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Processing silicon microparticles recycled from wafer waste via Rapid Thermal Process for lithium-ion battery anode materials



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HIGHLIGHTS

- Recycling silicon microparticles from wafer slicing waste for Si-based anodes.
- Designing an environmentally benign procedure to engineer the electrode.
- Enhancing performance of the anodes via Rapid Thermal Process.

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ABSTRACT

A vast quantity of waste sludge is generated during the silicon wafers slicing process in semiconductor and photovoltaic industries. Turning the waste powder into high-value products is of strategic importance for industrial processes. The purified Si microparticles (Si-MP) are recycled by a simple and fast procedure, Rapid Thermal Process (RTP). A prominent anodic material of Si-MP/Carbon composite with porous structure is obtained via in-spaced carbonization of water-soluble binder sodium carboxymethyl cellulose during RTP. This strategy provides buffer space, which is constructed by carbon porous continuous conductive framework throughout the entire electrode, to resist local stress and intense volume variation. In addition, a sufficiently electrochemically stable solid-electrolyte interphase layer is accomplished with the coating of SiO_x film and amorphous carbon on the surface of Si-MP. Under these circumstances, the enhanced electrodes achieve a first cycle efficiency of approximately 80% and a reversible charge capacity of 800 mAhg⁻¹ over 100 cycles at 0.5 Ag⁻¹ with good retention. Through a green and simple procedure, a remarkable Si-MP embedded carbon-matrix with porous structure is established to achieve commercially high performance Si-MP/C composite anodes and also to resolve the issues of waste disposal.

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1. Introduction

The rapid, ongoing growth of semiconductor and solar cell industries has invested a tremendous amount of raw materials. In the midst of the raw materials, Silicon (Si) is the most widely used material in these industries. However, a vast quantity waste sludge is produced from the Si wafers slicing process during the photovoltaics and electronic device manufacturing. This causes an enormous loss of valuable resource as well as pollution [1,2]. Due to the demand of high electrical storage systems and the environment impacts of waste disposal, recycling these Si micro-sized particles (Si-MP) from waste sludge into a valuable product is of great

interest to the researches and industries [2–7].

Si has been regarded as one of the promising next-generation anode materials because of its high theoretical gravimetric capacity of ~4200 mAhg⁻¹ and low electrochemical potential [8]. However, severe pulverization of Si and loss of electrical contact between active materials and current collector caused by its large volumetric change of over 300% during the insertion and extraction process of Li⁺ ions result in battery's severe capacity decay. As mentioned, large volume expansion and loss of electrical contact are the main issues for the failure of Si anode. For the purpose of improving the capacity retention of Si-based LIBs, nanostructured Si have been proposed as different structures such as core-shell nanowires [9,10], yolk-shell particles [11,12], hollow particles and tubes [13,14], porous Si [15,16], Si–C composites [17,18]. Among several approaches, preparing Si/Carbon composites by coating

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Carbon(C)-related materials on the surface of Si particles or dispersing Si within C, is one of the attractive methods to buffer the mechanical strain and build up a conductive continuous framework for active Si [10,19–22]. It takes advantage of the positive properties of Si and C to improve the overall electrochemical performance of the anode for LIBs. Through minimizing the straightforward exposure of Si particle to the electrolyte and further suppressing the volume expansion of active Si, the cycle stability of the battery thus could be enhanced. Therefore, the growth of SiO_x or carbon-related materials coatings with a thickness in 2–10 nm also have revealed significantly promoted formation of stable SEI film on the surface of the active particles, thus improving capacity retention during cycling [23–25]. Apart from coating a layer on the particles' surface, buffer space is needed to alleviate the drastic volume expansion of the active Si [26–29]. Furthermore, the suitable porosity in the carbon layer of the Si/Carbon composites has been reported to play a structural buffering role to minimize the mechanical stress induced by the huge volume change of Si and provided conductive framework to maintain the stability of the electrodes [30–32]. Combining the advantages of both surface coating and buffer space, it is possible to achieve stable silicon electrodes with a capacity that meets the requirements of commercial LIBs.

Obviously, the impressive advances in Si anodes are developed in the past. However, the high reliance on nanostructured Si material causes high production cost and thus not yet scalable owing to its complex synthetic procedures that should be conquered before their industrialization and commercialization. Therefore, attempts to use Si micro-sized particles as low-cost raw materials have showed intense interest [25–27]. This implies that, Si-MP is more promising as anode materials for the practical industrial application. It is because they are cheap and readily available to use. However, there is a big challenge associated with using the micro-sized waste sludge particles as the raw materials of the battery. The volume expansion occurs more drastically when Si particles are larger than ~150 nm [33,34]. The repeating volume change not only destroys the internal electrical contact, but also damages the as-formed SEI layer and exposes the new surface, which continuously reacts with the electrolyte. To resolve these problems, engineered void space and the electrolyte blocking layer were introduced and proven to be very powerful [35–38].

