



Chemically synthesized boron carbon oxynitride as a new cold cathode material



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ABSTRACT

Synthesis of boron carbon oxynitride (BCNO) nanosheets at different temperature from amorphous to crystalline regime has been reported. The synthesis was done by a simple molten salt process using sodium borohydride and urea as precursors.

Transmission electron microscopic study confirms the formation of sheet-like structure of the as-synthesized material. The performances of the as-synthesized BCNO nanosheets as cold cathode materials have been studied for the first time in the high vacuum electron field emission set up. It has been seen that the material gives considerable field emission current with turn on field as low as 2.95 V/ μm with good stability and thus a new cold cathode material can be postulated.

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1. Introduction

The beginning of 21st century will be long remembered both by the scientists as well as the technologists in the field of nanoscience and technology due to the discovery of a new two-dimensional carbon material called graphene by Novoselov and Geim [1]. As the newly discovered graphene readily proved its outstanding potential in many fields of applications like ultra-tough paper, super capacitor, gas adsorbent, cold cathode emitter, field effect transistor, etc. [2–5] the research on two-dimensional nanomaterials suddenly found a new boom thereafter. In this context the exfoliated layered boron nitride (often called white graphene) and boron carbon nitride (BCN) are two flagging materials that have higher oxidation resistance compared to graphene, very good electrical and mechanical properties and above all a finite band gap that can be tuned to get emission within a wide energy range [6–8]. It has been seen that when carbon is incorporated into the BN matrix the band gap, i.e. the optical properties can be tuned by varying the carbon content [9]. So far there are mainly three established methods for synthesizing oxygen contained BCN (BCNO) or boron carbon

oxynitride. They are thermal or microwave chemical vapour deposition (CVD) [10,11], solid state reaction [12] as well as chemical route [13] of which the last one is the most suitable due to its simplicity as well as the possibilities of a finer tuning of the product properties. There are reports for the synthesis of BCNO by salt melt process, e.g. by Lei et al. [9], or by chemical process as done by Singh et al. [13], Zhang et al. [14], or Fellinger et al. [15]. This material found its applications in different fields that include hydrogen storage, electronic devices, electrocatalysis, super capacitors [16–18] but mainly as tuneable nanophosphor [9,14,19]. Also its electrochemical behaviour has been studied by Lei and his co-workers [20]. However, so far the authors are concerned the study that can prove the possibilities of the material for being used as a potential material for cold cathode application has been largely ignored though there are reports of electron field emission properties of negative electron affine boron nitride [21,22].

Keeping the above-mentioned literature study in mind here for the first time, we have reported the FE characteristics of the BCNO nanosheet-like structure synthesized by a very simple molten salt process in both the amorphous and crystalline regimes. It has been shown that the field emission characteristics of the as-synthesized BCNO nanosheets are well-comparable to other established field emitters like carbon nanotubes (CNTs), graphene, zinc oxide (ZnO) having turn-on field as low as 2.95 V/ μm . The field emission is believed to be the wrinkle-induced field enhancement as reported in case of graphene.

This systematic study can add another new member in the family of the cold cathode emitter thus helping the display technologists and scientists in numbers of ways.

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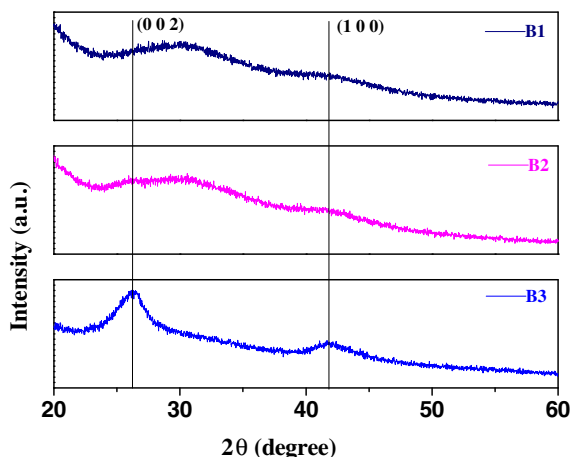


Fig. 1. XRD spectra of all the samples.

2. Experimental and characterization

The experimental was rather simple and similar to reported by Lei et al. [9]. It simply consists of preparation of a eutectic mixture by taking LiCl/KCl in (45/55) weight ratio; all taken with analytical grade. Sodium borohydride and urea was used as the precursor for boron and nitrogen, respectively.

