

Research Article

Electric Double-Layer Capacitor Fabricated with Addition of Carbon Nanotube to Polarizable Electrode

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Electrical double-layer capacitor (EDLC) was fabricated with addition of carbon nanotube (CNT) to polarization electrodes as a conducting material. The CNT addition reduced the series resistance of the EDLC by one-twentieth, while the capacitance was not increased by the CNT addition. The low series resistance led to the high electrical energy stored in the EDLC. In this paper, the dependence of the series resistance, the specific capacitance, the energy, and the energy efficiencies on the CNT addition is discussed.

1. Introduction

Electric double-layer capacitors (EDLCs), supercapacitors, or ultracapacitors have the same type of electrical energy storage device, which is based on a double electric charge layer effect [1, 2]. Commercial EDLCs recently have capacitance value up to thousands F and the power density up to thousands W/kg, which is higher than that of conventional batteries (<1000 W/kg). Higher capacitance and power density allow higher energy to be stored and discharged at higher rate. Moreover, EDLCs have longer cycle lives exceeding 500000 times. These features have generated great interest in the application of EDLCs such as consumer electronics, hybrid electric vehicle [3], and industrial power managements. On the other hand, the energy density of EDLCs is below 10 Wh/kg, which is lower than that of batteries (35–40 Wh/kg of lead-acid batteries, ~150 Wh/kg of lithium ion batteries). The electrical energy (L_{stored}) stored in the EDLC is calculated according to the following equation:

$$L_{\text{stored}} = \frac{1}{2} CV^2, \quad (1)$$

where C is capacitance and V is voltage. This equation indicates that an increase in the capacitance leads the electrical energy stored in the EDLC to be high.

The capacitance of the EDLC is theoretically proportional to surface area (S) of polarizable electrodes, as based on the following equation:

$$C = \int \frac{\epsilon_0 \epsilon_r}{d} S, \quad (2)$$

where ϵ_0 and ϵ_r are permittivity in vacuum and relative permittivity of the double layer, respectively, and d is thickness of double layer. Therefore, activated carbon is widely used as a constituent of polarizable electrode in order to obtain large capacitance, because it has high surface area of over 1000 m²/g.

In addition to the surface area of the polarizable electrodes, the energy density is also gravened by the series resistance of the EDLC, because the charge and the discharge currents are followed in the series resistance. The energy loss (L_{loss}), which is caused by the current flowing in the serial resistance during the charge or the discharge, is estimated using the following equation:

$$L_{\text{loss}} = I^2 R_s \cdot t, \quad (3)$$

where I is the charge or the discharge current, R_s is the series resistance, and t is the charge or the discharge time. In the application of the EDLC to the second power system in an electric vehicle, the discharge for the acceleration and the charge by the deceleration take place over a short

period. Therefore, these charge and discharge currents are of a particularly high value when compared with those of other applications. Higher charge and discharge currents cause the energy loss to be increased. For these reasons, there is a strong requirement for the series resistance to be decreased in order to reduce the energy loss.

A decrease in series resistance (R_s) also allows the power density of EDLC to be increased, because maximum output power (P_{\max}) of EDLCs is theoretically calculated to the following equation:

$$P_{\max} = \frac{V^2}{4R_s}, \quad (4)$$

where V is the voltage. Therefore, the series resistance of EDLCs is also required to be reduced in order to increase the energy and the power densities.

Activated carbon is the base material widely used for the fabrication of polarizable electrodes [4, 5]. However, the activated carbon in itself does not have a high enough electrical conductance to act as a fully suitable material for the polarizable electrodes. An EDLC fabricated exclusively from activated carbon shows a high energy loss during the charge and the discharge processes, because it has high series resistance. Therefore, a conducting material such as acetylene black is generally added to the polarizable electrodes in order to decrease the series resistance.

Carbon nanotube (CNT) is one of the candidate materials for polarizable electrodes of the EDLC. The CNT is tube shaped with a diameter of a few nm, which is expected to be suitable for forming the double layer between the surface and the electrolyte. Moreover, CNTs have a high electrical conductivity [6] and chemical stability. It has been reported that the EDLC fabricated from multiwalled CNT showed high specific capacitance in high range of 4–135 F/g [7, 8]. For single-walled CNT, the specific capacitance of 180 F/g and the power density of 20 kW/kg at the energy density of 7 Wh/kg were reported [9]. Moreover, CNT-based electrodes fabricated by direct synthesis of CNT on the bulk Ni substrates have been reported to have the specific capacitance of 38.7 F/g [10]. However, a comparative investigation of the specific capacitance, achieved with CNTs and activated carbon materials, revealed that activated carbon materials exhibited significantly higher capacitance [11]. On the other hand, the addition of CNT to activated carbon instead of acetylene black or graphite power for the polarizable electrodes also has been studied [12–15].

