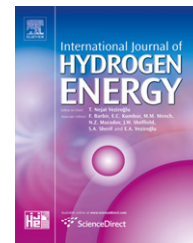


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Synthesis and hydrogen storage properties of different types of boron nitride nanostructures

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ABSTRACT

The present work reports the synthesis and hydrogen storage properties of different types of boron nitride (BN) nanostructures prepared by an *in situ* silica-assisted catalytic chemical vapor deposition technique. The BN nanostructures have been characterized by scanning electron microscopy (SEM), transmission electron microscopy (TEM), high-resolution transmission electron microscopy (HRTEM), Brunauer, Emmett, and Teller (BET), Raman spectroscopy and Fourier transform infrared (FTIR) spectroscopy studies. The hydrogen storage properties of BN nanostructures have been investigated using a high-pressure Sieverts' apparatus in the pressure range of 1–100 bar and at 298 K. The dependence of hydrogen storage capacity on the morphology of BN nanostructures is discussed in detail.

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1. Introduction

Hydrogen is a globally accepted clean fuel and has emerged as one of the most promising candidates for the replacement of current carbon-based energy services. Developing a viable hydrogen storage system is becoming increasingly important for promoting a “hydrogen economy”. Although many alloys are capable of storing hydrogen reversibly [1–7] the gravimetric storage density is too low for their use in practical applications. The recent focus of developing hydrogen storage materials has shifted to nanostructured materials which can store hydrogen by physisorption. Due to their highly porous structure, light mass density, and good interaction between carbon and hydrogen molecules, single walled carbon nanotubes are intensively studied and promoted as promising hydrogen storage materials [8–18]. However, due to their diversity in diameter and helicity, carbon nanotubes show highly complex electronic properties which lead to

controversy in hydrogen storage properties [19–22]. On the other hand, Boron Nitride Nanotubes (BNNTs) are particularly attractive because, as opposed to carbon nanotubes, their electronic properties are independent of helicity, diameter, and number of walls, and as well as they have a strong tendency to form zigzag nanotubes [23,24]. The dipolar nature of B–N bonds in BNNT can lead to stronger adsorption of hydrogen. Taking into account the chemical and thermal stability, BNNT can be considered as one of the stable lightweight hydrogen gas accumulators. Hence, it is valuable to study hydrogen storage properties of different types of BN nanostructures synthesized under different growth conditions.

In the present work, we report optimized parameters for the bulk synthesis of different types of BN nanostructures by an *in situ* silica-assisted catalytic chemical vapor deposition technique. The hydrogen storage properties of different types of BN nanostructures have been investigated using

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a high-pressure Seiverts' apparatus in the pressure range of 1–100 bar and at 298 K, and the results have been compared and discussed.

2. Experimental

2.1. Synthesis of different types of BN nanostructures

Synthesis of BN nanostructures was carried out by an *in situ* silica-assisted catalytic growth method within the framework of the vapor–liquid–solid (VLS) mechanism [25–27]. The catalyst preparation for different types of BN nanostructures involves two steps. The first step involves the preparation of the ferrous oxide (Fe_2O_3) catalyst by impregnating appropriate amounts of ferric nitrate and fumed alumina with methanol solution. The mixture is stirred, and then dried. The second step involves the ballmilling (20 h) of the pre-synthesized Fe_2O_3 catalyst together with amorphous boron and silica powder (1:1 molar ratio). Fine powders of the ball-milled Fe_2O_3 catalyst-containing mixture were directly placed in a quartz boat and placed at the center of a quartz tube, which was inserted into a tubular furnace. The furnace was heated to 1000 °C under a constant argon gas flow of 50 sccm. After reaching the desired temperature, pure ammonia gas was introduced at a flow rate of 50 sccm for 1 h. Then, the ammonia gas flow was stopped and the furnace was cooled to room temperature. Argon gas flow was maintained throughout the experiment. After cooling to room temperature, a gray product can be obtained. The product was washed with dilute hydrofluoric acid (1 M) and then filtered several times to remove the residual silica. To synthesize different types of BN nanostructures, the growth temperature was varied from 1000 °C to 1150 °C, while the flow of ammonia gas was kept constant at 50 sccm.

