Highly conducting fibrous carbon-coated silicon alloy anode for lithium ion batteries

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ABSTRACT
Carbon-coated silicon/iron silicide nanocomposite anodes developed for lithium ion rechargeable batteries present a large initial irreversible capacity owing to many pores in the carbon coating layer generated from the carbonization of polyfurfuryl alcohol (PFA) resin during the heat treatment. To overcome this issue of large initial irreversible capacity loss, we attempted to fill the pores via chemical vapor deposition (CVD) of carbon using acetylene as the source. The Brunauer-Emmett-Teller surface area is reduced from 51 to 7 m\textsuperscript{2} g\textsuperscript{−1} and the initial irreversible capacity also decreased from 197 mAh g\textsuperscript{−1} corresponding to a simple resin-coated sample to 164 mAh g\textsuperscript{−1} after CVD of carbon on the resin-derived carbon coating. The rate capability tests show an excellent ability to maintain a capacity of 500 mAh g\textsuperscript{−1} at the rate of 7 C (10.5 A g\textsuperscript{−1}), suggesting that the carbon nanofibers (CNFs) formed by the catalytic decomposition of acetylene on iron silicide grains aid in improving the electrical connection between the active anode particles during cycling.

1. Introduction
Lithium-ion batteries (LIBs) are being widely used in portable electronic devices since more than two decades, owing to their high energy density, good rate capability, long cycle life, and low self-discharge rate [1–7]. The capacity of graphite, which is commonly used as an anode material, is limited to 372 mAh g\textsuperscript{−1} (LiC\textsubscript{6}) and this is not high enough to satisfy the requirements of electric vehicles and large scale renewable energy storage systems. Among the advanced materials being studied as alternatives to graphite, silicon is considered one of the most promising [8]. Silicon has a high theoretical specific capacity of 3580 mAh g\textsuperscript{−1}, forming Li\textsubscript{15}Si\textsubscript{4}. It has a significantly higher capacity compared to graphite, but suffers from rapid deterioration of capacity in the early stage of cycling, owing to the volume expansion reaching up to 400% during lithiation and the resultant pulverization of the Si particles during cycling. An irreversible solid-electrolyte interphase (SEI) is formed on the surface of the active electrode particles owing to the electrochemical reaction with the liquid electrolyte during the initial lithiation process. As the pulverization of silicon continuously creates fresh silicon surfaces, SEI forms at these new surfaces. This in turn causes a loss of capacity after each cycle, leading to a poor long term cycle performance [9–12].

To circumvent this problem, researchers have adopted various strategies such as reducing the Si particle size to nanoscale [13–15], introducing adequate empty spaces to compensate for the volume expansion [16–23], and coating the Si particles with carbon. The easiest method among these is the carbon coating. Carbon coating prevents direct contact between the Si-based particles and the electrolyte solution, thereby promoting the formation of the SEI layer on the surface of the carbon coating layer in the first cycle, and also enhances the electrical connection between the silicon particles [24–27].

So far, various methods have been employed for preparing carbon-coated Si powders. Depending on the physical state of the carbon source, the coating method can be divided into three categories: Chemical vapor decomposition (CVD) [24,28,29], dry ball milling [30–33], and wet coating [34–37].

Among these, wet coating using polyfurfuryl alcohol (PFA) resin is known to produce a more uniform coating. However, the surface area becomes quite large owing to numerous pores within the coating layers formed during the carbonization process, which results in increased formation of SEI and thus a high loss in the initial irreversible capacity [38–40]. In order to reduce the surface area, the carbonization temperature should be relatively low (below 500 °C) or very high (above 900 °C) [41]. However, when the carbonization temperature is too low, the decomposition of the resin is far from completion and when the temperature is too high, the microstructure of the active material could

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change and/or silicon carbide could form by the reaction between silicon and the carbon coating layer, resulting in capacity loss [42]. That is, there is a temperature range in which appropriate carbonization can be achieved, and it is necessary to develop another method for reducing the surface area in this specific temperature range.

