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## Nitrogen- and Oxygen-Enriched 3D Hierarchical Porous Carbon fiber: Synthesis and Superior Supercapacity

Ying Li<sup>a,b</sup>, Chunxiang Lu<sup>a</sup>, Shouchun Zhang<sup>\*a</sup>, Fang-Yuan Su<sup>c</sup>, Wenzhong Shen<sup>d</sup>, Pucha Zhou<sup>a</sup>,  
Canliang Ma<sup>e</sup>

This supporting information contains the electrochemical experiment using a three-electrode cell, the SEM and TEM images, the porous structure parameters, the XRD patterns, the electrochemical capacitive performances using a two-electrode cell and a three-electrode cell.

### Electrochemical experimental for a three-electrode cell

The preparation of the working electrode in the three-electrode configuration was the same as in the two-electrode cell. Hg/HgO electrode served as the reference electrode, and a platinum sheet as the counter electrode. The gravimetric capacitance ( $C$ ,  $F g^{-1}$ ) for the three-electrode cells was calculated according to the GCD test by the following equation:

$$C = I \Delta t / m \Delta V \quad (2)$$

where  $I$  (A) is the discharge current,  $\Delta t$  (s) is the discharge time,  $m$  (g) is the mass of single carbon electrode and  $\Delta V$  (V) is the potential difference.

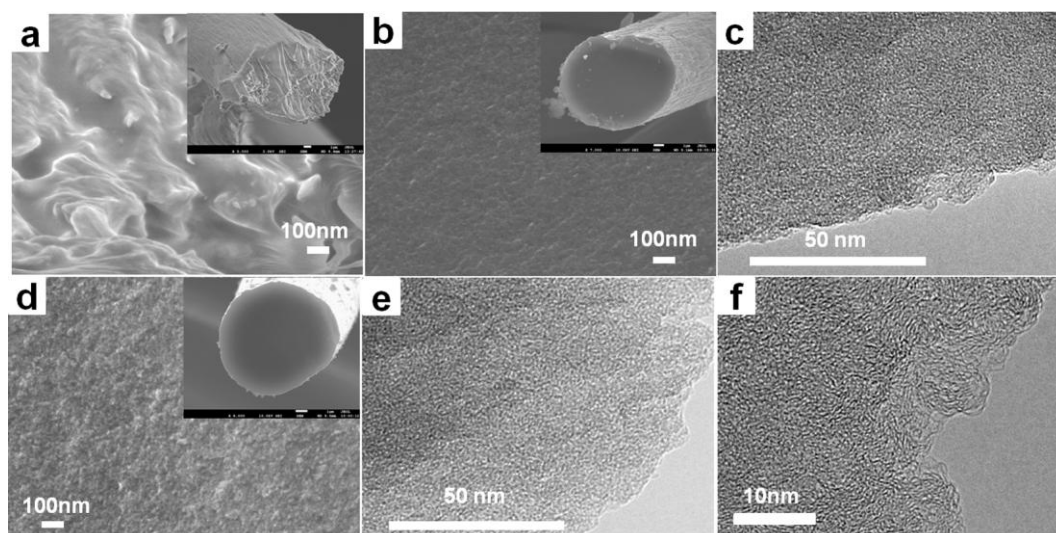


Fig. S1 SEM and TEM images of the cross section morphology of the control samples: (a) SEM image of NPF, the nonporous commercial PAN fiber; (b) SEM and (c) TEM images of NCF, the nonporous carbon fiber derived from NPF after carbonization; (d) SEM and (e, f) TEM images of ACF, the activation product of NCF. The insets in (a, b, d) are the low-resolution SEM images of NPF, NCF, and ACF, respectively.

Table S1 the detail information about pore structure of samples

Sample	$S_{\text{BET}}$ ( $\text{m}^2 \text{g}^{-1}$ )	$S_{\text{micro}}$ ( $\text{m}^2 \text{g}^{-1}$ )	$V_{\text{total}}$ ( $\text{cm}^3 \text{g}^{-1}$ )	$V_{\text{BJH}}$ ( $\text{cm}^3 \text{g}^{-1}$ )
PF	72.5	–	0.522	0.522
NPF	0.2	0.1	–	–
CF	98.9	13.3	0.129	0.128
NCF	8.5	–	0.026	–
HPCF	2176.6	1730.7	1.272	0.740
ACF	1002.9	827.9	0.521	0.208

$S_{\text{BET}}$ : Total surface area calculated by the BET method.