2.2. Scanning electron microscopy (SEM)

The morphology of BN nanostructures was analyzed using a FEI Quanta 400 ESEM FEG scanning electron microscope (SEM) operating at 20 kV. The samples were coated with gold using a sputter coating unit, to avoid charging during the recording.

2.3. Transmission electron microscopy (TEM)

Transmission electron microscopy (TEM) studies were carried out using JEOL JEM-2100F transmission electron microscopes. The sample was initially dispersed in acetone by ultrasonication for 1 h. It was then allowed to settle and a drop of the supernatant liquid was transferred onto a carbon coated copper grid and mounted onto the microscope and the micrographs were recorded.

2.4. Raman spectroscopy

Vibrational properties of BN nanostructures were collected in a backscattering geometry at room temperature using a Renishaw Raman spectrometer equipped with a 514.5 nm Ar-ion laser source.

2.5. Fourier Transform Infrared Spectroscopy (FTIR)

Variable angle attenuated total reflectance (ATR)-FTIR spectroscopy (Bruker Vertex-70LS) was employed to characterize the lattice vibrations of all BN nanostructures.

2.6. Hydrogen storage studies

Hydrogen adsorption studies were carried out for the different types of BN nanostructures in the pressure range of 1–100 bar and at 298 K, using a high pressure Seiverts' apparatus [28]. Prior to hydrogen storage measurements, experiments were conducted to make sure that the system was leak free. About 0.2 g of the sample was loaded in the sample cell which was subsequently evacuated to 10^{-6} Torr after flushing with hydrogen 2–3 times. Activation of the sample was done by heating it at 120 °C for about 4 h, following which the hydrogen at a particular pressure was allowed to react with the sample and then cooled to the desired temperature. The equilibrium pressure drop was monitored for 2 h. After each cycle the sample was degassed for 3 h at 300 °C. The hydrogen adsorption capacity of each BN nanomaterial is expressed in terms of wt % (weight of the hydrogen molecules/weight of the material \times 100). In the present work, the hydrogen storage capacity was determined by a gasometric technique. In this technique, the number of moles of hydrogen atoms adsorbed per mole of the sample is calculated from the pressure change observed before and after hydrogen adsorption at a constant calibrated volume and at a constant temperature. At higher hydrogen gas pressures, the ideal gas law was corrected using the van der Waals equation for the volume of gas molecules and molecular interactions. The low sample density of the BN materials was also taken into account while calculating the adsorption capacity.

3. Results and discussion

Fig. 1 shows TEM images of flower-type BN nanostructures (flower-type BN) grown at a temperature of 1000 °C and an ammonia gas flow of 50 sccm. These flower-type BN nanostructures exhibit hair pin structures on their outer surfaces. Whereas, by changing growth temperature to 1050 °C and keeping the ammonia gas flow constant (50 sccm), a short bamboo BN nanostructure was observed. Fig. 2 shows SEM and TEM images of short bamboo BN nanotubes (bamboo BNNTs), which clearly indicate insufficient growth temperature for the long bamboo-type BNNTs. Hence we examined the morphology of BN structures obtained at increased temperatures; long bamboo-type BN nanostructures were obtained by increasing the growth temperature to 1100 °C. SEM and TEM images show well-developed bamboo-type BNNTs having diameters of around 70–100 nm and lengths greater than 15 μm (Fig. 3). Straight-walled boron nitride nanotube (BNNT) structures were obtained with increased growth temperatures to 1150 °C and at an ammonia gas flow of 50 sccm. In Fig. 4, the SEM and TEM images of BNNTs clearly evidence a straight-walled tubular structure having an outer diameter of 70–100 nm. Furthermore, the HRTEM

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