In this study, a silicon/iron silicide nanocomposite is used as the anode active material, in which iron silicide serves as a buffer for volume expansion [22,40,43–45]. PFA resin was used as the precursor in the wet coating process and the resultant carbon coating has a large initial irreversible capacity due to the presence of numerous micropores [46–48]. Therefore, we attempted to fill the pores by combining the wet-coating process with a CVD method for coating carbon by flowing acetylene gas during carbonization so as to reduce the initial irreversible capacity [49]. Apart from the filling of the pores of the resin coating layer, carbon nanofibers (CNF) were also formed during the CVD process; these played a crucial role in enhancing the electrochemical performance. A decrease in the initial irreversible capacity loss of the active material was observed after CVD of carbon on the resin-coated anode and the CNFs resulting from CVD process contributed to maintaining a stable electrical property at high Coulombic rates.

2. Experimental procedures

2.1. Sample preparation

PFA resin (14,500 cps, Poly Sciences Inc.) and acetylene (C2H2) were used as carbon precursors for the wet coating and CVD processes, respectively, for coating the Si/iron silicide nanocomposite. Si/iron silicide nanocomposite which is made by ball milling a ferrosilicon at 24 rpm for 2 weeks in tumbling ball mill were provided by MKE company, Korea (particle size distributions: D10 = 0.5 μm, D50 = 1.4 μm, and D90 = 3.0 μm). Hereafter, the Si/iron silicide nanocomposite powder is referred as “as-milled FeSi.” It is a nanocomposite powder of finely dispersed nanocrystalline iron silicide and amorphous Si which is not detectable in X-ray diffraction patterns. The powder consists mainly of agglomerates of submicron sized nanocomposite Si/iron silicide primary particles. The chemical composition of the as-milled FeSi, as analyzed by X-ray fluorescence, is given in Table 1. For the wet-coating of the resin, the PFA resin was dissolved in propylene monomer ethanol and then dried at 80 °C in an oven for 2 h. It was subsequently placed in an alumina boat which was loaded into a horizontal quartz tube furnace. The tube was first purged with 6 N purity Ar gas (80 mL/min) for 30 min and then heated to 800 °C for 30 min at a heating rate of 1 °C/min. For CVD coating, a mixture of acetylene and high-purity argon (argon: acetylene = 8:2 by volume) was blown between 700 and 800 °C. The resultant active materials were labeled according to the coating method used (e.g., Resin + CVD@FeSi for the as-milled FeSi coated by carbon from the resin and then via CVD using acetylene). The carbonized powder was ball-milled for 5 min at 30 Hz using a mixer mill (Retsch MM 400) to crush the hard aggregates formed during the carbonization into a fine powder. The resin + CVD coating of the sample was designed to achieve 5 wt% carbon from resin and 5 wt% carbon from CVD. For comparison with the Resin + CVD sample, specimens coated with only resin were also prepared, and their total carbon content was maintained at ~10 wt%.

2.2. Characterization

The phase compositions were analyzed by XRD using a Bruker D8 Advance diffractometer with Cu-Kα radiation operating at 40 kV and 40 mA. The exposure time was 2 s/step with a step size of 0.03° in 2θ. The carbon content of the samples was evaluated by thermogravimetric analysis (TGA). The TGA was conducted using a TA SDT Q600 in the temperature range of 50–800 °C at a heating rate of 5 °C/min under the flow of dry air. The specific surface area of the samples was estimated through N2 adsorption measurements performed using a Micromeretics Tristar II 3020 and the Brunauer-Emmett-Teller (BET) method. Samples were degassed at 300 °C for 4 h before the BET measurement. The micropore surface area and micropore volume were calculated from the thickness-plot (also known as t-plot), while the mesopore volume was done from the desorption branch of the isotherm using Barrett-Joyner-Halenda (BJH) method. The morphology of the coating and particle size of the anode were investigated using a field emission scanning electron microscope (SEM, Hitachi S-4300SE). SEM images of the cross-section carbon-coated specimens were obtained using a JEOL 2010 SEM (Tokyo, Japan) at an accelerating voltage of 200 kV for detailed analysis of the microstructure of the carbon-coated nanocomposite powders. The structure of the carbon coating was analyzed using Raman spectroscopy (Renishaw InVia Micro-Raman, λ = 532 nm, 45 mW). Each spectrum was collected thrice with an exposure time of 30 s using a 50 x objective lens. The electrical conductivities of the active material and anode were measured with a Keithley 2000 multimeter. The electrical conductivities of the active materials were measured after compacting the powders at 80 MPa using a home-made mold. The electrical conductivities of the anodes prepared by mixing the active materials, binder, and a conductive agent were measured at 10 kPa. Both measurements were carried out using a two-probe system.

