

Supporting Information:

## Free-standing Highly Ordered Mesoporous Carbon-Silica Composite Thin Films

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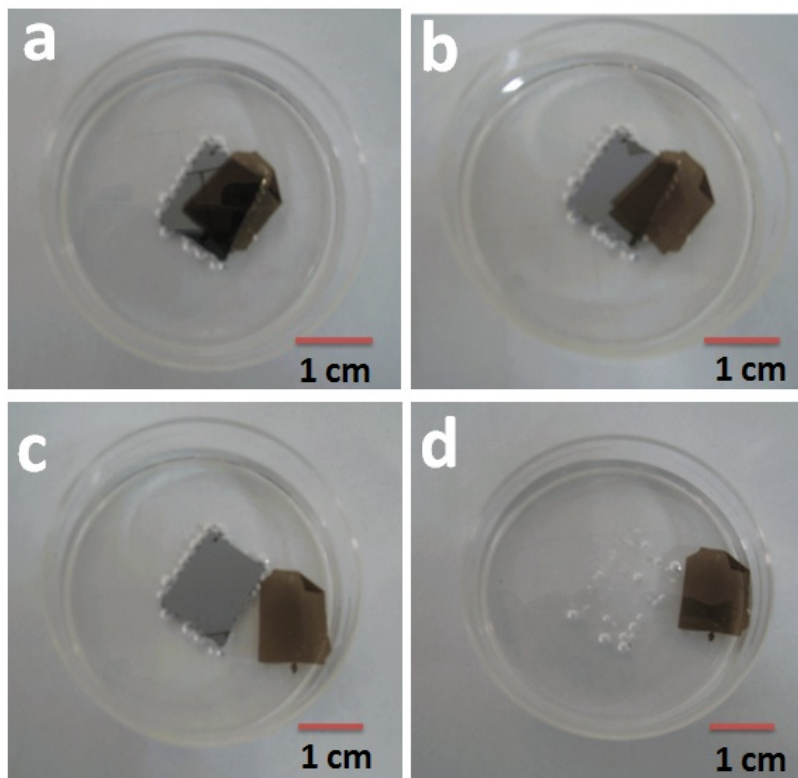
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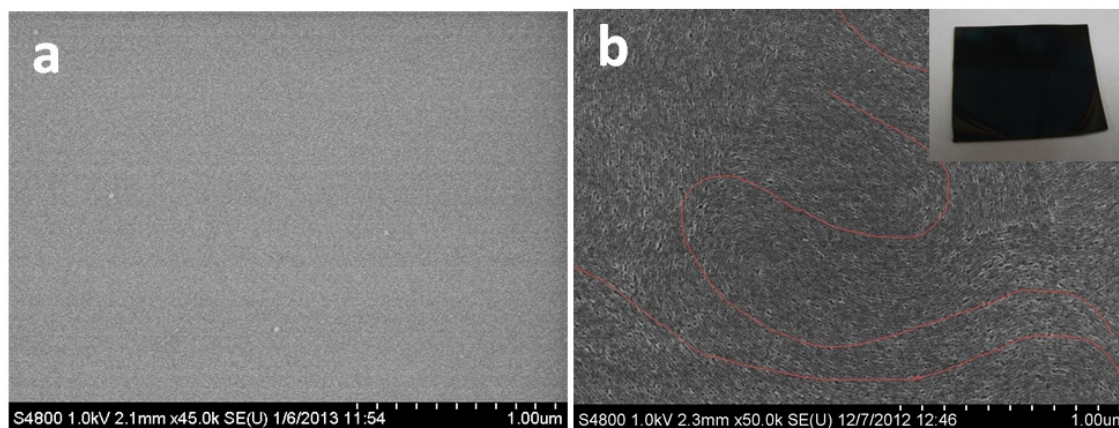
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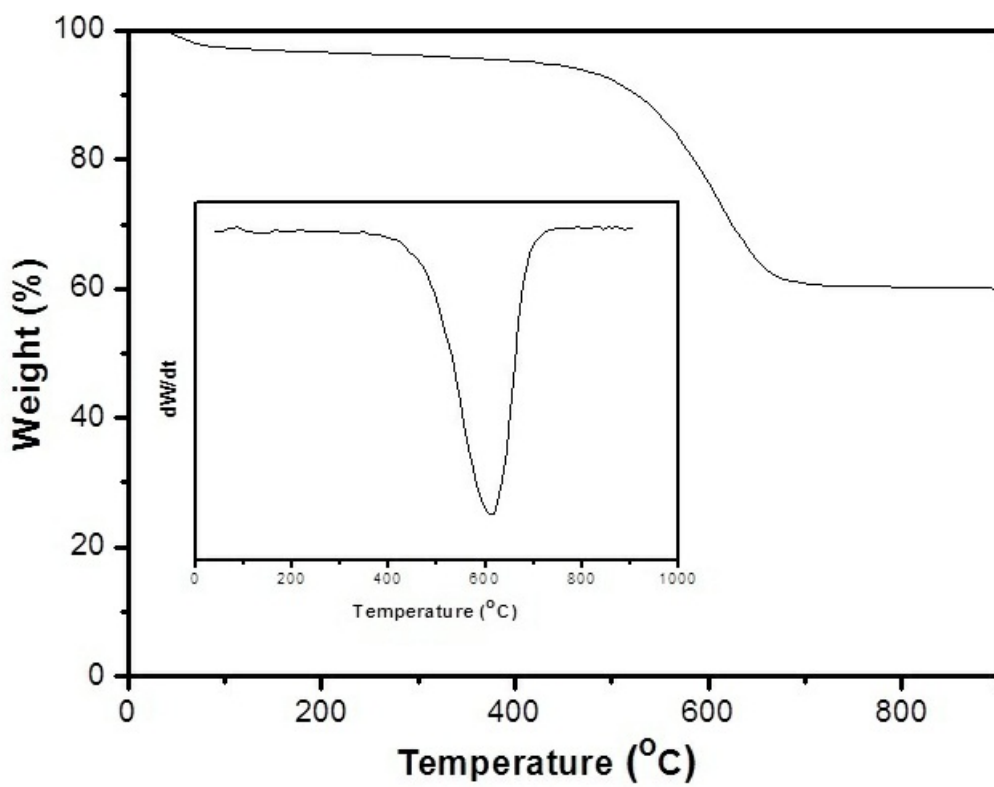
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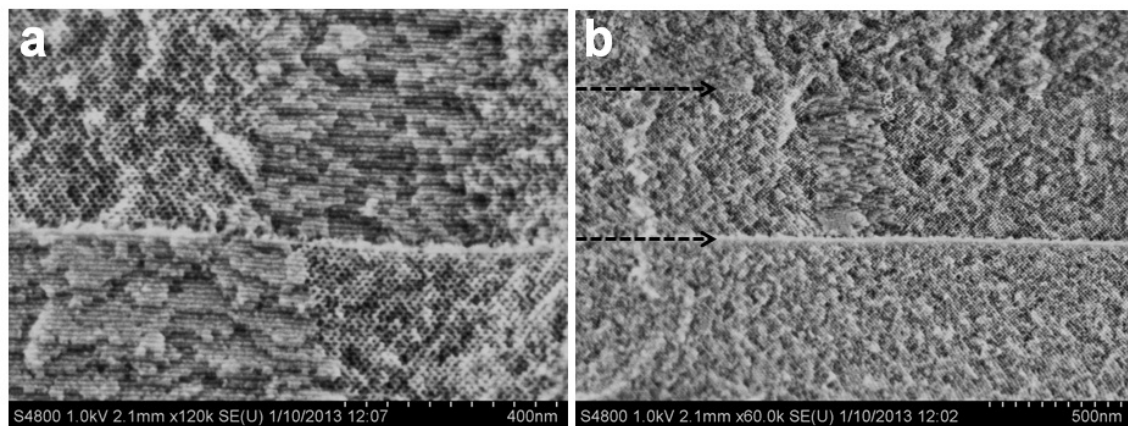
**Figure S1.** (a-d) Optical pictures in sequential of the etching process to obtain the free-standing mesoporous carbon-silicon composite thin films.



