

Supporting Information

Fabrication and pseudocapacity behavior of elm flower branch-like nickel@graphene fibers

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Part I: Experimental Section

1.1 Preparation of PAN nanofibers by electrospinning

10 wt.% PAN solution was prepared by mixing with polyacrylonitrile (PAN) in N,N-dimethylformamide (DMF, CP). A variable high-voltage power supply was used to provide high voltage of approximately 17 kV for electrospinning. The positive electrode of the high-voltage power supply was connected to the needle tip with 1 mm in diameter. The flow rate and tip-collector distance was 160 $\mu\text{L}/\text{h}$ and 20 cm, respectively. The grounded electrode was connected to a metallic collector wrapped with aluminum foil.

1.2 Preparation of Elm flower branch-like nickel@graphene fiber

Elm flower branch-like nickel@graphene fiber (ENGF) was synthesized via solvothermal reaction followed by annealing. The precursor solution was prepared by mixing 0.7 g of $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ and 0.2 g of urea with 40 mL of ethanol. Electrospinning PAN

nanofibers (~30 mg) were then placed in the preceding solution, and the mixture was transferred into a Teflon-lined autoclave with a capacity of 100 mL. The solvothermal reaction time and reaction temperature were set for 15 h and at 170 °C, respectively.

After the solvothermal reaction, the fiber-like product was rinsed with deionized water and dried at 60 °C for 12 h. The dried nanofibers were completely immersed into 1 wt.% PMMA solution (DMF) for 2 h, picked out, and dried at 70 °C for 24 h. Then the fibers coated with PMMA were annealed at 900 °C in Ar (5% H₂) atmosphere for 3 min. Nickel@graphene structural fiber was thus obtained.

1.3 Characterization

The samples were characterized by field-emission scanning electron microscopy (FESEM; Sirion 200), TEM (EM-200CX), and X-ray diffraction (XRD; Philips X'Pert) using Cu-K α radiation. Raman spectra were performed with an Intelligent Fourier transform infrared – Raman spectroscopy (NEXUS). The specific surface area was examined by N₂ adsorption–desorption (Quantachrome Autosorb 3B).

The electrochemical measurements were carried out in a three-electrode system with 1 M KOH solution as the electrolyte at room temperature on an electrochemical working station (IM6 & Zennium, Zahner, Germany). A working electrode was constructed by pressing the prepared electrode materials onto nickel foam (0.5 cm \times 1.0 cm) under 10 MPa. The electrode materials containing poly(vinylidene fluoride) (PVDF) as a binder, and the mass ratio of the prepared composites to PVDF was 8:1. Typical weight of the active materials of each sample was controlled within 2.0 ± 0.2 mg. A Pt foil and an Ag/AgCl electrode served as counter and reference electrodes, respectively. Cyclic voltammetry (CV) and galvanostatic charge–

discharge were performed to evaluate the electrochemical behavior of the mesoporous graphitic carbon/nickel nanoparticle composites.

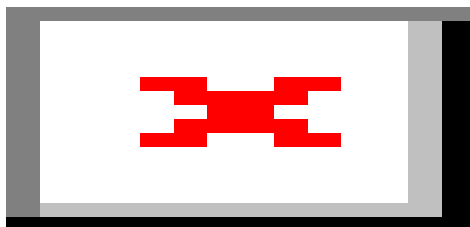


Fig. S1 SEM images of the first group of as-prepared products after PAN nanofibers were dealt with solvothermal reaction at 100 °C for 15 h (A) and 130 °C for 15 h (B).

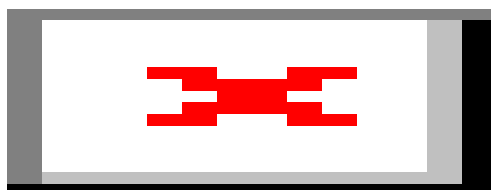


Fig. S2 FESEM images of the second group of as-prepared products after PAN nanofibers were dealt with solvothermal reaction at 170 °C for 3 h (A), 170 °C for 7 h (B), and 170 °C for 12 h (C).

Part II: Supplementary Figures

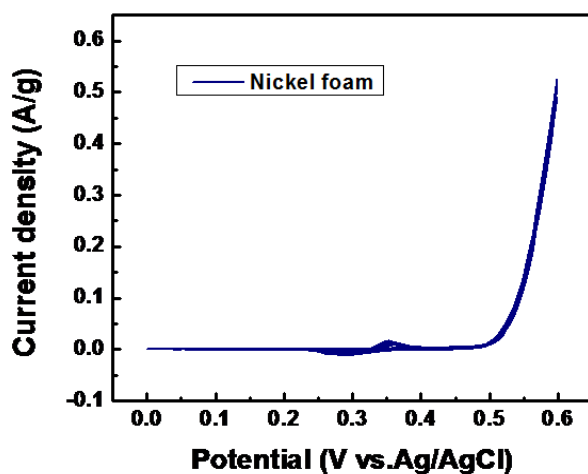


Fig. S3 CV curves of nickel foam at a scan rate of 5 mV/s within a potential window of 0.0 V to 0.6 V vs. SCE in 1.0 M KOH aqueous

