



# Novel carbon nanofibers build boron carbonitride porous architectures with microwave absorption properties



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## ARTICLE INFO

### Article history:

Received 18 August 2014

Received in revised form

15 January 2015

Accepted 1 February 2015

Available online 10 February 2015

### Keywords:

BCN

Porous architectures

Microwave absorption

## ABSTRACT

Boron Carbonitride (BCN) porous architectures build by carbon nanofibers and BCN precursor were successfully synthesized via a simple precursor pyrolysis method. Scanning electron microscope (SEM) and BET results showed the BCN porous architectures having the pore size distribution ranging from 50 nm to 24 microns in diameter. Fourier transform infrared (FT-IR) and X-ray diffractometer (XRD) results indicate that the products have the chemical composition of h-BCN and graphite. The minimum reflection loss and the frequency bandwidth below  $-20$  dB for BCN are  $-56.2$  dB and 7.7 GHz respectively, indicating better microwave absorption properties.

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## 1. Introduction

In recent years, hexagonal ternary BCN (h-BCN) materials, the analogues of graphite, have attracted tremendous interests because its dielectric properties can be tailored by changing the composition. Comparing with graphite, h-BCN materials have better oxidation resistance property, more excellent thermal conductivity and better chemical stability [1–4]. BCN materials are widely used in large numbers of applications, such as electronic devices [5], supercapacitor [6], hydrogen storage [7], electronic field emission [8] and electrocatalysts [9]. It is expected that the BCN porous architectures may have interesting properties and applications complementary to its analogues h-BN and graphite. However, there are only a few reports on the synthesis of the porous BCN architectures since it is a better candidate in the fields of hot gas separation, sensors, and microwave absorption [10,11].

Herein we have fabricated BCN porous architectures in high purity by PAN nanofibers and BCN organic precursor. The as-prepared BCN porous architectures exhibit promising microwave-absorbing properties with the minimum reflection loss of  $-56.2$  dB at 7.7 GHz, much better than traditional microwave-absorbing materials. The oxidation resistance property of the as-prepared BCN porous architectures is exciting reaching to 816°C

higher than graphene and the reported BCN nanotubes [12]. This fabrication method may contribute to exploring other new porous materials.

## 2. Experimental

The organic precursor (Equation (1)) as one of starting materials was synthesized from Boron trichloride ( $\text{BCl}_3$ ) (purity 99.9%, purchase from Guangzhou Ruihe Chemical Science and Tech. Co. Ltd) and Aniline ( $\text{Ph-NH}_2$ ) (purity  $\geq 98\%$ , obtained from Tianjin Yongda Chemical Reagent Co. Ltd) (molar ratio 1:1).  $\text{BCl}_3$  was piped into  $\text{Ph-NH}_2$  for 2 h at the flowing rate of 0.35 L/min which was located into a flask keeping at 100°C, and then continued to react 3 h at 100°C after turning off the  $\text{BCl}_3$ . Finally the reacted product was cooled to the room temperature naturally forming the dark brown precursor. The as-prepared BCN organic precursor was injected into the PAN nanofibers sheets layer by layer that were prepared by electrospinning method. The BCN organic precursor–PAN nanofibers hybrid was delivered into a tube sintering furnace and heated to 1200°C at a heating rate of 5°C/min for 2 h in a flowing nitrogen atmosphere (purity 99.99%, flowing rate 4.5 L/min.), and then cooled to the room temperature naturally with flowing nitrogen atmosphere. In the end, the black products were collected without any further purification.

The morphology of the products was observed using a field emission scanning electron microscopy (SEM, MX2600FE). The chemical characteristics were examined by Fourier transform

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infrared (FTIR, Bruker VECTOR-22). The structures were examined using RIGAKU D/Max 2000 VPC powder X-ray diffractometer (XRD,  $\text{CuK}\alpha$  radiation,  $\lambda = 1.5418 \text{ \AA}$ ) and Raman spectroscopy (Renishaw, RM-1000). Thermogravimetric analysis (TGA) was recorded on a NETZSCH STA 449C under ambient atmosphere. The Barrett-Emmett-Teller (BET) surface area was performed at  $-196 \text{ }^\circ\text{C}$  using nitrogen in a conventional volumetric method by Micromeritics ASAP 2020. The reflection loss, RL (dB), of the BCN porous architectures were simulated and obtained using our self-programmed software.

### 3. Results and discussion

A group of typical SEM images show in Fig. 1. Fig. 1a and b shows the uniform pore structure of the as-prepared products with the pore size of  $2\text{--}5 \text{ }\mu\text{m}$  built by the carbon nanofibers. Fig. 1c and d shows another kind of waterweeds-like pore morphology of the products with the pore size about  $5\text{--}30 \text{ }\mu\text{m}$  in diameter. The waterweeds-like backbone is probably contributed by the carbonized PAN nanofibers. The membrane like structure between the fibers may be attributed to the BCN organic precursor pyrolysis during the high temperature.

