

Silicon/flake graphite/carbon anode materials prepared with different dispersants by spray-drying method for lithium ion batteries

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Abstract: Silicon/flake graphite/carbon (Si/FG/C) composites were synthesized with different dispersants via spray drying and subsequent pyrolysis, and effects of dispersants on the characteristics of the composites were investigated. The structure and properties of the composites were determined by X-ray diffractometry (XRD), scanning electron microscopy (SEM) and electrochemical measurements. The results show that samples have silicon/flake graphite/amorphous carbon composite structure, good spherical appearances, and better electrochemical performance than pure nano-Si and FG/C composites. Compared with the Si/FG/C composite using washing powder as dispersant, the Si/FG/C composite using sodium dodecyl benzene sulfonate (SDBS) as dispersant has better electrochemical performance with a reversible capacity of 602.68 mA·h/g, and a capacity retention ratio of 91.58 % after 20 cycles.

Key words: lithium ion battery; Si/C composite; spray drying; anode

1 Introduction

Lithium ion batteries are widely used in portable appliances due to a favorable combination of voltage, energy density, and cycling performance [1–7]. Because of its high theoretical capacity (3570 mA·h/g) [8,9], silicon is considered a prospective alternative to commercial graphite (with a theoretical capacity of 372 mA·h/g) anode for Li-ion batteries [10]. However, many obstacles exist for commercialization of Si-based anode materials. Major problems include the huge volume change (>300%) during Li⁺ insertion/ extraction and the low electrical conductivity [9–12]. The huge volume change can lead to loss of electrical contact of Si particles in the electrode, even losing Li⁺ storage ability. Silicon/carbon composite anodes attract great interest because of good electrical conductivity and stress-buffer effect of carbon [13–17]. The preparation method of Si/C composite anode is also important for producing a Si/C composite anode with good electrochemical performance. A spray drying and subsequent pyrolysis approach is thought to be an effective way in preparing spherical particles with homogeneous distribution of chemical components and improving tap density and electrochemical performance of the electrode materials

for lithium ion batteries [16,18,19]. Besides, an effective dispersant can also facilitate the even dispersion of nano-sized or submicron particles into their matrix in the preparation of composites [14,20–22].

In this work, nano-Si, flake graphite, glucose and different dispersants were used to synthesize Si/FG/C composites by a spray drying and subsequent pyrolysis approach. The structure, morphology and electrochemical performance of Si/FG/C composites were investigated in detail.

2 Experimental

1.78 g glucose (C₆H₁₂O₆·H₂O) was dissolved in 250 mL deionized water under continuous stirring. Then, nano-Si (1 g, ~ 30 nm) and flake graphite (2 g, ~ 0.5 μm, called as colloidal graphite powder) were added in the solution. In order to avoid the agglomeration of nano-sized Si and the flotation of submicron flake graphite on the liquid surface [14,20–22], two kinds of dispersants (~ 0.9 g) were also used separately. One was pure sodium dodecyl benzene sulfonate (SDBS), and the other was Diaopai washing powder (WP) which is a composite of 30% SDBS, 30% Na₂SO₄, 25% micro-sized 4A zeolite, 7% Na₂CO₃, 5% Na₂SiO₃·9H₂O, 2% enzyme and 1% carboxymethyl cellulose in mass fraction. As a

result, stable suspension can be obtained after ultrasonic vibration for 2 h.

Subsequently, the suspension obtained was spray dried in a spray dryer at a rate of 15 mL/min with the inlet and outlet temperatures maintained at 160 °C and 110 °C, respectively. The as-prepared precursor was calcined at 800 °C for 2 h in a tube furnace with a flowing argon atmosphere, then cooled naturally to ambient temperature. The final substance was the silicon/flake graphite/carbon composite (Si/FG/C composite) with a corresponding mass ratio of 1:2:1. The flake graphite/carbon composite (FG/C composite) with a corresponding mass ratio of 2:1 was prepared by the same process described above except the addition of nano-Si.

