In recent years, electrochemical capacitors have attracted increasing attention not only for use in portable electronic devices like smart phones and tablet PCs, but also as large power sources in electric vehicles. The advantages of electric double-layer capacitors (EDLCs) are their high rate capability and semi-permanent long cycle life, which fit the requirements for smart grids and energy harvesting. The favorable performance of EDLCs is attributed to the fast charge/discharge mechanism of activated carbon (AC) electrodes involving an electric double layer regardless of the faradaic reaction (electrochemical reaction). However, EDLCs possess low energy density compared with those of other energy storage devices like Li-ion batteries (LIBs), and nickel–hydrogen batteries.1–4 To overcome this weakness, many hybrid capacitors with asymmetric electrode systems have been studied. Recently, Li-ion capacitors (LICs) using a Li pre-doped graphite or hard carbon negative electrode (NE) have been developed and released as commercial products.5 Figures 1a and 1b outline the principles of an EDLC and LIC with a graphite NE, respectively. In the case of an EDLC, the cell voltage changes linearly during charging and discharging because of the potential change of both AC electrodes. This limits the energy density of the EDLC because of electrolyte decomposition, especially at the positive electrode (PE) side. The cell voltage for the LIC system is much higher than that for the EDLC because the LIC contains a Li pre-doped graphite NE with a low redox potential around −0.02 V, resulting in an energy density ca. three times higher than that of the reference EDLC.6

To extend LICs to applications such as stationary energy storage and electric vehicles, further improvement of their discharge capacity and energy densities is strongly desired. Therefore, many researchers have devoted considerable effort to increasing the performance of LICs by using the other NE materials; e.g., Li4Ti5O12,8–10 and TiO2(B).11–13 Some of these LICs exhibited better performance, especially safety and rate capability, than that of LICs using graphite and hard carbon NEs. However, titanium oxide-type NEs have relatively high redox potentials of around 1.6 V vs. Li/Li+ and lower theoretical capacities (175 and 335 mAh g−1 for LTO and TiO2(B), respectively) than that of a graphite NE (372 mAh g−1). Therefore, the discharge capacity and energy densities of LIC cells do not increase much even if the operating range of the NE is expanded by adjusting the capacity balance between the NE and PE.

Recently, Si NEs have been intensively researched, mainly in the field of LIBs, because their theoretical capacity (ca. 4200 mAh g−1) is higher than that of graphite NEs and their redox potential is suitable for electrolyte decomposition. Therefore, LICs using Si NEs as the negative electrode have been attractively researched for use in hybrid capacitor systems. The advantages of silicon (Si) NEs are their high theoretical capacity (ca. 4200 mAh g−1), low redox potential around −0.3 V vs. Li/Li+, and high energy density of ca. 700 mAh gSi−1, corresponding to a high energy density of 114 Wh kg(NE + positive electrode)−1. The Si pre-doping method also improved the rate capability; >60 Wh kg−1 was attained even at 1.0 mA cm−2 (ca. 2500 mA g(Si)−1), which was three times larger than that of the reference EDLC.

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low (below 0.5 V vs. Li/Li$^+$). However, it is difficult to use pure Si NEs in the LIB systems because of their large volume change during the charge/discharge (i.e., alloying/dealloying) process. This large volume change causes rapid capacity fading originating from crack formation and pulverization of the Si active material and continuous electrolyte decomposition on the newly exposed bare Si surface. To alleviate the physical stress in Si active materials, Si continuous electrolyte decomposition on the newly exposed bare Si surface. To alleviate the physical stress in Si active materials, Si continuous electrolyte decomposition on the newly exposed bare Si surface.

Thin films and thin flakes, as well as three-dimensional porous Si materials, have been prepared and their basic morphology studied. Composite materials with carbon or SiO$_x$ ($Si/C$ or $Si/SiO_x$), thin films, and three-dimensional porous Si materials have also been examined as candidate materials for NEs.

It has been reported that Si NEs exhibited good cyclability if the capacity was limited to under 1000 mAh g$^{-1}$ by narrowing the operating range of charge/discharge, implying control of the range of volume change. This enabled excellent capacity retention for up to a few hundred cycles. Even when the capacity of Si NEs was limited in this manner, their capacity was still over 744 mAh g$^{-1}$, which is twice that of a graphite one. This is quite an attractive approach to increase the discharge capacity and energy density of LICs because it decreases the amount of NE material required in addition to improving cyclability. It also decreases the thickness of NE coating layer, which facilitates homogeneous Li pre-doping in a short time. Therefore, the combination of Si NE and Li pre-doping is a good idea from the viewpoint of industrial production to shorten the pre-doping time and cell assembly. Moreover, Li pre-doping of the Si NE used in hybrid capacitor systems can lower or basically cancel out the large initial irreversible capacity of Si NEs caused by the reduction decomposition of the electrolyte during the 1st charging process.

