



# Synthesis of nanostructural turbostratic and hexagonal boron nitride coatings on carbon fiber cloths by dip-coating



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## ABSTRACT

The fabricating and characterization of boron nitride coating onto carbon fiber cloths via dip-coating process with boric acid and urea as precursors, followed by nitriding in a nitrogen flow were done in this study. Temperature and dip-coating cycles were considered as variables to find out optimum amounts. Dip-coating process has done in 1, 2 and 4 cycles. Also, nitridation process was done in different temperatures of 650 and 1000 °C. The results show that boron nitride coatings exhibited a pebble-like structure. The SEM micrographs indicated that the thickness of nanostructural coatings among 1, 2 and 4 cycles of dip-coating is about 55, 120 and 210 nm, respectively. By increasing dip-coating cycles, smoother surface was developed. The FT-IR and XRD results revealed that the boron nitride film fabricated at 650 °C has turbostratic microstructure and by increasing the nitriding temperature to 1000 °C, hexagonal boron nitride phase was indicated through the film. Thermal analysis revealed that boron nitride crystallization starts at temperature of 600 °C.

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## 1. Introduction

Boron nitride has distinct physical and chemical properties such as low density, high melting and boiling point, superb thermal conductivity and high electrical resistivity [1]. In particular, h-BN has a layer structure like that of graphite that causes good interface lubricity [2]. It can also help to solve the strong adhesion problem between fiber and ceramic matrix by creating a weak interface between them, thereby developing mechanical properties of composites [3,4].

Carbon fiber cloths have extensive application in military and air craft brakes due to their unique thermal, mechanical and wear properties. However, their natural tendency to be oxide around 450 °C limits their probable applications [5]. So, providing the carbon fibers a continuous coating that oxide at higher temperatures is very necessary. Boron nitride (BN) has been suggested as a coating material on carbon fiber cloths, because of its graphite like structure and its better oxidation resistance. Higher oxidation resistance is due to the fact that at higher temperature, BN forms a protecting B<sub>2</sub>O<sub>3</sub> layer [6].

Boron nitride coatings had been manufactured primarily by chemical vapor deposition (CVD) method [7]. Some researchers had successfully used dip-coating method to prepare coatings on fibers [8]. The dip-coating method, when compared with the CVD method, was simpler in procedure, more economical and might obtain uniform coatings on every fibers of 2.5D fabric, which had been widely used in ceramic matrix composites.

In this study, besides preparing boron nitride coating on carbon fiber cloths by dip-coating method, which is a simple, economical and controllable rout to obtain continuous coatings, the effect of nitridation temperature and different cycles of nitridation on coating morphology, structure and thickness was also investigated.

## 2. Experiments

### 2.1. Sample preparation

The carbon fiber cloths (T300, 3K, Torayca Co.) with an average size of 5 × 10 cm were used as substrates for dip-coating. Six (6) carbon fiber cloths were considered as samples for applying the coating processes. Firstly, samples were ultrasonically cleaned in acetone for 10 min. Then, the samples were heated dry at 110 °C in an oven for 1 h. Finally, samples were prepared by heat treatment at 350 °C in air for 2 h to reduce porosity of primary samples.

### 2.2. Preparation of BN coating

Boric acid (H<sub>3</sub>BO<sub>3</sub>, Merck, Germany) was used as boron source while urea (N<sub>2</sub>H<sub>4</sub>CO, Merck, Germany) and nitrogen gas (Technical Gas Services, 99.999%) were used as nitrogen sources of BN. To prepare dip-coating solution, boric acid and urea (4:1 mol. ratio) were dissolved in a mixture of methanol and distilled water (3:1, vol. ratio), then the solution was ultrasonically stirred for 15 min in order to disintegrate the agglomerates. The samples were dipped into the solution for approximately 5 min. Then the samples were drawn from the solution with a low speed (3 cm/min) by a low speed motor and dried in air for 10 h.

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The dip-coated samples were heated to 120 °C for 1 h to evaporate the residual solvent. Later, the samples were heated to a temperature of 150 °C in tube furnace by vacuum of 400 m Torr. Finally, half of the samples were nitrided at temperature of 650 °C and others were nitrided at 1000 °C at a rate of 2.5 °C/min under nitrogen flow for 2 h. In order to obtain different thicknesses, three dipping-drying-nitridation cycles of 1, 2 and 4 were considered in this study.

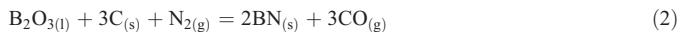
### 2.3. Characterization

The morphology and thickness of BN coatings were characterized by scanning electron microscopy (FESEM, ZEISS, Germany). The surfaces topography and roughness were examined using atomic force microscope (AFM, CP-Research, USA). The chemical compositions of coating were analyzed by EDS equipped with SEM. An investigation of bonding in the nitridation production from BN precursor was performed via Fourier transform infrared spectroscopy (FTIR, Perkin Elmer Spectrum, United States). The crystal structure of the coatings was characterized by X-ray diffraction (XRD, STOE-Study-MP, Germany). The chemical reactions to form BN and its crystallization behavior were characterized with a TG/DTA instrument (STA, NETZSCH 409, Germany) in the nitrogen atmosphere.