The phase components of the materials were investigated by powder X-ray diffractometry (XRD, Rint-2000, Rigaku) with Cu K_{α} radiation. The morphologies of the samples were examined by scanning electron microscopy (SEM, JSM-6360LV, JEOL). The nitrogen adsorption and desorption isotherms at 77 K were measured using an automated surface area and pore size analyzer (Autosorb/Monosorb, Quantachme).

The working electrodes were prepared by pasting a slurry of 80% (mass fraction) active material, 10% acetylene black, and 10% PVdF dissolved in *N*-methyl-2-pyrrolidone on a copper foil and dried at 120 °C for 12 h. Pure nano-Si electrode was prepared at a mass ratio of 60:20:20 (Si to acetylene black to PVdF). The electrodes were punched into round pellets with a diameter of 14 mm and then were pressed under the pressure of 20 MPa. The CR2032 coin cells were assembled in an argon-filled glove box employing lithium foil as a counter electrode and 1 mol/L $\text{LiPF}_6/\text{EC-EMC-DMC}$ (1:1:1 by volume) as electrolyte.

The charge (Li^+ extraction) and discharge (Li^+ insertion) tests were measured by Neware Tester (CT-3008W-5V1mA-S4, Shenzhen Neware Electronics Co., Ltd., China). The cells were galvanostatically discharged at 60, 30, 15 and 6 mA/g step by step, and charged at 60 mA/g between the cut-off voltage of 0.02–1.5 V. Cyclic voltammetry (CV) and the electrochemical impedance spectra (EIS) were measured on the CHI 600 Electrochemical Workstation. CV tests were conducted at 0.1 mV/s and EIS measurements were conducted in a frequency ranging from 0.01 Hz to 100 kHz with AC amplitude of 5 mV.

3 Results and discussion

Figure 1 shows the XRD patterns of silicon/flake graphite/carbon composite with washing powder dispersant (labeled as Si/FG/C composite-WP), silicon/flake graphite/carbon composite with sodium

dodecyl benzene sulfonate dispersant (labeled as Si/FG/C composite-SDBS), flake graphite/carbon composite with washing powder dispersant (labeled as FG/C composite-WP) and flake graphite/carbon composite with sodium dodecyl benzene sulfonate (labeled as FG/C composite-SDBS). In the FG/C composites, there only exist graphite crystalline diffraction peaks and a broad diffraction peak around 23° attributed to the amorphous glucose-pyrolyzed carbon. For the Si/FG/C spherical composites, the crystalline diffraction peaks of silicon can be observed besides those of graphite and amorphous carbon, and no other peaks of the impurities such as SiC or SiO_2 can be found. All of those indicate that the FG/C samples are the composites of flake graphite and amorphous glucose-pyrolyzed carbon, while the Si/FG/C samples are the composites of nano-Si, flake graphite and amorphous glucose-pyrolyzed carbon.

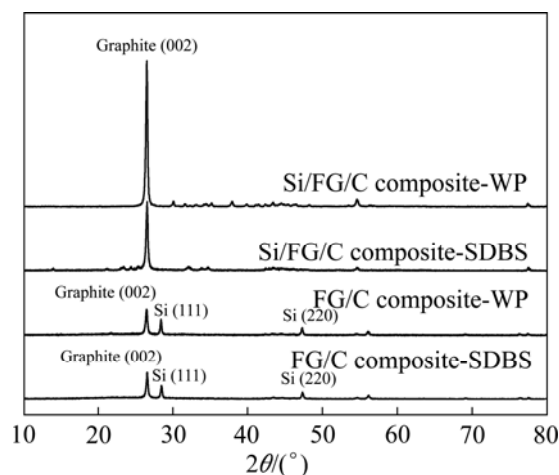


Fig. 1 XRD patterns of FG/C composites and Si/FG/C composites

Figure 2 shows the SEM images of FG/C composites and Si/FG/C composites. FG/C composites are irregularly shaped particles composed of flake graphite and glucose-pyrolyzed carbon as the binder. Si/FG/C composites present good spherical appearance with some pores. The BJH pore size distribution curves for Si/FG/C composites in Fig. 3 indicate that Si/FG/C composites have a wide pore size distribution of 0.3–150 nm. As shown in Table 1, the average BJH pore sizes of Si/FG/C composite-SDBS and Si/FG/C composite-WP are separately 3.216 nm and 3.423 nm, while Si/FG/C composite-SDBS has a larger BET surface area (12.038 m^2/g) and pore volume (0.027 cm^3/g). It is considered that those pores mainly arise from the decomposition of glucose during pyrolysis [23], and the differences of the BET surface area and pore volume between Si/FG/C composite-SDBS and Si/FG/C composite-WP may be mainly attributed to the different frameworks formed by

