Supporting Information

Low temperature synthesis of silicon carbide nanomaterials using solid-state method

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Experimental

Materials: Tetraethoxysilane (TEOS, 99%, Sigma-Aldrich), ammonium hydroxide (NH_4OH, 28%, Caledon), magnesium powder (Mg, 99%, BDH), glassy carbon powder (C, 98%, sigma-aldrich), polyvinylpyrrolidone (PVP, M_w = 55,000, Sigma-Aldrich), sodium citrate dihydrate (ACS grade, Sigma-Aldrich), *n*-butanol (ACS grade, Sigma-Aldrich), ethanol (ACS grade, Sigma-Aldrich) and hexanes (ACS grade, Sigma-Aldrich) were used as received.

Synthesis of Stöber silica particles: Stöber silica particles were synthesized via base catalyzed sol-gel method. 10 mL (45 mmol) TEOS were stirred with ethanol (10 mL), deionized water (20 mL) and NH_4OH solution (28 %, 1 mL) for predetermined times (tabulated below) to yield different size particles. The white precipitate was collected by vacuum filtration and washed with deionized water and ethanol multiple times (4 × 25 mL). The solid was transferred to an oven and was kept there for 24 hours at 100 ^oC to drive off any residual water and ethanol.

Sample #	**Reaction time**	**Silica particle size**
1	12 hours	30.6 ± 2.2 nm
2	36 hours	66.8 ± 5.7 nm
3	120 hours	213. 8 ± 5.4 nm

Synthesis of silica nanorods: Silica nanorods were prepared using a modified procedure reported by Kuijk *et al*. PVP (30 g, M_w = 55000) was dissolved in *n*-butanol (300 mL) by sonicating for 40 minutes. Absolute ethanol (30 mL), deionized water (8.4 mL) and sodium citrate dihydrate solution (0.18 M, 2 mL) were added to the butanol solution and the flask was manually shaken to mix the reagents. This was followed by addition of NH_4OH solution (6.5

mL, 28 %) and TEOS (3 mL, 13.5 mmol) to the reactions mixture. The mixture was manually shaken for 30 seconds and left undisturbed for 12 hours. The reaction mixture turns from a clear solution to cloudy suspension as the reaction proceeds. The white precipitate was collected by centrifuging at 4000 rpm for 15 minutes. The supernatant was discarded and the white solid washed with deionized water (50 mL) three times using centrifugation (4000 rpm). The solid was finally washed with ethanol (50 mL) and dried in oven overnight at 100^{o}C. The resulting rods exhibited an average length of 1.5 ± 0.2 μm and diameter of 0.22 ± 0.04 μm.

Synthesis of silica nanofibers: The nanofibers were prepared using the same procedure described above (for nanorods), however the reaction time was increased from 8 hours to 72 hours. The resulting fibers were longer than 10 μm and 0.20 ± 0.05 μm thick.

Synthesis of silicon carbide (SiC) nanomaterial: 0.25 g (4.2 mmol) of the silica precursor (i.e., particles, nanorods or fibers) were manually mixed with 0.22 g (9.2 mmol) of magnesium powder and 0.05 g (4.2 mmol) of carbon powder and transferred to an alumina boat. The mixture was heated to 600 ^{o}C at a rate of 16 ^{o}C/min in a tube furnace under argon atmosphere and maintained at that temperature for 15 hours. The sample was cooled to room temperature and reheated to 500 ^{o}C for 30 minutes in air. The resulting grey solid was exposed to 5 M aqueous HCl (10 mL) for an hour to remove magnesium oxide. The solution was subsequently filtered and the grey solid was washed repeatedly with water. It was then dried in oven overnight at 100 ^{o}C.