In this paper, CNTs are added to the polarizable electrode which is made of activated carbon. The CNT concentration in the polarizable electrode is from 0 to 100%. The dependence of the series resistance, the specific capacitance, the electrical energy, and the energy efficiency on the CNT addition is studied.

2. Experimental Conditions

CNT (Ohashi Kasga Tsusho Inc.) and activated carbon (Hohsen Corp.) were used as the base material for the

polarizable electrode. The ratio of the CNT to the activated carbon was varied from 0 (pure activated carbon) to 100% (pure CNT). These electrode materials were mixed in a mortar made of agate. Polytetrafluoroethylene (PTFE) was added to the mixture of CNT and activated carbon as binder at 17% in weight. Acetylene black (Kanto Chemical Co., Inc) was also mixed to some of the polarizable electrodes instead of the CNTs for reference/comparison purposes. The total mass of the polarizable electrode was varied from 30 mg to 120 mg.

The mixture was pressed at 20 MPa and formed into a disk shape with an area of 1 cm² (diameter of 11.5 mm) using a steel mold-type pressure apparatus. The thickness of the polarizable electrode hardly depends on the ratio of the constituents. In this experiment, it was varied from 0.5 to 2 mm by increasing its weight from 30 to 120 mg.

The constituents of the polarizable electrode were characterized by the scanning electron microscope (SEM) (Hitachi High-Technologies Corp., HF-2200TU and S-4800). The capacitance measurements were performed using a symmetrical cell configuration (a two-electrode system). The platinum plates were used as a collector electrode, because contact resistance between the polarizable electrode and the platinum plate is lower than any other metal such as stainless steel. A pair of polarizable electrodes was set up in a glass beaker or a can cell to provide both an anode and a cathode. An organic solvent (1.5 M SBP-BF₄/PC, Japan Carlit Co., Ltd.) was used as the electrolyte. This electrolyte has high electrical conductivity of 16 mS/cm.

The constant current charge-discharge measurement was carried out at 5 mA in the voltage range between 0.1 and 1.2 V using the source meter (Advantest Corp., R6243). The voltage change during the charge-discharge was measured. The series resistance and the capacitance of the EDLC were estimated from the voltage change by the mathematical fitting method described in Result and Discussion.

3. Result and Discussion

Figure 1 shows SEM images of (a) activated carbon (AC), (c) carbon nanotube (CNT), and (e) acetylene black (AB), which are constituents of the polarizable electrodes. The activated carbon was a particle with a size of 2–3 μm. A high-resolution SEM image shows that the activated carbon contains a great number of mesopores on its surface. The nitrogen adsorption measurements indicated that the activated carbon was 2000 m²/g in the specific surface area. The CNT was cylindrical-shaped material as shown in Figure 1(c). The diameter and the length were approximately 10 nm and 100 μm, respectively. No traces of the metal catalyst, which had been used for CNT growth, were found at the tips of the CNT in this SEM image. Furthermore, very little amorphous carbon, which is a byproduct of the CVD process, was found. This SEM investigation suggests that the CNT used is well purified by the post-acid washing process used after the CNT growth. The acetylene black used was a particle with a diameter of 50–60 nm, as shown in Figure 1(e).