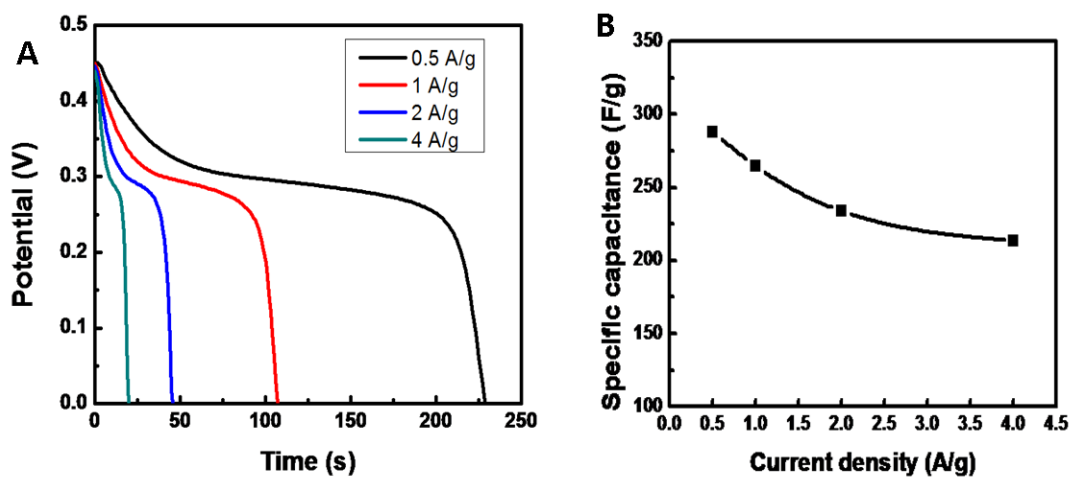


Fig. S4 Discharge curves of nickel@graphene structural fibers at different current densities (A) and the specific capacitance as a function of the current densities (B).

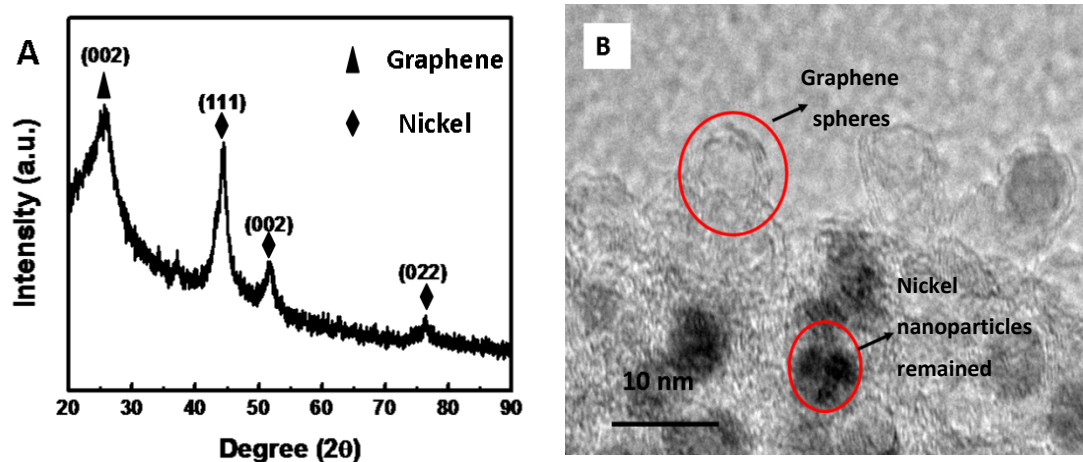


Fig. S5 XRD pattern (A), TEM image (B) of the obtained product after ENGF was immersed in nitric acid solution.^[1-2]

Part III: Calculations

The mass-specific capacitance was calculated from the galvanostatic discharge curves using an equation as below:

$$C = I \times \Delta t / (\Delta V \times m) \quad (1)$$

where I was the discharge current (A), Δt was the time spent in discharge (s), ΔV was the potential drop during discharge (V), and m was the mass of active electrode materials (g).

The specific capacitance of the electrodes could also be calculated from the CV curves according to the following equation :

$$C = Q / (m \cdot \Delta V) \quad (2)$$

where Q (C) was the average charge during the charging and discharging processes, m (g) was the mass of the active materials in the electrodes, and ΔV (V) was the potential window. ^[3-4]

Notes and references

1 The analysis of XRD pattern is based on JCPDS card: 10-2279 and the results of the following literature:

Z. J. Fan, J. Yan, L. J. Zhi, Q. Zhang, T. Wei, J. Feng, M. L. Zhang, W. Z. Qian and F. Wei, *Adv. Mater.*, 2010, **22**, 3723–3728

2 The preparation of A-ENGF: A certain amount (20 mg) of ENGF was put into 50 ml nitric acid ($V_{\text{acid}}:V_{\text{wat}}=1:4$) in 100ml Teflon-lined autoclave. The solvothermal reaction time and reaction temperature were set for 10 h and at 60 °C, respectively. After the solvothermal reaction, the product was washed with deionized water until neutral, and dried.

3 J. Yan, E. Khoo, A. Sumboja and P. S. Lee, *ACS Nano*, 2010, **4**,4247

4 K. B. Xu, R. J. Zou, W. Y. Li, Y. F. Xue, G. S. Song, Q. Liu, X. J. Liu and J. Q. Hu, *J. Mater. Chem. A*, 2013, **1**, 9107–9113