



Industrial waste silica preparation of silicon carbide composites and their applications in lithium-ion battery anode



Kaixiong Xiang^{a,b}, Xianyou Wang^{a,*}, Manfang Chen^a, Yongqiang Shen^a, Hongbo Shu^a, Xiukang Yang^a

^a Key Laboratory of Environmentally Friendly Chemistry and Applications of Ministry of Education, Hunan Province Key Laboratory for Electrochemical Energy Storage and Conversion, School of Chemistry, Xiangtan University, Hunan, Xiangtan, 411105, China

^b College of Metallurgy Engineering, Hunan University of Technology, Zhuzhou, 412008, China

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ABSTRACT

Silicon particles with uniform sizes were formed by cleaning solid organic wastes from the silicon industry with ether and nitric acid and ball milling the products. The resulting silicon was then used to prepare anode materials for lithium-ion batteries by mixing the silicon with graphite microspheres (GMs) and sintering the mixture in the presence of sucrose. X-ray diffraction, scanning electron microscopy, transmission electron microscopy, and thermo-gravimetric analysis were used to characterize the composites. The silicon and GM mixtures contained tightly bound amorphous carbon and ball-like composites. These P-Si/C composites had high capacities (520 mAh/g at 50 mA/g and 320 mAh/g at 500 mA/g) and excellent charge-discharge cycling stabilities, displaying only a 8.8% loss after 100 cycles at 50 mA/g.

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1. Introduction

Lithium-ion batteries are widely used as power supplies for portable electronic devices [1–3]. Therefore, the development of improved cathode materials for lithium-ion batteries is needed. The most common anode materials employed in lithium-ion batteries are lithium intercalation compounds like graphite [4]. However, the limited theoretical capacity of graphite (372 mAh g⁻¹) as an anode material cannot meet the needs of lithium-ion batteries [5]. Therefore, research on anode materials has become a major focus in the field of lithium-ion batteries [6].

After many studies, silicon, which has a high theoretical capacity (4200 mAh/g), has emerged as a potential candidate material for lithium-ion battery anodes [7–9]. However, silicon-lithium alloys increase in size during the electrochemical reactions associated with lithium-ion battery anodes. As a result, cracks form in the electrode, and contact failures reduce conductivity. The resulting cycling performance is poor, making it difficult to use silicon for commercial applications [10].

In this work, silicon/carbon composite anode materials were prepared using amorphous carbon from commercial fossil ink microspheres, recycled silicon, and pyrolysis in the presence of sucrose. At present, the use of industrial wastes containing solid organic silicon requires dissolution in acid and precipitation in an alkaline solution to recover copper compounds, but the remaining silicon particles are not fully used. In this work, the silicon carbon anode materials were prepared by uniformly mixing the low-cost silicon particles, graphite microspheres (GMs), and sucrose. GMs and amorphous carbon can be used as buffers in lithium-silicon alloys during charge cycling [11,12]. Amorphous carbon effectively connects silicon with the GMs, forming a highly conductive network [13–15]. This approach uses solid industrial waste for silicon recovery and utilization, while providing a low-cost and high-performance silicon-based anode material for lithium-ion batteries.

2. Experimental section

2.1. Synthesis of samples

Waste silicon (W-Si) was provided by the Dongyue Group Co.,

* Corresponding author.

E-mail address: wxianyou@yahoo.com (X. Wang).

Table 1
XRF analysis of W-Si P-Si, Fe-Si and FeCu-Si.

Sample	Si (%)	O (%)	Cu (%)	Fe (%)	Cl (%)	Ca (%)	Al (%)
W-Si	37.17	19.39	19.92	9.46	3.49	1.83	1.69
FeCu-Si	40.50	7.63	26.57	16.39	1.50	1.34	1.50
Fe-Si	80.32	5.40	0.14	12.05	0.17	1.07	0.49
P-Si	97.56	1.71	0.02	0.25	0.05	0.16	0.07

Ltd. The silicon purification process involved crushing the W-Si into a fine powder using a high-speed universal mill and washing the resulting powder for 1 h under ultrasonic agitation. The resulting dry granules are referred to as FeCu-Si. The FeCu-Si was cleaned in a nitric acid solution with ultrasonic agitation, and the static half hour was divided into upper and lower layer. Ball milling was performed, and the FeSi₂-containing Fe-Si particles were collected after the upper and lower particles were dried. Pure silicon particles are referred to as P-Si. The composites were prepared using 1.2 g of sucrose, which was added to the appropriate amount of deionized water to form a solution. Subsequently, 0.3 g of raw silicon and 0.9 g of graphite were added to the solution. The mixture was mixed evenly until dry and heated for 5 h in argon at 800 °C. Finally, the carbonization product was crushed and filtered through a 200 mesh sieve.