As the matter of fact, the development manufacturing process for the batteries has been imposed due to the growing concerns on environment issue in recent years. Because of that, the water-soluble sodium carboxymethyl cellulose (CMC) is used as binder to replace the toxic organic solvent. CMC binder can cover the particles rather uniformly and it can maintain a stable and perfect surface coating after drying [39–41]. Therefore, CMC binder can be a good C-precursor [42–44]. In this work, a green, economical, and short duration strategy was introduced to modify the waste-sludge Si-MP.

Rapid Thermal Process (RTP) is a process used in semiconductor industry which heats the material to high temperatures within a time scale of several seconds. Then the process cools slowly to avoid the breakage due to thermal shock. The heat treatment is directly practiced on the electrodes after the slurry-coating process. The RTP engineered the conductive framework as the buffer space throughout the electrodes by the in-spaced carbonization of the CMC binder and modified the surface architecture of Si-MP, simultaneously. To underline this ordinary procedure, the advantages of the RTP include: (1) the construction of electrode blocking layer (SiO_x and amorphous C) around Si-MP which can prevent the direct contact between active Si and the electrolyte; (2) the formation of unique porous structure to provide sufficient buffer space which can alleviate the drastic volume expansion; and (3) the

carbonized CMC provides a conductive framework throughout the electrode to enhance the electrical conductivity and further improves the capacity retention of the battery. Besides, it allows better dispersion of Si-MP powder in the binder since the conductive additive is excluded. The RTP approach potentially presented a relatively low-cost and environmentally benign procedure in converting slicing waste to new commercial energy application.

2. Experimental section

2.1. Source of active materials

The silicon slicing sludge used in this work was obtained from cutting waste produced during the wafer-sawing process in a Taiwan semiconductor company. Fig. S1 illustrates that the particle size distribution of the purified waste powder, indicating that the size distribution of waste particles mainly ranged from 500 nm to 10 μm. Through a certain purification process (Patent publication number: US20130230445 A1) [45], SiC particles and others metal impurities obtained from the saw-wiring process were excluded from the waste sludge.

2.2. Fabrication and assembling active electrodes

For the working electrodes, the as-received silicon waste powders (60 wt%) were fabricated on copper foil using a blade coater with sodium carboxymethyl cellulose (CMC, 40 wt%) (Daicel Co. Ltd) as a binder. The electrodes were dried for 12 h at 110 °C under vacuum. Subsequently, the dried electrodes were subjected to the rapid thermal annealing process (RTP, using Premtek RTP T41) at a heating rate of 50 °C s⁻¹, then held for 20 min at 680 °C under vacuum (1 Torr). The rapid thermal shock rendered the so-called in-spaced carbonization of CMC binder on copper foil, and thus created a carbon matrix with Si-MP confined in between (denoted as Waste-RTP). For the comparison, an electrodes (Waste-Normal, 60:30:10 wt% = Waste: Super-P: CMC) were developed. These electrodes were used without RTP treatment. The comparative electrodes emphasized the positive effect of RTP on the electrochemical properties that were evaluated on CR2032 coin half-cells with lithium metal as the counter/reference electrode. The as-prepared electrodes were dried in the vacuum oven for overnight, then fabricated in an argon-filled glovebox containing separator (polypropylene) and electrolyte (1 M LiPF₆ in ethylene carbonate/diethyl carbonate = 1:1, Samsung Co.). The schematic diagram of fabrication procedure and hierarchical structure of Si-MP/C composite electrodes are shown in Fig. 1.

2.3. Materials characterization and electrochemical analysis

To determine the phase of the as-received waste sludge, X-ray diffraction (XRD, Bruker D8 advance) with Cu K α radiation was applied. The morphological assessments of the waste sludge, Waste-RTP and Waste-Normal were performed by field emission scanning electron microscopy (FE-SEM, JEOL 7600F, Japan) and high-resolution transmission electron microscopy (HR-TEM, JEOL JEM-3000F). Raman scattering spectra (Horiba IHR-550, 532 nm laser) and X-ray photoelectron spectroscopy (XPS, ULVAC-PHI PHO 5000 Versaprobe II) were conducted to investigate bonding state and the surface elemental information of the electrode before and after RTP. Meanwhile, the direct observations of the cross section for the electrodes before and after cycling were applied by Focused Ion Beam-Scanning Electron Microscopy (FIB-SEM, Helios Nanolab 600i System). The cells were cycled by galvanostatic charging/discharging between 0.01 V and 1.2 V versus Li⁺/Li on a multichannel testing system (Arbin BT-2000) at 0.2 Ag⁻¹ for first 2 cycles and

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