RAPID COMMUNICATION

Three dimensionals α-Fe$_2$O$_3$/polypyrrole (Ppy) nanoarray as anode for micro lithium ion batteries

Jilei Liu[a,b], Weiwei Zhou[a], Linfei Lai[a], Huanping Yang[b], San Hua Lim[b], Yongda Zhen[b], Ting Yu[a], Zexiang Shen[a,*], Jianyi Lin[b,*]

[a]Division of Physics and Applied Physics, School of Physical and Mathematical Sciences, Nanyang Technological University, 21 Nanyang link, SPMS-PAP-03-18A, Singapore 637371, Singapore
[b]Heterogeneous Catalysis, Institute of Chemical Engineering and Sciences, A*star, 1 Pesek Road, Jurong Island, Singapore 627833, Singapore

Abstract
Three dimensional, self-supported α-Fe$_2$O$_3$/Ppy composite electrode with enhanced specific areal capacity and rate performance was successfully fabricated by a simple, low-cost, two-steps process consisting of direct heating of iron foil in air and subsequent coating of conducting polymer Ppy on the α-Fe$_2$O$_3$ nanoflakes. By using α-Fe$_2$O$_3$/Ppy as the anode materials with iron foil as the current collector, the unique structure affords a highly conductive pathway for electron, a short diffusion length for ions, a fast mass transport channel for electrolyte, and sufficient void space for accommodating large volume variations during Li intercalation/diintercalation for Li-ion battery. A relatively high specific capacity of 0.42 mA h/cm^2 can be achieved at 0.1 mA/cm^2 even after 100 charge/discharge cycles, with a plateau potential of 1 V and nearly 100% Coulombic efficiency, suggesting the feasibility to use this unique 3D nanostructured hybrid composite for microbattery in both small and large scale applications.
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Introduction

Rechargeable Li-ion batteries are now dominating the portable electronic market and showing promising applications in hybrid electric vehicles (HEV) as well as micro-electro-mechanical systems (MEMS) [1]. Microelectronic devices require rechargeable batteries with dimensions on the scale of 1–10 mm^3, high energy capacity and high power density, which are difficult to achieve for conventional thin film batteries because of insufficient power and energy related to state-of-the art two dimensional configuration [2]. Compared to 2D design, three dimensional (3D) nanostructured electrode architecture offers the opportunity to achieve large

*Corresponding authors.
  E-mail addresses: zexiang@ntu.edu.sg (Z. Shen),
lin_jianyi@ices.a-star.edu.sg (J. Lin).

areal energy capacities without sacrificing power density in terms of small areas footprint, short ion diffusion length, enhancement in active materials loading along vertical direction [1]. 3D nano-architectured electrodes based on various kinds of current collectors, such as copper nanopillar arrays [3], nickel nanorod arrays [4], gold nanoporous nanorods [5], porous polymer scaffold [6] and carbon nanotubes [7] have been demonstrated. Improvements in both energy and rate performance have been observed.

The fabrication of 3D nanostructured electrode usually starts with preparation of self-supported nano-architectured current collector, and then followed with electrochemical/chemical deposition of active materials. Template method is among the mostly used methods to prepare self-supported nano-architectured current collector [8]. Although quite attractive, it is difficult for practical microbattery applications due to high production cost. Therefore, it is highly desirable to explore simple, cost-effective, self-supported electrode elaboration process compatible with the use of low cost and high performance materials, growing on noble-metal-free current collectors.

α-Fe₂O₃ has been demonstrated to be an attractive anode material because of its high theoretical capacity, low cost, low toxicity, and ease of fabrication [9]. However, iron oxide always suffers from poor cyclability caused by drastic volume change during lithium ion intercalation/deintercalation process. Many approaches have been proposed to improve energy and rate capacity, such as fabricating Fe_2O_3 nanomaterials into optimized nanostructure and shapes (nanotubes, [10] nanoflakes [11] or branched nanostructure [12]), or embed the active materials in a cushioning medium (carbon nanotubes [13], graphene [14], carbon fibers or conducting polymer [9]) to form hybrid nanostructure, etc. Several Fe_2O_3/carbon hybrid nanostructures have been designed and lead to improvement in charge/discharge rate.