In a hybrid capacitor system, it is quite easy to adjust the utilization ratio (state of charge) of Si NEs by varying the amount of AC in the PE and circumventing the initial irreversible capacity loss of the Si NE by Li pre-doping. In this study, we propose an idea for the effective usage of Si NEs by fabrication of a new hybrid capacitor with a Li pre-doped Si NE (denoted Si-CAP). At the same time, we try to improve the discharge capacity and energy density of the Si-CAP cell, as shown in Fig. 1c. By focusing on the Li pre-doping methods used to treat the Si NE, the relationship between the Li pre-doping conditions and cell performance is investigated to improve the discharge capacity and energy density, cyclability, and rate capability of the capacitor. First, the basic operation of the Si-CAP cell is demonstrated, and then two kinds of methods to improve Li pre-doping, i.e., additives and vacuum pressure impregnation (VPI), are examined to determine their effects on cell performance. Furthermore, to elucidate the effects of capacity limitation of the Si NE by the AC PE, a Li pre-doped Si half-cell [Li pre-doped Si | Li metal foil] (capacity limitation: 700 mAh g(Si)$^{-1}$) and a three-electrode Si-CAP cell [Li pre-doped Si | AC] are also fabricated and their charge/discharge properties evaluated.

Experimental

Si NEs (Alfa Aesar, average particle size of Si NPs: $\leq$ 50 nm, Figure 2a) were used as the Si NE material of Si-CAP. The NPs were relative size-like and their crystallinity was high even though their particle size was nanoscale (Figure 2b). A slurry was prepared by mixing 83.3 wt% Si NPs, 5.6 wt% Ketjen Black (Lion, EC600JD) as a conductive agent, and 11.1 wt% carboxymethyl cellulose sodium salt (Nacalai Tesque) as a binder using water as a solvent. The slurry was coated on Cu foil (Hohsen, thickness: 20 μm) as a current collector, dried overnight at 80°C under vacuum, and then cut into circles ($Φ = 16$ mm; i.e., area of NE was ca. 2 cm²). The thickness of the Si NE coating was ca. 12 μm, which corresponded to a loading weight of 0.32–0.43 mg(Si) cm$^{-2}$ for the Si NEs as an active material. Li pre-doping and cell assembly were performed in Ar-filled glove boxes (VAC, NEXUS II and Miwa, MDB-1BK-NT1). For Li pre-doping, the Si NE was contacted with Li metal foil (Honjo Metal, thickness: 0.05 mm), and then immersed in 1.0 M LiBF$_4$ dissolved in propylene carbonate (PC, Kishida Chemicals, Battery Grade) at room temperature for 1 h. The morphology change of the Si NEs before and after Li pre-doping was observed by a scanning electron microscopy (SEM, Philips, XL-30). Coin-type cells were constructed using a Li pre-doped Si NE, separator...
(TEF4030, Nippon Kodoshi), and AC PE (Hohsen, thickness: ca. 100 μm) in the same glove boxes used for Li pre-doping. The average weight of AC in each AC PE was 11.44 mg, which was 11.0–14.8 times larger than the amount of Si NPs to adjust the capacity balance between the Si NE and AC PE. In the Si-CAP cell, 1.0 M LiBF4/PC was used as the electrolyte and the Li pre-doping process was performed. In some experiments, 10 wt% vinylen carbonate (VC, Aldrich) or fluoroethylene carbonate (FEC) was added to the electrolyte solution prior to Li pre-doping after the electrolyte was dried with molecular sieves (4A 1/16, Wako). Moreover, VPI was also conducted prior to Li pre-doping using 1.0 M LiBF4/PC as the electrolyte in the absence and presence of 10 wt% FEC. Charge/discharge tests were carried out in constant current mode at a current density of 0.1 mA cm−2 between 2.0 and 4.2 V with a battery test system (Hokuto Denko, HJ1001SM8). As a reference, an EDLC cell composed of [AC] | [AC] was also fabricated and evaluated in the same way as the Si-CAP cells. The rate capability of the cells was evaluated in the current density range between 0.1 and 1.0 mA cm−2. In addition, a Li pre-doped Si half-cell [Li alloyed Si | Li metal foil (Honjo Metal, thickness: 0.5 mm)] with both 10 wt% FEC and VPI was also examined to understand the effects of Li pre-doping and capacity limitation by the AC PE on the performance of the hybrid capacitor system. The capacity limitation was 700 mAh g(Si)−1.