$S_{\text{micro}}$ : Micropore surface area derived from t-Plot method.

$V_{\text{total}}$ : Total pore volume of pores calculated from single point adsorption at the relative pressure of  $P/P_0$  of 0.995.

$V_{\text{BJH}}$ : Mesopore and macropore volume calculated using the BJH method.

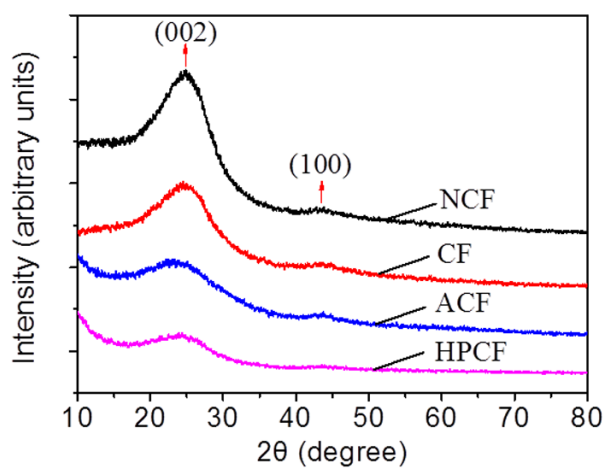


Fig. S2 XRD patterns of NCF, CF, ACF and HPCF.

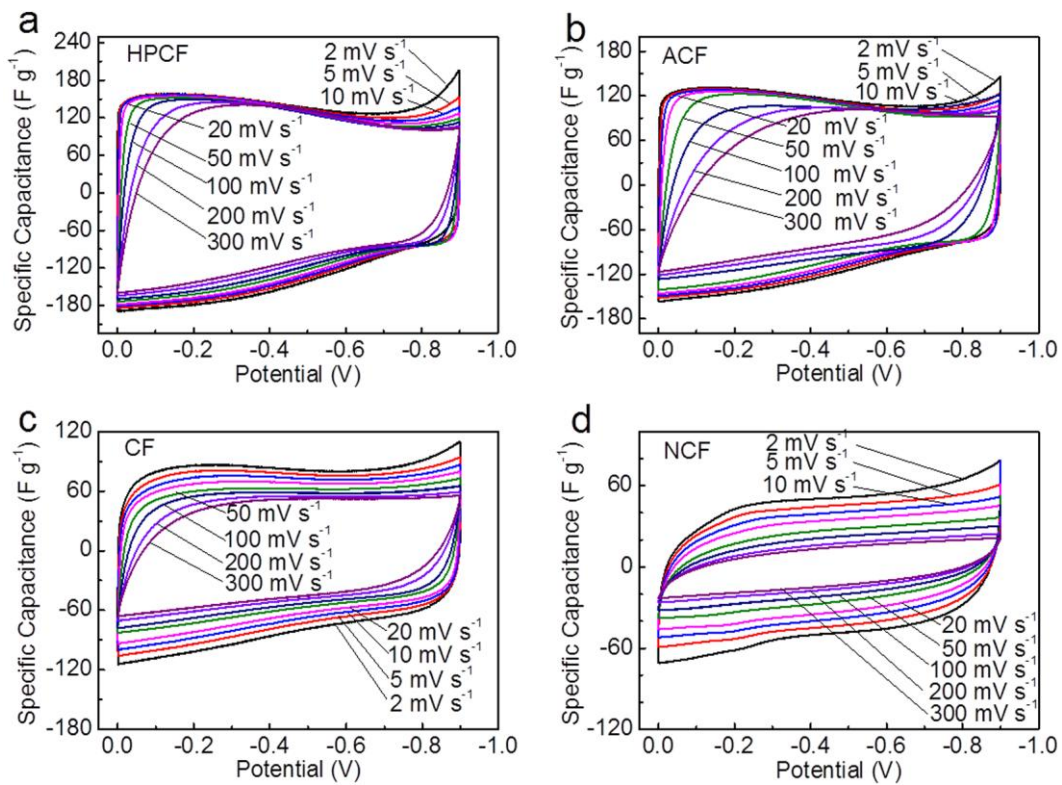


Fig. S3 CV curves at the scan rate range of 2-300  $mV s^{-1}$  for (a) HPCF, (b) ACF, (c) CF and (d) NCF using a two-electrode cell.