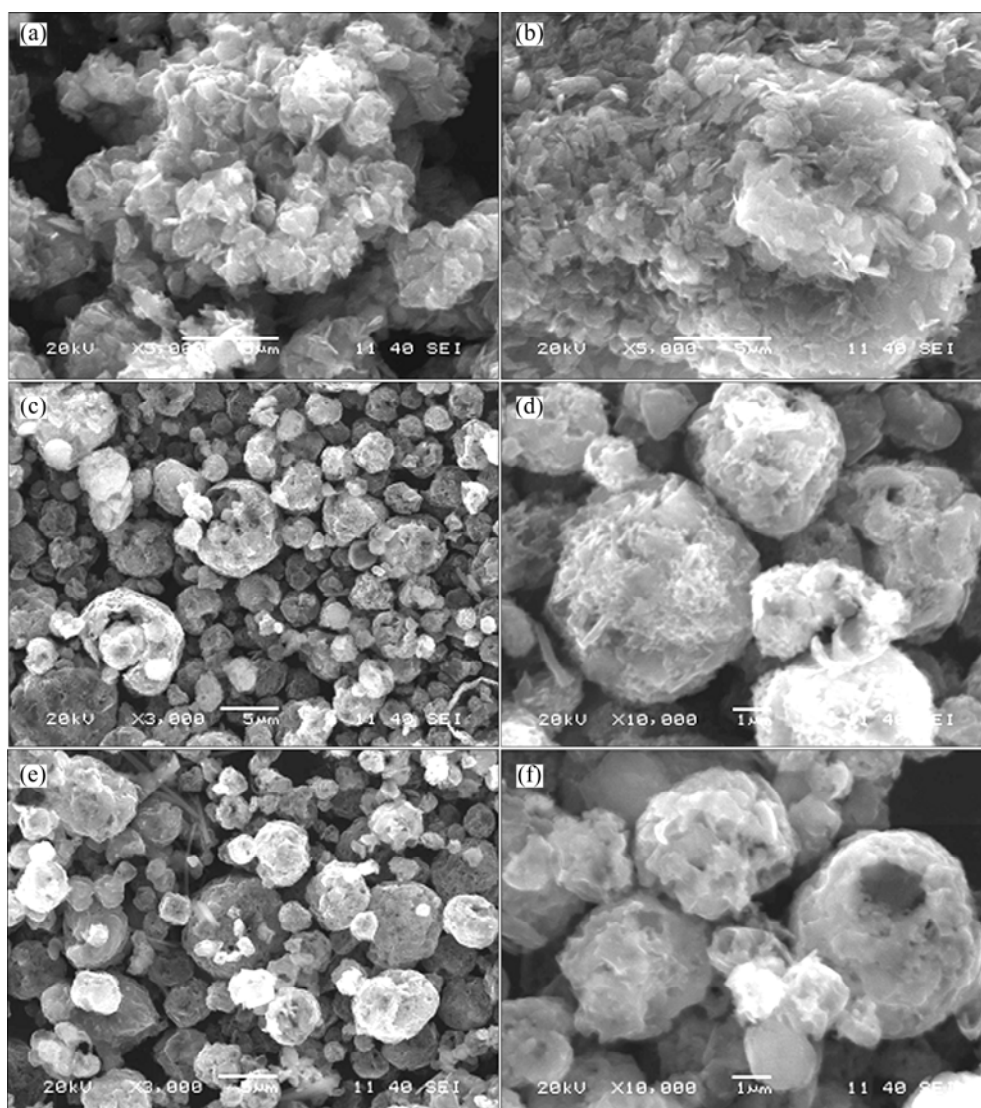


Fig. 2 SEM images of FG/C composite-WP (a), FG/C composite-SDBS (b), Si/FG/C composite-WP (c, d) and Si/FG/C composite-SDBS (e, f)