2.3. Electrochemical measurements

The working electrodes were prepared by mixing the active materials, Ketjen black (ECP600JD, Surface area: 1270 m2 g−1, Lion Specialty Chemicals) and a water-soluble binder (AST9005, provided by Aekyung Chemical, Korea) in a weight ratio of 89.5:3.5:7 in distilled water to form a slurry using a planetary mixer (ARE-310, Thinky Corp., Japan). The resulting slurries were pasted on 18 μm copper foils and then dried at 80 °C for 2 h. The pasted foil was cut into 14 mm diameter disks and then dried overnight at 120 °C under vacuum. 2032 coin-type cells were assembled in a glove box using lithium coins as the counter electrodes and ceramic-coated polypropylene membranes (MTI Inc.) as separators. LiPF6 (1.0 M) in a mixture of ethylene carbonate, diethyl carbonate, and fluoroethylene carbonate (5:70:25 in v/v) was used as the electrolyte. The galvanostatic charge and discharge cycle tests were carried out using an automatic battery cycler (WBCS3000L, Wonatech Co., Korea). All the cells were first discharged (for Li insertion into the working electrode) in the constant current (CC) mode to 0.01 V vs. Li/Li+ at a constant current of 150 mA g−1 (0.1 C) and then further discharged in the constant voltage mode until the current reached 0.01 C. The cells were charged to 1.5 V vs. Li/Li+ in only CC mode with a constant current of 1 C. Both discharging and charging cycles were conducted.

3. Results and discussions

The XRD patterns of as-milled and carbon-coated FeSi specimens are shown in Fig. 1. The as-milled FeSi consists of β-FeSi2 (PDF: 71-0642) and α-Fe2Si2 (PDF: 73-1843) as well as amorphous Si. However, there is no carbon peak corresponding to the (0 0 2) plane at 2θ = 24° in the
XRD pattern of carbon-coated FeSi because carbon has an amorphous structure or the amount of crystalline carbon is small [50,51]. This will be further discussed in conjunction with Raman analysis. The two peaks corresponding to Si (1 1 1) and β-FeSi₂ between 28° and 30° are difficult to differentiate owing to peak broadening, because both Si and β-FeSi₂ crystallites are very small after hours of ball milling. No structural difference could be identified between the resin@FeSi and resin + CVD@FeSi samples, and the carbon peak was not observed either. In the coated FeSi, the silicon and β-FeSi₂ peaks between 28° and 30° are separated because of the crystallization of the amorphous Si and/or crystal growth of β-FeSi₂ induced by the heat treatment. In addition, most of the α-Fe₂Si₃ phase has transformed into the β-FeSi₂, which is consistent with the equilibrium phase diagram of the Fe-Si binary system. According to this, β-FeSi₂ is stable below the eutectoid temperature (937 °C) [52].

As shown in the TGA thermograms (Fig. 2), the carbon content of the coated sample is ∼ 10 wt%. The combustion temperatures of the coated specimens are different depending on the coating method. The study on the specific surface area will be described later in detail, but it seems that the increase in the specific surface area has lowered the combustion temperature.