Inspired by these needs, we choose Iron foil as current collector and precursor for fabricating Fe_2O_3 electrode. A simple, two-step process is developed, which includes coating of conducting polymer, polypyrrole (Ppy) additive layer on α-Fe₂O₃ to fabricate α-Fe₂O₃/Ppy electrode.

Experimental

Electrode synthesis

Three dimensional α-Fe₂O₃/Ppy electrode was fabricated via a simple, two-step approach which consists of growing of 3D α-Fe₂O₃ nanoflakes onto iron foil by direct thermal heating, and subsequent coating of Polypyrrole via chemical polymerization in neutral solution. Briefly, α-Fe₂O₃ nanoflakes were first grown onto an iron foil substrate uniformly by thermal heating of the iron foil (Aldrich, 0.1 mm thick, 99.9+%) in air at 425 °C for 10 h. The Fe foil was polished by sand paper, prior to the growth, to form scratching, which acts as growth center of α-Fe₂O₃ (Figures S1 and S2). Then, a thin layer of Ppy was coated on the surface of α-Fe₂O₃ nanoflakes uniformly via chemical polymerization process with sodium p-toluenesulfonate (pTSNa) as the dopant, a mixture of DI water/ethanol (1:1, v/v) as solvent, and

ammonium persulfate (APS, $(NH_4)_2S_2O_8$) as oxidant. The molar ratio of APS: pTSNa: monomer is 1.25:0.5:1. The polymerization was carried out at 0-5 °C for 15 min. The resulted α-Fe₂O₃/Ppy hybrid was then washed with DI water and dried overnight at 80 °C in a vacuum oven.

Material characterization

Scanning electron microscopy (SEM, JEM-6700F, 10.0 kV), X-ray diffraction (XRD, Bruker D-8 Avance), Raman system (Renishaw, 532 nm excitation laser) were used to characterize samples.

Electrochemical characterization

Electrochemical performance was evaluated on CR2016 coin-type cells using the aforementioned self-supported α-Fe₂O₃/Ppy composites as working electrode and lithium foil as the counter electrode. The electrolyte was 1 M LiPF₆ in a mixed organic solvent consisting of ethylene carbonate (EC) and dimethyl carbonate (DMC) (1:1, v/v). Coil cells were assembled in an argon-filled glove box with both moisture and oxygen contents below 0.1 ppm. Galvanostatically charge/discharge tests were performed using a NEWARE cell tester over the potential range 0.01–3.0 V (vs. Li⁺/Li). Cyclic Voltammetry (0.01–3.0 V) tests and Electrochemical Impedance measurements (0.1–100000 Hz) were carried out using a CHI660D electrochemical workstation (CH Instrument Co. China).

