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Single-handed helical SiC fiber preparation using 1,4-phenylene-bridged polybissilsesquioxane

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ABSTRACT

Single-handed helical mesoporous 1,4-phenylene-bridged polybissilsesquioxane fibers were synthesized using a previously reported supramolecular templating approach by increasing the concentration of NaOH. They exhibit optical activity and the size of the mesopores is approximately 2.5 nm. Single-handed helical SiC fibers, which consisted of β -SiC nanoparticles, were prepared by carbothermal reduction of the 1,4-phenylene-bridged polybissilsesquioxane fibers. The handedness of the SiC fibers was the same as that of the mesoporous 1,4-phenylene-bridged polybissilsesquioxane fibers. The left- and right-handed helical SiC fibers showed opposite optical activities. Signals were identified in the diffuse reflectance circular dichroism spectra. Since the β -SiC phase belongs to the non-chiral $\bar{F}43m$ space group, we suggest that the optical activity originates from chiral defects on the SiC nanoparticle surfaces.

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1. Introduction

Helical nanomaterials have attracted much attention because of their mechanical, optical, and catalytic properties [1,2]. Self- and external-templating approaches have been developed for controlling the handedness of these nanomaterials [3,4]. To date, single-handed helical polybissilsesquioxane, silica, TiO₂, Ta₂O₅, ZrO₂, and CdS nanotubes and nanofibers have been successfully prepared [3-9]. Template removal results in the formation of tubular structures and pore channels. Optical activity is the rotation of the plane of polarization of linearly polarized light as it travels through chiral materials. These helical nanomaterials show optical activity which is proposed to originate from chiral defects on the inner surfaces of the nanotubes or pore channels. Generally, optical activity of them is determined based on circular dichroism (CD) or diffuse-reflectance CD (DRCD). It is also reported that optical activity of anisotropic materials (especially for UV-inactive ones) can be determined using Mueller matrix ellipsometry [10].

Post-synthesis methods have been developed for the preparation of other single-handed nanotubes; for example, LiTaO₃ nanotubes have been prepared by impregnation of LiOH into Ta_2O_5 nanotubes [11]. Carbonaceous nanotubes have been prepared by the carbonization of the 4,4'-biphenylene-bridged polybissilsesquioxane nanotubes and removal of silica [12]. SiC

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http://dx.doi.org/10.1016/j.matlet.2016.04.100 0167-577X/© 2016 Elsevier B.V. All rights reserved. nanotubes have been prepared by carbothermal reduction of 1,4phenylene-bridged polybissilsesquioxane nanotubes [13]. The nanotubes retain their optical activities after these post-treatments; the optical activities probably originate from chiral defects on the inner surfaces. Because the intensity of the CD or DRCD signal is weak, the chiral defects on the surfaces are partially destroyed during the post-synthesis process. In this study, optically active SiC fibers were prepared by carbothermal reduction of single-handed helical mesoporous 1,4-phenylene-bridged polybissilsesquioxane fibers, which had high surface areas. The DRCD signal intensities were higher than those previously reported for single-handed helical SiC nanotubes.

2. Materials and methods

2.1. Characterization

Details of the characterization methods used are shown in the Supporting Information.

2.2. Materials

1,4-Bis(triethoxysilyl)benzene was purchased from Suzhou Soochiral Co., Ltd. Hydrochloric acid (35.0–37.0 wt%), sodium hydroxide, and anhydrous ethanol were purchased from the Sinopharm Chemical Reagent Co. Ltd. low-molecular-weight gelators, namely LL- and DD-12Val6PyBr, were synthesized as reported [14] (Fig. S1 Supporting Information).





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2.3. Preparation of single-handed helical mesoporous 1,4-phenylenebridged polybissilsesquioxane fibers

The gelator, i.e., IL- or DD-12Val6PyBr (100 mg, 0.11 mmol), was dissolved in 0.5 M aqueous NaOH solution (1.0 mL) to form a viscous solution. 1,4-Bis(triethoxysilyl)benzene (120 mg, 0.30 mmol) was added to the solution under vigorous stirring at room temperature. Stirring was stopped when the reaction mixture had been completely converted to a gel. The gel was kept at 0 °C for 1 d and then at 80 °C for 4 d under static conditions. The gelator was removed by extracting with a mixture of ethanol (100 mL) and 36.0 wt% aqueous HCl (5.0 mL) for 48 h. The sample was dried under vacuum at 60 °C.

2.4. Preparation of single-handed helical SiC fibers

Single-handed helical mesoporous 1,4-phenylene-bridged polybissilsesquioxane fibers were heat treated at 1400 °C for 4 h at a heating rate of $4 \,^{\circ}C \min^{-1}$ under Ar. The resultant materials were then heat treated in air at 700 °C for 2 h to afford the SiC fibers.

3. Results and discussion

LL- and DD-12Val6PyBr can self-assemble into helical bundles in water [15]. It has been reported that straight 1,4-phenylenebridged polybissilsesquioxane fibers can be prepared using IL- and DD-12Val6PyBr under weakly basic conditions [9]. In this study, we obtained helical fibers by increasing the concentration of NaOH. Left- and right-handed helical mesoporous 1,4-phenylene-bridged polybissilsesquioxane fibers are denoted by M-PS and P-PS, respectively. Moreover, the left- and right-handed helical SiC fibers are denoted by M-SiC and P-SiC, respectively. M-PS and P-PS consist of bundles of nanofibers. Field-emission scanning electron microscopy (FESEM) and transmission electron microscopy (TEM) images of M-PS and P-PS are shown in Fig. 1. The diameters of these fibers are approximately 70–600 nm. The mesopores run along the long axes of the fibers. The nitrogen sorption isotherms of *M*-PS show two hysteresis loops at $P/P_0 = 0.44-1$; these originate from the mesopores in and among the nanofibers (Fig. S2a Supporting Information). The Barrett–Joyner–Halenda pore size distribution plot calculated from the adsorption branch shows one shoulder peak at 2.5 nm (Fig. S2b Supporting Information). The size of the mesopores in the nanofibers is approximately 2.5 nm.



Fig. 1. (a) FESEM and (c) TEM images of M-PS; (b) FESEM and (d) TEM images of P-PS.

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