Supporting Information

Simple Fabrication of Micro/Nano-porous SiOC Foam from Preceramic Polysiloxane

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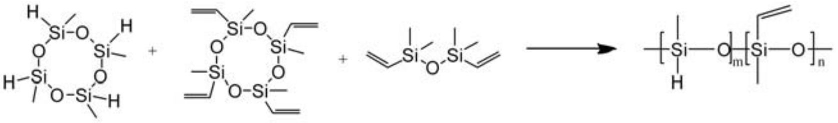
Experimental section

Methods

A typical synthesis of precursor polysiloxane (PSO):

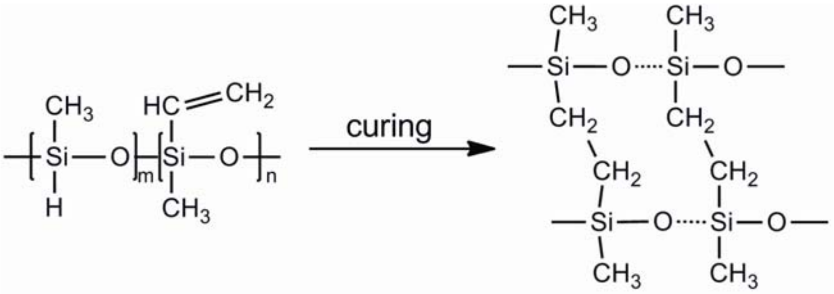
To a mixture of tetramethyl-tetravinyl-cyclotetrasiloxane (TMTVS, 650 g), tetramethyl-cyclotetrasiloxane (TMS, 776 g), and tetramethyl-divinyl-disiloxane (TMDVS, 3.4 g) was added CF_3SO_3H (2.0 g) under stirring. The reaction mixture was then heated at 65 °C for 5 hours. After neutralized with bubbled ammonia gas, the mixture was then vacuum distilled at 125 °C/2 mmHg over 3 h to remove low boiling components. The removal of $CF_3SO_3NH_4$ by filtration produced 1300 g of PSO as a colorless transparent liquid.



TMS **TMTVS** **TMDVS** **PSO**

Fabrication of SiOC porous materials:

To a homogeneous mixture of PSO and PDMS with appropriate mass ratio was added into 3 ppm of Karstedt's catalyst. The well stirred mixture was then poured into a cylindrical graphitic mold. After removal of trapped air by vacuum, the mixture was heated to 175 °C at a rate of 0.25 °C/min (for S1), 0.5 °C/min (for S2−S6) or 5 °C/min (for S7−S10) and hold for 2 hours to form crosslinked polymeric gels.



Then, the demoulded polymeric gels were pyrolyzed at a Clock Hood-type furnace under Ar atmosphere to produce porous foams. The typical ramp rate was as follows: 20–250 °C, 2 °C /min; 250–600 °C, 0.5 °C /min; 650–750 °C, 1 °C /min; 750–1000 °C, 2 °C /min; 1000–1450 °C, 3 °C /min, and holding for an hour at the maximum temperature. The maximum temperature for samples in this communication is 850, 1000, 1250, or 1450 °C.

Materials

Tetramethyl-tetravinyl-cyclotetrasiloxane (TMTVS) and tetramethyl-divinyl-disiloxane (TMDVS) were purchased from Haoyou Industry & Chemical. Co (Jilin, China) and used as received. Tetramethyl-cyclotetrasiloxane (TMS) was purchased from Runhe Chemical. Co. (Ningbo, Zhejiang, China) and used as received. Methyl-terminated polydimethylsiloxane (PDMS) was purchased from Hangping Chemical. Co. (Beijing, China). Karstedt's catalyst (platinum-divinyltetramethyldisiloxane complex in divinyltetramethyldisiloxane, 1.5 wt% Pt) was prepared by general procedure.

Characterization

FTIR spectra were measured with a Bruker Tensor-27 FTIR spectrometer. Mercury pressure porosimetry were carried out on a mercury porosimeter (Pore Master 60GT, Quantachrome Instruments, USA). Nitrogen adsorption/desorption isotherms were measured on ASAP 2020 (Micromeritics Instrument Corp., Atlanta, USA) Thermal gravimetric analyses (TGA) were carried out on a SII EXSTAR TG/DTA6300 instrument under N_2 atmosphere at a heating rate of 10 °C /min. XRD measurements were carried out on a Rigaku D/M4X 2500 diffractiometer with Cu-Kα radiation. Scanning electron micrographs (SEM) investigation and its associated energy-dispersive X-ray microanalysis (EDX) were run on a HITACHI JEOL S-4300 instrument or S-4800 instrument.