A three-electrode Si-CAP cell in which Li metal foil was used as a battery test system (Hokuto Denko, HJ1001SM8). As a reference, an EDLC cell composed of [AC] | [AC] was also fabricated and evaluated in the same way as the Si-CAP cells. The rate capability of the cells was evaluated in the current density range between 0.1 and 1.0 mA cm−2. In addition, a Li pre-doped Si half-cell [Li alloyed Si | Li metal foil (Honjo Metal, thickness: 0.5 mm)] with both 10 wt% FEC and VPI was also examined to understand the effects of Li pre-doping and capacity limitation by the AC PE on the performance of the hybrid capacitor system. The capacity limitation was 700 mAh g(Si)−1. A three-electrode Si-CAP cell in which Li metal foil was used as a reference electrode was also prepared to separate the electrode potentials of the PE and NE from the cell voltage and investigate the charge/discharge (Li alloying/dealloying) reaction at the Si NE. All charge/discharge tests were performed at 30 °C.

Results and Discussion

Charge/discharge properties of the Si-CAP cell.—Prior to the charge/discharge testing of Si-CAP cells, the morphology change of the Si NEs before and after Li pre-doping was confirmed by SEM observation. Although the surface of Si NE was relatively flat and smooth before the Li pre-doping, after Li pre-doping with no additives and VPI, the Si NPs aggregated to form numerous islands. The average size of Si NP islands was 20–30 μm. However, the exfoliation of the Si NPs was not observed. The similar morphology change was also observed for the Li pre-doped Si NEs with additives and/or VPI.

Figure 3a presents the 1st charge/discharge curves of the Si-CAP cell with a Li pre-doped Si NE and EDLC with both AC electrodes. The Si-CAP cell enabled the charging up to 4.2 V and the discharge curve was convex upward (not linear), while the EDLC cell showed a low operating voltage range between 0 and 2.7 V and a linear discharge curve. This implies that the Li pre-doped Si NE worked at sufficiently low redox potential to raise the cell voltage of Si-CAP. In fact, the cell voltage was not raised up to 4.2 V when a pristine Si NE without Li pre-doping was used; only current originating from electrolyte decomposition at the electrode was observed (data not shown). The magnitudes of the irreversible capacities (Qirr) of the 1st charging process of the two cells were compared. The Si-CAP cell exhibited a much larger Qirr than the EDLC one. This indicates that the current originating from electrolyte decomposition was mainly caused by electrolyte reduction at the Si NE surface, which suggests that the Li pre-doping of Si NE was not sufficient under the condition used here. However, the Si-CAP cell exhibited a high initial discharge capacity of 223.6 mAh g(Si)−1, which was ca. 5.5 times larger than that of the EDLC one. Figure 3b shows plots of the charge/discharge capacities and coulombic efficiency (CE) against cycle number for the Si-CAP and EDLC cells. The 1st discharge capacity of the EDLC cell was ca. 40 mAh g(AC)−1, and discharge capacity remained stable for 50 cycles. The Si-CAP cell possessed a higher discharge capacity than that of the EDLC one over 50 cycles, although it gradually decreased during cycling. The CE value of the Si-CAP cell was relatively low compared with that of the EDLC one, but rapidly increased during the initial cycles to reach 96.3% after 50 cycles. These results suggest that the Li pre-doped Si NE can work in a hybrid capacitor system and improve both discharge capacity and energy density with a relatively good cycle life. Also, the volume change of the Li pre-doped Si NE in the hybrid capacitor system was sufficiently small to prevent critical damage such as cracks and pulverization. The Si-CAP cell with a Li pre-doped Si NE maintained a relatively high discharge capacity (>100 mAh g(Si)−1) even after 50 cycles, remaining higher than that of the EDLC one.