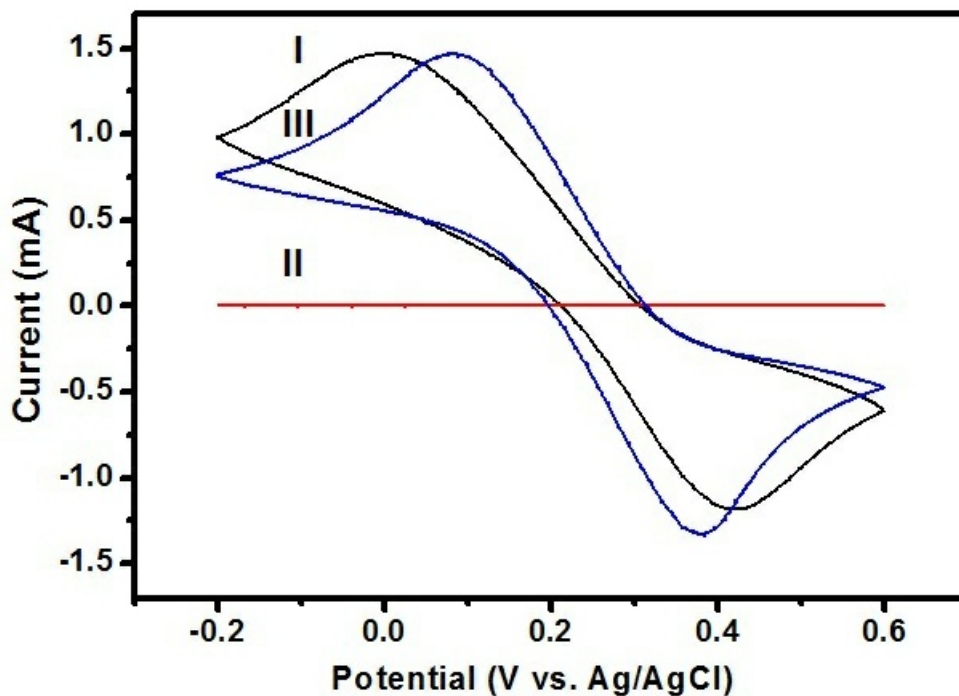
**Figure S2.** SEM images of (a) the Al<sub>2</sub>O<sub>3</sub> layer by atomic layer deposition (ALD) method, and (b) mesoporous carbon-silica composite film (MF-CS-61) on the Al<sub>2</sub>O<sub>3</sub>-coated Si substrate. Inset: optical photo of the thin film on substrate.