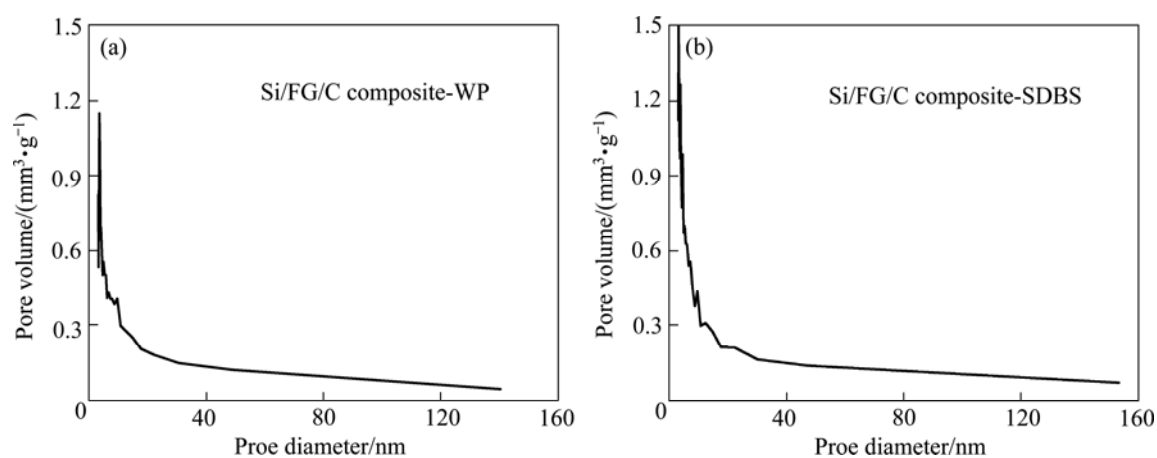


Fig. 3 BJH pore size distribution curves of Si/FG/C composites: (a) Si/FG/C composite-WP; (b) Si/FG/C composite-SDBS

nano-Si and flake graphite under the effects of SDBS and WP dispersants, respectively [20–22]. It is well known that different dispersants can change the interface tension,

viscosity and dispersivity of the suspension [20–22], which is composed of graphite ($\sim 0.5 \mu\text{m}$), nano-Si ($\sim 30 \text{ nm}$) and glucose solution. Thus, different

Table 1 BET surface area, BJH pore size and pore volume of Si/FG/C composites

Sample	BET surface area/($\text{m}^2\cdot\text{g}^{-1}$)	BJH pore size/nm	BJH pore volume/($\text{cm}^3\cdot\text{g}^{-1}$)
Si/FG/C-WP	4.756	3.423	0.018
Si/FG/C-SDBS	12.038	3.216	0.027

frameworks may be formed after spray drying and pyrolysis.

The charge–discharge profiles at the first two cycles and the cycling performance of different anodes are given in Fig. 4 and Fig. 5, respectively. The

corresponding charge/discharge capacities and coulombic efficiencies at the first two cycles are listed in Table 2. Owing to the large specific capacity of nano-Si, the initial reversible capacities of both Si/FG/C spherical composites are about twice those of FG/C composites. Compared with pure nano-Si, whose initial coulombic efficiency is only 45.38 % and the reversible capacity decays rapidly from the initial 1509.79 $\text{mA}\cdot\text{h/g}$ to 160.88 $\text{mA}\cdot\text{h/g}$ at the 20th cycle, the initial coulombic efficiencies and cycleabilities of Si/FG/C spherical composites are obviously improved. It is also interesting that the initial coulombic efficiencies of both Si/FG/C spherical composites increase by about 14% compared

