



Spectral properties of spherical boron nitride prepared using carbon spheres as template



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ABSTRACT

Spherical boron nitride nanoparticles have been successfully fabricated by temperature-controlled pyrolysis procedure in a N_2 atmosphere, using boron acid and urea as the precursors. The carbon spheres were prepared from glucose ($C_6H_{12}O_6$) by a hydrothermal method as a template to be used. Comprehensive scanning electron microscopy (SEM), X-ray diffraction (XRD), X-ray photoelectron spectroscopy (XPS) and Fourier infrared spectrum (IR) characterizations all confirm that the obtained products are spherical boron nitride. The amount of $C_6H_{12}O_6$ and reaction time were found to affect the morphology and structure of the as-prepared products. The average diameter of the spherical boron nitride nanoparticles synthesized with the addition of $C_6H_{12}O_6$ is about 0.3–1 μm . The spherical boron nitride has a high surface area of $176.78 m^2 g^{-1}$ and $\sim 3.5 nm$ pore size. The as-synthesized nanospheres also exhibit strong photoluminescence (PL) bands at 436, 454, 486, and 616 nm under 312 nm excitation, indicating that they could have potential application in novel optical devices.

1. Introduction

Hexagonal boron nitride (*h*-BN) as an artificially synthesized inorganic material, an analogue of graphite, has the advantages of light mass, loosening, lubricating characteristics [1]. Besides, it has unique physical and chemical properties including high thermal conductivity, electrical insulation, chemical durability, oxidation resistance, etc [2–5]. Apart from these properties, BN nanomaterials have been explored for their potential as a luminescent material which is used in ultraviolet light emission devices, light-emitting diodes and lasers [6–8].

With the development of science and technology, a variety of BN micro/nanostructures have been reported, where the representative ones include nanotubes [9], nanosheets [10], nanobelts [11], and nanofibers, etc [12]. Among the different BN nanostructures, little has been specially mentioned in the literature on the preparation and characterization of the spherical BN ensembles. Several publications about BN spheres have occasionally been reported [13–17] that nanospheres are conventionally used as fillers, pigments, coatings, and catalyst carriers due to their relatively low densities and high specific surface area [18]. In addition, BNPSs have potential applications in series of technologies and engineering. The spherical boron nitride nanoparticles were synthesized by the chemical vapor deposition (CVD) reaction of trimethoxyborane ($B(OMe)_3$) with ammonia

[14]. However, the CVD technique requires strict operating conditions on expensive equipment. Lin et al. reported a simple method to fabricate BN particles with an average diameter of 70 nm, although there was serious agglomeration of the *h*-BN particles encountered [19]. Hexagonal boron nitride nanoparticles could be synthesized through the reaction of boric acid and ammonium chloride [20]. A certain amount of copper oxide is crucial for the formation of uniform BN spherical nanoparticles with an average size of 30 nm. Furthermore, the BN nanostructures via the catalyst inevitably contain metallic or metal oxide impurities, which is not easy to eliminate. Wen et al. synthesized novel BN hollow microspheres with open mouths by a facile chemical vapor reaction approach employing ammonia-borane as a precursor [21]. The hollow microspheres exhibit intense cathodeluminescence emissions in the region of 200–400 nm, indicating that they could be potentially used as compact ultraviolet laser emitters. Bernard and coworkers reported that hollow boron nitride nanopolyhedrons (HNPHs) had been successfully prepared at 1800 °C under N_2 without a metal catalyst [22]. It is clear that the synthetic method can improve purity of HNPHs, but the service temperature is too high. BN hollow spheres were synthesized by using $NaBF_4$ and NaN_3 in autoclaves at 300 °C for 20 h [23]. However the NaN_3 is deadly poisonous compound. Moreover, hollow sphere nanostructure of *h*-BN was also investigated by using the synergic nitrogen sources [24]. Although the synthesis methods of BN spheres are multifarious, finding

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a green route to produce the BN nanospheres with high purity and high yields is still a challenge at present.

