

Full Length Article

Preparation of high-content hexagonal boron nitride composite film and characterization of atomic oxygen erosion resistance

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ABSTRACT

Space aircrafts circling in low earth orbit are suffered from highly reactive atomic oxygen (AO). To shield AO, a flexible thin film with 80 wt.% hexagonal boron nitride (h-BN) and h-BN/epoxy film were fabricated through vacuum filtration and adding nanofibrillated cellulose fibers. H-BN nanosheets were hydroxylated for enhancing interaction in the films. Mass loss and erosion yield at accumulated AO fluence about 3.04×10^{20} atoms/cm² were adopted to evaluate the AO resistance properties of the films. A carpet-like rough surface, chemical oxidations and change in crystal structure of h-BN were found after AO treatment, and the degrading mechanism was proposed. The mass loss and erosion yield under AO attack were compared between h-BN film and h-BN/epoxy film, and the comparison was also done for various types of shielding AO materials. Excellent AO resistance property of h-BN film is shown, and the reasons are analyzed.

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1. Introduction

Strong oxidizing atomic oxygen (AO), which accounts for 80% of neutral gases in low earth orbit (LEO), is the predominant factor that drives materials failure in LEO [1], especially for polymeric materials. Current protective materials, such as metals, inorganic particles and composite materials, are applied in satellites, space stations and other spacecrafts used in LEO space environment [2–4]. Among these options, multifunctional composite materials with low density and high mechanical property have been focused in recent years. In this area, utilizing nanomaterials, including graphene, carbon nanotubes (CNTs), polyhedral oligomeric silsesquioxane (POSS) and boron nitride (BN) in polymer composite, is prevailing tendency for enhancing AO resistance [5–8].

Hexagonal boron nitride (h-BN) with large band gap [9], excellent mechanical property [10], thermal and chemical stability [11,12], has been regarded as potential candidate in terms of water cleaning [13], luminescent device [14] and reinforced phase in composite materials [15]. Simple and feasible synthetic approaches such as chemical vapor deposition [16–18] have made it reality

for production of h-BN with thickness ranging from mono layer to a few atomic layers. Thin h-BN is proved to have superior anti-oxidation property even at high temperatures. Liu et al. transferred a 5 nm thick h-BN thin film onto the surface of stainless steel, which confirmed excellent performance on shielding stainless steel from erosion at 1000 °C for 30 min [19]. Such outstanding property of h-BN is not only in air oxidation, but also can be extended to resistance to highly reactive AO. Yi et al. found that with only 0.5 wt.% addition of h-BN, the mass loss of epoxy composite after AO exposure would decrease by 70% compared with undoped epoxy specimens [20]. Therefore, h-BN and its composite are promising candidate materials for shielding structure against AO.

H-BN materials in forms of powder [21], fiber [15] and thin film [11] have been investigated and successfully applied. In the area of AO protection, h-BN powder/epoxy resin bulk composite was prepared by blending method [20], and h-BN polymer composite film was obtained via solution casting [22]. There are some works about AO resistance of h-BN composite with h-BN content varying from 0.2 wt.% to 4 wt.% [22,23], but limited studies were launched on AO exposure behavior of h-BN film with higher h-BN concentration. Considering the outstanding chemical property of h-BN, we believe that h-BN film with high content of h-BN should exhibit superior performance when exposed to AO. However, since h-BN is chemical inert and easy to agglomerate, it is difficult to obtain flexible and free-standing h-BN film with high h-BN content. Herein, researches

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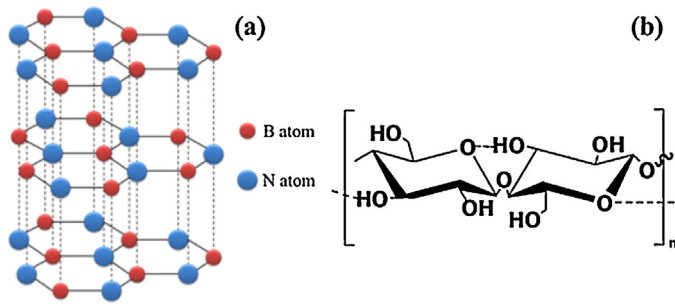


Fig. 1. Schematic illustrations of (a) h-BN and (b) NFC.

on the fabrication technique of h-BN film containing high level of h-BN are of great significance.

Our previous work reported good performances of light weight, strong CNT and CNT/epoxy film against AO erosion. Due to chemical inertness, high CNT content and compacted packing structure of CNT film, its AO resistance was better than carbon fiber reinforced composites, polymers and nanoparticle modified polymers [24]. Considering excellent chemical stability and anti-oxidation property of h-BN over CNT, we believe that film materials made of h-BN should possess much better AO erosion resistance than CNT films and can be used as surface protective layer. Research about AO resistance property of high h-BN content film has not been reported on the known literature.