The surface morphology of resin@FeSi and resin + CVD@FeSi specimens are similar except for the carbon nanofibers (CNFs) formed in the sample subjected to CVD process (Fig. 3). Acetylene is known to catalytically decompose over iron particles, resulting in the growth of CNFs [53–57]. Formation of an amorphous carbon coating layer on nano-silicon particles has also been reported [58]. Moreover, the CNFs were formed only when acetylene gas was flown into the furnace containing as-milled FeSi (Fig. S2). Therefore, it seems that iron silicide on the surface of the active material acts as a catalyst for the formation of CNFs and the silicon on the surface of the active material is coated with a layer of amorphous carbon during CVD process. CNFs could be found in the resin + CVD@FeSi specimen because acetylene gas could reach the surface of the iron silicide grains through the open pores in the carbon coating layer derived from the resin (see Table 2) [41,59,60].

The microstructures of resin + CVD@FeSi are shown in Fig. 4. As mentioned in the experimental section, the nanocomposite powders are agglomerates of submicron-sized Si and iron silicide nanocomposite primary particles, and the particles seem to be densely agglomerated, as shown in TEM images. After carbonization, the voids between the particles are filled with carbon (Fig. 4). In the high-resolution TEM image, the carbon coating layer with a rather uniform thickness of ∼ 20 nm on the surface of Si/iron silicide particles and CNFs grown on the surface of the active material through the resin coating layer are observed.

The N₂ adsorption/desorption isotherms of the samples are shown in Fig. S1. As-milled FeSi and resin + CVD@FeSi show only type IV isotherms but resin@FeSi exhibits both type I and type IV isotherms. This means resin@FeSi have both micropores and mesopores [61] while the other two samples have mainly mesopores. It is clearly shown in Table 2 that almost all the micropores and mesopores formed by resin coating in resin@FeSi have been disappeared after CVD coating. Therefore, the SEM and BET analysis results reveal that the CVD process not only promotes the formation of CNFs but also leads to filling of the micropores.

As observed in the SEM images of the anodes, the particles tend to aggregate with increasing resin content (Fig. 5). Even though the same ball-milling process was applied to the both coated samples, the sizes of the aggregated active particles in the anode are different. This can be explained by the fact that the carbon obtained by carbonizing PFA resin has high strength [62], and therefore, the resin-coated samples have larger particles and a non-uniform particle size distribution compared to the sample coated with carbon via CVD. Resin@FeSi presents the largest aggregate size and the least uniform size distribution because it contains the highest resin content among the samples.

The electrochemical performance of the as-milled FeSi and the coated Si/iron silicide anodes was characterized via cycle performance and rate capability tests (Fig. 6). The 1st discharge capacity of as-milled FeSi is higher than those of the other coated FeSi samples (Table 3). This implies that the Si/iron silicide has a higher specific capacity than the carbon coating layer. Between the coated FeSi samples, the resin + CVD@FeSi exhibits higher initial efficiency than resin@FeSi. This is because, as mentioned in Table 2, the BET surface area of the coated sample is significantly reduced by carbon coating via CVD, and therefore, the initial SEI formation and thus the initial irreversible capacity loss is reduced.

In the cycle performance test, the cycle characteristics of resin@FeSi are not better than that of as-milled FeSi (Fig. 6a).
owing to the formation of larger aggregates and a non-uniform size distribution of the resin@FeSi sample, as mentioned above. The resin@FeSi particle was sieved through a 400 mesh sieve to adjust the particle size. Resin@FeSi with a controlled particle size exhibits better cycle characteristics than the original sample (Fig. S3).

Rate capability tests were also carried out at various current densities ranging from 1 C to 7 C to examine the influence of the CVD coating on the electrochemical properties. The resin@FeSi samples displayed higher capacity than the as-milled FeSi at the rate of 7 C, despite their larger particle size (Fig. 6b). At the high rate conditions, the improvement in the electrical connection between the active particles by the carbon coating seems to be a dominant factor for enhanced capacity. The resin + CVD@FeSi anode exhibits excellent rate capability and even maintains a capacity above 500 mA h g\(^{-1}\) at 7 C. The capacity is restored when the current is lowered again to 1 C, implying that the structure of the anode is not significantly damaged even at a high rate. The reason for the better characteristics of the samples coated with carbon via CVD can be explained by electrical conductivity analysis, because the rate capability would be affected by the differences in the electrical conductivity of the anodes.