2.2. Sample characterization

The samples' crystal phases, morphologies, and specific surface areas were characterized using powder X-ray diffraction (XRD,

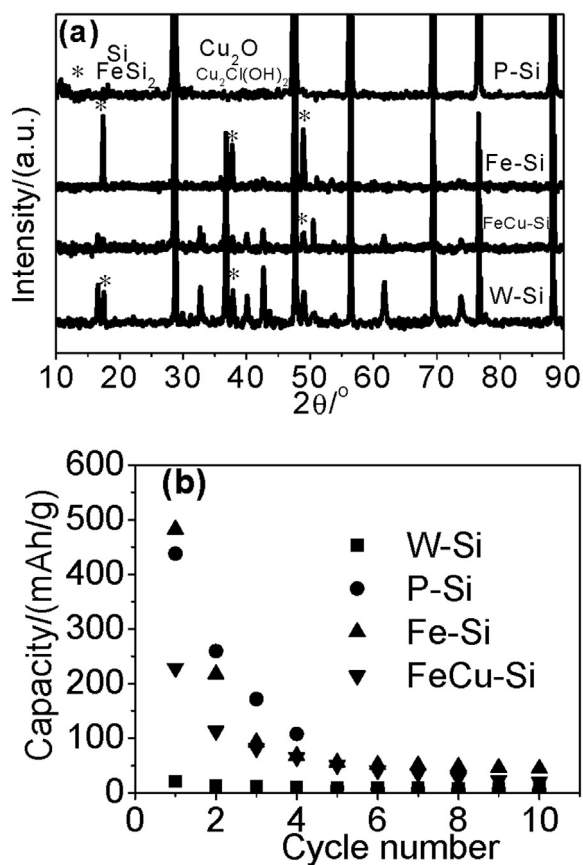


Fig. 1. XRD patterns (a) and charge capacities at 50 mA/g (b) for W-Si, FeCu-Si, Fe-Si, and P-Si.

Siemens, D5000) with Cu $\kappa\alpha$ radiation, scanning electron microscopy (SEM, JSM-6700F, JEOL, Tokyo, Japan), and transmission electron microscopy (TEM, JSM-3010), respectively. Thermogravimetric analysis (TGA) was carried out using an STA 449C instrument (NETZSCH, Germany) at a heating rate of 5 °C/min in air.

2.3. Electrochemical measurements

Electrochemical performances were evaluated in coin-type cells using lithium metal as the reference electrode. The working electrode was prepared from an 8:1:1 (by mass) mixture of silicon-carbon composite/acetylene black (ATB)/polyvinylidene fluoride (PVDF) in *N*-methylpyrrolidone (NMP). The resulting slurries were cast onto copper foils and dried at 120 °C under vacuum for 12 h. The CR2016 coin-type cells were assembled in an argon-filled glove box. Polypropylene microporous films (Celgard 2400) were used as separators, and the electrolyte was 1 mol/L LiPF₆ in a mixture of ethylene carbonate (EC) and dimethyl carbonate (DMC) (1:1, v/v). Cyclic voltammetry (CV) and electrode capacity measurements were performed with an electrochemical workstation (Princeton, Parstat2273) and an electrochemical test instrument (Land, CT2001A). Galvanostatic tests were carried out with an LHS testing instrument between 0 V and 2 V at various initial charge ratios.

3. Results and discussion

Table 1 shows the results of XRF analyses of the W-Si, P-Si, Fe-Si, and FeCu-S samples. Aside from silicon, W-Si contained many

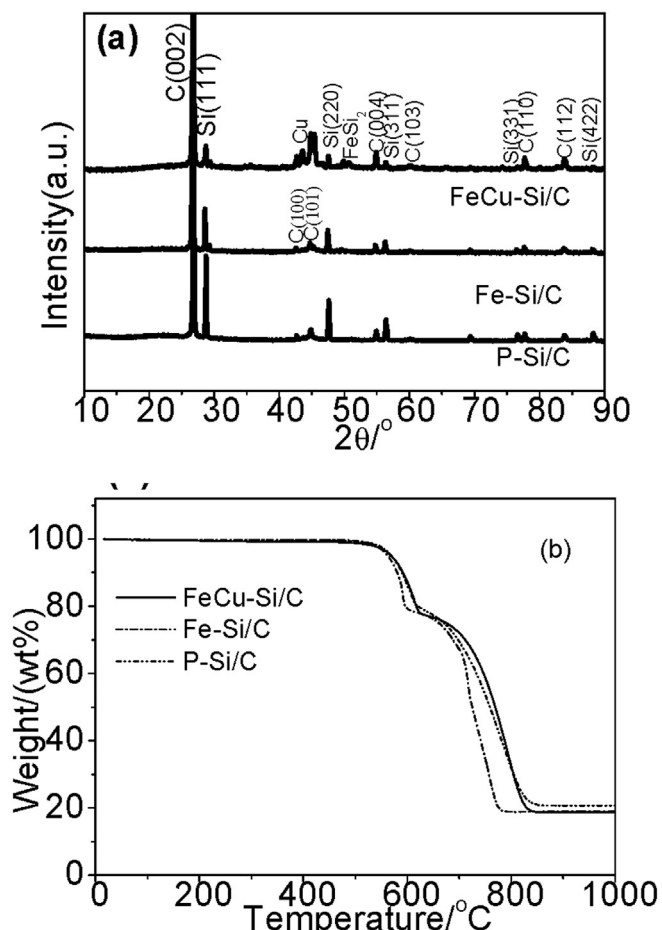


Fig. 2. XRD patterns (a) and TGA curves (b) of the composites.

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