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Hydrogen adsorption on graphene foam synthesized by combustion of sodium ethoxide

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ABSTRACT

Hydrogen storage is a crucial technology for the realization of a carbon-neutral society. However, few materials have been able to approach useful hydrogen storage capacity at reasonable temperatures and pressures. Graphene has an extremely high surface-area-to-weight ratio, is strong, cheap, chemically inert, and environmentally benign. As such it may be an ideal substrate for hydrogen storage. Here we present synthesis of graphene foam by combustion of sodium ethoxide. This technique is low-cost, scalable, and results in a three-dimensional graphene network with a surface area of more than 1200 m²/g. It is applied as a hydrogen storage material at liquid nitrogen temperature, with a capacity of 2.1 wt%.

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1. Introduction

Graphene is a single sheet of sp²-bonded carbon atoms, which was famously isolated from graphite in 2004 using sticky tape [1]. It is the strongest material ever measured, has large surface area (calculated to be 2630 m²/g), and has high electronic and thermal conductivity [2,3]. Potential applications include sensors [4], electronic devices [5], energy applications [6–8], and displays/touch-screens [9,10].

Hydrogen gas from renewable sources could provide a suitable energy vector for a carbon-neutral and sustainable

society. Storage and transport of hydrogen is a crucial part of this process. Hydrogen gas has an extremely high gravimetric energy density, however the volumetric energy density at room temperature and pressure is very low. The volumetric energy density can be improved by compression, but this results in the need for expensive and bulky storage vessels to increase safety at high pressure. To overcome this problem, there have been numerous attempts to bind hydrogen molecules onto high surface-area support materials by physisorption or chemisorption, in order to reduce the pressure needed to reach a given hydrogen capacity. However, suitable

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materials have yet to be found. Graphene offers a potential new solution, owing to its extremely high surface area, and the low atomic mass of carbon.

Graphene has been explored as a hydrogen storage material in various theoretical studies. In one of the first theoretical studies, Patchkovskii et al. calculated that the hydrogen storage capacity of graphene would be limited to <3 wt % at 5 MPa, and <4 wt % at 10 MPa at room temperature; and that 6.5 wt % should be achievable at 200 K and 5 MPa [11]. These values are well within the DOE targets for hydrogen storage. Dimitrakakis et al. calculated the hydrogen storage capacity for a pillared 3D graphene-like structure doped with lithium cations, concluding that the DOE targets for mobile applications at room temperature could be achieved with this layered material [12]. Titanium anchored to graphene oxide (GO) substrates has been calculated to have a 4.9 wt % gravimetric capacity at room temperature [13]. Two groups have independently proposed calcium-decorated graphene with calculated room temperature hydrogen storage capacities of 5 wt % and 8.4 wt %, respectively [14,15]. Lithium-doped porous graphene was calculated to have a capacity of 12 wt. % [16], and aluminium-doped graphene was calculated to have a capacity of 13.8 wt % [17], although these extremely high capacities seem optimistic.

Few experimental results have been published on the hydrogen adsorption properties of graphene. The spacing

between graphene sheets was tailored via chemical functionalization, resulting in a maximum hydrogen storage capacity of 1.4 wt% at 77 K and 0.2 MPa [18]. Chemical bonding of hydrogen to graphene to produce *graphane* resulted in 5 wt% hydrogen content. The hydrogen was liberated by heating the sample to 500 °C, or by laser irradiation [19]. Pd- and Pt-decorated graphene were measured to have a capacity of just 0.15 wt% at room temperature and 6 MPa in one study [20], whilst a capacity of 1.2 wt % at room temperature and 3.2 MPa was measured in similar samples [21]. A three-dimensional network of NiB-doped graphene displayed a hydrogen capacity of 4.4 wt% at 77 K and 0.1 MPa [22,23]. The above results vary widely, and so far the DOE targets have not been reached in experimental studies. However, it seems that there is great promise for graphene-based hydrogen adsorption in the future.