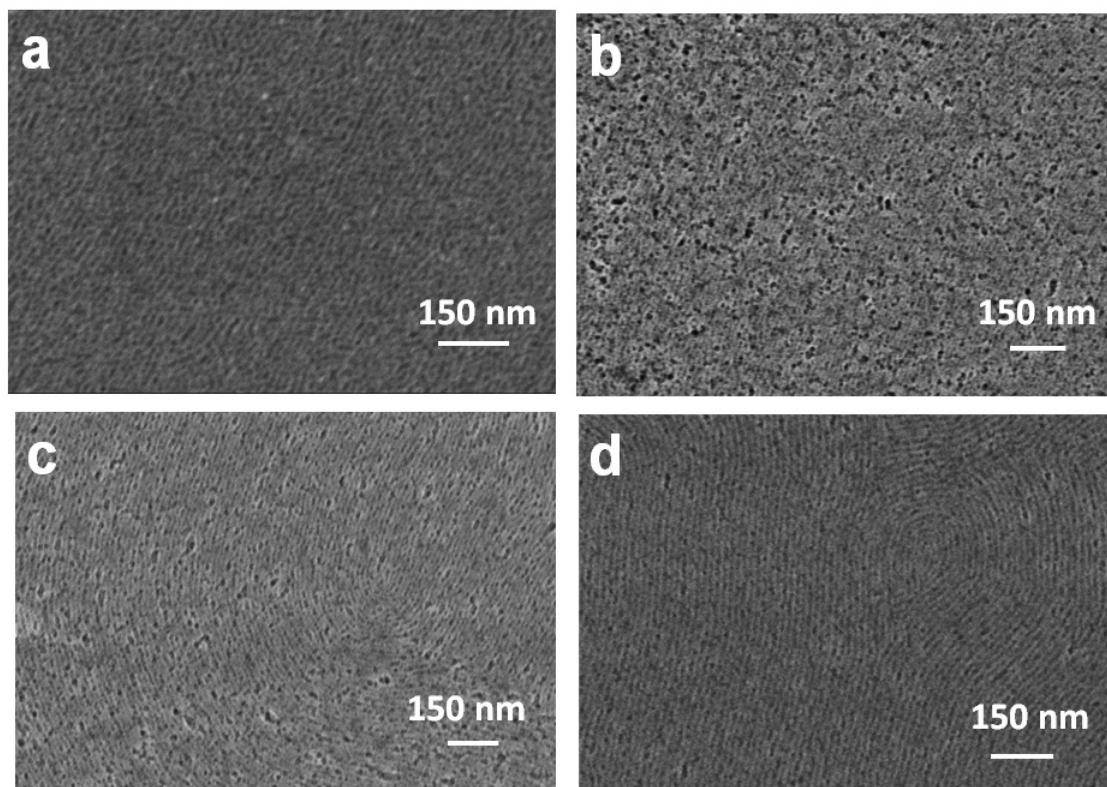
**Figure S3.** Thermal gravimetric (TG) and derivative thermal gravimetric (DTG, inset) curves of free-standing carbon-silica composite film (MF-CS-61) measured in air.