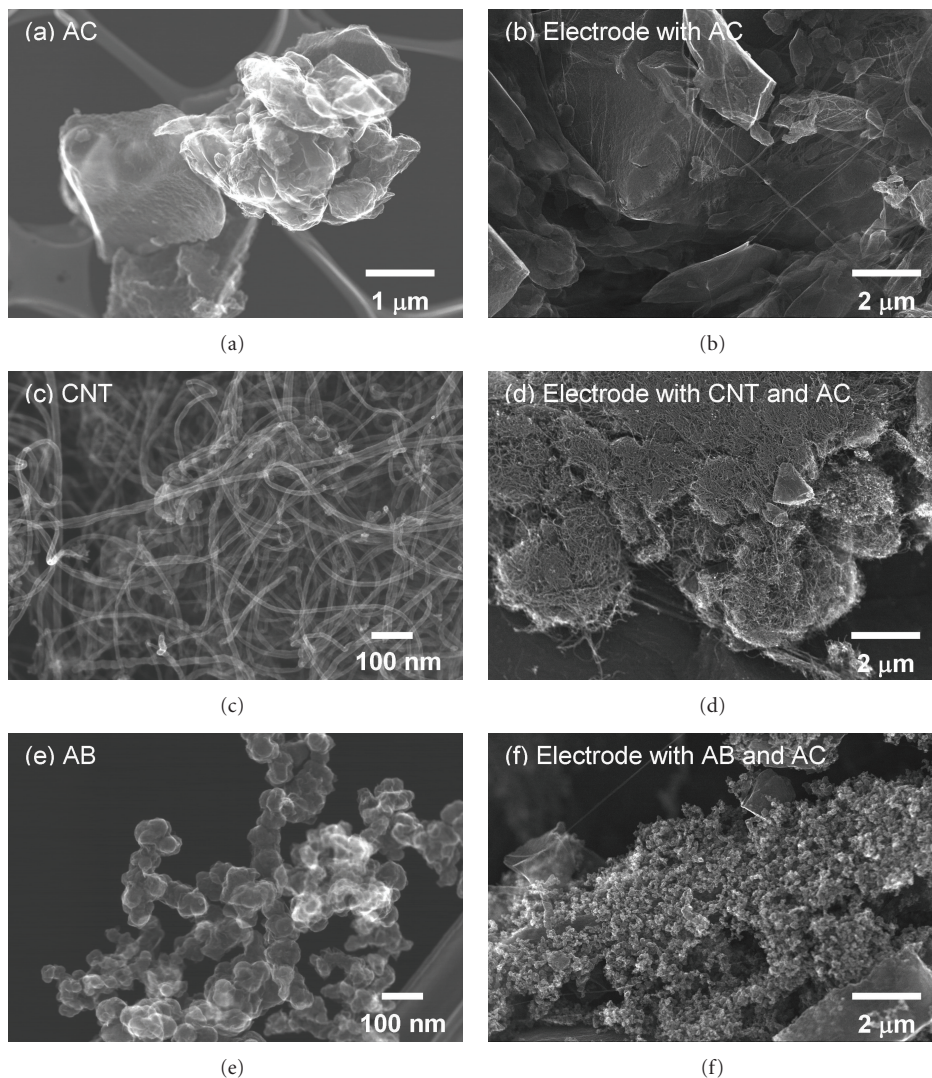


FIGURE 1: SEM images of (a) activated carbon (AB), (c) carbon nanotube (CNT), and (e) acetylene black (AB) as constituents of the polarizable electrodes. SEM images of polarizable electrodes fabricated from (b) exclusively activated carbon, (d) CNT and activated carbon, and (f) acetylene black and activated carbon.

Figure 1 also shows SEM images of polarizable electrodes fabricated from (b) exclusively activated carbon, (d) CNT and activated carbon, and (f) acetylene black and activated carbon. The concentration of CNT or acetylene black in polarizable electrode is 20%. Particles of activated carbon are stacked each other by the polytetrafluoroethylene (PTFE), which was added as binder as shown in Figure 1(b). Thin fiber-shaped PTFE was observed in this image. On the other hand, the activated carbon particles were covered with the CNT in the polarizable electrode containing CNT, as shown in Figure 1(d). The CNTs kept their long shape and were intertwined on many particles of the activated carbon in the polarizable electrode. The SEM image of the acetylene black in Figure 1(f) shows their particles are distributed on the activated carbon surface.

Figure 2 shows the charge-discharge curves of EDLCs fabricated with the following three types of polarizable electrodes: (a) those of exclusively activated carbon (no

CNT) and those containing (b) 10 and (c) 50% CNTs. Those polarizable electrodes were fixed at the weight of 60 mg. The charge-discharge measurements were carried out at a constant current of 5 mA. A rapid voltage change at the initial stage of the charge and the discharge, which is called the IR drop, was observed for all types of EDLCs. The IR drops of 0.45 V and 0.04 V were observed for (a) the EDLC without and (b) the EDLC with the 10% CNT addition, respectively. At 0.04 V the IR drop of the EDLC containing CNT at 10% was lower than that of the EDLC fabricated without using CNT.