In addition, BN micro-nano structures were reported to exhibit good optical properties and have potential applications in information storage technology, medical treatment field-emission displays and nanodevices [25,26]. Li et al. reported the un-milled h-BN nanosheets had 224 nm and weak defect-related UV (~300 nm) PL emissions [27]. The deep ultraviolet (DUV) peak profile slightly changes with the increase of milling time. High-purity h-BN single crystals exhibited intense ultraviolet emission, demonstrating their promise for use as deep ultraviolet-light emitters [28]. Light in DUV emission at 225 nm for boron nitride nanotubes were observed [29]. The obtained hBN crystals emitted intense 215 nm luminescence at room temperature [30]. Chen et al. reported that the PL spectrum of the yard-glass shaped boron nitride nanotubes (YG-BNNTs) was composed of three individual luminescent emissions centered at 352, 470 and 680 nm [31]. However, the optical properties of spherical BN have rarely been reported.

In this work, we demonstrate a new synthesis strategy for the fabrication of spherical BN structures by reacting boric acid with urea in the presence of glucose. The role of glucose is investigated in the formation of BN nanoparticles, the determination optimum condition for the synthesis of spherical boron nitride nanoparticles from the amount of glucose and the heating reaction time. The as-prepared spherical BN nanostructures are characterized with different techniques including X-ray diffraction (XRD), X-ray photoelectron spectroscopy (XPS), Fourier transform infrared (FT-IR) spectroscopy, field emission scanning electron microscopy (FE-SEM). In addition, the photoluminescence (PL) properties of the spherical BN are investigated.

2. Experimental

2.1. Materials and reagents

Boric acid (H_3BO_3 , analytically pure), urea ($CO(NH_2)_2$, analytically pure) and glucose ($C_6H_{12}O_6$, analytically pure) (Sinopharm Chemical Regent Co, Ltd, China) were used to prepare the boron nitride (BN). C_2H_5OH (75.0% purity) and HCl (36.0–38.0% purity) were used to remove the byproducts of as-synthesized products. All the reagents were of analytical purity grade, and were directly used without further purification.

Oven (DUG-9036A, Shanghai Jinghong Experimental Equipment Co., Ltd, China) were used to dry products. Ultrasonic washing device (KQ-100DB, single transducer, Kunshan Company, China) was used to disperse samples, operating at ultrasonic frequency of 40 kHz and output power of 100 W through manual adjust. Centrifugal machine (TGL-16B Changzhou Zhongjie Experimental Instrument Manufacture Co., Ltd, China) was used to remove residual impurities.

2.2. Synthesis of BN powders

In a typical procedure, boric acid, urea and glucose were selected as the raw materials. Firstly, a mixture of glucose, boric acid and urea with a molar ratio of 2:1:6 were dissolved in distilled water (H_2O) and stirred for 24 h at 40 °C using a hot magnetic stirrer. The obtained product was loaded in an autoclave. After it was sealed, the autoclave was heated at 180 °C for 24 h, followed by cooling to room temperature outside the furnace. The products in an autoclave were washed with absolute ethanol and distilled water for several times. After that, a homogeneous precursor was obtained. The precursor was loaded in the middle of an alumina boat in the tube furnace with the protection of static N_2 atmosphere (~1 atm), heated at 500 °C for 1 h and 1100 °C for 2 h with heating rate of 5 °C/min. The tube furnace was cooled naturally to ambient temperature under the protection of N_2 flow. The sample was put into an aqueous solution of HCl/ H_2O (V/V =1:3)

and stirred for 12 h. Afterwards, the powder was centrifuged five times (i.e. three times water and two times in ethanol) for 10 min at 3000 rpm in order to remove all residual impurities. Finally, the sample was dried in air at 110 °C for 12 h.