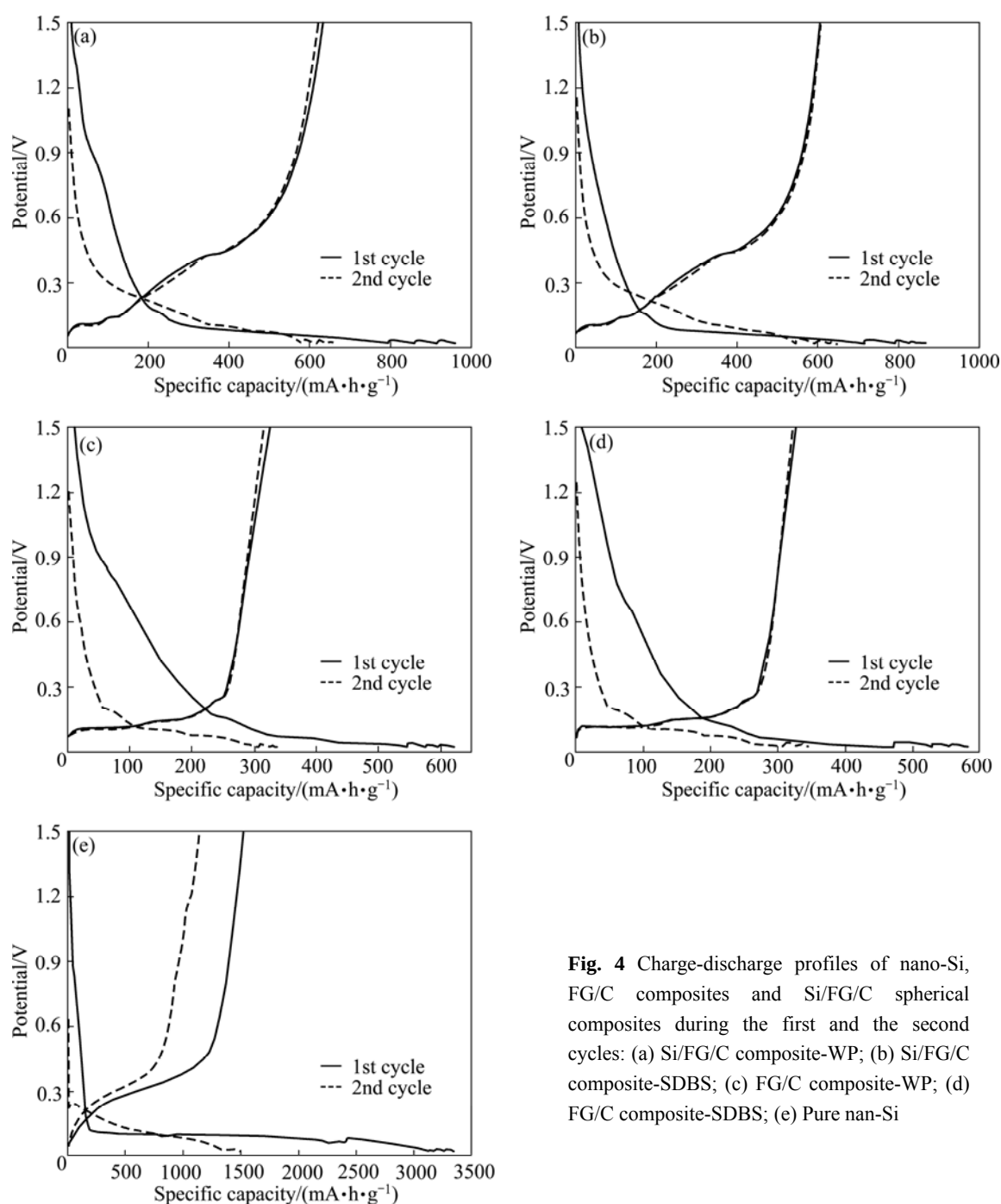
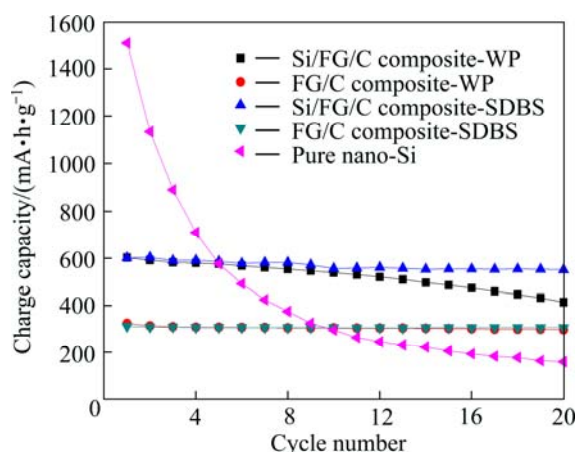