Synthesis of SiC using magnesium acetylide (MgC_2): 0.25 g (10.4 mmol) of magnesium powder was manually mixed with 0.25 g (20.8 mmol) of carbon powder and transferred to an alumina boat. The mixture was heated to 400 ^{o}C at 15^{o}C/min in a tube furnace and maintained at that temperature for 12 hours. The resulting product was used within 24 hours of the synthesis. 0.25 g (4.2 mmol) of the silica precursor was manually mixed with 0.21 g (4.4 mmol) of MgC_2. The mixture was transferred to an alumina boat and was heated to 500 ^oC at 15 ^oC/min and maintained at that temperature for 15 hours. The resulting composite was treated with 5 M HCl (10 mL) for an hour and the black-grey product was collected by filtration. The solid was washed multiple times with water and dried in the oven overnight at 100 ^oC. The reaction products were characterized using XRD (**SI Fig. 4**).

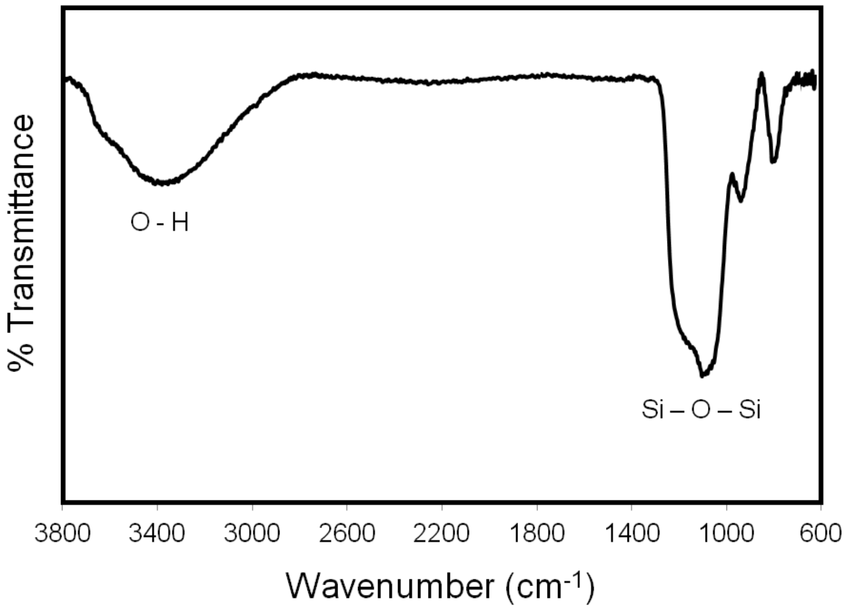
Characterization: Fourier Transformation Infrared Spectroscopy (FTIR) was performed on Nicolet Magna 750 IR spectrometer. X-ray powder diffraction (XRD) patterns were collected using an INEL XRG 3000 X-Ray diffractometer with CuK_α radiation (λ = 1.54 Å). Photoluminescence spectra for the solution phase samples were acquired using a Varian Cary Eclipse Fluorescence Spectrometer.

Transmission electron microscopy (TEM) analyses were performed using a JOEL-2010 (LaB_6 filament) with an accelerating voltage of 200 keV. The particle sizes were measured using Image J software. High resolution (HR) TEM images were obtained from Hitachi-9500 electron microscope with an accelerating voltage of 300 kV. The HRTEM images were processed using Gatan DigitalMicrograph software (version 2.02.800.0). The samples were prepared by drop coating solutions of composite dispersed in ethanol onto a carbon coated copper grid (400 mesh) and allowing the solvent to evaporate in air.

