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In-situ Wrapping Si Nanoparticles with 2D Carbon Nanosheets as High-Areal-Capacity Anode for Lithium-Ion Batteries

Lijing Yan¹, Jie Liu¹, Qianqian Wang¹, Minghao Sun¹, Zhanguo Jiang³, Chengdu Liang¹, Feng Pan²,* and Zhan Lin¹,³,*

¹Zhejiang Provincial Key Laboratory of Advanced Chemical Engineering Manufacture Technology, College of Chemical and Biological Engineering, Zhejiang University, Hangzhou 310027, China
²School of Advanced Materials, Peking University, Shenzhen Graduate School, Shenzhen 518055, China
³College of Light Industry and Chemical Engineering, Guangdong University of Technology, Guangzhou 510006, China

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ABSTRACT: Silicon (Si) has aroused great interest as the most attractive anode candidate for energy-dense lithium-ion batteries (LIBs) in the last decade due to its significantly high capacity and low discharge potential. However, the large volume change during cycling impedes its practical application, which is more serious in the case of high mass loading. Designing Si anode
with high mass loading and high areal capacity by a simple, scalable, and environmentally friendly method is still a big challenge. Herein, we report in-situ one-pot synthesis of Si/C composite, where Si nanoparticles are wrapped by graphene-like 2D carbon nanosheets. After 500 cycles at 420 mA g\(^{-1}\), the Si/C anode displays a gravimetric capacity of 881 mAh g\(^{-1}\) with 86.4% capacity being retained. More specially, a high areal capacity of 3.13 mAh cm\(^{-2}\) at 5.00 mg cm\(^{-2}\) after 100 cycles is achieved. This study demonstrates a novel route for the preparation of the Si/C composite with high material utilization and may expand the possibility of future design Si-based anode with high areal capacity for high energy LIBs.

The advent of lithium-ion batteries (LIBs) for consumer electronics has greatly changed human being's life, while the pursuit of energy-dense batteries for electric vehicles (EVs) has never stopped.\(^1\) Currently EV battery cells have a energy density of 200–250 Wh kg\(^{-1}\), the objective of 300-350 Wh kg\(^{-1}\) for EV batteries or even an aggressive goal of 500 Wh kg\(^{-1}\) in 2020 has been proposed.\(^2\)\(^-\)\(^3\) Extensive research activities have been conducted on the search of high-capacity electrode materials for LIBs. Silicon (Si) is a feasible anode candidate for next-generation LIBs with the merits of environmentally friendly, abundant, and possessing theoretical capacity ten times higher than those of commercialized graphite and lithium titanium oxide anodes.\(^4\)\(^-\)\(^5\) Moreover, another newly emerging anode candidate lithium metal, including Li-O\(_2\) and Li-S system, has been widely studied. However, the lithium metal has high reactivity and easily forms dendrite which may cause the safety issues.\(^6\)\(^-\)\(^8\) Si has not only the highest theoretical gravimetric capacity but also relatively low voltage for delithiation (~0.4 V vs. Li/Li\(^+\)). Considering the merits mentioned above, the Si is the most attractive anode material for LIBs.
Till now, successful implementation of Si for LIBs is still impeded by large volume change (~400%) during full lithiation (up to Li$_{4.4}$Si), resulting in structural degradation and capacity fading.$^{4-5}$ Many breakthroughs have been achieved by utilizing advanced nanotechnologies to prepare Si anode materials with different morphologies, such as Si nanowires,$^9$ Si nanotubes,$^{10}$ hollow Si nanospheres,$^{11}$ and so forth. However, the poor electrical conductivity of Si seriously limits its capacity utilization and hinders subsequent rate performance.$^{4-5}$ Modifying or decorating with other materials, especially carbon-based materials,$^{12-15}$ has been widely considered as one of the most effective strategies.

Among different kinds of carbon materials, two-dimensional (2D) layered graphene has been extensively utilized as an effective supporting material in Si/C composites.$^{16-19}$ On account of superior electronic conductivity, excellent mechanical and chemical properties, graphene can accommodate large volume expansion, form stable solid electrolyte interphase (SEI), and improve electronic conductivity for Si anodes. However, the pre-step for the obtainment of graphene or reduced graphene oxide is a necessity,$^{20}$ resulting in complex multi-processes for Si/graphene composites with high cost and risky pollution. Besides, both of high areal capacity and high mass loading are great concerns to evaluate the performance of anode material in LIBs, a successful synthesis of Si/graphene composite anode with high areal capacity and high mass loading for practical application still remains a great challenge.