The voltage gradually increased with an increase in the charging time after the initial stage of the charge. The inclination of the voltage changes for the EDLC without and EDLC with the 10% CNT was almost the same. This result suggests that these EDLCs have almost the same electric capacitance. However, the voltage of the EDLC without the CNT addition reached 1.2 V in a shorter time than that of

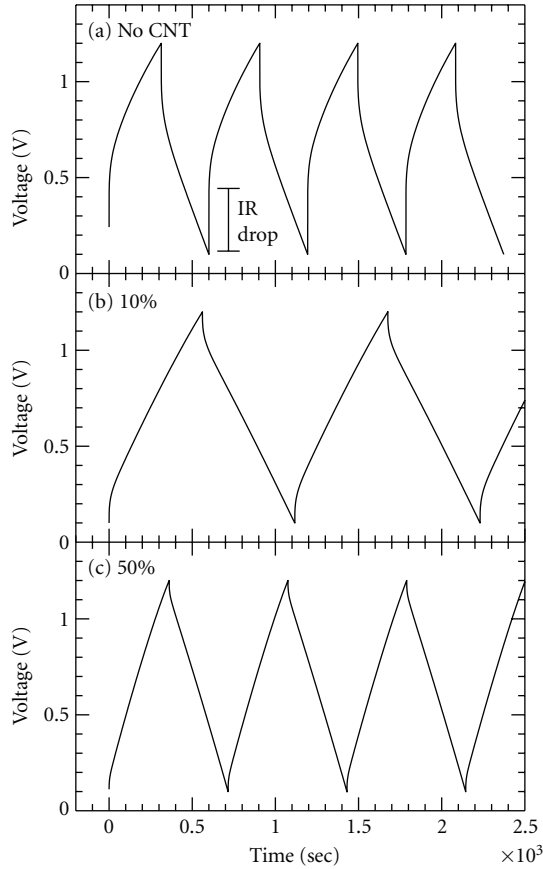


FIGURE 2: The charge-discharge curves of EDLCs fabricated with the following three types of polarizable electrodes: (a) those of exclusively activated carbon (no CNT) and those containing (b) 10 and (c) 50% CNTs.

the EDLC with the CNTs, because the IR drop is bigger. One cycle of the charge and the discharge on the EDLC without the CNT addition was 600 s. On the other hand, that of the EDLC with the CNT addition was 1200 s. A longer cycle time indicates that a higher amount of electric energy is stored in the EDLC, because this measurement is carried out using a constant current.

The EDLC containing CNT at 50% was 0.02 V in the IR drop, which was lower than that of 10%. However, one cycle of the charge and the discharge on this EDLC was as short as 700 s, because the inclination of the voltage changes was higher than that of 10%. Higher inclination suggests that the EDLC with the 50% CNT concentration is lower than that of 10% in the electrical capacitance.

An equivalent electric circuit model for the EDLCs, shown in Figure 3, was used to analyze the above charge-discharge curves. In this circuit, the equivalent capacitance (C) is originated from the formation of the electrical double layer at the interface between surface of the polarizable electrodes and the electrolyte. Larger surface area of the polarizable electrode generally generates larger capacitance. The parallel resistance (R_p) and the series resistance (R_s) are connected to the capacitance. The parallel resistance

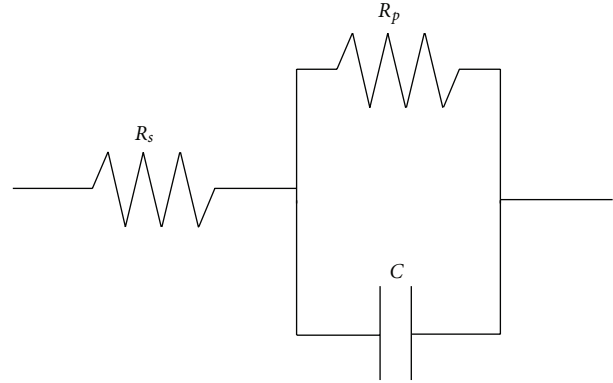


FIGURE 3: An equivalent electric circuit model for the EDLCs.

expresses the current leakage of the electrical double layer, which influences long-term energy storage. The series resistance consists of resistances of the polarizable electrodes and electrolyte, and contact resistance between the polarizable electrode and the collector electrode. The series resistance causes the IR drop and energy loss during the charge-discharge.