By using the same method, various boron nitride products were prepared by changing the amount of $C_6H_{12}O_6$ and the heating time. BN_m-T , where m represents glucose and boric acid with a molar ratio of 0:1, 1:1, 2:1, 3:1 and T represents the heating time of at 1, 2, 3, 4 h, respectively.

2.3. Characterization of the prepared BN powders

The structure and morphology of the h-BN samples were measured using X-ray powder diffractometer (XRD, D-8, Bruker-axs, Germany, Ni filtered Cu K α radiation in the range of 2θ from 10° to 70°) with graphite monochromatized Cu K α radiation ($\lambda \sim 1.54 \text{ \AA}$). Fourier transform infrared spectroscopy (FT-IR) was obtained as KBr disks on America Elmer Spectrum One Spectrometer from 4000 to 500 cm^{-1} and scanning electron microscopy (SEM, JEOL JSM-6701F, Hitachi Corporation, Japan) were used to determine the crystalline phase and surface morphology. X-ray photoelectron spectroscopy (XPS) technique was applied to detect the surface elemental compositions of the hybrids, which was carried out on a Thermo Scientific ESCA Lab 250Xi using a 200 W monochromated Al K α as radiation. The 500 μm X-ray spot was used for XPS analysis. The base pressure in the analysis chamber was about 3×10^{-10} mbar. The ultraviolet spectrum of BN was detected by UV-Vis spectrophotometer (Cary 50, Varian Incorporation, USA) in the wavelength range of 200–500 nm. Nitrogen adsorption-desorption isotherms were obtained with a Micromeritics ASAP 2010 instrument (USA) at -196 °C. A photoluminescence spectrophotometer attached to the Hitachi F-4500 (312 nm excitation) was used to investigate optical properties of the synthesized BN products.

3. Results and discussion

Fig. 1 presents the XRD patterns of BN samples collected during different amount of glucose and heating times, including $BN_{2:1-1}$, $BN_{2:1-2}$, $BN_{2:1-3}$, $BN_{2:1-4}$, $BN_{0:1-2}$, $BN_{1:1-2}$, $BN_{3:1-2}$. All of the three peaks were observed at 26.86°, 41.82°, and 55.52°, can be indexed as hexagonal BN (0 0 2), (1 0 0), and (0 0 4), both of which are in close agreement with the reported values of hexagonal boron nitride from the Joint Committee on Powder Diffraction Standards (JCPDS) card no.34-0421. No other diffraction peaks were observed, indicating that the synthesized products have a high purity of BN without impurities.

To confirm the chemical characteristics of synthesized products, the Fourier transformation infrared spectroscopy investigations were car-

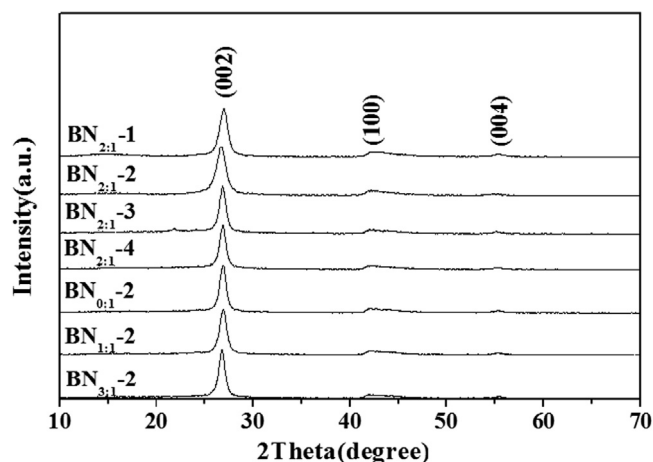


Fig. 1. XRD patterns of $BN_{2:1-1}$, $BN_{2:1-2}$, $BN_{2:1-3}$, $BN_{2:1-4}$, $BN_{0:1-2}$, $BN_{1:1-2}$, $BN_{3:1-2}$.

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