Results and discussion

Three dimensional, self-supported α-Fe₂O₃/Ppy composites were fabricated via a simple, two step process consisting of growing of 3D α-Fe₂O₃ nanoflakes onto iron foil and subsequent coating of Polypyrrole. As illustrated in Scheme 1, in the first step, α-Fe₂O₃ nanoflakes are grown onto iron foil uniformly by thermal heating of the polished iron foil in air at 425 °C for 10 h (Figure S2). Secondly, a thin layer of polypyrrole is coated on the surface of α-Fe₂O₃ nanoflakes uniformly via chemical polymerization (Figures S3 and S4). The conversion of α-Fe₂O₃ nanoflakes to α-Fe₂O₃/Ppy composites is evidenced by scanning electron microscopy (SEM) images (Figure 1b–e & Figures S3 and S4). An obvious color change can be viewed from low magnification SEM images in Figure 1(b). The image of bare Iron foil on the left is bright while Fe₂O₃/Iron on the right is dark due to the growth of α-Fe₂O₃ nanoflakes. In Figure 1(c), it is clear that micrometer-sized flakes with thickness around 10-30 nm are generally pointed perpendicular to the plane of Fe foil. The nanoflakes are separated by distance in the range of 60-300 nm. A thin Ppy film is uniformly deposited onto the surface α-Fe₂O₃ nanoflakes array, forming a conformal coating around each nanoflakes without any coalescence between them (Figure 2d). The enlarge SEM image in Figure 2 clearly shows self-supported discontinuous nanoarchitectured α-Fe₂O₃/Ppy composite. This unique 3D nanostructure processes a large active surface area and preserves the space between nanoflakes. It affords a highly conductive pathway for electron, a short ion diffusion length for ions, a fast mass transport
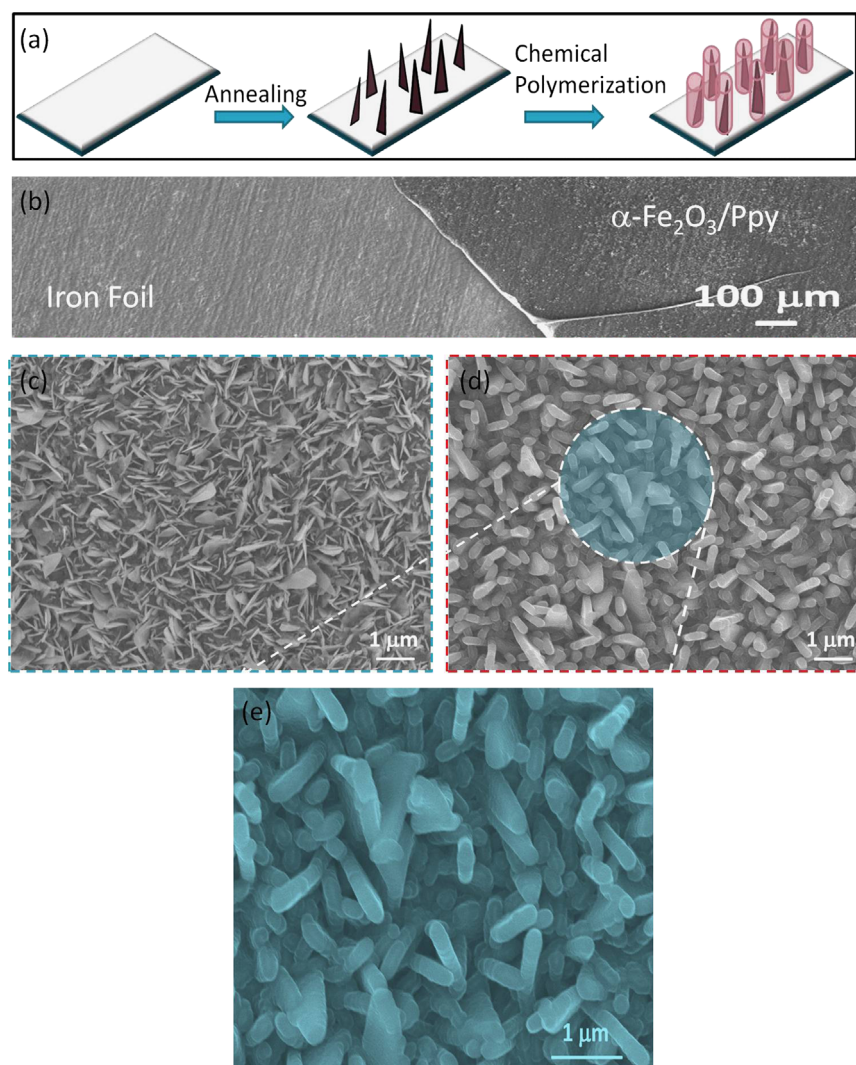
Figure 1 (a) Schematic illustration of the formation of α-Fe$_2$O$_3$/Ppy composite: (left) direct growth of α-Fe$_2$O$_3$ nanoflakes onto the surface of iron foil current collector; (right) uniform deposition of polypyrrole via chemical polymerization process. (b) Low-magnification SEM images of Iron foil (left) and α-Fe$_2$O$_3$/Ppy composite (right). (c) Typical SEM image of bare α-Fe$_2$O$_3$ nanoflakes. (d) SEM image of α-Fe$_2$O$_3$/Ppy heterostructures and (e) High-magnification SEM image of α-Fe$_2$O$_3$/Ppy heterostructures (blue area in d).

channel for electrolyte, and sufficient void space among neighboring nanoflakes for accommodating large volume variations during Li intercalation/disintercalation when serve as anode for Li-ion battery [3,5]