The equivalent series resistance (R_s) of the EDLC was estimated from the value of the IR drop according to the following equation:

$$R_s = \frac{V_{IR}}{(I_{charge} - I_{discharge})}, \quad (5)$$

where V_{IR} is the voltage change at the time that the flowing current changes from the discharge current $I_{discharge}$ (negative value) to the charge current I_{charge} (positive value). In this study, the value of $(I_{charge} - I_{discharge})$ is 10 mA. Therefore, the series resistance (R_s) was estimated to be 45, 4, and 2 Ω for the EDLCs without and those with CNT addition of 10% and 50%, respectively. No dependence of the series resistance on the charge-discharge current was observed in the range between 0.5 and 50 mA.

The inclination of the voltage change during the charge-discharge depends on the capacitance of the EDLC. Therefore, the capacitance (C) of the EDLC was estimated using the mathematical fitting technique according to the following equation explaining the relationship between the charging time and the voltage:

$$V(t) = R_p \cdot I_{charge} \left\{ 1 - \exp\left(-\frac{t}{C \cdot R_p}\right) \right\} + V_{IR}, \quad (6)$$

where $V(t)$ is the voltage as a function of the charging time (t), the capacitance (C), and the equivalent parallel resistance (R_p).

The capacitance was estimated to be 2.4 F for both EDLC without and that with CNT at concentration of 10% from the above method. On the other hand, the EDLC with 50% CNT was estimated to be as low as 1.2 F, because the inclination of the voltage change was high and the charge-discharge cycle was short. No dependence of the capacitance on the charge current was observed in the range from 0.5 to 50 mA.

Figure 4(a) shows the dependence of the series resistance on the ratio of CNT to activated carbon in the polarizable electrode. Moreover, those of the EDLC containing the acetylene black, instead of the CNT, are also shown. In this figure, the ratio of 0 means the EDLC is fabricated exclusively with the activated carbon without using the CNT and acetylene black. The series resistance of the EDLC fabricated with exclusively of the activated carbon showed as high as value 45Ω . When the CNTs were mixed with the activated carbon in the polarizable electrodes, the series resistance of the EDLC decreased with an increase in CNT concentration. The EDLC fabricated with a CNT concentration of 17% has a series resistance as low as 2Ω . This series resistance was approximately one quarter of an EDLC fabricated at the same concentration of acetylene black. No series resistance changed with an increase in CNT concentration above 20%. However, it increased to 2.5Ω in the face of the EDLC fabricated from 100% CNT, because clacks were formed in the polarizable electrodes by sinking them in the electrolyte.

The addition of the acetylene black to the polarizable electrode also decreased the series resistance of the EDLC. Both the EDLCs for concentration of the acetylene black at 50 and 100% are 4Ω . No clacks were formed in the polarizable electrodes formed from exclusively acetylene black. However, the series resistance was higher than EDLCs with addition of the CNT.

Figure 4(b) shows the dependence of the specific capacitance (F/g) on ratio of the CNT or the acetylene black to the activated carbon in the polarizable electrode. In this figure, the value was indicated as the capacitance per the weight of polarizable electrode. For example, the EDLC fabricated from exclusively the activated carbon showed the 2.4 F in the capacitance. This EDLC is fabricated with two pieces of polarizable electrode containing carbon materials, such as activated carbon, CNT, or acetylene black (120 mg in total weight). Therefore, the specific capacitance of this EDLC was calculated to be 24 F/g .

No significant change in the capacitance was observed with mixture of the CNT and the acetylene black up to 17%. The capacitance decreased with an increase in their ratio above 20%. The EDLC fabricated exclusively from the CNT showed the capacitance of 4 F/g . This value is higher than that of the EDLC fabricated exclusively from acetylene black. However, these values are extremely lower than that of the EDLC fabricated from exclusively activated carbon.

The specific capacitances for the CNT and the activated carbon in the polarizable electrode were estimated by the mathematical fitting technique according to the following equation:

$$C(x) = C_{\text{CNT}} \frac{x}{100} + C_{\text{AC}} \frac{(100-x)}{100}, \quad (7)$$

where $C(x)$ is specific capacitance for the fabricated polarizable electrode. C_{CNT} and C_{AC} are the specific capacitances for the CNT and the activated carbon, respectively. x is ratio of the CNT to activated carbon. The result of the mathematical fitting is also shown in Figure 4(b).