Herein, we adopt in-situ one-pot synthesis of 2D carbon nanosheets wrapped Si nanoparticles (denoted as Si/C) as anode material for LIBs with high areal capacity and high mass loading. This in-situ synthesis is an easy, economic, and scalable synthetic route for mass production. The 2D carbon nanosheets have the structure similar to that of graphene, which not only accommodate tremendous volumetric expansion of silicon nanoparticles (SiNPs) during cycling,
but also preserve the electronic conductivity of the whole electrode with long-term stability. After 500 cycles at 420 mA g\(^{-1}\), the as-prepared composite anode displays a gravimetric capacity of 881 mAh g\(^{-1}\) with about 86.4\% capacity being retained. Even the mass loading reaches to 5.00 mg cm\(^{-2}\), the electrode still has areal capacity of 3.13 mAh cm\(^{-2}\) at 2.10 mA cm\(^{-2}\) over 100 cycles. When matching the Si/C composite anode with commercial Nickel-Manganese-Cobalt (NMC 532) cathode, the full cell displays reversible capacity of 171 mAh g\(^{-1}\) at 0.5 mA cm\(^{-2}\) and maintains at 132 mAh g\(^{-1}\) after 20 cycles. This novel route has a great potential to be used for high-capacity Si anodes in practical application for LIBs.

**Results and Discussion.** The 2D carbon nanosheets wrapping SiNPs composite was prepared via one-pot salt melt synthesis, using glucose as carbon source in a eutectic NaCl-KCl compound system. Due to chemical inertness and low cost, NaCl and KCl were selected as the “solvent”\(^{21}\). NH\(_4\)Cl was added to generate NH\(_3\) for Maillard reaction\(^{22}\) i.e., reactive carbonyl groups of glucose react with nucleophilic ammonia. Figure S1 shows photographs captured by a digital camera of materials in ceramic boats before (left) and after (middle) carbonization. The color changes obviously. After ball-milling for homogenization, the mixture appears canary yellow mainly because of the existence of SiNPs. By carbonizing at 1050 °C in Ar for 1 h, the gray black products were obtained, which is the synergistic effect of carbon and SiNPs. According to the measurement, the tap density of those gray black Si/C composite is 0.141 g cm\(^{-3}\). Pure 2D carbon nanosheets (denoted as Pure C) were also synthesized by using the same procedure without the addition of SiNPs for comparison.
**Figure 1.** SEM images of (a) Si/C composite and (b) the red box in (a) at higher magnification. (c) TEM image of Si/C composite. The inset is SAED pattern taken from the black box. (d) HRTEM image of the red box in (c). The inset is SAED pattern taken from the red box in (c).

**Figure S2** is the scanning electron microscope (SEM) image of the Si/C composite before removing NaCl and KCl. The brick-like crystals are KCl and NaCl, Si/C composites intersperse disorderly at the surface of the crystals. **Figures 1a and 1b** demonstrate SEM images of Si/C composite with different magnifications. Spherical SiNPs are covered by a thin, wrinkled, and crumpled carbon layer, which has 2D layer structure similar to that of graphene. The structure of the carbon layer is consistent with the observation of Pure C (Figure S3). Average diameter of SiNPs is approximate 50 nm, which is in accord with raw commercial SiNPs (Figure S4).
encapsulation of SiNPs by 2D carbon nanosheets is further confirmed by transmission electron microscopy (TEM) characterization. As shown in Figures 1c, SiNPs are well wrapped by wrinkle-like carbon nanosheets. Selected area electron diffraction (SAED) pattern (the inset) taken from the black box shows the diffraction rings of amorphous carbon. Figure 1d is the high resolution TEM (HRTEM) image of the red box in Figure 1c. The D-spacing of the (111) plane is 0.31 nm. The inset is taken from the red box in Figure 1c. The diffraction pattern matches well with the planes of Si. Moreover, a thin layer of amorphous SiOx (around 1.7 nm) can be observed. Figure S5 also shows that a thin layer of amorphous SiOx because the SiNPs materials with high specific surface area are relatively reactive, which is easy to suffer from oxidation when exposed to air.