Since the bonding structure of the carbon coating layer affects the electrical conductivity [63], the structure of carbon was analyzed by Raman spectroscopy (Fig. 7). Raman spectra of all specimens showed two broad peaks assigned to D and G peaks at \(\sim 1360\) and \(\sim 1590\) cm\(^{-1}\), respectively, which are the typical bands of carbonaceous materials. The D band is associated with the vibration of disordered carbon, while the G band is attributed to the vibration of sp\(^2\)-bonded graphitic carbon [64]. The relative electrical conductivity can be correlated to the difference in the intensity of D and G bands (\(I_D/I_G\)) because the higher the sp\(^2\)-bonded carbon, the greater the electrical conductivity [65]. A previous study has shown that the Raman spectrum of carbon obtained from PFA resin appears to show a larger D band than the G band, however, the G band is actually larger because the D band appears larger due to the presence of the M band between 1320 and 156 cm\(^{-1}\), which might be the related to various compounds formed during the pyrolytic degradation of the resin [66]. Resin@FeSi is also estimated to have a larger G band, but the electrical conductivity is expected to be lowered owing to the M band. In the case of resin + CVD@FeSi, the amorphous carbon coating formed on the silicon surface by CVD affects the intensity of the D band [58]. However, the CNFs formed on the surface of iron silicide would have different \(I_D/I_G\) depending on the degree of graphitization of the CNF [67]. The Raman spectrum of resin + CVD@FeSi shows a similar intensity of the D and G bands despite the additional amorphous coating layer. This may indicate that CNF has a graphitic structure but the amount of crystalline CNF seems very small, because its presence is not clearly manifested in the XRD pattern (Fig. 1). In resin + CVD@FeSi, amorphous carbon from the CVD process is deposited on top of the carbon coating from the resin as well as within the pores containing silicon grains at the bottom, and therefore three types of carbon, viz., CNFs and amorphous carbon from CVD and carbon from the resin are present simultaneously. The difference in the electrical conductivity of the carbon coating layer itself would not be large because all the coated samples show similar Raman spectra.

Table 4 shows the electrical conductivity of a pellet made by the coated powder only and the anode on a Cu foil. Compared to as-milled

<table>
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<th>(S_{\text{total (BET)}}) (m(^2) g(^{-1}))</th>
<th>(S_{\text{ext}}) (m(^2) g(^{-1}))</th>
<th>(S_{\text{micropore}}) (m(^2) g(^{-1}))</th>
<th>(V_{\text{meso}}) (mm(^3) g(^{-1}))</th>
<th>(V_{\text{micro}}) (mm(^3) g(^{-1}))</th>
</tr>
</thead>
<tbody>
<tr>
<td>As-milled FeSi</td>
<td>4.1</td>
<td>3.2</td>
<td>0.9</td>
<td>16.0</td>
<td>0.0</td>
</tr>
<tr>
<td>Resin@FeSi</td>
<td>50.5</td>
<td>16.7</td>
<td>33.8</td>
<td>29.6</td>
<td>11.7</td>
</tr>
<tr>
<td>Resin + CVD@FeSi</td>
<td>7.2</td>
<td>4.3</td>
<td>2.9</td>
<td>15.5</td>
<td>0.7</td>
</tr>
</tbody>
</table>
FeSi, the electrical conductivity of the pellet of resin + CVD@FeSi is more than double while that of the anode is larger than 20 times. Such difference originates from the high compaction pressure (80 MPa) used for fabricating the pellet. The contact resistance between the particles is sufficiently small so that the pellet exhibits an electrical conductivity similar to that of a single particle. The results of the electrical conductivity of the pellets support those of the Raman analysis. However, the anode is a mixture of the active material, binder, and conductive agent, and the electrical conductivity is more likely controlled by the electrical contact between the active material particles rather than by the electrical conductivity of the active material itself. Therefore, the electrical conductivity of the anode should be considered when comparing the rate capability. As observed in the SEM image of the resin + CVD@FeSi anode (Fig. 5d), CNFs connect the active particles, thereby contributing to the increase in the conductivity and maintenance of a relatively high capacity at a high rate.