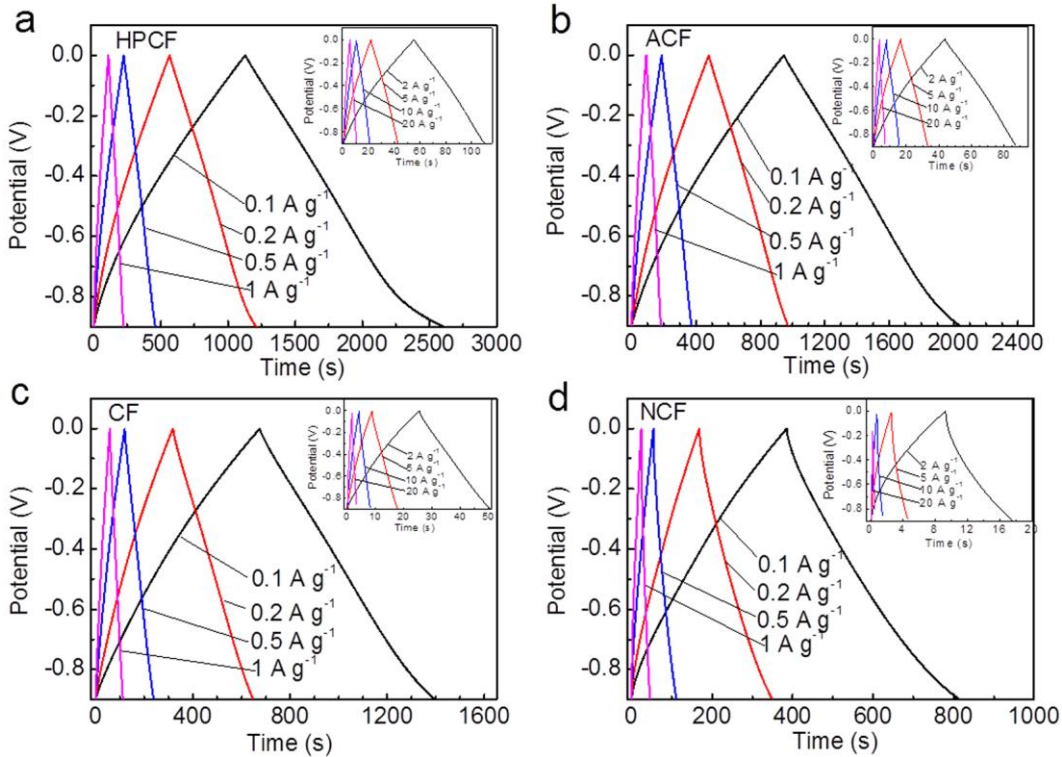


Fig. S4 GCD curves at the current density range of 0.1-20  $A g^{-1}$  for (a) HPCF, (b) ACF, (c) CF and (d) NCF using a two-electrode cell.

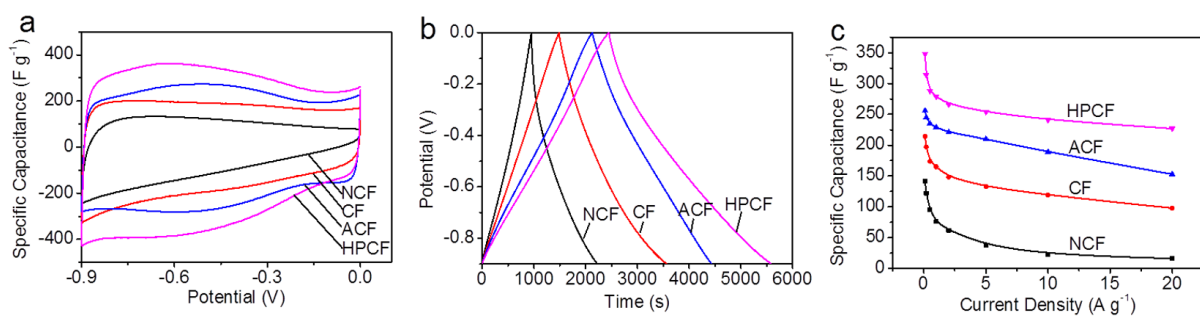


Fig. S5 CV curves at  $5 \text{ mV s}^{-1}$  (a), GCD curves at  $0.1 \text{ A g}^{-1}$  (b), and specific capacitances as a function of current densities at the range of  $0.1\text{-}20 \text{ A g}^{-1}$  (c) for NCF, CF, ACF, and HPCF, derived in a three-electrode configuration.