## 3. Results and discussion

### 3.1. FESEM, EDS and AFM analysis

The number of coating cycles and nitridation temperature have numerous effects on the morphology and thickness of coatings. Figs. 1, 2 and 3 show the effect of coating cycle numbers on coating thickness and morphology in constant temperature. After one cycle of dip-drying treatment and nitriding at 650 °C, a non-continuous coating can be found (Fig. 1a). By increasing the number of coating cycle, a more uniform coating is obtained; although it doesn't have a fully smooth and uniform surface, which could be due to low temperature and time of nitridation (Fig. 1b and c). There is another reaction (2) for the formation of BN from boric oxide, carbon, and nitrogen according to Aydoğdu and Sevinç [9] which completes at higher temperature compared to reaction of boric acid and urea (1) (more than 600 °C) [10]. In low temperature coated samples the dominant mechanism is due to reaction (1); however, at 1000 °C coated samples reaction (2) accomplishes too that cause uniform layer due to melting of B<sub>2</sub>O<sub>3</sub> and reaction with N<sub>2</sub>. By increasing temperature to 1000 °C the viscosity of liquid B<sub>2</sub>O<sub>3</sub> increases and helps the coatings to be more uniform.



By increasing nitridation temperature to 1000 °C in same conditions, uniform nanostructured coating can be formed even in first cycle of dipping-drying-nitridation treatment. As shown in Fig. 2, every single carbon fibers are fully covered by coatings and the surface is smooth and uniform. Increasing the number of coating cycles causes an approximately linear increase in coating thickness (Fig. 3a, b and c) [8]. As shown in Fig. 4, the nano size pebble-like BN coating can be considered as nanostructured coating.

Fig. 5 shows the energy dispersive spectroscopy (EDS) spectrum of the heat treated samples which nitridated at 1000 °C in four dip-drying cycles. As shown in Table 1, theoretical weight ratio of N:B compare to N:B weight ratio obtained from EDS, can prove the formation of boron nitride. Fig. 6 shows the existence of nitrogen and boron elements in the coating which synthesized at 650 °C, however, the weight ratio of N:B is not close enough to its theoretical value, as shown in Table 2. This

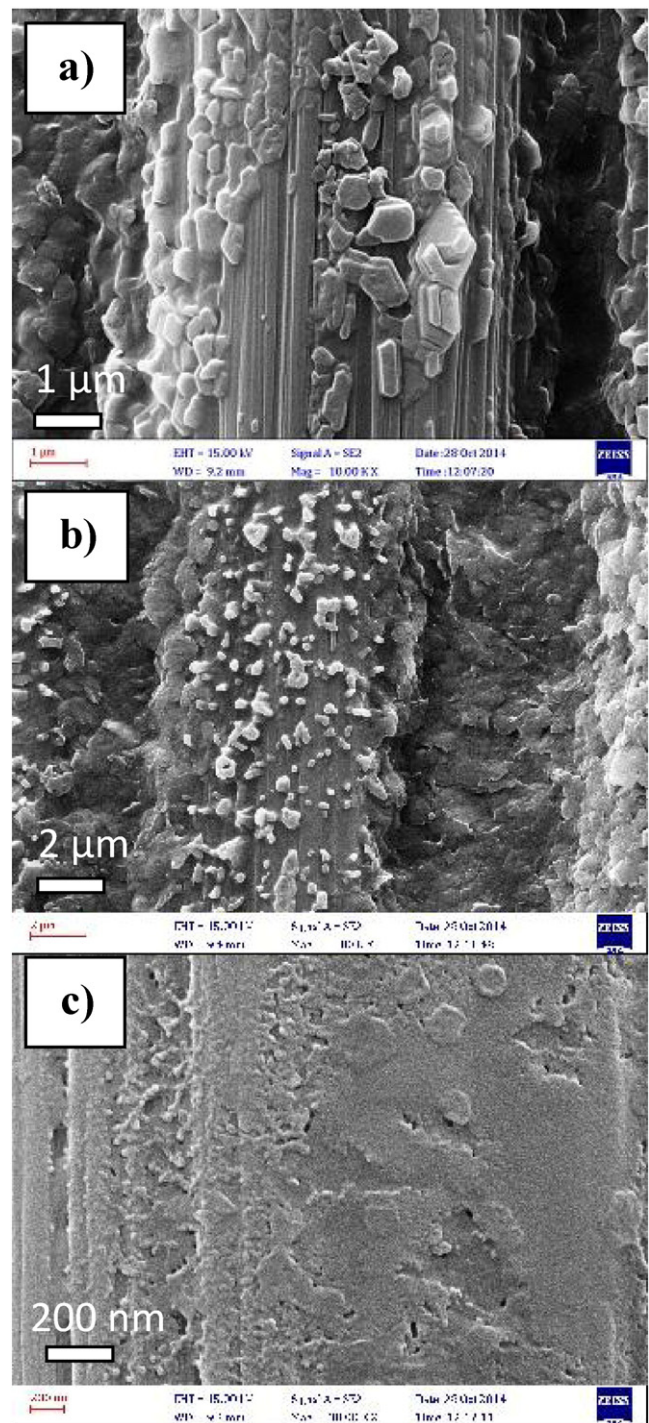


Fig. 1. Heat-treated samples and coated at 650 °C a) 1 cycle, b) 2 cycles, c) 4 cycles.

mismatch could be shown in complete reaction of forming BN from precursors due to low temperature and/or inadequate time of nitridation. It can be considered that the high temperature reaction (1) would use residual B<sub>2</sub>O<sub>3</sub> to form BN and make a more uniform coating.

For roughness and 3D morphology investigation, the atomic force microscopy (AFM) analysis was performed. As shown in Fig. 7 and Fig. 8, the heat treated samples which dip-coated once at 1000 °C, have rougher surface compare to those heat treated samples which dip-coated 4 times at the same temperature. It shows that increasing the dip-dry-nitridation cycles, causes more uniform and smoother surface. The average roughness of one cycle coated samples is 30 and 25 nm long to area 1 and 2, respectively. Though, the average roughness

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