Table 1 shows the specific capacitance for the CNT, the acetylene black, and the activated carbon estimated by the

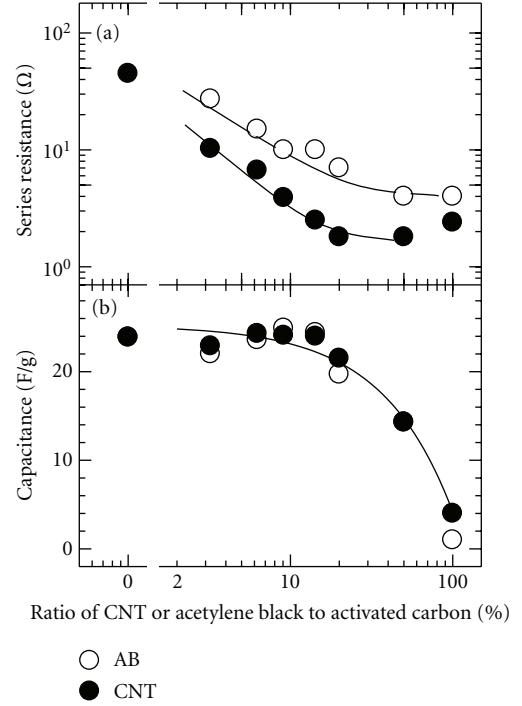


FIGURE 4: (a) The dependence of (a) the series resistance and (b) the specific capacitance on the ratio of the CNT or the acetylene black to the activated carbon in the polarizable electrode.

TABLE 1: Specific capacitance of various materials used for the fabrication of EDLCs.

Material	Specific capacitance (F/g)
Carbon Nanotube	4.5
Acetylene black	1.7
Activated carbon	24

above method. The CNT and the acetylene black were 4.5 and 1.7 F/g in specific capacitance, respectively. The CNT was higher than the acetylene black in the specific capacitance. However, they were lower than the activated carbon (24 F/g).

Figure 5 shows the dependence of (a) the electrical energy and (b) the energy efficiency on ratio of the CNT or the acetylene black to the activated carbon in the polarizable electrode, whose weight is fixed at 60 mg. The electric energy L stored in the EDLC was estimated by the discharge curve ranging between 1.2 V and 0.1 V with a constant current of 5 mA using the following equation:

$$L = \int V(t) \cdot i dt. \quad (8)$$

On the other hand, the energy efficiency η was defined by the ratio between the discharged and the charged electric power using the following equation:

$$\eta = \frac{L_{\text{discharge}}}{L_{\text{charge}}} \times 100, \quad (9)$$

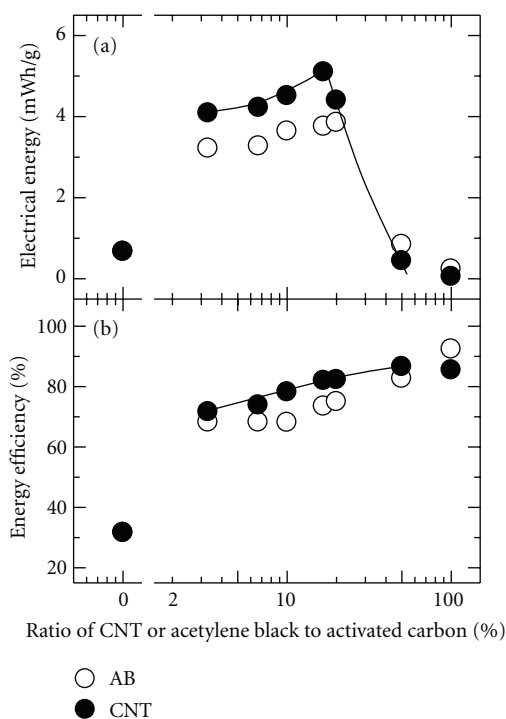


FIGURE 5: The dependence of (a) the electrical energy and (b) the energy efficiency on ratio of the CNT or the acetylene black to the activated carbon in the polarizable electrode.

where L_{charge} and $L_{\text{discharge}}$ are, respectively, the electric power charged and discharged between the voltage of 0.1 V and 1.2 V with a constant current of 5 mA.

The EDLC fabricated using exclusively activated carbon without the addition of any conducting materials showed a low electric energy of 0.7 mWh/g. When the CNTs were added to the polarizable electrodes up to 15%, the electric energy increased up to 5.1 mWh/g according to the increase in CNT concentration. The electric energy was on the contrary decreased by increasing the CNT concentration above 20%. The EDLCs with addition of the acetylene black also have the same trend in the electrical energy. However, the electric energy for EDLCs fabricated with CNTs was higher than that of those fabricated with acetylene black.