Proposed methods to improve the performance of the Si-CAP cell.—In the above section, we demonstrated that it is possible to use the Li pre-doped Si NE in a hybrid capacitor system. However, we identified two problems that need to be solved for the practical use of Si-CAP; one is the large Qirr of the 1st charging process and the other is poor cyclability. These problems may be caused by the Si NE. In particular, the large Qirr and relatively unstable behavior in the initial cycles (Figures 3a and 3b) indicate that the degree of Li pre-doping was not sufficient to form a suitable solid–electrolyte interphase (SEI) film on the Si NE surface. Therefore, methods to improve Li pre-doping are needed to achieve high-performance Si-CAP cells. We next investigated improving strategy of Li pre-doping by two methods; one was the use of the additives (VC or FEC) to form stable SEI films on the Si NE surface (surface stabilization), and the other was VPI with the same electrolyte solution prior to Li pre-doping to promote uniform Li doping of all the Si NPs to increase the Si utilization ratio.

Figure 4a depicts the discharge curves of Si-CAP cells with a Li pre-doped Si NE using 1.0 M LiBF4/PC with 10 wt% VC or FEC along with those of a reference cell fabricated without additives. The discharge capacities of the Si-CAP cells were markedly increased by using both additives. Especially for the FEC addition, the discharge capacity per weight of Si, i.e. Si utilization ratio, rather increased at 50th cycle compared with those at 1st cycle, and the value was reached up to 405 mAh g(Si)−1. The details of this capacity increase are shown in Figure 4b. The discharge capacities of the cells without additives and with VC gradually decreased during charge/discharge cycling. In contrast, the discharge capacity of the cell with FEC increased during cycling. This indicates that a suitable SEI film containing FEC-decomposed components was gradually forming and growing at the Si/electrolyte interface. The discharge capacity of 405 mAh g(Si)−1 for the 50th cycle reveals that the utilization ratio of the Si NE
increased during charge/discharge cycling, reaching up to 10% of the theoretical capacity (4200 mAh g$^{-1}$). As a result, the estimated energy density was 76 Wh kg(NE + PE)$^{-1}$ after 50 cycles. Figure 4b shows that CE values were also improved by using the additives. The highest CE value of the investigated cells of >99.0% was achieved after the 50th cycle for the cell containing FEC.

It is generally known that the charge/discharge properties of Si NE are quite improved by adding VC$^{26,47}$ or FEC$^{48}$ into the electrolyte solutions for LIBs. This is because these additives decompose prior to the electrolyte solution on the Si NE, and form more suitable SEI films on the surface of Si NE for the Li alloying/dealloying reaction. The SEI films prevent direct contact between the Si NE and electrolyte, and suppress continuous electrolyte decomposition. Also, the SEI films formed by the additives accelerate the Li alloying/dealloying reaction at the Si NE surface because of their uniformity and low electrical resistance.$^{49}$ This increases the reversibility of the charge/discharge reaction. Therefore, addition of VC or FEC during Li pre-doping is an effective method to improve the electrochemical properties of the Si NE in the hybrid capacitor system. Meanwhile, these additives did not have a negative effect on the AC PE.

Although the observed discharge capacity was 405 mAh g(Si)$^{-1}$, this value was not still limited by the available capacity of around 800 mAh g(Si)$^{-1}$ due to the capacity and loading amount of AC PE (vide infra). To increase the utilization ratio of the Si NE and form a more uniform SEI film on the surface of the Si NPs, we immersed the Si NE in 1.0 M LiBF$_4$/PC containing FEC prior to Li pre-doping under vacuum conditions; i.e., VPI treatment. Figure 5a illustrates the charge/discharge curves of the Si-CAP cells treated by VPI with 1.0 M LiBF$_4$/PC in the absence and presence of 10 wt% FEC prior to Li pre-doping. Surprisingly, $Q_{\text{irr}}$ of the 1st charging process disappeared upon using this Li pre-doping method. This means that the electrolyte decomposition to form SEI films on the Si NE surface was successfully finished by Li pre-doping. That is, sufficient and uniform Li pre-doping of all the Si NPs was obtained by VPI. Also, the charge/discharge capacities of the cells were increased markedly by VPI. The Si-CAP cell with FEC and VPI exhibited the highest 1st discharge capacity of 734 mAh g(Si)$^{-1}$ among the investigated cells. This indicates that the utilization ratio of Si NE reached ca. 700 mAh g(Si)$^{-1}$ by using the improved Li pre-doping method, approaching the limit of around 800 mAh g(Si)$^{-1}$ determined by the AC PE. As a result, a high initial energy density of 114 Wh kg(NE + PE)$^{-1}$ was achieved. Figure 5b plots charge/discharge capacities and CE values against cycle number for these Si-CAP cells. For both cells, high charge/discharge capacities were retained for 50 cycles. The CE values of the Si-CAP cells using VPI were relatively low compared with those of the cell with only FEC addition, but remained over 95% after the 4th cycle.