The main factors to be considered if graphene is to be utilized as a practical hydrogen storage material are cost, scale-up, and surface area. Commercially available graphene is currently very expensive. Economies of scale will play a part in reducing this, however cheaper methods of graphene synthesis are required. The surface area of commercially available graphene is also an issue, with most samples being limited to around 700 m²/g. A promising technique to produce cheap graphene with large surface area at the gram scale is by combustion of sodium ethoxide. [24] Such graphene foam has already found application as a platinum support [25], and as a metal-free catalyst for the electrochemical oxygen reduction reaction in polymer electrolyte membrane fuel cells [26].

Here, we synthesize graphene foam via combustion of sodium ethoxide, and explore its hydrogen adsorption properties.

2. Experimental

5 ml of ethanol and 2 g of sodium were added to a PTFE melting pot, under inert atmosphere. The vessel was tightly sealed and heated to 180 °C for 24 h, to yield sodium ethoxide. This was ignited in air in an ignition dish, and allowed to burn. Note that extreme care must be taken when working with high pressure, sodium, or open flames. The carbonized product was collected, crushed in a pestle, mixed with deionized water, and sonicated for several hours. It was then washed in deionized water by vacuum filtration, and dried under vacuum at 120 °C overnight, yielding a fine black powder. Subsequently, this was pyrolysed at 1000 °C for 1 h, under flowing nitrogen, in order to improve graphitization and decompose any remaining sodium ethoxide impurities. Commercially obtained graphene was used as a reference sample (GNPs Grade 3, Cheap Tubes Inc.).

The product was characterized using scanning electron microscopy (SEM) (S-5200, Hitachi, Japan) in tandem with energy dispersive X-ray analysis (EDX) (Genesis 4000, EDAX Japan K.K.); X-ray photoelectron spectroscopy (XPS) (ESCA-3400SPQ, Kratos Analytical Ltd.); Raman spectroscopy (DM2500M, Renishaw); and nitrogen adsorption (Belsorp mini II-VS, Bel Japan, Inc.). Hydrogen sorption isotherms (Belsorp-HP, Bel Japan, Inc.) were carried out at 77 K, up to 1 MPa.

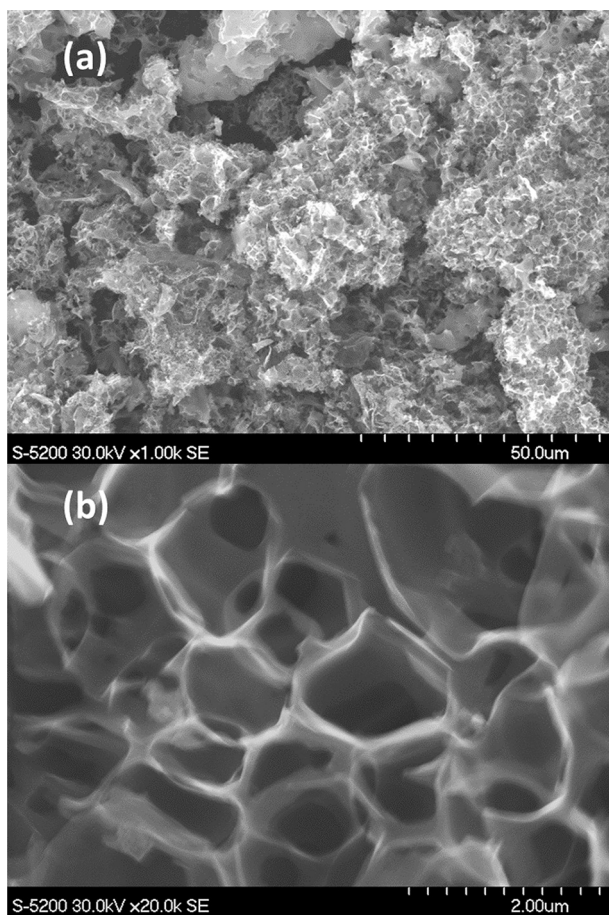


Fig. 1 – Scanning electron microscopy of graphene foam at different magnifications.

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