In a typical experiment, LiCl (1.35 g) + KCl (1.65 g) are mixed in a mortar. Then the mixture was heated in an open air tube furnace at 360 °C for 2 h. The solid was collected and mixed with sodium borohydride (2.5 g) and urea (2.5 g). The mixture was heated in N₂ atmosphere at 750 °C for 1 h (B1), at 850 °C for 1 h (B2) and at 850 °C for 2 h (B3) under nitrogen flow which when cooled gives a greyish powder. The final products were obtained only after washing the powder with de-ionized water, filtering and drying the filtrate overnight at 80 °C.

The as-synthesized samples were characterized by X-ray diffraction (XRD Bruker, D8 Advance), using a diffractometer with Cu K α radiation ($\lambda = 0.15406$ nm) operating at 40 kV, 40 mA with a normal θ – 2θ scanning. Field emission scanning electron microscope (FESEM, Hitachi, S-4800) and high resolution transmission electron microscopy (HRTEM, JEOL-JEM 2100; operated at 200 kV) were used to study the morphology of the as-synthesized sample as well as to have ideas about the crystallinity of the sample. Energy dispersive X-ray analysis (EDX, Oxford, model-7582 operated at 15 kV) and X-ray photoelectron spectroscopy (XPS, Specs-Germany using Mg K α radiation) analysis were done to do the compositional analysis. For better understanding of different bonds, Fourier transformed infrared (Shimadzu FTIR-8400S) spectroscopic analysis were done.

The field emission study has been carried out in our home-made high vacuum field emission set up. Field emission measurements were carried out by using a diode configuration consisting of a

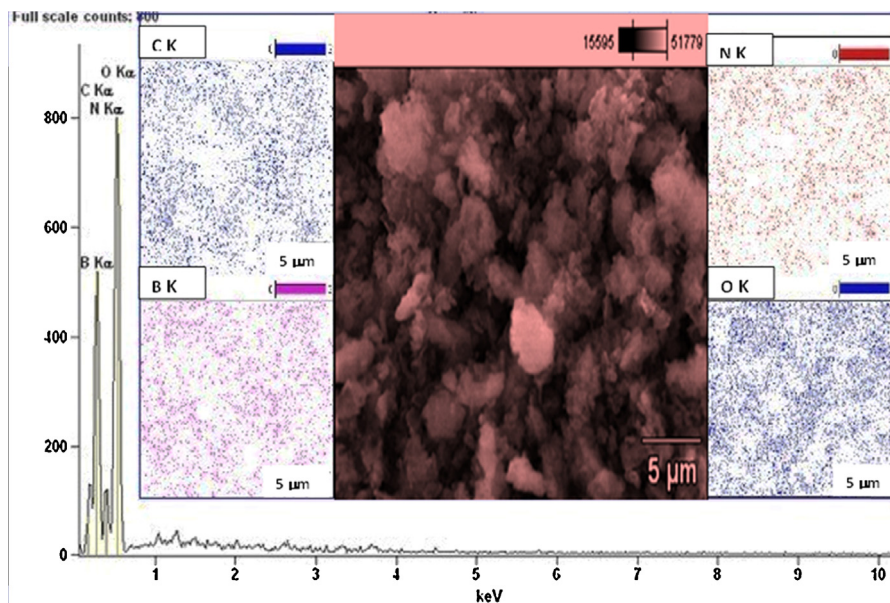


Fig. 2. EDX spectrum of sample B1.

Table 1

Percentage of all the elements present in the samples as obtained from EDX analysis.

Sample	Atomic percentage		Weight percentage		N/B (at.)	C/B (at.)	O/B (at.)
B-3	B	16.94	B	20.81	1.51	1.53	1.85
	C	26.07	C	28.84			
	N	25.66	N	24.34			
	O	31.33	O	26.01			
B-2	B	19.99	B	19.99	0.8	1.58	1.61
	C	31.64	C	31.64			
	N	16.07	N	16.07			
	O	32.3	O	32.3			
B-1	B	30.3	B	28.6	0.38	0.48	1.43
	C	14.5	C	15.73			
	N	11.71	N	11.2			
	O	43.49	O	44.47			

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