resin infiltration process, phenolic resin solutions of various concentrations were infiltrated into the open pores of the porous ceramic fibrous media. The resin coated samples were subsequently pyrolysed at 800 °C for 1 h in nitrogen under atmospheric pressure to convert the resin to pyrolytic carbon.

A typical bar sample of ~9 mm thickness, 31 mm width and 33 mm length L with a rectangular cross section S was cut from disk samples with a bulk density of 0.26~0.34 g/cm³. The electrical resistance R of the carbon contained sample was measured by a two-point method using Hewlett-Packard 34401A multimeter. The ends of the sample were completely coated with silver paint to promote consistently good electrical contact with the probes. The electrical conductivity $\sigma = (1/R)(L/S)$ was calculated by taking the value of resistance and dimensional measurements. The temperature rise of the sample was measured as a function of time during constant voltage application (Model HP 6010A DC Power Supply) by using a K-type thermocouple located in the middle of the top surface of the sample. The temperature dependence of the volume electrical resistivity for the sample was measured using a shunt resistor. Adsorption and electrothermal desorption behavior of gaseous adsorbates such as water molecules in air and ethyl acetate was investigated by in situ measurement of the sample weight during Joule heating using an electronic balance (Model Ohaus Explorer) with a resolution of ±0.1 mg. The electrothermal desorption experimental apparatus consisted of an electronic balance, a sample


Fig. 1 Micrographs of carbon contained ceramic fiber media prepared by (a) slurry mixing and (b) resin infiltration method. Microcracks are indicated by *arrows*

connected to a DC power supply and a data acquisition unit [8]. The Brunauer-Emmett-Teller (BET) specific surface area and pore volume of samples containing carbon were calculated by N_2 adsorption. Carbon content of samples was

Table 1 Electrical and physical properties of carbon contained porous ceramic fiber media.

Designation	S1	S2	S3	S4
Carbon content (mg/g sample)	25	192	106	216
BET area (m²/g carbon)	2,368	1,145	267	48
Resistance (Ω) at 23 °C	19.5	2.0	15	16
Electrical resistivity (Ω·cm) at 150 °C	10.6	0.9	14.4	11.1
Bulk density (g/cm³)	0.26	0.34	0.30	0.28
Porosity (%)	91	87	90	91
Surface area (m²)	143	476	267	144
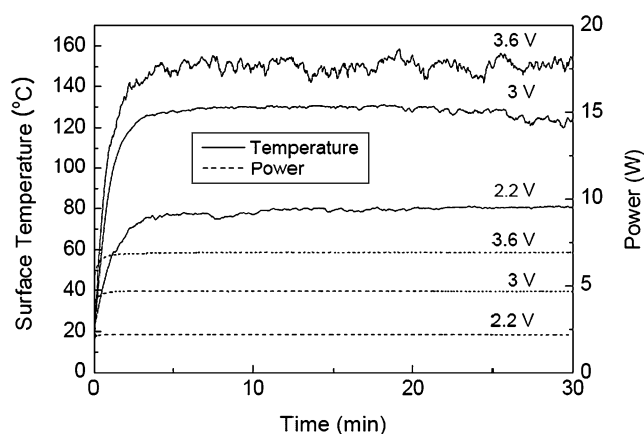
Fig. 2 Surface temperature rising and power input of S2 sample as a function of heating time for three different applied voltages, 2.2, 3 and 3.6 V
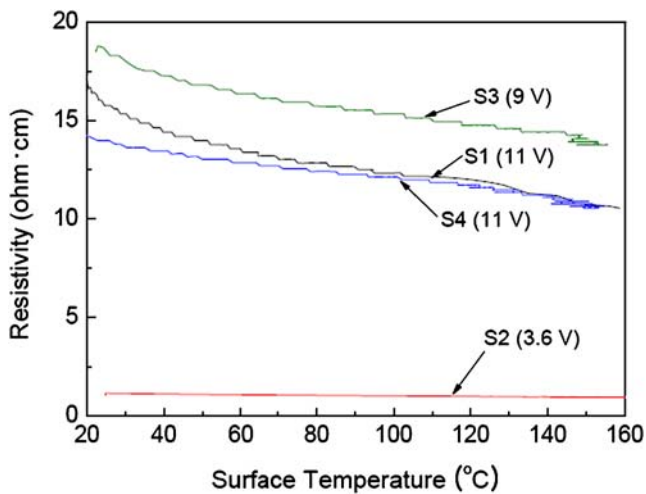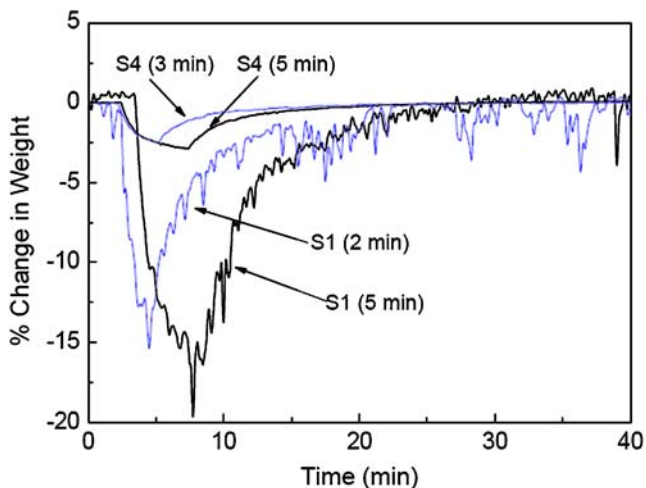
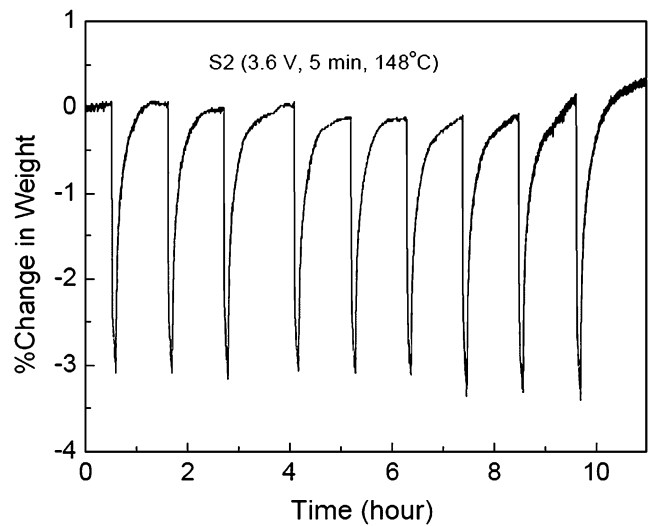**Fig. 3** Electrical resistivity of carbon contained samples as a function of the surface temperature