Fig. 2 shows the FT-IR transparency spectra for the BCN precursor and BCN porous architectures. Fig. 2a shows the typical peaks of the basic structures of the BCN precursor ( $\text{cm}^{-1}$ ): Phenyl N–H (2858, 2589); Phenyl (1498, 1596) and B–N ring (1380, 683), B–N (1182), B–Cl (1022, 745) [13,14]. It can be indicated that the B–N ring and Phenyl bondings build the backbones of the precursor. Fig. 2b shows the typical spectrum of the products, the wave numbers at  $745 \text{ cm}^{-1}$  and  $1380 \text{ cm}^{-1}$  are corresponding to out-of-plane vibrations and in-plane vibration of B–N ring, respectively. The basic chemical structure of the products ( $\text{cm}^{-1}$ ): hexagonal B–N (1402, 615), B–N (1148) [15]. And the spectrum also shows the peaks of H–O ( $3440 \text{ cm}^{-1}$ ,  $1632 \text{ cm}^{-1}$ ) and C=O ( $2354 \text{ cm}^{-1}$ ) because of the absorption in the atmosphere [16], indicating that

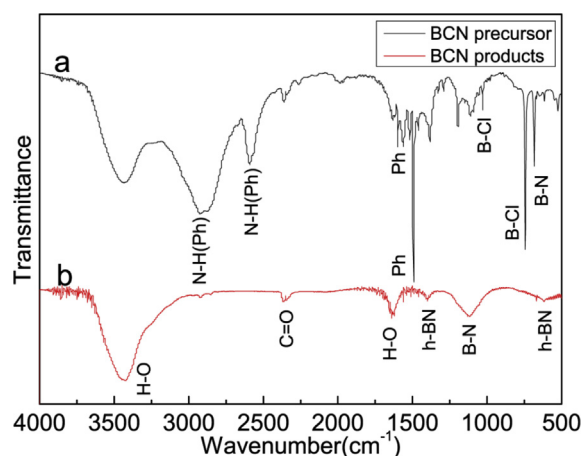


Fig. 2. The FT-IR spectra of (a) the organic precursor and (b) BCN porous architectures.

the products have excellent absorption owing to its high surface area and porosity.

Fig. 3 shows the X-ray diffractogram from the as-prepared products. It shows characteristic peaks centered at about  $25.6$  and  $43^\circ$  ( $2\theta$ ) (with a full width at half maximum (FWHM) of  $3.6^\circ$  and  $4.5^\circ$ ) belonging to the (002) and (100) interlayer reflections of graphite (ICDD card 751621), respectively. The broadening of the peaks indicates the formation of nanosized domains and low correlation lengths [11]. The Scherrer formula applied to the (001) reflection yields a crystallite size about  $10.7 \text{ nm}$ . The peak at  $2\theta = 13.2^\circ$  slightly shifting to lower angles from  $14^\circ$  with FWHM of  $1.1^\circ$  could be ascribed to the diffractive plane of (001) of hexagonal BN phase (ICDD card 741978). It is nothing worth that the shift from  $14^\circ$  to  $13.2^\circ$  may reveal the larger interplanar distance compared with h-BN and graphite due to the ternary atomic mixing in the  $\text{sp}^2$

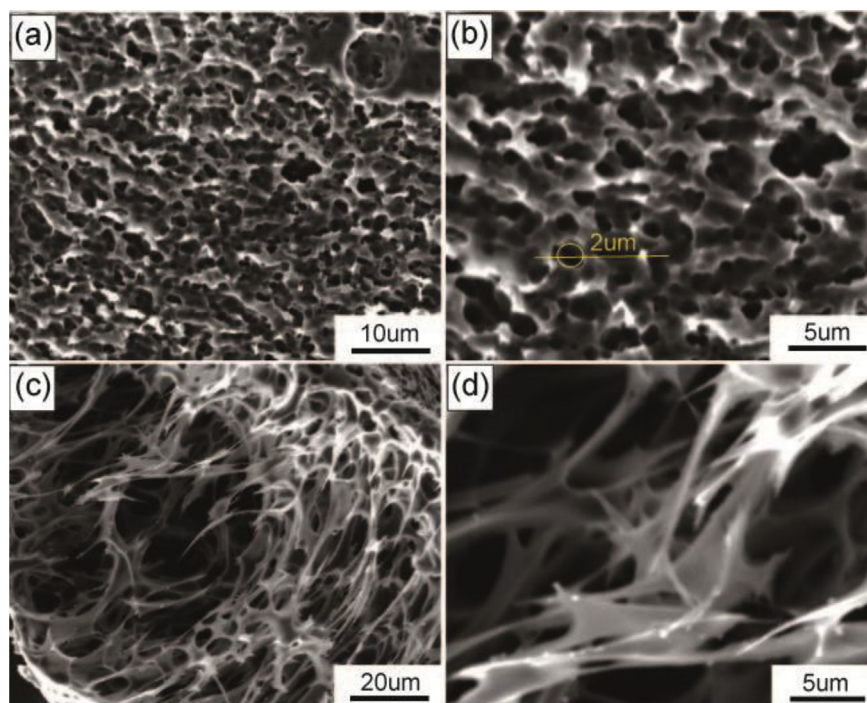


Fig. 1. The SEM images of the as-prepared BCN porous architectures at different magnification of (a, c) low magnification and (b, d) high magnification.

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