![Figure 2](image)

**Figure 2.** (a) XRD results of Si/C composite and pure 2D carbon nanosheets. (b) Raman spectrum of the Si/C composite.

The composition of the as-synthesized Si/C composite was investigated using X-ray diffraction (XRD) and Raman spectroscopy. As shown in **Figure 2a**, the diffraction peaks at 28.5, 47.4, 56.2, 69.2, and 76.4° can be well-indexed to the Si phase (JCPDS 27-1402). An additional broad peak at ~24° is the amorphous carbon phase. Figure 2b shows the Raman
spectrum from 400 to 1800 cm\(^{-1}\) for the Si/C composite. Four distinct peaks were observed at \(\sim 497, 923, 1354,\) and \(1590\) cm\(^{-1}\), respectively, which are associated with Si and C. Carbon displays a disorder band (D-band) at \(1354\) cm\(^{-1}\) and an apparent graphitic band (G-band) at \(1590\) cm\(^{-1}\). The \(I_D/I_G\) is 0.921, indicating that amorphous carbon sheets contain a significant amount of defects. The defects can provide Li\(^{+}\) diffusion channels and react electrochemically with SiNPs during charge/discharge. From the measurement of \(N_2\) adsorption-desorption isotherms in Figure S6 and the calculation on the basis of Brunauer-Emmett-Teller (BET), the specific surface area of Pure C is computed to be 737.5 m\(^2\) g\(^{-1}\), the SiNPs exhibit relatively smaller surface area of 60.9 m\(^2\) g\(^{-1}\), while the Si/C composite is in the middle, having the specific surface area of 104.8 m\(^2\) g\(^{-1}\).

Figure 3. (a) The CV curves of the first three cycles at 0.1 mV s\(^{-1}\) between 0.01 and 3.0 V. (b) Galvanostatic charge–discharge voltage profiles of the Si/C composite. (c) Long-term cycling
capability of the SiNPs, Pure C and Si/C composite electrode. All the cells are charged/discharged between 0.01 and 1.2 V at 100 mA g\(^{-1}\) for the first cycle and then cycled at 420 mA g\(^{-1}\) for subsequent cycles.

The electrochemical performance of the as-synthesized Si/C composite anode for LIBs were systematically studied. Figure 3a shows the cyclic voltammetry (CV) curves of the Si/C composite. In the first cathodic scan of the CV curve, a peak centered at about 1.24 V is associated with the decomposition of fluoroethylene carbonate (FEC).\(^{23}\) A broad cathodic peak at about 0.70 V can be ascribed to the SEI formation during cycling. They both disappeared in the following cycles. A sharp redox peak at ~0.01 V corresponds to the formation of the Li\(_x\)Si phase.\(^{12, 24}\) 0.35 and 0.51 V in the anodic scan are corresponding to lithium extraction from the Li\(_x\)Si phase. Meanwhile, a broad cathodic peak at 0.20 V, anodic peaks at 0.35 and 0.51 V both gradually increase with the cycles due to the activation of SiNPs. Figure 3b shows the 1\(^{st}\), 2\(^{nd}\), and 3\(^{rd}\) charge/discharge voltage profiles of the Si/C anode, whose voltage plateaus is consistent with the the CV curves. A small bulge at ~1.24 V in the 1\(^{st}\) discharge process is assigned to the decomposition of FEC. A flat and long voltage plateau blow 0.11 V is caused by lithium insertion into the Si. The first discharge capacity is 1815 mAh g\(^{-1}\) with the ICE of 71.6\%, which is comparable to most of Si/C composites reported. The irreversible SEI layer leads to the capacity loss during the 1\(^{st}\) cycle. In the charge process from 0.25 to 0.65 V, a sloping plateau can be observed. Due to smaller current density, the first charge capacity is much higher than those of the 2\(^{nd}\) and 3\(^{rd}\) cycles.