This paper started with the fabrication of flexible and free-standing film with high h-BN content, followed by studying AO exposure behaviors of h-BN and its composite films. This film about 13 μm thick with 80 wt.% h-BN and 20 wt.% cellulose fiber was obtained through vacuum filtration method, and h-BN/epoxy resin composite film was produced by hot-pressing process. In order to improve interaction between h-BN and cellulose fiber, hydroxyl treatment of h-BN was carried out and composite film using treated h-BN was prepared. The specimens were tested in a ground simulation system where AO fluence was 3.04×10^{20} atoms/cm². Changes in surface morphology, chemical composition and structure of the films after AO experiment were analyzed. Based on the results, the erosion process and shielding mechanism of the films were discussed.

2. Experimental

2.1. Materials

H-BN (percentage of 99%, crystal size $<3 \mu\text{m}$) was purchased from Zibo Jonye Ceramic Technologies Co., Ltd. and used as received without further purification. Hydroxyl treatment of h-BN via substitution reaction [25] was performed in a 5 mol/L sodium hydroxide (NaOH, Beijing Chemical Works, analytically pure) solution under mechanical stirring at 100 °C for 12 h at 5 mg/ml concentration. The resulting h-BN-OH particles were rinsed with deionized (DI) water and filtered several times until the pH of the suspension was 7. Next, the particles were dried in a culture dish at 70 °C for 12 h, allowed to cool to ambient temperature, and stored in a desiccator.

Nanofibrillated cellulose (NFC, diameter 10–20 nm, length 0.8–2.0 μm , substituted ratio of carboxyl 0.46, charge density 1.5 meq/g) was obtained from Tianjin Haojia Cellulose Co., Ltd. The chemical structures of h-BN and NFC [26] are shown in Fig. 1. 4, 4'-tetradiglycidyl diaminodiphenyl methane epoxy resin, named as AG80, with 4, 4'-diaminodiphenyl sulfone (DDS) as hardener (Zhenzhengfeng New Materials Co., Ltd.) was used. DI water and acetone (Beijing Aoli general chemical reagent sales co., Ltd.) was used as dispersant and solvent respectively.

2.2. Specimen preparation

H-BN-OH film was prepared through vacuum filtration on top of cellulose membrane (aperture diameter: 0.45 μm) at room temperature and atmospheric pressure (Fig. 2a). Firstly, the proportionally calculated NFC was dispersed in DI water under sonication for 30 min to achieve a homogenous stabilized dispersion. Then h-BN-OH was added into the dispersion at 4: 1 equivalent weight ratio to NFC. The mixture was sonicated for 30 min and diluted to the concentration of h-BN-OH at 1.3 mg/ml. A certain amount of the mixture was vacuum filtered and the membrane with h-BN-OH film was transferred onto a heating platform to evaporate residual DI water at 40 °C. After dried for 10 min, h-BN-OH film was carefully peeled off from the membrane.

H-BN-OH/epoxy film containing 28 wt.% of epoxy resin was prepared using one layer of h-BN-OH film via hot-pressing process

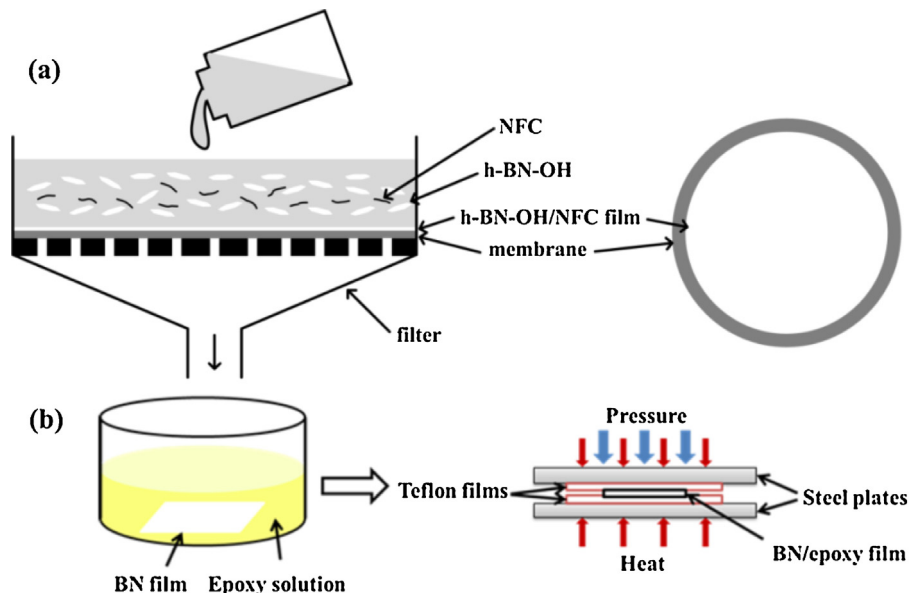


Fig. 2. Schematic illustrations of (a) h-BN-OH and (b) h-BN-OH/composite films preparation.

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