Fig. 8 schematically illustrates the results described so far. The resin-coated composite particles have many pores and thus lead to the formation of an increased number of SEIs, resulting in increased initial irreversible capacity. The initial irreversible capacity could be reduced by filling up the pores with carbon deposited by CVD. The carbon obtained by the acetylene gas was deposited on the surface of the iron silicide grains through the open pores of the resin-derived carbon coating layer. In addition, the iron silicide surface catalyzes the formation of CNFs. This is believed to significantly improve the conductivity of the anode, and lead to better charge/discharge characteristics at high rates.

4. Conclusions

The electrochemical properties of silicon/iron silicide nanocomposites were improved by carbon coating derived from PFA resin, followed by CVD of carbon using acetylene. The reduction in the BET surface area owing to the filling of the pores in the PFA resin-derived carbon coating layer by the additional CVD carbon coating led to a decrease in the initial irreversible capacity. We believe that CNFs formed by CVD coating on iron silicide grains increase the electrical connection between the active particles in the anode. Therefore, a high capacity could be maintained even at a higher current density. Significant improvements in the initial capacity and Coulombic efficiency are expected along with higher capacity retention after long cycles, which can be realized by selecting appropriate coating.
conditions.

Acknowledgment

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Table 3
Initial discharge capacity, irreversible capacity, and Coulombic efficiency of different FeSi samples.

<table>
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<tr>
<th></th>
<th>Initial discharge capacity (mAh g(^{-1}))</th>
<th>Initial irreversible capacity (mAh g(^{-1}))</th>
<th>Coulombic efficiency (%)</th>
</tr>
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<tbody>
<tr>
<td>As-milled FeSi</td>
<td>1521</td>
<td>163</td>
<td>89.3</td>
</tr>
<tr>
<td>Resin@FeSi</td>
<td>1359</td>
<td>197</td>
<td>85.5</td>
</tr>
<tr>
<td>Resin + CVD@FeSi</td>
<td>1338</td>
<td>164</td>
<td>87.7</td>
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Table 4
Electrical conductivity of the powdered pellet and electrode.

<table>
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<tr>
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<th>Conductivity (10(^{-2}) Sc m(^{-1}))</th>
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<tr>
<td>Powder pellet</td>
<td></td>
</tr>
<tr>
<td>As-milled FeSi</td>
<td>11</td>
</tr>
<tr>
<td>Resin@FeSi</td>
<td>17</td>
</tr>
<tr>
<td>Resin + CVD@FeSi</td>
<td>24</td>
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<tr>
<td>Electrode</td>
<td>0.011</td>
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<td></td>
<td>0.064</td>
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Fig. 6. (a) Cycle performance and (b) rate capability of the anodes derived from as-milled FeSi, resin@FeSi, and resin + CVD@FeSi.

Fig. 7. Raman spectra of carbon-coated FeSi samples, showing peaks of $\beta$-FeSi\(_2\) at 170, 189, 247, and 281 cm\(^{-1}\); Si at 520 cm\(^{-1}\); D band and G band due to carbon at 1360 cm\(^{-1}\) and 1590 cm\(^{-1}\), respectively.

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Fig. 8. Schematic illustrating the (a) resin and (b) resin + CVD coated composite.

Appendix A. Supplementary material

Supplementary data associated with this article can be found, in the online version, at https://doi.org/10.1016/j.apsusc.2018.05.165.

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