In order to explore the impact on battery performance caused by different weight ratios of Si and carbon, three composites abbreviated Si/C-0.2, Si/C-0.25 and Si/C-0.3, respectively, were prepared. To make certain the Si content in Si/C composites, thermal gravimetric analysis (TGA)
was carried out under air atmosphere from 100 to 700 °C (Figure S7). The mass of the SiNPs increases slightly because the surfaces of SiNPs were covered by a compact SiO\(_x\) layers (Figure 1d), preventing SiNPs from being further oxidized.\(^\text{12}\) The Pure C lose 97.80\% of its initial weight at 700 °C, indicating there is 2.20\% unknown impurities existed. According to the TGA analysis, the Si/C-0.3, Si/C-0.25, and Si/C-0.2 composites contain 18.70, 23.70, and 28.30 wt.\% of carbon, respectively. Considering impurities in the pure carbon, the weight percentages of impurities in three systems are 0.42, 0.53, and 0.64\%, respectively. Hence, the weight percentages of Si in the Si/C-0.3, Si/C-0.25, and Si/C-0.2 composite are 80.88, 75.77, and 71.06\%, respectively.

The long-term cycling performance of commercial SiNPs, the Si/C composites with various ratios of Si to carbon, and the Pure C in FEC-containing electrolyte are shown in Figure 3c. Obviously, commercial SiNPs shows the poorest cycling stability that their capacity quickly decrease to 388 mAh g\(^{-1}\) after 45 cycles, which is ascribed to huge volumetric change with unstable electrode structure during cycling. After 500 cycles, Pure C shows a very flat discharge profiles with specific capacity of 166 mAh g\(^{-1}\). The Si/C-0.3, Si/C-0.25, and Si/C-0.2 deliver reversible capacity of 2137, 1723, and 1423 mAh g\(^{-1}\) in the 1\(^{st}\) cycle at 100 mA g\(^{-1}\), corresponding to ICES of 76.1, 70.4, and 67.1\%, respectively. The ICE increases with reducing carbon weight percentage from TGA results because of relatively higher specific surface area of carbon. Good cycling capability is observed at 420 mA g\(^{-1}\) for subsequent cycles. The Si/C-0.3, Si/C-0.25, and Si/C-0.2 anode maintain capacities of 983, 881, and 602 mAh g\(^{-1}\), respectively, after 500 full charges and discharges. The capacity retentions are 65.5, 86.4, and 71.9\% from the 2\(^{nd}\) to 500\(^{th}\) cycle, with an average loss over 500 cycles of 0.069, 0.027, and 0.038\% per cycle, respectively. Taking specific capacity and cycling performance into consideration, the Si/C-0.25
anode is the best. Therefore, except the special statement, material characterizations and electrochemical tests in this study are all based on the Si/C-0.25 composite.

![Figure 4](image-url)

**Figure 4.** (a) Rate capability of the Si/C electrode under increasing current density from 0.1 to 3.2 A g\(^{-1}\). (b) Nyquist plots of the SiNPs and Si/C electrode obtained from EIS within 100 kHz to 0.01 Hz before cycling.

In Figure 4a, the rate performance of the Si/C composite was investigated from 0.1 to 3.2 A g\(^{-1}\). The Si/C electrode renders the discharge capacities of 1018, 984, 867, 686, 529, and 401 mAh g\(^{-1}\) at 0.1, 0.2, 0.4, 0.8, 1.6, and 3.2 A g\(^{-1}\), respectively. A capacity more than 1051 mAh g\(^{-1}\) is retained when current density reduces back to 0.1 A g\(^{-1}\) after 60 cycles. The Si/C electrode can obtain a capacity of 1031 mAh g\(^{-1}\) at 0.1 A g\(^{-1}\) after 40 cycles. The capacity retention can reach to 98.1%, demonstrating that the Si/C composite has good rate capability and excellent cycling stability. Electrochemical impedance spectroscopy (EIS) were measured before cycling, after the first cycle and after the 20\(^{th}\) cycle of SiNPs and Si/C composite electrodes. The Randles equivalent circuit (the inset in Figure 4b, Figure S8a, and Figure S8b) was used to measure the impedance spectra. The fitted impedance data was demonstrated in Table S1. In
Figure 4b, both Nyquist plots show a semicircle in high frequency region and a diagonal in low one. According to the fitting, the charge transfer resistance of the Si/C composite is 183.10 Ω, while the SiNPs have much higher resistance of 233.50 Ω. The difference in resistance indicates that the Si/C composite improves the electronic conductivity. Compared Nyquist plots of the SiNPs with the Si/C electrode after the 1st cycle and the 20th cycle in Figure S8, the SiNPs change much while the Si/C composites keep relatively stable, which proves that the Si/C electrode can keep structure stable and hold conductive integrity during cycling.