A low energy efficiency of 31% was observed for the EDLC fabricated exclusively from activated carbon as shown in Figure 5(b). The energy efficiency was improved by the addition of CNTs to the polarizable electrodes and linearly increased according to the increase in the CNT concentration. A high energy efficiency of 86% was observed for the EDLC with a 50% CNT concentration. The energy efficiency of the EDLCs with addition of the CNT was higher than that of the acetylene black.

Figure 6 shows the dependence of (a) the capacitance and (b) the series resistance on the weight and the thickness of the polarizable electrode in EDLC. The thickness of the polarizable electrode is increased by an increase in its weight, since the polarizable electrode is fixed at 11.5 mm in the diameter. The CNT or the acetylene black in the

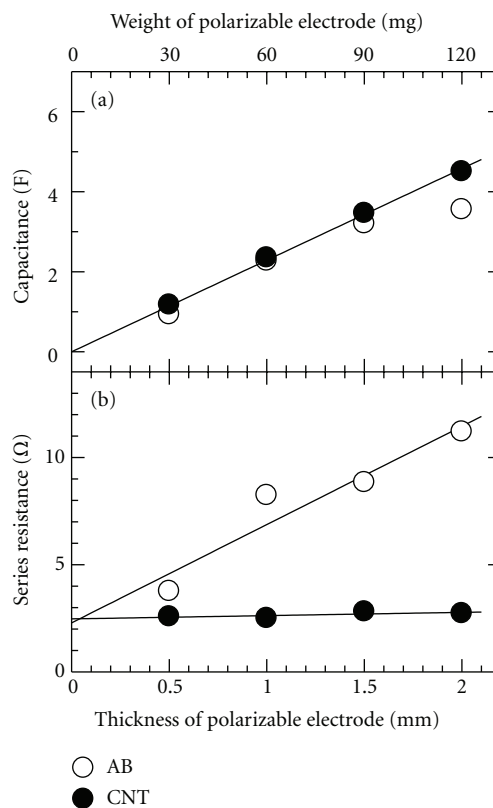


FIGURE 6: The dependence of (a) the capacitance and (b) the series resistance on the weight and the thickness of the polarizable electrode.

polarizable electrodes was fixed at 10% in the weight ratio. Both of the EDLCs fabricated with the CNT or the acetylene black linearly increased from 1.5 to 4.5 F in capacitance with an increase in the thickness of polarizable electrodes. The specific capacitances at various thicknesses were almost of the same value of 24 F/g. The series resistance of the EDLCs added with the acetylene black was increased from 3.7 to 11 Ω with increasing the thickness of polarizable electrode. On the other hand, the series resistance of the EDLCs fabricated with the CNT slightly increased from 2.5 Ω to 2.8 Ω with an increase in the thickness of polarizable electrode. The series resistance at the thickness of 0 mm was estimated to be 2.5 Ω by extrapolation method. The EDLC formed with the acetylene black also showed the same value. These series resistances represent the contact resistance between the polarizable electrode and the counter electrode, because the resistances of the polarizable electrode and the electrolyte are neglected at the thickness of 0 mm. Therefore, these results indicate that the series resistance of the EDLCs fabricated using the CNT are governed by the contact resistance, since the resistance of the polarizable electrode containing CNT is low enough.

Figure 7 shows the schematic model comparing the electrical conduction in the polarizable electrodes with addition of (a) the acetylene black or (b) the CNT. Acetylene black is particle with a diameter of 50–60 nm, which is smaller than activated carbon by a one-30th to a one-60th approximately.

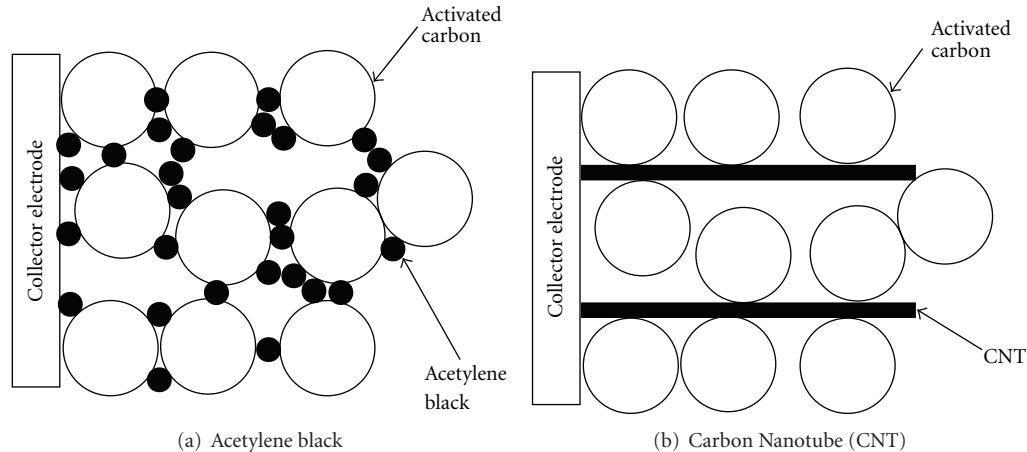


FIGURE 7: The schematic model comparing the electrical conduction in the polarizable electrodes with addition of (a) the acetylene black and (b) the CNT.