Raman spectroscopy and X-ray diffraction (XRD) further confirm the presence of Ppy, α-Fe$_2$O$_3$ and a small amount of Fe$_3$O$_4$ in the nanoflakes. In Figure 2(a) bare α-Fe$_2$O$_3$ shows characteristic Raman peaks at 224, 290, 409 and 1300 cm^{-1}. After Ppy coating these α-Fe$_2$O$_3$ peaks decrease in intensity, which is attributed to uniformly coating of polypyrrole onto the surface of α-Fe$_2$O$_3$ nanoflakes [15]. The appearance of the strong peak at 1590 cm^{-1}, weak peaks at 982 cm^{-1} and 930 cm^{-1}, which are assigned to C=C backbone stretching and ring-in plane deformation of PPY, respectively, indicating the successful polymerization of polypyrrole [16]. Similarly, the decrease in intensity of the typical X-ray diffraction peaks of α-Fe$_2$O$_3$ is clearly observed in Figure 2(b) for α-Fe$_2$O$_3$/Ppy hybrid composite, indicating the formation of Ppy onto the surface of α-Fe$_2$O$_3$ nanoflakes [9,11]. The uniform coating of PPy

was further verified by Energy dispersive X-ray (EDX) spectroscopy analysis (shown in Figures S5 and S6). Significant amount of carbon was detected only for α-Fe$_2$O$_3$/Ppy, homogenously dispersed throughout the whole area of α-Fe$_2$O$_3$/Ppy [17].