Fig. 4 Charge-discharge profiles of nano-Si, FG/C composites and Si/FG/C spherical composites during the first and the second cycles: (a) Si/FG/C composite-WP; (b) Si/FG/C composite-SDBS; (c) FG/C composite-WP; (d) FG/C composite-SDBS; (e) Pure nan-Si

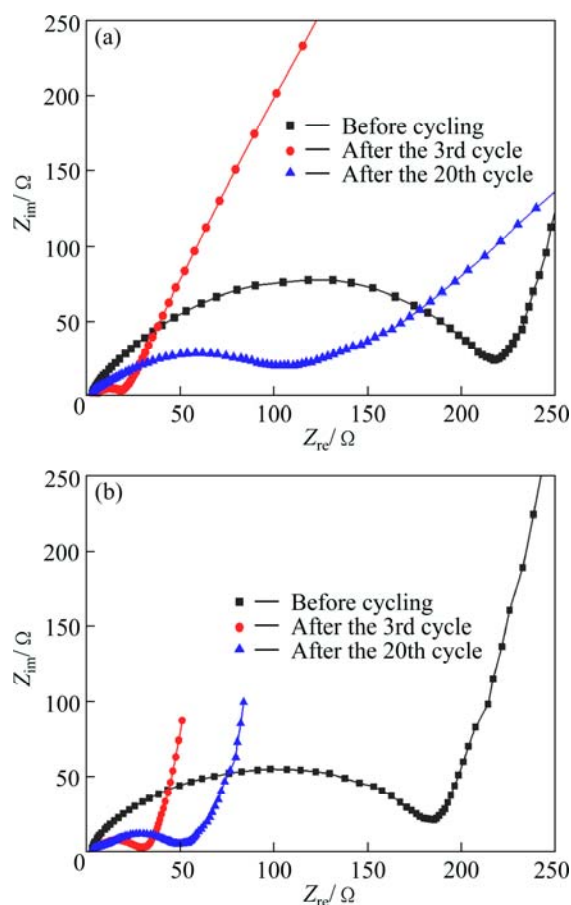
Table 2 Electrochemical characteristics of nano-Si, FG/C composites and Si/FG/C composites

Electrode	The 1st cycle			The 2nd cycle		
	Charge capacity/ (mA·h·g ⁻¹)	Discharge capacity/ (mA·h·g ⁻¹)	Coulombic efficiency/%	Charge capacity/ (mA·h·g ⁻¹)	Discharge capacity/ (mA·h·g ⁻¹)	Coulombic efficiency/%
Si/FG/C-WP	630.80	958.28	65.82	618.68	660.46	93.68
Si/FG/C-SDBS	602.68	864.54	69.71	604.80	644.23	93.88
FG/C-WP	323.73	621.22	52.11	313.82	338.12	92.81
FG/C-SDBS	325.22	581.27	55.95	321.76	345.03	93.26
Nano-Si	1509.79	3326.83	45.38	1134.40	1457.61	77.83

**Fig. 5** Cycle performance of nano Si, FG/C composites and Si/FG/C composites

with the FG/C composites (Table 2). With the assumption that the reversible capacities of flake graphite and pyrolytic carbon in the Si/FG/C spherical composites are the same as those in the FG/C composites, and the reversible capacity delivered by Si (ca. 25%) in Si/FG/C composite (ca. 610 mA·h/g) for the 2nd cycle can be estimated to be about 380 mA·h. Therefore, the specific capacity of Si in the composite is calculated to be 1520 mA·h/g, which is about 390 mA·h/g higher than that of pure Si (ca. 1134 mA·h/g). All of those mentioned above may be caused by the synergistic effect of nano-Si, flake graphite and glucose-pyrolyzed carbon [24].

