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Functionalization of hexagonal boron nitride in large scale by a low-temperature oxidation route

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ABSTRACT

A simple and effective method to functionalize hexagonal boron nitride (hBN) was demonstrated by low temperature (600 °C) oxidation of hBN powders in air. An oxygen-rich amorphous layer was observed on the surface of oxidized hBN particles. Compared to raw hBN, the oxidized hBN particles showed better affinity with ethanol. In addition, the oxidation process and mechanism were also investigated in detail. The results showed that the longer oxidation time, the thicker oxidation coating would be formed. In the beginning of oxidation, oxygen could insert into the lattice ascribe to the surface defects of hBN. With oxidation time increasing, more oxygen would embed in the inner of h-BN particles, causing the intense lattice distortion in local zone. With the oxidation process proceeded, the amorphization behavior would appear, and form a coating of amorphous boron oxides on the surfaces of h-BN particles. This method holds great promise for the application of hBN materials.

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1. Introduction

Analogous to graphite, hBN has superior thermal, mechanical and tribological properties, and has been used as reinforced phases in composites to enhance their performances [1–5]. However, the thermal stability and chemical inertness makes it hard to combine with others [6,7]. Hence, great interest has been attracted on the improvement of its surfactivity or wettability. In contrast to hBN, graphene has performed certain activity, but it has not yet been applied in large-scale until the widely fabrication of graphene oxide (GO), which shows higher reactivity due to the extended oxygen-containing functional groups on the surface [8–10]. Many similar efforts have also been devoted to functionalization of hBN recently [11]. Sonication of hBN in hydrazine, piranha solution or molten hydroxides has been confirmed to be an effective treatment for the functionalization of hBN [12–14]. The

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functionalized hBN in above process shows good affinity with various organic solvents, but this liquid-phase method often requires complicated operation process, and the chemical reagents used are harmful to the environment. Hence, a facile and ecofriendly way to functionalize hBN needs to be explored.

In this paper, a simple and effective method for the surface functionalization of hBN is proposed. By treating of hBN powders at 600 °C for 144 h in air, an oxygen-rich amorphous layer was decorated on hBN, although the thermodynamics reaction temperature between hBN and oxygen is above 800 °C [15,16]. The functionalized hBN showed better affinity with ethanol. And the oxidation process and mechanism of hBN were also investigated in detail.

2. Experimental

Commercial hBN powders with the purity of 99.9% and average size of 10 μ m were used as raw material. The powders were homogenously spread on a high purity alumina plate. The oxidation process was conducted at 600 °C for 144 h in a muffle furnace equipped with ventilation equipment (Nabertherm GmbH, Germany). The dry air was supplied from the bottom of the furnace with a flowing rate of 50 mL/min.





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Fig. 1. (a) XRD patterns, (b) Raman spectra and (c) FTIR spectra of r-BN and o-BN, respectively, (d) UV-vis spectra of r-BN and o-BN in ethanol, with the inset showing photograph of ethanol, r-BN and o-BN in ethanol (e) TGA curves of r-BN and o-BN in different atmosphere.



Fig. 2. SEM images of r-BN (a) and o-BN (b) with EDS.

Crystalline phases of the raw hBN (r-BN) and oxidized hBN (o-BN) were identified by X-ray diffraction (XRD, X'Pert Pro) with Cu K_{α} radiation. Raman spectra (HR 800), Fourier transform infrared spectrometer (FTIR, IRPrestige-21) and UV–vis absorption spectra (TU-1900) were used to analyze the surface structure of the powders. High temperature stability of the powders was investigated by thermogravimetric analyzer (TGA, SDT Q600). Microstructures were observed by field emission scanning electron microscopy (FE-SEM, FEI Quanta600) and high resolution transmission electron microscopy (HRTEM, JEOL JEM-2100F).

3. Results and discussion

Fig. 1a–c illustrates XRD patterns, Raman spectra and FTIR spectra of r-BN and o-BN powders, respectively. Only hBN peaks were detected in r-BN, but when r-BN was oxidized, an amorphous boron oxide phase was also observed. In addition, compared with

the diffraction peaks of r-BN, the hBN peaks of o-BN move to the left slightly, indicating the increase of the crystal plane spacing. Raman analyses show that, the peak intensity in o-BN is weaker but its FWHM is wider than that is in r-BN, which can be considered to the formation of functional groups, furthermore, the E_{2g} mode for r-BN is centered at 1366 cm⁻¹, while the peak of o-BN has blue shifted to 1369 cm⁻¹, indicating the formation of BNO layers [17,18]. FTIR results show that compared with r-BN, some new peaks appear in o-BN, which can be attributed to the B-O-H band (3200 cm⁻¹ and 883 cm⁻¹) and B-O band (1210 cm⁻¹ and 927 cm⁻¹) [12,14].

To demonstrate the change before and after oxidation of hBN, UV–vis absorption spectroscopy (Fig. 1d) and TGA (Fig. 1e) were used. UV–vis absorption spectroscopy shows that r-BN has a scattering profile while o-BN has an absorption peak at \sim 203 nm, suggesting that the surface functional groups are formed in o-BN. The photograph in the inset identifies that these powders have different dispersibility in ethanol. TGA curves indicate that when

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