To evaluate the electrochemical behavior, coin cells based on self-supported α-Fe$_2$O$_3$/Ppy composites, without using binder and additional conductive additives, were assembled against a lithium metal counter electrode and subjected to galvanostatic charge–discharge cycling as well as cyclic voltammetry testing. Figure 3(a) shows the representative discharge/charge voltage profiles of α-Fe$_2$O$_3$/Ppy hybrid electrodes at a current density 0.10 mA/cm^2 with a cutoff window of 0.01-3 V. A voltage plateaus is observed at 0.85 V during lithium intercalation, which is assigned to decomposition of Fe$_2$O$_3$ to Fe with the formation of amorphous Li$_2$O [18]. The initial discharge capacity is 1.33 mA h/cm^2. It gradually reduces to 0.82 and 0.78 mA h/cm^2 for the second and fifth charge/discharge cycles, respectively. The irreversible capacity loss is obviously due to the formation of solid electrolyte interface (SEI) [10,14,18]. The
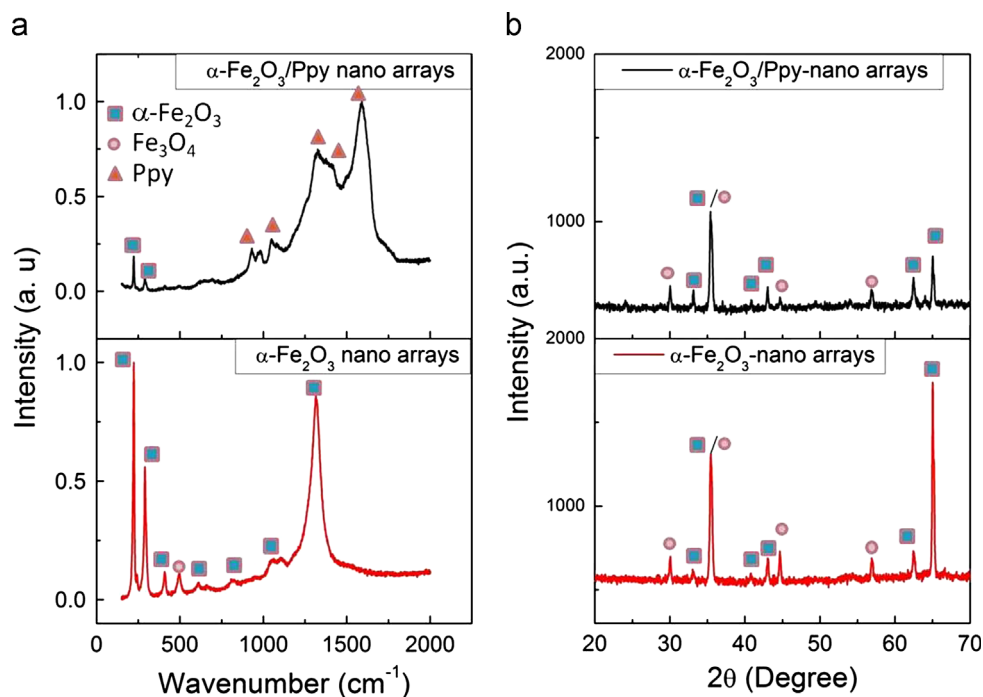
Figure 2 Raman spectra (a) and XRD patterns (b) of bare α-Fe$_2$O$_3$ (bottom) and α-Fe$_2$O$_3$/Ppy composite (top), respectively.
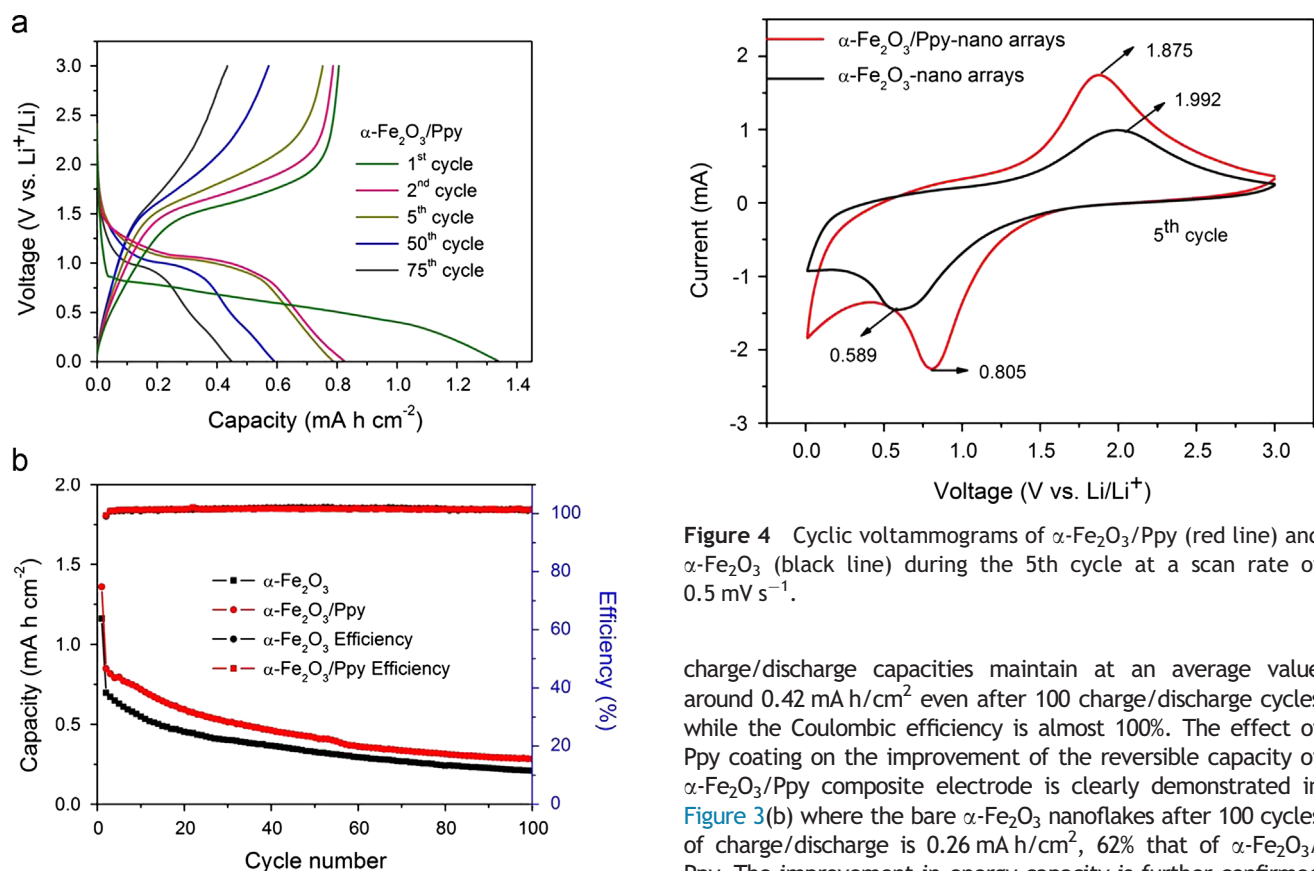