Figure 5. Schematic drawing of the charge/discharge process of the Si/C electrode. (a) the model of SiNPs (yellow balls) wrapped by 2D carbon nanosheets (black coatings). (b) the lithiated process of Si/C electrode, the defects in carbon nanosheets provide more diffusion channels for the facile lithium ion transport and the carbon nanosheets are good electronic conductor. (c) the delithiated process of Si/C electrode, the carbon nanosheets can accommodate the volume expansion of SiNPs during cycling and keep good electrical contact.

No doubt that the coating of 2D carbon nanosheets on the surface of the SiNPs has significantly influenced the rate performance and cycling stability. One key function of the 2D carbon nanosheets is to form a conductive network that can facilitate electron transport and improve the electronic conductivity of Si/C composites. This is validated by our EIS measurements in Figure 4b. Another key function of the carbon nanosheets is to generate a stable
structure by accommodating the volume expansion of SiNPs and providing a stable SEI film during lithiation/delithiation cycles. EIS results in Figure S8 reveal more stable structure of Si/C electrode than SiNPs electrode. Plenty of researches about Si/C composites as LIBs anodes have verified important functions of carbon layers mentioned above.\textsuperscript{25-27} In our work, in-situ wrapping SiNPs by 2D carbon nanosheets is much more simple and efficient. In Figure 5, the Si/C composite can effectively provide electron transfer pathways, keep good electrical contact, and buffer accommodate volume expansion of SiNPs.

![Figure 5](image)

**Figure 6.** SEM characterization of electrode swelling. (a) SiNPs electrode before cycling. (b) fully lithiated SiNPs electrode after the 1\textsuperscript{st} discharge. (c) SiNPs electrode after the 1\textsuperscript{st} cycle. (d) Si/C electrode before cycling. (e) fully lithiated Si/C electrode after the 1\textsuperscript{st} discharge. (f) SiNPs electrode after the 1\textsuperscript{st} cycle.

In order to implement Si anodes into full cells for practical application, electrode swelling is a key challenge.\textsuperscript{28-30} Limited swelling of the as-prepared Si/C electrode was corroborated by cross-section SEM images. **Figures 6a-6f** are characterizations in the thickness of the pure SiNPs and
Si/C electrodes before cycling, fully discharged to 0.01 V, and fully charged to 1.20 V at the 1st cycle, respectively. The pristine electrodes of the SiNPs (Figure 6a) and Si/C (Figure 6d) have an average thickness of ~13 µm and ~23 µm. The cycled electrodes at lithiated state are ~48 µm (Figure 6b) and ~38 µm (Figure 6e) thick, while they are ~17 µm and ~26 µm at the delithiated state, respectively. The electrode swelling at the lithiated state of the SiNPs and Si/C are 269% [(48-13)/13] and 65% [(38-23)/23], which suggests that the 2D carbon nanosheets can effectively limit the volumetric expansion of the electrode. The electrode thickness of the SiNPs and Si/C increase by 31% [(17-13)/13] and 13% [(26-23)/23] after first-delithiated, which also proves that the Si/C electrode is superior to the SiNPs electrode. The electrode surface of the SiNPs and the Si/C after the 1st cycle can be observed in Figure S9. Obviously, the surface of the Si/C is much more compact and smooth than the SiNPs. Keeping the electrode surface integrated is beneficial for the SEI formation. The structural stability of the SiNPs and Si/C electrode were characterized by SEM. As show in Figure S10a and S10b, the electrode of pure SiNPs after the 1st cycle seriously cracks. While the Si/C electrode clearly reveals no signature of damage after the 1st cycle (Figure S10d) and after the 120th cycle (Figure S10e), including no pores and cracks without electrical contact loss. Figure S10f shows the surface morphology of the electrode after 500 cycles. In spite of some small cracks are present, the electrode still maintains well, no active material peels off. This Si/C composite can well accommodate the volumetric expansion of SiNPs and ensure a stable electrode structure for long term cycling.
Figure 7. Areal capacities of the Si/C electrodes with different areal mass loadings (0.75, 2.47, 3.11, and 5 mg cm\(^{-2}\)). All electrodes are tested at 0.5 mA cm\(^{-2}\) for the 1\(^{st}\) cycle and 2.1 mA cm\(^{-2}\) for the subsequent cycles.