Figures 6a–6c present the discharge curves at different current densities for the Si-CAP cells with only FEC addition and both FEC addition and VPI along with those of a reference EDLC cell. The rate capability of the Si-CAP cell was greatly enhanced by the Li pre-doping method involving both VPI and FEC addition. The relatively large IR drops, i.e. cell resistance including charge transfer and SEI film resistances, observed for the Si-CAP cell with FEC addition only was effectively lowered by the combination of FEC addition and VPI. Consequently, the energy density of the Si-CAP cells produced by two treatments (FEC addition with/without VPI) are much higher than that of the EDLC even at a current density of 1.0 mA cm$^{-2}$ (corresponding to a rate capability of around 2500 mA g(Si)$^{-1}$). An energy density of over 60 Wh kg(NE + PE)$^{-1}$ was attained for the Si-CAP cell with FEC and VPI even at a high charge/discharge rate; this value was three times larger than that of the EDLC.

**Investigations on the stability of the Li pre-doped Si NE.—**To examine the electrochemical stability of the Li pre-doped Si NE, two types of charge/discharge tests were performed; one using a Li pre-doped Si half-cell [Li-Si alloy | Li metal] (capacity limitation: 700 mAh g(Si)$^{-1}$) and the other using Si-CAP in a three-electrode cell with Li metal foil as a reference electrode [Li-Si alloy | AC]. Figure 7a shows the cyclability of the Li pre-doped Si half-cell. The half-cell with the Li pre-doped Si electrode exhibited no capacity fading at the capacity limitation of 700 mAh g(Si)$^{-1}$ during 50 cycles. In contrast, a pristine Si electrode (without capacity limitation) exhibited rapid capacity fading to around 200 mAh g(Si)$^{-1}$ during the first 10 cycles (data not shown). Overall, capacity limitation together with Li pre-doping using 10 wt% FEC and VPI provides Si NEs with a quite
Figure 6. Discharge curves at different current densities for the Si-CAP cells with (a) FEC and (b) FEC and VPI treatment. (c) Discharge curves of a reference EDLC. (d) Plots of the discharge capacities of the Si-CAP and EDLC cells against the common logarithm of current density.

A stable charge/discharge (Li alloying/dealloying) reaction and good reversibility, resulting in high cell performance of Si-CAP. Figure 7b shows the potential profiles of both AC PE (P_{PE}) and Li pre-doped Si NE (P_{NE}) together with the cell voltage (V_{cell}) of the Si-CAP during the initial 3 cycles. All the potential profiles exhibited a reversible redox reaction. This indicates that the Li pre-doped Si NE operated stably in the hybrid capacitor system in the potential range of 0.02 to 0.5 V. The profile of charge/discharge curve of Li pre-doped Si NE exhibited an amorphous Si although the Si NPs was clearly shown the crystalline structure in Figure 2b. Therefore, Li pre-doping make the crystalline Si NPs amorphous during Li pre-doping and stabilized the Si NE also by forming suitable SEI film on the surface. However, for a much longer cycle life, we have to determine the performance factors of additives and VPI with their relationship. Anyhow, these methods in the Li pre-doping were quite effective to activate and stabilize most Si NPs in the NE. Optimization of these conditions and further treatment methods for Li pre-doping of Si NEs is in progress.

Conclusions

A new type of high energy storage system (Si-CAP) based on a hybrid capacitor concept was fabricated using a Li pre-doped Si NE, and its cell performance, including discharge capacity and energy density, cyclability and rate capability, was investigated. To stabilize the Li alloying/dealloying reaction at the Si NE surface, VC or FEC was added to the electrolyte used in the Li pre-doping process. Both additives enhanced discharge capacity and cyclability. In particular, FEC increased the discharge capacity of the 50th cycle up to 405 mAh g(Si)^{-1}. However, the capacity was not still limited by the AC PE. Therefore, VPI prior to Li pre-doping was performed to increase in the utilization ratio of the Si NE. Consequently, the utilization ratio of Si NE reached up to 734 mAh g(Si)^{-1} in the 1st cycle, and a high energy density of 114 Wh kg(NE + PE)^{-1} was attained. Therefore, it is concluded that Li pre-doping of Si NEs is an important factor determining the performance of Si-CAP cells. In particular, the degree and uniformity of Li pre-doping and surface protection with a suitable SEI film govern the stabilization of the Si NE, resulting in enhancement of cell performance.

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