Supplementary information:

Synthesis of Nanostructured Silicon Carbide Spheres from Mesoporous C-SiO₂ Nanocomposites

By Kun Wang, Huanting Wang and Yi-Bing Cheng*

[*]Corresponding author: Prof. Yi-Bing Cheng Department of Materials Engineering, Monash University, VIC 3800, Australia E-mail: yibing.cheng@eng.monash.edu.au Tel: +61 3 9905 4930, Fax: +61 3 9905 4940.

Synthesis of mesoporous C-SiO₂

First, 5 g of H₂O, 3.28 g of ethanol (anhydrous, Aldrich) and 0.5 g of 1 M HCl (Merck) were mixed in a capped polypropylene bottle with a magnetic stirrer. To this solution, 4.1 g of P123 ($EO_{20}PO_{70}EO_{20}$, MW5800, Sigma-Aldrich) was added under continuous agitation to obtain a P123 solution. Then 10 g of tetraethoxysilane (TEOS) (99%, Sigma-Aldrich) and 7.06 g of furfuryl alcohol (FA, 99%, Aldrich) were added into the P123 solution. The resulting mixtures were rigorously stirred at room temperature for 3 h, followed by aging at room temperature for 4 days, and drying at 90 °C for 3 days. The black monoliths obtained were carbonized at 550 °C for 5 hours with flowing nitrogen, leading to mesoporous C-SiO₂ composites. To determine the C/SiO₂ ratios of C-SiO₂ composites, thermogravimetric analysis (TGA, Perkin-Elmer, Pyris 1 thermogravimetric analyser) was used to record the mass loss of the samples at a heating rate of 5 °C /min under flowing oxygen. Up to 700 °C all carbon was burned off. The C/SiO₂ ratio was then calculated by taking the total mass loss as the mass of carbon and the residual mass as the mass of SiO₂. The mesoporous C-SiO₂ composite had a C/SiO₂ molar ratio of 3.54/1. By increasing the amount of FA to 11.77 g, the C/SiO₂ molar ratio could be increased to 6.87/1.

Heat treatments

The C-SiO2 composites were ground by an agate mortar and sorted within 50-100 μ m by Keiq Sieves (bought from Keison International Ltd.), then transferred into a sealed tube furnace equipped with a vacuum pump. Before heating, the furnace was vacuumed to evacuate air for 10 min. The C-SiO2 composites were heated under argon atmosphere with a flowing rate of 120 ml/min and at a heating rate of 2 °C /min up to a setting temperature. The samples were kept at the setting temperature for different time and then cooled down to room temperature at a cooling rate of 2 °C /min. The oxidization of the obtained products took place in air at 700 °C for 5 hours with a heating rate of 5 °C/min.

Characterization

X-ray diffraction (XRD) patterns were recorded on a Philips PW 1140/90 diffractometer with Cu K α radiation at a scan rate of 2 °/min and a step size of 0.02°. Scanning electron microscopy (SEM) images were taken with a JSM-6300F microscope (JEOL). The samples were degassed at 250 °C for 5 hours prior to examination. Nitrogen adsorption-desorption experiments were performed at 77 K by a Micromeritics ASAP 2020MC instrument. The pore volume was estimated from the desorption branch of the isotherm at P/P₀ = 0.98 assuming complete pore saturation. The pore size distribution was calculated from the desorption branch of the isotherm by using the Barrett-Joyner-Halenda (BJH) method.



Fig. S1 EDX analysis of mesoporous C-SiO₂ (a) before heat treatment, (b) heated at 800° C for 0min, (c) heated at 1350° C for 0min



Fig. S2 Nitrogen-sorption results of mesoporous C-SiO₂ heated at different temperatures (a) N₂ adsorption-desorption isotherms, (b) Pore size distributions



Fig. S3 TEM image of the edge of C sphere in the mesoporous C-SiO₂ heated at 1200° C for 0min



Fig. S4 High resolution SEM image of the surface of SiC spheres



Fig. S5 Nitrogen adsorption and desorption isotherm and pore size distributions (inserted) of the SiC-encapsulated C core-shell structure



Fig. S6 SEM image of SiC hollow spheres after oxidization in air at 700 °C for 5 hours



Fig. S7 TEM image of SiC hollow spheres with a thicker shell obtained from mesoporous C-SiO₂ heated in Argon at 1400 °C for 10 hours and then oxidized in air at 700 °C for 5 hours



Fig. S8 TEM image of SiC hollow spheres with a smaller diameter obtained from mesoporous C-SiO₂ with a C/SiO₂ ratio of 6.87 heated in Argon at 1400°C for 5 hours; and then oxidized in air at 700 °C for 3 hours