The particles of the acetylene black cover activated carbon particles in the polarizable electrode. The charge and the discharge currents flow through a large number of the acetylene black particles on the activated carbon. In this conduction mechanism, the acetylene black particles must connect to each other in order to effectively decrease the resistance of polarizable electrode. Although CNT has cylindrical shape with the diameter of 10 nm which is size a similar to that of acetylene black the length of CNT is as long as 100 μm . Therefore, one piece of CNT connects large number of activated carbon particles, because the length of CNT is longer than activated carbon by approximately 30–50 times. Therefore, CNT forms the electrical conduction mechanism in the polarizable electrode, which has higher conductivity comparing with acetylene black.

Activated carbon is a particle with the specific surface area of 2000 m^2/g . Therefore, it is widely used as a constitution of the EDLC. In this study, the specific capacitance for the activated carbon was 24 F/g. On the other hand, CNT also seems to have large surface area, because it has cylindrical shape with small diameter. The small diameter expects that CNT is more suitable constitution of the EDLC than activated carbon. However, the CNT showed 4.5 F/g in specific capacitance, which was smaller than the activated carbon. Small capacitance in this study suggests that only small surface area of CNT contributes to the double-layer capacitor. One possibility of this reason is the hydrophobic surface of CNT that causes its surface to be hardly wet with electrolyte.

EDLC fabricated from exclusively activated carbon shows high capacitance of 2.4 F. However, the electric energy stored in its EDLC is as low as 0.7 mWh/g, because its series resistance has value as high as 45 Ω . High series resistance loses electric energy during the charge and the discharge and also decreases the energy efficiency. The CNT addition into the polarizable electrode decreases the series resistance of the EDLC up to 2 Ω . Moreover, the EDLC with CNT addition increases to 5.1 mWh/g in the electric energy, because lower series resistance causes lower IR drop and longer charging

time. The small series resistance prevents losing electric energy during the charge and discharge. Therefore, the higher electric efficiency is observed from the EDLC with the addition of CNT. The addition of acetylene black also has the same trend in these characteristics. However, CNT is more effective than acetylene black as a conducting material.

The significant decrease in the capacitance of the EDLC with the addition of CNT was observed above 20%, because the specific capacitance of CNT is lower than that of activated carbon. The decrease of the capacitance leads to the lower stored electric energy. However, the efficiency increases with an increase in the CNT concentration, since the series resistance is decreased.

The CNT addition decreases the series resistance of EDLCs, because of its high aspect ratio. However, the CNT addition above 20% does not decrease the series resistance, because the series resistance becomes governed by the contact resistance between the polarizable electrode and the counter electrode. On the other hand, the capacitance decreases with an increase in the CNT concentration above 20%, because of low specific capacitance of CNT. These results suggest that the CNT is suitable material as the conducting material under the concentration of 20%.

4. Summary

The CNT addition into the polarizable electrodes up to 20% reduces the series resistance of the EDLC from 45 to 2 Ω . The reduction of the series resistance increases the electrical energy stored in the EDLC, and also prevents losing the energy loss during the charge and the discharge. However, the CNT addition does not increase the capacitance, because most of surface area of the CNT does not contribute to the electrical double layer capacitor. The EDLC with the 20% CNT addition shows the capacitance of 24 F/g, which is mainly contributed by activated carbon.

The EDLC with the CNT addition has lower than that of acetylene black in the series resistance. Since the series

resistance of the EDLC was decreased by the CNT addition, the EDLC achieves to increase the electric energy stored in it. Moreover, the energy efficiency for the charge and the discharge is also improved.

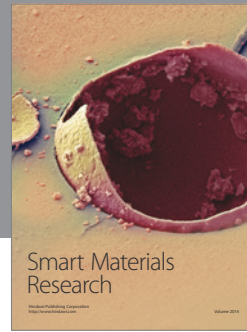
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