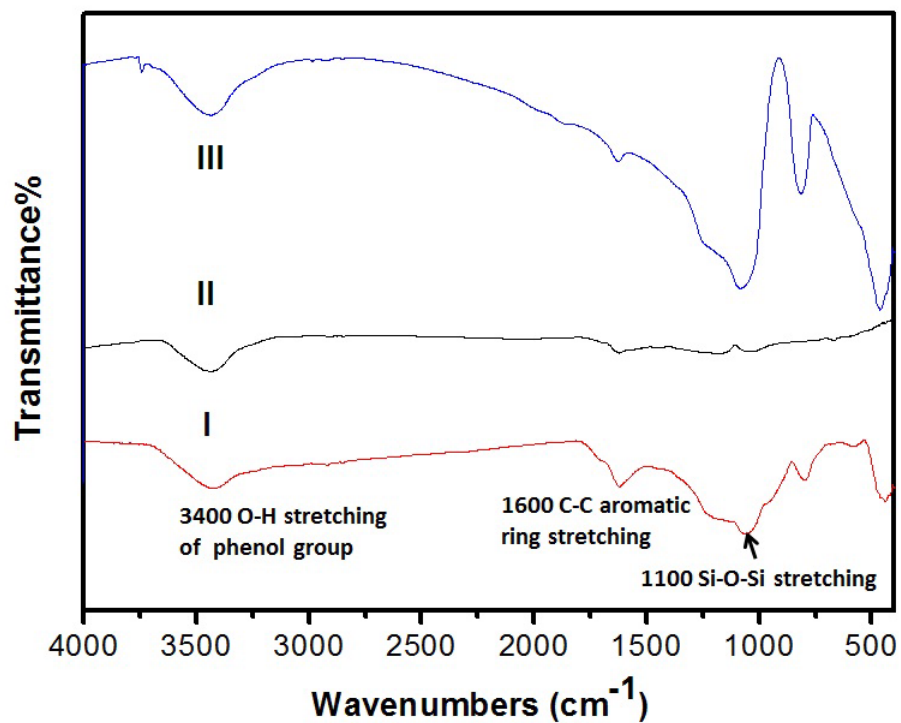
**Figure S4.** Cross-section SEM images of (a) two-layered and (b) three-layered mesoporous carbon-silica films. Black arrows indicate the interface between different layers.



**Figure S5.** Cyclic voltammograms (CVs) recorded in 10 mM  $K_3Fe(CN)_6$  using (I) a bare FTO, (II) a FTO electrode coated with the as-synthesized film aged at 100 °C for 24 h, and (III) a FTO electrode covered by mesoporous carbon-silica composite film (MF-CS-61) after calcination. The electrochemical measurements were carried out using a three-electrode cell, in which the FTO was used as the working electrode, a Pt wire and Ag/AgCl were used as the counter and reference electrodes, respectively. CV plots were recorded at a scan rate of 20  $mV s^{-1}$  at room temperature.