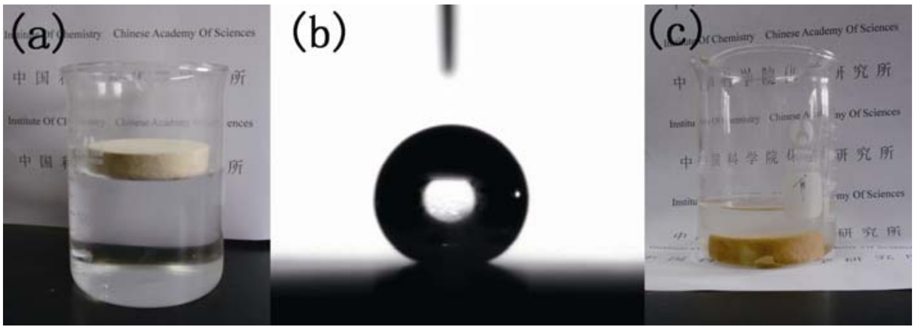


Figure S1. Hydrophobicity of the porous hybrid material prepared by pyrolysis of a mixture of PSO and PDMS (20 cSt/85 wt %) at 550 ^{o}C. (a) Floating on water, (b) The water contact angle for the sample was 152°. (c) Immerged in ethanol.

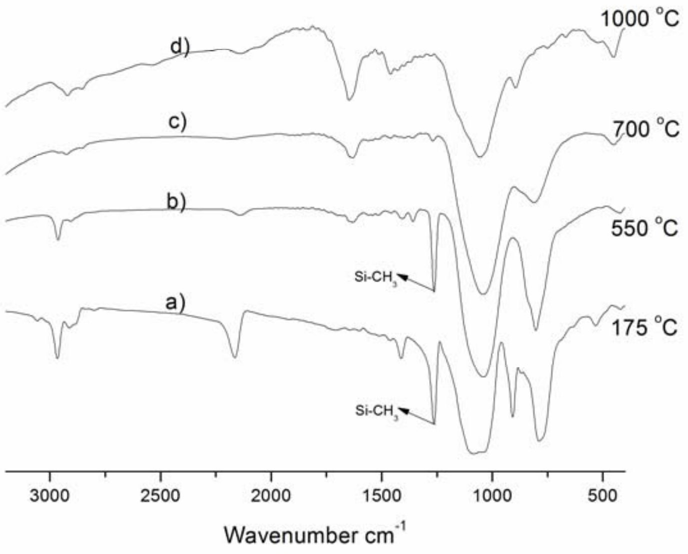


Figure S2. FTIR spectra of a) PSO cured at 175 ^{o}C for 2 hours, b), c), d) specimens produced from a mixture of PSO and PDMS (20 cSt/85 wt %) at maximum pyrolysis temperature 550, 700, 1000 ^{o}C, respectively.

Figure S3. Typical large-sized SiOC aerogel-like monolith produced from a mixture of PSO and PDMS (20 cSt/85 wt %), (pyrolysis temperature: 1000 ^{o}C).

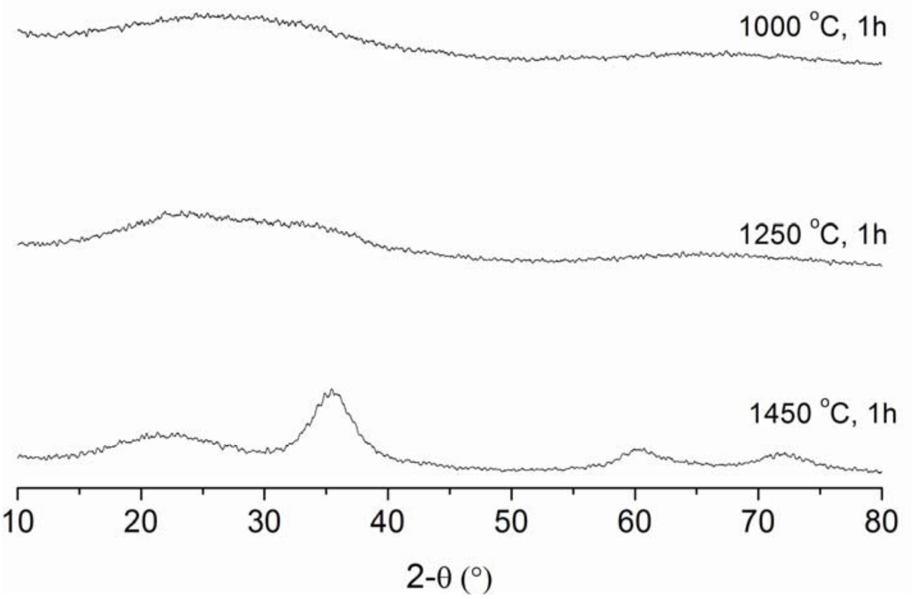


Figure S4. XRD powder diffraction pattern of samples pyrolyzed at different temperatures.