Figure 3 (a) Discharge-charge voltage profiles of α-Fe$_2$O$_3$/Ppy composite at a current rate of 0.1 mA/cm^2 and (b) Comparative cycling performance of the α-Fe$_2$O$_3$/Ppy composite electrode (red) and bare α-Fe$_2$O$_3$ (black) at a current rate of 0.1 mA/cm^2.

Figure 4 Cyclic voltammograms of α-Fe$_2$O$_3$/Ppy (red line) and α-Fe$_2$O$_3$ (black line) during the 5th cycle at a scan rate of 0.5 mV s^{-1}.

charge/discharge capacities maintain at an average value around 0.42 mA h/cm^2 even after 100 charge/discharge cycles while the Coulombic efficiency is almost 100%. The effect of Ppy coating on the improvement of the reversible capacity of α-Fe$_2$O$_3$/Ppy composite electrode is clearly demonstrated in Figure 3(b) where the bare α-Fe$_2$O$_3$ nanoflakes after 100 cycles of charge/discharge is 0.26 mA h/cm^2, 62% that of α-Fe$_2$O$_3$/Ppy. The improvement in energy capacity is further confirmed by the CV curve of α-Fe$_2$O$_3$/Ppy composite electrode vs. bare α-Fe$_2$O$_3$ electrode (shown in Figure 4). The anodic and cathodic peaks of α-Fe$_2$O$_3$/Ppy composite electrode are shifted to lower (1.992–1.875 V) and higher (0.589–0.805 V) potentials

respectively, while the intensity of both the peaks becomes stronger compared to bare α-Fe$_2$O$_3$ electrode, indicating higher capacity and lower polarization due to the Ppy coating which can improve the conductivity of α-Fe$_2$O$_3$ electrode. This was further verified by impedance measurements carried out before and after 200 charge/discharge cycles for both α-Fe$_2$O$_3$/Ppy and α-Fe$_2$O$_3$ at current rate of 0.10 mA cm^{-2} (Figure S8). The initial charge transfer resistance for α-Fe$_2$O$_3$/Ppy is 47.9 Ω, which is much smaller than that of bare α-Fe$_2$O$_3$ nanoflakes (65.3 Ω). Moreover, in contrast to the sharp resistance increase for bare α-Fe$_2$O$_3$ (from 65.3 to 85.5, after 200 cycles), α-Fe$_2$O$_3$/Ppy composite electrode exhibited a smaller changes (from 47.9 to 61.0, 13.1 Ω).

In order to confirm the feasibility of α-Fe$_2$O$_3$/Ppy nano-architectured electrode for microbattery application, cycle rate capability tests were conducted at various current rates ranging from 0.10 to 1.0 mA/cm^2. As demonstrated in Figure 5, the capacity progressively decreases with increasing rates. However the capacity remains fairly stable after 10 cycles and 0.32 mA h/cm^2 can be achieved at current of 1.0 mA/cm^2, which is much better than those (0.03–0.2 mA h/cm^2) of many previous reports (such as 0.14 mA h/cm^2 at current density of 0.05 mA/cm^2 over 50 charge/discharge cycles for crystalline TiO$_2$ nanotube/ SnO nanowire [19], 0.2 mAh/cm^2 at current density of 0.025 mA/cm^2 over 45 charge/discharge cycles for TiO$_2$ nanotube/Fe$_2$O$_3$ nanowire composite [20], 0.113 mA h/cm^2 at current density of 0.1 mA cm^{-2} after 50 charge/discharge cycles for coaxial SnO$_2$/TiO$_2$ nanotube hybrids [21], 0.032 mA h/cm^2 at current density of 0.04 mA/cm^2 up to 75 charge/discharge cycles for Au/Polyanilne hybrid nanorods [5], and 0.1 mA h/cm^2 at current density of 0.006 mA/ cm^2 up to 100 charge/discharge cycles for carbon micro-net films [22]). Moreover, when the current rate decreases back to 0.1 mA/cm^2, specific area capacity of around 0.46 mA h/ cm^2 can be restored after 60 cycles including deep cycling at high C-rate of 1.0 mA/cm^2, indicating good stable rating capability.