In the practical application, the mass loading of the active material on a unit area of the electrode is a key index of evaluating the battery performance. High areal mass loading can minimize the weight percentage of metal foil current collectors in the battery. However, in order to achieve stable cycling, many publications used low areal mass loading, which have not been apparent enhancements in areal storage capacities. In our work, the cycling performances of batteries with different mass loadings and areal capacities are showed in Figure 7. When the mass loading is 0.75 mg cm\(^{-2}\), excellent cycling performance is achieved with 5.0% capacity decay after 100 cycles. At increased mass loading of 2.47 and 3.11 mg cm\(^{-2}\), 1.81 and 2.47 mAh cm\(^{-2}\) with stable cycling for 100 cycles can still be retained. When the mass loading is as high as 5.00 mg cm\(^{-2}\), the high initial areal capacity of 5.06 mAh cm\(^{-2}\) is obtained at 0.50 mA cm\(^{-2}\). High mass loading inevitably places a much heavier burden on cycling stability. The resulting capacity retention is only 67.3% compared with the 2\(^{nd}\) cycle. The areal capacity of 3.13 mAh cm\(^{-2}\) at 2.10 mA cm\(^{-2}\) after 100 cycles can be achieved. However, this value is still superior to the commercial
graphite anode (3.00 mAh cm\(^{-2}\), the dashed line in Figure 7). As far as we know, such a mass loading level have rarely been reported for Si/graphene, and even for Si/C based anodes. Figure S11, Tables S2 and S3 compare our Si/C composite to representative outstanding research results in the past three years about Si/graphene and Si/C composites as anodes for LIBs. The result definitely indicates the performance of our Si/C composite is comparable or even superior to most of the excellent researches of Si/graphene and Si/C composites reported before. Except the highest mass loading of our work in Tables S2 and S3, the current density of 2.1 mA cm\(^{-2}\) is also relatively large. High current density is a critical factor affecting the electrochemical performance of active materials. Last but not least, when matching the Si/C composite anode with commercial NMC 532 cathode, the full cell displays a reversible capacity of 171 mAh g\(^{-1}\) at 0.5 mA cm\(^{-2}\) and maintains at 132 mAh g\(^{-1}\) after 20 cycles (see Figure S12). The only fly in the ointment is the ICE of as-prepared material is about 70% due to the large specific surface area (104.8 m\(^2\) g\(^{-1}\)). Further studies on how to improve the ICE need be carried out in the near future. In spite of this, our work still demonstrate a novel route to prepare high-capacity Si anodes in practical applications for LIBs.

**Conclusion.** A rational strategy has been demonstrated to prepare graphene-like 2D carbon nanosheets wrapped Si nanoparticles as high areal capacity anode for LIBs. This special structure ensures outstanding electrochemical performance during charge–discharge. After 500 cycles at 420 mA g\(^{-1}\), the Si/C composite can achieve 881 mAh g\(^{-1}\) with about 86.4% capacity being retained. When the mass loading reaches to 5.00 mg cm\(^{-2}\), the Si/C shows an area-specific-capacity of 3.13 mA h cm\(^{-2}\) at 2.1 mA cm\(^{-2}\) after 100 cycles. Anode with high mass loading and high areal capacity is important for powering future high-energy-density LIBs. Compared with some excellent researches about Si/graphene and Si/C composites, our work is competitive as
well. By combining simple and efficient experimental procedure, environmentally friendly and easily obtainable raw materials, and favorable areal capacity, our work demonstrates a promising method to prepare high-energy-density and low-cost battery materials for EVs.

ASSOCIATED CONTENT

Supporting Information.

The Supporting Information is available free of charge on the ACS Publications website at DOI:

Detailed description of the experiments. Additional figures about TGA/BET/SEM/TEM /EIS characterization and full cell electrochemical performance. Additional table about the performance comparison with other Si/C, Si/graphene based materials (PDF)

AUTHOR INFORMATION

Corresponding Authors:
Email: panfeng@pkusz.edu.cn (F.P.)
Email: zhanlin@zju.edu.cn (Z.L.)

ORCID
Zhan Lin: 0000-0001-5009-8198
Feng Pan: 0000-0002-8216-1339

Notes
The authors declare no competing financial interest.

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The Si/C composite is prepared by in-situ wrapping Si nanoparticles by graphene-like 2D carbon nanosheets, which exhibits a high areal capacity of 3.13 mAh cm\(^{-2}\) after 100 cycles at the mass loading of 5.00 mg cm\(^{-2}\) as the anode for lithium-ion batteries.