Furthermore, compared with the FG/C composite-WP and Si/FG/C composite-WP, both the FG/C and Si/FG/C composites using SDBS as dispersants have correspondingly higher first coulombic efficiencies (about 4% higher) and the lower irreversible capacities (Table 1). As shown in Fig. 5, the charge capacity after 20 cycles for the Si/FG/C composite-WP retains only 60.11% of the initial capacity, while the capacity retention ratio of the Si/FG/C composite-SDBS after 20 cycles is 91.58%. Better cycling performance of the Si/FG/C composite-SDBS can be explained by the difference in specific pore volume of the composite particles. The Si/FG/C composite-SDBS is obviously more porous than the Si/FG/C composite-WP (Figs. 3(a) and (b)). The micropores or/and micro-tunnels in the

**Fig. 6** Nyquist plots of Si/FG/C composite-WP (a) and Si/FG/C composite-SDBS (b)

particles can accommodate the huge volume expansion/contraction of the Si-based anode materials [25,26]. Thus, good cycling performance can be achieved for the porous materials.

To verify the difference of degradation, the electrochemical impedance spectra of the Si/FG/C composite-WP and Si/FG/C composite-SDBS electrodes were investigated. In all Nyquist plots, there is a depressed semicircle section in the medium and high frequency range and an angled straight line in the low frequency range. The diameter of the depressed semicircle corresponds to the charge transfer resistance and the angled straight line is related to a diffusion controlled process [27,28]. For the Si/FG/C

composite-WP electrode, the semicircle diameter—after the 3rd cycle is about 1/10 that before cycling. But the resistance of the electrode presents significant difference after the 20th cycle. The semicircle diameter increases by about four times. However, the semicircle diameter for the Si/FG/C composite-SDBS electrode after the 3rd cycle is about 1/6 that before cycling, while the semicircle diameter after the 20th cycle increases only about 0.7 times. The previous studies [24,28,29] have shown that the resistance of the electrode will decrease after the electrode is activated under the condition that the conductive network of the electrode is maintained. These results indicate that the integrity of conductive network for the Si/FG/C composite-WP electrode has been destroyed in 20 cycles, whereas that for the Si/FG/C composite-SDBS electrode is kept much better, which is consistent with their cycling performance illustrated in Fig. 5.

Figure 7 shows the cyclic voltammograms of FG/C composites and Si/FG/C spherical composites for the 1st and the 2nd cycle. In the 1st cycle, the broad and weak cathodic peaks at about 0.8 V for all the four composites disappear in the 2nd cycle, corresponding to the reductive decomposition of electrolyte and the formation of the SEI film on the surface of the electrodes [30]. The sharp cathodic peaks below 0.15 V are due to the Li^+ insertion

into the active materials. The anodic peaks between 0.15 and 0.35 V in the four curves are mainly related with the Li^+ extraction from flake graphite of the composites. The anodic broad peaks around 0.45 V, only existing in the curves of the Si/FG/C spherical composites, is related to the extraction of Li^+ from nano-Si. In the 2nd cycle, all anodic peaks for the four composites become higher, which is related to the activation of the active materials [14,30]. All the results agree with the conclusions from the charge–discharge profiles of the composites during the 1st and the 2nd cycle, as shown in Fig. 4.

4 Conclusions

1) Si/FG/C composites were synthesized with dispersants by spray drying and subsequent pyrolysis. The samples are the composites of nano-Si, flake graphite and amorphous carbon, which present good spherical appearance with some pores (0.3–150 nm) in the particles.

2) The electrochemical properties of Si/FG/C composites are much better than both pure nano-Si and FG/C composites. Compared with Si/FG/C composite-WP, Si/FG/C composite using SDBS as dispersant has a better electrochemical performance with a reversible