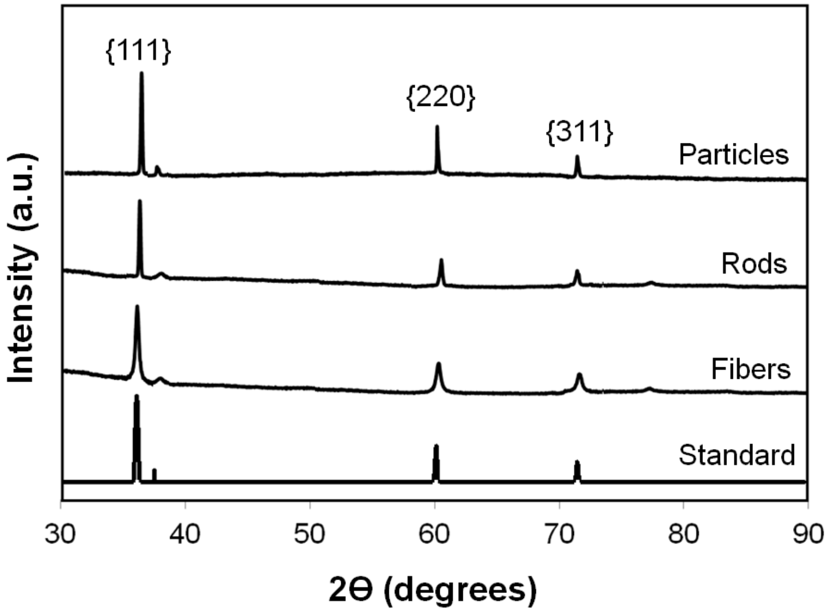
Scanning electron microscopy (SEM) images were recorded in a Field Emission Scanning Electron Microscope, JEOL 6301F. Ethanol dispersion samples were deposited on silicon wafer which was attached with aluminum pin stubs using double sided carbon tape. Conductive coatings of chromium was applied on dry samples using Xenosput XE200 sputter coaters before loading them into SEM holder. All SEM images were recorded using secondary electron imaging with an accelerating voltage of 5.0.

X-Ray photoelectron spectroscopy (XPS) analysis was performed using a Kratos Axis Ultra instrument operating in energy spectrum mode at 210 W. The base pressure and operating chamber pressure were maintained at 10^{-7} Pa. A monochromatic Al K_α source (λ = 8.34 Å) was used to irradiate the samples, and the spectra were obtained with an electron takeoff angle of 90°. To minimize sample charging, the charge neutralizer filament was used when required. Survey spectra were collected using an elliptical spot with major and minor axis lengths of 2 and 1 mm, respectively, and 160 eV pass energy with a step of 0.33 eV. CasaXPS software (Vamas) was used to interpret high-resolution (HR) spectra. All of the spectra were internally calibrated to the O 1s emission (532.9 eV). After calibration, the background was subtracted using a Shirley-type background to remove most of the extrinsic loss structure.

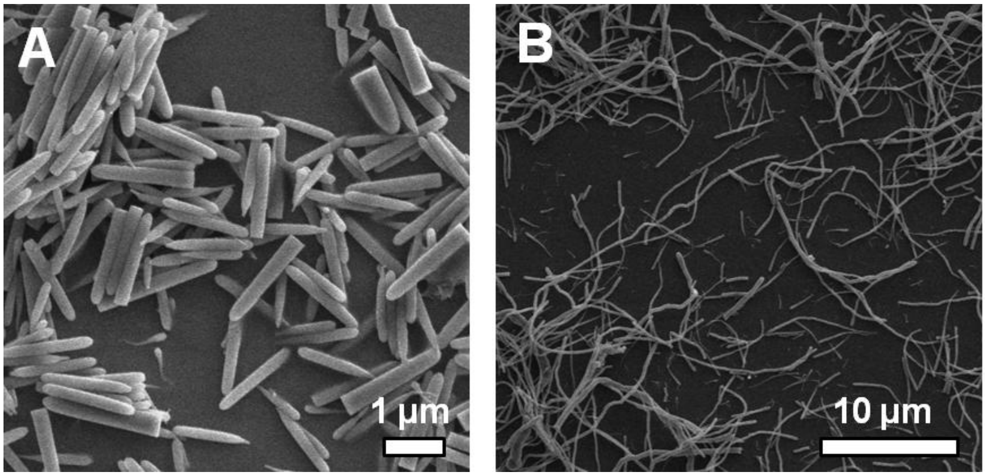
SI Figure 1: Representative FT-IR spectrum of Stöber silica particles.



SI Figure 2: XRD pattern of various different SiC structures.



SI Figure 3: SEM images of silica (A) rods and (B) fibers.



SI Figure 4: XRD pattern of MgC_2 and SiC formed from reacting SiO_2 and MgC_2.