Robust 3D host for sodium metal anodes with excellent machinability and cycling stability

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Experimental

Deposition of 3D interconnected porous α-Fe_2O_3 on the carbon textiles

Firstly, carbon textiles (CTs, CeTech W0S1002, through-plane resistance < 5 mΩ cm^{-2}) were cleaned by sonication in acetone, ethanol, and deionized (DI) water for 30 min, respectively. Then the CTs were refluxed in 3 M HNO_3 solution for 5 h at 80 ˚C and washed by DI water, followed by drying under vacuum at 60 ˚C overnight. The acid-treated CTs were used as the substrates for the assembly of β-FeOOH on the CTs via a facile chemical bath deposition (CBD) process. After the sonication for 15 min, the CTs were immersed in the 0.1 M $FeCl_3$ aqueous solution at 100 ˚C for 5 h, the obtained CTs with the assembly of β-FeOOH nanocrystals (FeOOH@CTs) were rinsed with DI water for several times, followed by annealing in ambient air condition at 450 ˚C for 8 h to convert into α-Fe_2O_3. The final product was labelled as Fe_2O_3@CTs (FCTs).

Infiltration of molten sodium into the 3D FCTs

The infiltration of sodium into the obtained 3D FCTs was carried out in an argon-filled glove box with O_2 and H_2O level below 0.1 ppm. Firstly, metallic Na was

heated to melt at 300 °C in a nickel boat on a hotplate. Subsequently, the edge of the 3D FCTs piece was dipped into the molten Na, which infuses into the 3D scaffold steadily and wet the whole matrix. The final obtained sodium infiltrated 3D FCTs were labelled as SFCTs. In the final SFCTs, the mass percent of metallic Na, Fe_2O_3, and carbon textiles is approximately 70.2%, 1.3%, and 28.5%, respectively. The mass loading of Na in the SFCTs is approximately 30 mg cm^{-2}.

Characterizations

SEM images of all samples were observed by a field-emission scanning electron microscopy (FE-SEM, HITACHI SU8010) at 3 kV. The energy-dispersive X-ray elemental mapping was obtained on a IXRF system 550i SDD detector. X-ray diffraction (XRD) measurements were performed on a PANalytical XRD system (X'Pert Powder) with Cu *K*α radiation (λ = 1.54056 Å). TEM and high-resolution TEM (HR-TEM) were carried out using a FEI Tecnai G20. Symmetric type-2032 coin cells were assembled with two identical electrodes in an argon-filled glove box to evaluate the stripping/plating process of Na. The common electrolyte consists of 1.0 M $NaClO_4$ in ethylene carbonate (EC) and dimethyl carbonate (DMC) was employed for the symmetric cells. The separator used for all the cells was a polypropylene membrane (Celgard). Galvanostatic cycling was conducted on a LANHE test system (CT2001A) at different current density with the stripping/plating capacity of 1 mAh cm^{-2} in all cases. Electrochemical impedance spectroscopy (EIS) was carried out using a electrochemical workstation (Zahner, Zennium) at room temperature between 100 kHz and 0.01 Hz with an amplitude of 5 mV before initial cycle and after 10 cycles.

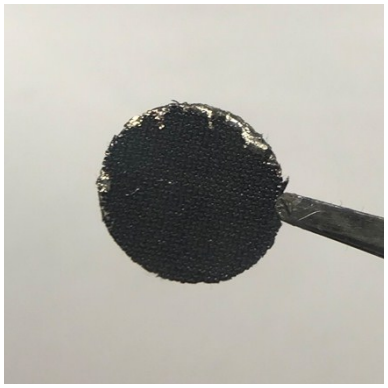

Fig. S1 The photograph of pure CTs after molten Na infiltration.

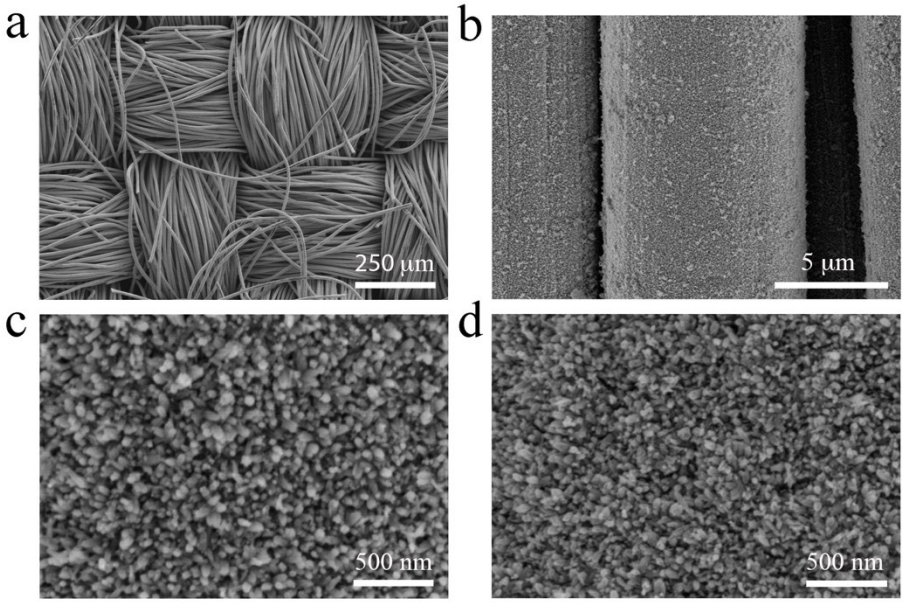

Fig. S2 FE-SEM images of (a, b, c) FeOOH@CTs and (d) Fe_2O_3@CTs.

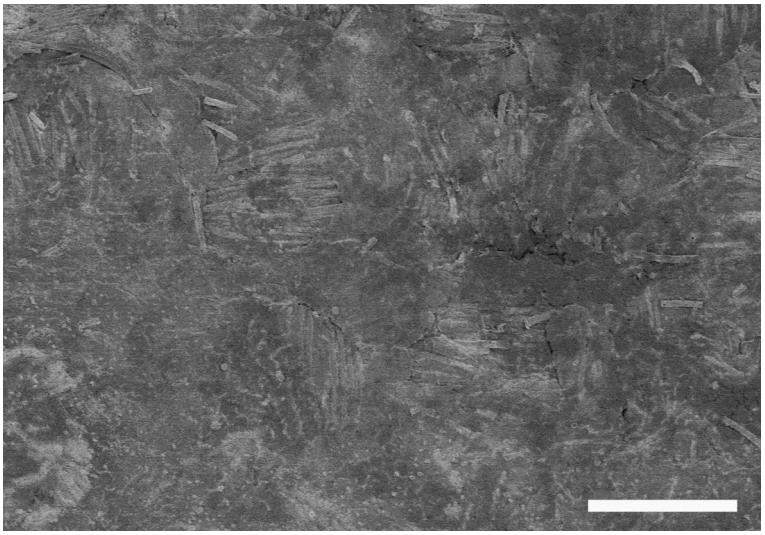

Fig. S3 The top-view SEM image of the SFCTs anode after 300 cycles at a current density of 1 mA cm^{-2} with a stripping/plating capacity of 1 mAh cm^{-2}. The scale bar is 250 μm.