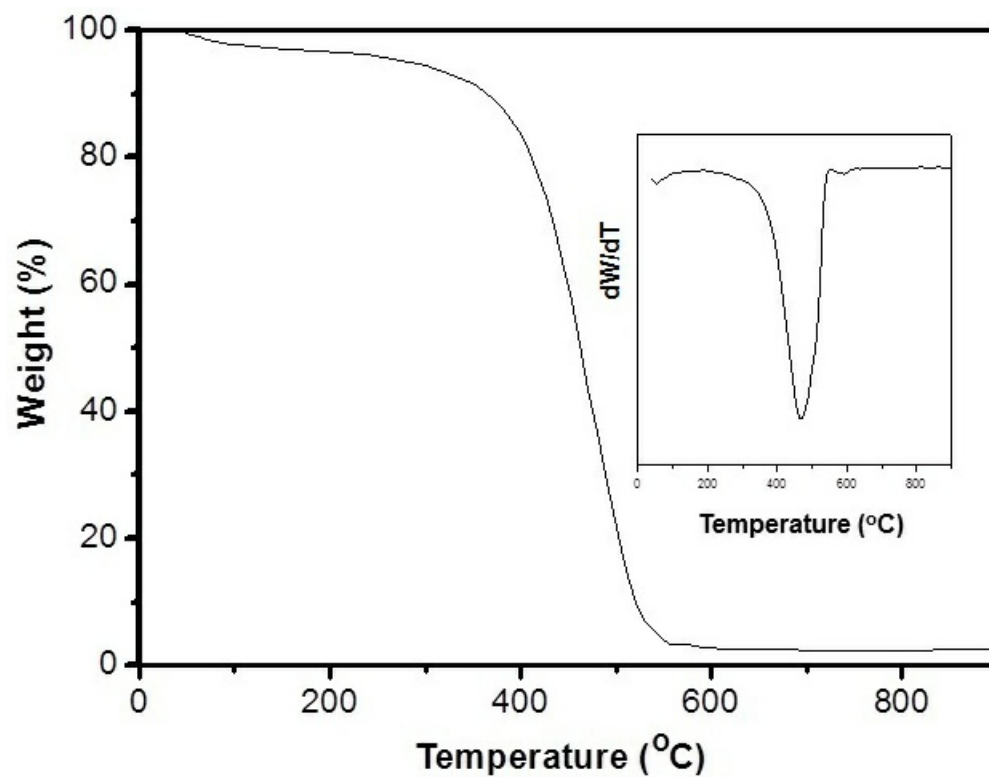


**Figure S6.** SEM images of the free-standing mesoporous carbon-silica composite films with different silica contents: (a) MF-CS-35, (b) MF-CS-51, (c) MF-CS-61, (d) MF-CS-72.



**Figure S7.** FT-IR spectra of (I) free-standing mesoporous carbon-silica composite film MF-CS-51; (II) free-standing mesoporous carbon film MF-C-51; (III) free-standing mesoporous silica film (MF-S-51).





**Figure S8.** TG and (inset) DTG curves of free-standing carbon film MF-C-51 recorded in air.

**Table S1.** Fabrication conditions and compositions of different free-standing mesoporous carbon-silica composite films

| Sample <sup>a</sup> | resol<br>(20wt%) | TEOS<br>(g) | F127 (g) | Reaction<br>time (h) | SiO <sub>2</sub><br>(wt%) <sup>b</sup> |
|---------------------|------------------|-------------|----------|----------------------|--|
| MF-CS-35            | 0.8              | 1.0         | 0.5      | 2                    | 35                                     |
| MF-CS-51            | 1.25             | 1.0         | 0.5      | 2                    | 51                                     |
| MF-CS-61            | 2.5              | 1.0         | 1.0      | 2                    | 61                                     |
| MF-CS-72            | 5.0              | 1.0         | 2.0      | 2                    | 72                                     |

**Table S2.** Physicochemical properties of free-standing mesoporous carbon-silica composite films with different silica contents

| Sample name | S <sub>BET</sub> <sup>2</sup><br>(m <sup>2</sup> /g) | V <sup>3</sup><br>(cm <sup>3</sup> /g) | D (nm) |
|-------------|--|--|--------|
| MF-CS-35    | 515  | 0.45                                   | 4.9    |
| MF-CS-51    | 440  | 0.34                                   | 5.8    |
| MF-CS-61    | 385  | 0.39                                   | 6.2    |
| MF-CS-72    | 365  | 0.41                                   | 6.6    |