Fig. 5 Percent change in weight of electrothermally cycled S2 sample. During each cycle, heating and cooling times were 5 min and 1 h, respectively

estimated by weight change after burn off of carbon in air at 800 °C for 1 h.

3 Results and discussion

The measured electrical and physical properties for S1, S2, S3 and S4 samples are compared and shown in Table 1. S2 sample has the lowest electrical resistivity value of 0.9 Ω·cm at 150 °C. On the other hand, S1 sample contains nanoporous carbon which shows the highest BET surface area of 2,368 m^2 per gram of carbon. This value is more than two and 49 times larger than that for the S2 and S4 samples, respectively. Two samples, S1 and S2, were

prepared using slurry mixing method while S3 and S4 were obtained using the resin infiltration process. The electrical resistivities of the S1 and S2 are lower than those of the S3 and S4 although the electrically conductive carbon content of the former is less than or similar to that of the latter. The higher electrical resistivity shown by S3 and S4 samples could be due to the presence of a significant number of microcracks in the carbon layer as shown in Fig. 1. Apparently, the resin infiltrated process produced more cracks than the slurry mixing method.

Figure 2 shows the change in temperature and power with time for S2 sample with different applied voltages. For a given voltage, the temperature increases with time, since the power increases, but it levels off gradually. As expected, a higher power gives a higher temperature at the same time
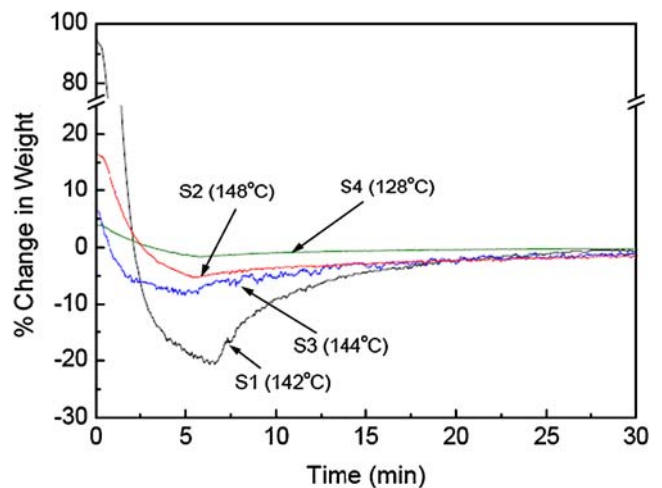

Fig. 4 Percent change in weight of electrothermally heated S1 and S4 samples as a function of Joule heating time. The weight change is expressed as weight percent of gas desorbed or adsorbed based on the total mass of carbon in the sample. Three different heating times, 2, 3 and 5 min, were employed. Preadsorbed species were water vapor from ambient air. Applied voltages were 11 V for S1 and 9 V for S4, respectively

Fig. 6 Electrothermal desorption/adsorption curves for S1, S2, S3 and S4 samples initially saturated with ethyl acetate vapor. The value in *parenthesis* denotes the maximum surface temperature of the sample during Joule heating

of heating. At the power of 6.86 W (3.6 V, 1.91 A), the surface temperature rises to about 150 °C in 5 min. In Fig. 3, electrical resistivities of carbon contained samples in Table 1 are drawn as a function of the surface temperature. A different voltage was applied on each sample to reach the maximum surface temperature of about 150 °C. In this way we can compare the electrical resistivity between samples and show characteristics of the electrical resistivity dependent on temperature more clearly. Results of measured electrical resistance of all samples showed a decrease in resistivity with increasing temperature. For example, electrical resistivity for S3 sample in Fig. 3 decreased from 18.0 to 13.8 Ω·cm with increasing surface temperature from 23 to 144 °C. It has been known that the electrical resistivity of carbon decreases with increasing temperature [6, 7].