Conclusions

In conclusion, three dimensional, self-supported α-Fe$_2$O$_3$/ Ppy composite electrode with enhanced specific areal capacity and rate performance was successfully fabricated by a simple, low-cost, two-step process consisting of direct heating of iron foil in air and subsequent coating of conducting polymer Ppy on the α-Fe$_2$O$_3$ nanoflakes. By using α-Fe$_2$O$_3$/Ppy as the anode materials with iron foil as the current collector, the unique structure affords a highly conductive pathway for electron, a short diffusion length for ions, a fast mass transport channel for electrolyte, and sufficient void space for accommodating large volume variations during Li intercalation/deintercalation for Li-ion battery. A relatively high specific capacity of 0.42 mAh/cm^2 can be achieved at 0.1 mA/cm^2 even after 100 charge/ discharge cycles, with a plateau potential of 1 V and nearly 100% Coulombic efficiency. All these show the feasibility to use this unique 3D nanostructured hybrid composite for microbattery in both small and large scale applications.
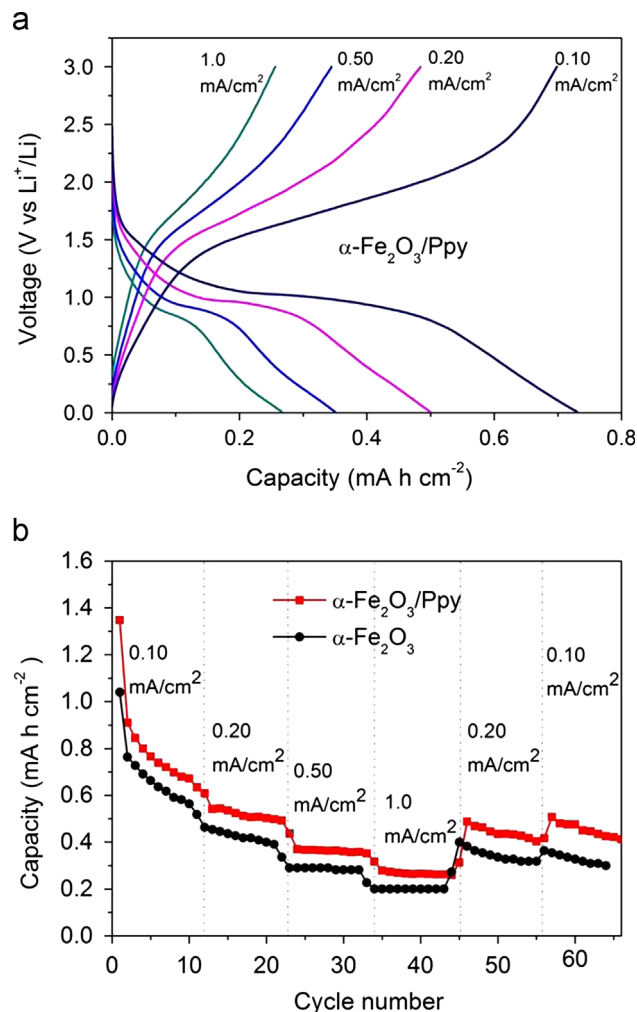

Figure 5 (a) Discharge–charge voltage profiles of α-Fe$_2$O$_3$/Ppy composite at different current rates and (b) Comparative rate performance of the α-Fe$_2$O$_3$/Ppy composite electrode (red) and bare α-Fe$_2$O$_3$ (black) with current rate increasing from 0.1 mA/cm^2 to 1.0 mA/cm^2.

Appendix A.   Supporting information

Supplementary data associated with this article can be found in the online version at http://dx.doi.org/10.1016/ j.nanoen.2012.12.008.

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His current research interests are synthesis, characterization of carbon materials (graphene and CNTs) and their applications in electrocatalysts, electrochemical capacitors, rechargeable batteries and Li-air battery.