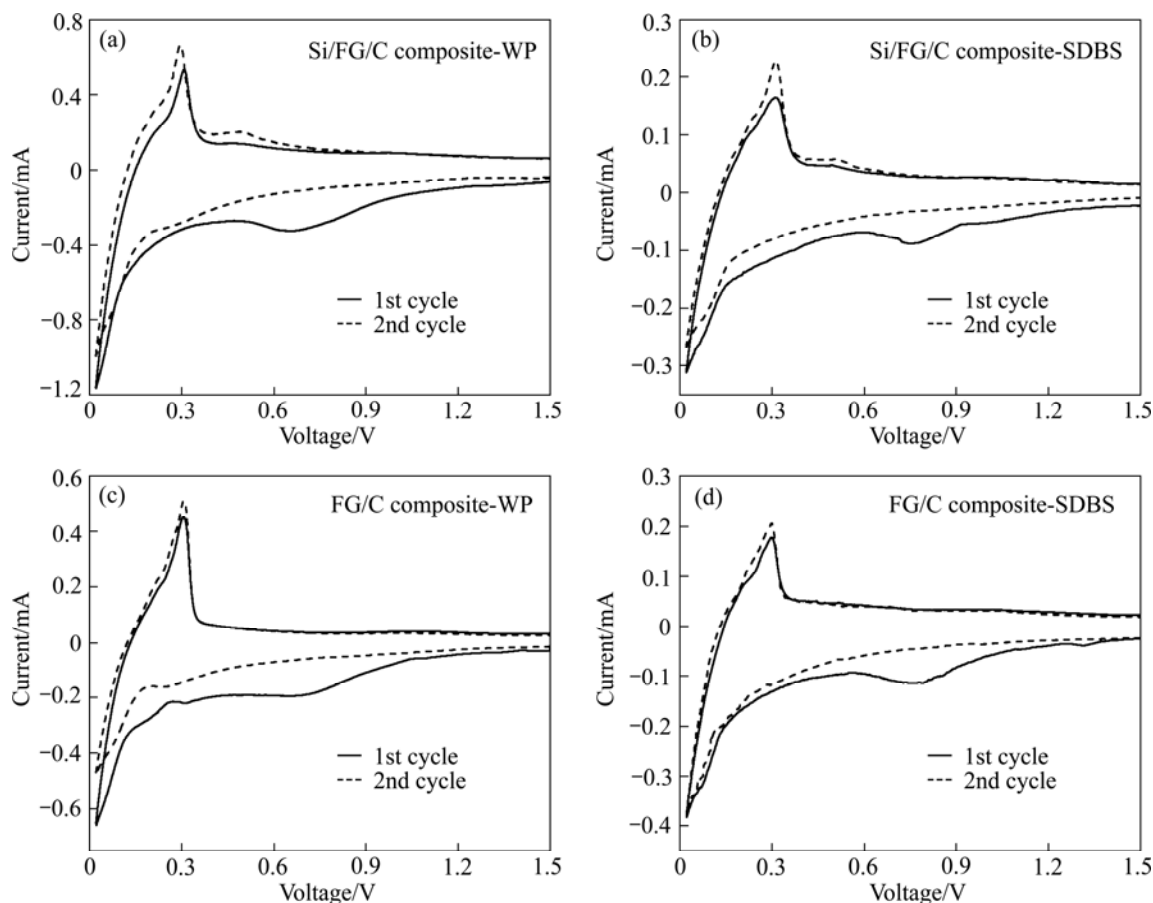


Fig. 7 Cyclic voltammograms of FG/C composites and Si/FG/C spherical composites

capacity of 602.68 mA·h/g, an initial coulombic efficiency of 69.71 %, and a capacity retention ratio of 91.58 % after 20 cycles.

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分散剂对喷雾干燥制备 硅/鳞片石墨/热解炭复合负极材料的影响

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摘 要: 采用喷雾干燥及热解法制备硅/鳞片石墨/热解炭复合负极材料, 研究分散剂对材料的影响。材料结构与性能通过 XRD、SEM 及电化学测试来表征。实验结果表明: 材料具有硅/鳞片石墨/热解炭复合结构及规则的球形表面, 其电化学性能比纯硅有明显的改善。与采用洗衣粉作为分散剂制备的复合材料相比, 采用 SDBS 作为分散剂制备的复合材料具有更好的电化学性能, 其可逆比容量达到 602.68 mA·h/g, 经 20 次循环后容量保持率高达 91.58%。

关键词: 锂离子电池; Si/C 复合材料; 喷雾干燥; 负极

(Edited by Hua YANG)