Figure 4 shows the typical dependence of the percent change in weight of electrothermally heated samples on the time for different heating periods, i.e., sample temperatures. The weight change was expressed as weight percent of gas desorbed or adsorbed based on the total mass of carbon in the sample. Two different sets of desorption/adsorption curves for the S1 and S4 samples at ambient condition are shown in Fig. 4. The first decreasing curve for the S4 shown on the left side of Fig. 4 resulted from desorption of adsorbates by Joule heating with an applied voltage of 9 V for 3 min. The primary adsorbate is presumed to be water vapor initially adsorbed to the sample exposed to ambient air. Desorption of adsorbates resulted in decrease of the sample weight. The increase in heating time from 3 to 5 min increased the weight loss of S1 from 2.5% (8.5 mg) to 3.0% (9.7 mg) due to sample temperature increase from 123 to 128 °C. Such an increase with increasing Joule heating is shown by another curves plotted for the S1 in Fig. 4 and again attributed to the increased thermal energy available for desorption. The increase in weight after the power is switched off occurs immediately and is completed usually within 30 min. This is due to regain of presumably water vapor from ambient air. It is noted that the water vapor adsorption capacity of S1 is six times higher than that of S4, due to its higher BET surface area of contained carbon. Moisture present in the pore of carbon adsorbent is known to reduce adsorption efficiency.

A series of nine experiments were conducted for S2 sample to investigate the effect of repeated cycles of desorption and adsorption on water vapor in ambient air. Results from this series of experiments are drawn in Fig. 5. By Joule heating with an applied voltage of 3.6 V for 5 min, the sample temperature rose to about 150 °C, and desorption of adsorbed water resulted in the weight loss of about 3% of carbon weight in S2. Water vapor from ambient air was re-adsorbed for 1 h after the power was switched off. Results shown in Fig. 5 clearly indicate that

the carbon contained ceramic fiber media in this work is electrothermally regenerable.

Figure 6 shows four different desorption/adsorption curves for S1, S2, S3 and S4 samples initially saturated with ethyl acetate vapor. In this figure it should be noted that the change in weight of all samples is not equals zero for t=0 since their initial weight represents the sum of their own weight plus the adsorbed ethyl acetate. The adsorption capacity of each sample was different from each other. For example, as shown in Fig. 6, the amount of ethyl acetate vapor preadsorbed on S1 sample is almost 15 times higher than the one on S3 sample. As shown in Table 1, BET surface area of S1 has nine times larger than that of S3 due to activation treatment. Therefore, it can be understood that the adsorption capacity of S1 is much higher than that of S3. By Joule heating, ethyl acetate was desorbed first, followed by preadsorbed water as shown in Fig. 6. As expected, the increase in weight after power-off occurred immediately and was completed usually within 30 min as mentioned earlier.

4 Conclusion

Two sets of carbon contained ceramic fiber media were prepared, one using slurry mixing method and the other via resin infiltration process. The higher values of electrical resistivity of the media prepared by the latter can be explained by the existence of microcracks in coated carbon. Results of multiple adsorption and electrothermal desorption experiments confirmed the regenerability of the carbon contained ceramic fiber media. The adsorption capacity of the media depended on the BET surface area of carbon.

References

1. Y. Li, T. Nomura, A. Sakoda, M. Suzuki, J. Membr. Sci. **197**, 23 (2002)
2. C.L. Mangun, J.A. DeBarr, J. Economy, Carbon, **1689**, 39 (2001)
3. J.C. Lee, H.J. You, H.S. Lee, M.C. Shin, J.S. Cha, S. Park, Colloids Surf. A Physicochem. Eng. Aspects, **241**, 185 (2004)
4. J.C. Lee, J.S. Yu, J.H. Sung, S. Park, S.C. Choi, Key Eng. Mater. **317–318**, 491 (2006)
5. J.C. Lee, H. Kwon, Y. Kwon, S. Park, H. Lee, J. Lee, J. Kim, J. Electroceram. **17**, 1109 (2006)
6. A. Subrenata, J.N. Bale'oa, P. Le Cloirea, P.E. Blanc, Carbon, **39**, 707 (2001)
7. L. Luo, D. Ramirez, M.J. Rood, G. Grevillot, K.J. Hay, D.L. Thurston, Carbon, **44**, 2715 (2006)
8. S. Park, Y. Pil Kwon, H.C. Kwon, J.H. Lee, H.W. Lee, J.C. Lee, J. Nanosci. Nanotechnol., **7**, 3776 (2007)