Weiwei Zhou is currently pursuing her Ph.D. in Division of Physics and Applied Physics at the Nanyang Technological University (NTU) under the supervision of Asst. Prof. Ting Yu and Asst. Prof. Hong Jin Fan. She received her M.Sc in Inorganic Chemisty from University of Science and Technology of China (Hefei, China, 2008). Her current research interests are design, synthesis of metal oxide based heterostructures and their applications in electrochemical energy storage devices.

Linfei Lai is currently pursuing her Ph.D. in Division of Physics and Applied Physics at the Nanyang Technological University under the supervision of Prof. Zexiang Shen and Prof. Jianyi Lin. She received her M.S. degree in College of Materials, Xiamen University in 2009. Her current studies include the application of transition metal nitrides, transition metal oxides, conducting polymers and their graphene composites in PEM fuel cell, supercapacitor, and Li-ion battery.

Huanping Yang received her Master's degree from College of Chemistry and Moleculer Sciences from the Wuhan University of China in 2005. After she received her Ph.D. in Chemistry and Biological Chemistry from the Nanyang Technological University of Singapore in 2012, she joined the Institute of Chemical and Engineering Sciences, A∗STAR, Singapore, as a Scientist I. Her main research interest focuses on carbon-based electrode materials for electrochemical energy storage applications.

San Hua Lim studied Physics at the National University of Singapore (NUS). He is currently working as a Research Scientist at the Institute of Chemical and Engineering Sciences (ICES). His research interests are synthesis, characterization and applications of nanowires, nanorods, nanobelts, carbon nanotubes, and graphene for energy storage and conversion. His work also involves the development of cathode materials for Li-ion batteries and catalytic conversion of butene to butadiene.

Jilei Liu is currently pursuing his Ph.D. in Division of Physics and Applied Physics at the Nanyang Technological University (NTU) under the supervision of Prof. Zexiang Shen and Prof. Jianyi Lin. He received his B.Sc in Materials Physics from Hunan University (Changsha, China, 2008) and M.Sc from Shanghai Institute of Ceramics, Chinese Academy of Sciences (Shanghai, China, 2011).

Yongda Zhen received his Ph.D. in manufacture engineering from the Nanyang Technological University (Singapore) in 2007 under the supervision of Prof. San Ping Jiang and Prof. Sam Zhang. After one-year of postdoctoral research training at the same university, he joined the Institute of Chemical and Engineering Sciences, Agency for Science, Technology and Research (Singapore), as a research scientist. His current research interests involve novel nanostructured materials, carbon-rich materials and their applications in energy-related areas.

Ting Yu is an Assistant Professor in the Division of Physics and Applied Physics, School of Physical and Mathematical Sciences at Nanyang Technological University (NTU); a joint assistant professor of Department of Physics, Faculty of Science, National University of Singapore (NUS) and a National Research Foundation (NRF) fellow. He obtained his B.Sc. from Jilin University (PR China), Ph.D. degree from National University of Singapore (NUS). His research group focuses on Raman spectroscopy and nanocarbons, carbon-metal oxide nanohybrids for nanoelectronics, nanophotonics and sustainable energy.

Zexiang Shen is a professor of physics at the School of Physical and Mathematical Sciences, Nanyang Technological University (NTU). He holds a joint appointment at the school of Materials and Engineering. He received his B.S. degree in physics from Jilin University in 1982 and Ph.D. in physics from King's college, University of London in 1989. His research interests include near-field Raman microscopy; fabrication, characterization and simulation of graphene and other nano-materials; applications of graphene in energy storage such as graphene-based supercapacitors and Li-ion batteries. He published more than 350 journal papers.

Jianyi Lin is currently a project consultant at Institute of Chemical and Engineering Sciences (ICES), A∗STAR, Singapore, and an Adjunct Professor in the Department of Physics, National University of Singapore (NUS). He graduated from Xiamen University, China and received Ph.D. in Chemistry from Stanford University in 1991. His research and expertise areas lie in surface science, heterogeneous catalysis and nano-materials, which include hydrogen production and storage, PEM fuel cell, supercapacitor and Li-ion battery studies.