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Growth of GaN by nitridation of seed/catalyst free electrodeposited Ga-based compound materials on graphene on insulator

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ABSTRACT

The nitridation of the electrochemically deposited Ga-based compound material on graphene on insulator towards the formation of GaN/graphene hybrid structure was studied by varying the nitridation time and temperature. First, the growth of Ga-based compounds which contains GaON and Ga_2O_3 on multi-layer graphene on SiO_2/Si using a mixture of NH_4NO_3 and $Ga(NO_3)_3$ by a simple two terminal electrochemical deposition at room temperature was performed. Then, the conversion of the grown structures to the crystalline GaN structure was carried out by nitridating the grown structures in NH_3 gas. The properties of the grown structures were critically influenced by the studied nitridation parameters. The complete transformation to hexagonal GaN was achieved at nitridation temperature of 1100 °C and time of above 60 min due to the observation of significant diffraction peaks which correspond to hexagonal GaN planes. Meanwhile no diffraction peaks of GaON and Ga_2O_3 structure were observed. Temperature and time are the key parameters in a nitridation process where the ammoniation rate of GaON and deoxidization rate of Ga_2O_3 to generate gaseous Ga_2O, increase with temperature and time. It was speculated that a complete transformation can not be realized without a complete ammoniation of GaON and deoxidization of Ga_2O_3. The change of morphological structures was also observed due to both reactions. The presented method demonstrates the feasibility to realize GaN/ graphene hybrid structure on insulator which is highly demanded in fabricating optoelectronic and sensing devices.

1. Background

Over these few decades, gallium nitride (GaN) has been proven as the promising candidate for various applications such as in electronic [1–8], optoelectronic [9–16] and sensing devices [17–24]. It is due to its superb properties such as wide direct energy bandgap (3.4 eV), high thermochemical stability, high carrier mobility, high breakdown electric field and high optical transparency. However, most of these GaN-based devices is fabricated on sapphire substrate which is not only very expensive but also possesses poor thermal conductivity and dielectric property. Graphene is considered as the promising material to overcome such limitations in sapphire substrate due to its well-known excellent electrical [25], thermal [26–29], optical [25], mechanical [30] and carrier transport [31–33] properties. In addition, graphene is also capable to realize the so-called flexible [34,35] and transferable [34,36] devices with excellent performance.

There are several reports on the growth of GaN films or nanostructures on seeded graphene or graphite [34,35,37–39]. However, the epitaxial growth of GaN directly on non-seeded graphene is very challenging and thus, it is not well reported in the literature. This may due to less chemical reaction or low nucleation rate of GaN on the inert sp^2-hybridized graphene [40,41]. Recently, we reported the electrochemically deposited gallium oxide (Ga_2O_3) on silicon (Si) [42], and its nitridation [43]. However, the direct growth on graphene have resulted to the formation of not only Ga_2O_3 structure but also gallium oxynitride (GaON) structure [44]. In this paper, we report the growth of GaN nanostructures by the nitridation of the electrochemically deposited Ga-based compounds on non-seeded graphene for the first time. It was found that the formed GaN structures were hexagonal (h-) polytype and their properties were strongly controlled by the nitridation time and temperature.

2. Experimental methods

The cathodic two-terminal electrochemical deposition (ECD) under galvanostatic control was performed at room temperature (RT) on commercial CVD grown multilayer graphene (MLG) on SiO_2/Si substrate (Graphene Laboratories Inc, Calverton, NY, USA) [44]. A mixture

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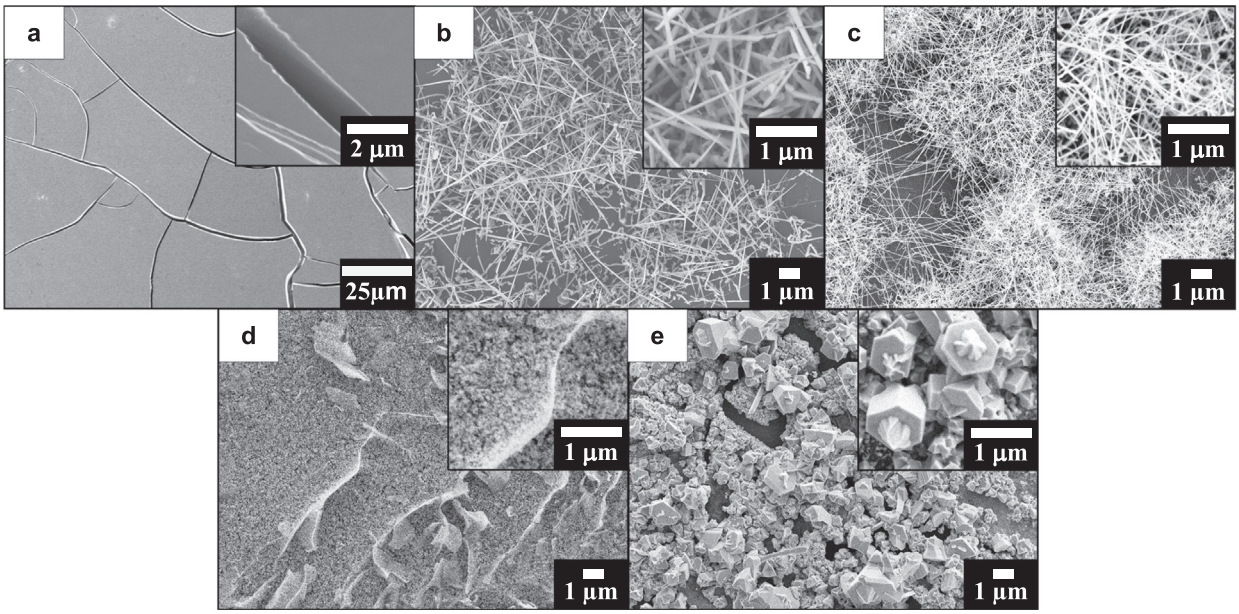

Fig. 1. Top view FESEM images of the **(a)** electrochemically deposited Ga-based structure, and nitridated structures for 20 min at temperature **(b)** 800, **(c)** 850, **(d)** 1000 and **(e)** 1100 °C.

of ammonium nitrate (NH_4NO_3) (Sigma Aldrich, ≥98% purity) and gallium nitrate ($Ga(NO_3)_3$) (Sigma Aldrich, ≥99.9% purity) dissolved in deionized (DI) water was used [44]. A complete ECD setup and details of sample treatment can be found in Ref. [44]. The electrodeposition was done at the current density of 3.5 mA/cm^2 for 6 h. The molarities of NH_4NO_3 and $Ga(NO_3)_3$ solutions were fixed at 7.5 M and 0.8 M, respectively.

The nitridation of the ECD sample was done in a single-zone quartz tube furnace at different temperatures ranging from 800 to 1100 °C for 20–100 min at atmospheric pressure. A flow of ammonia (NH_3) gas was fixed at 200 sccm. The quartz tube was purged with nitrogen (N_2) gas to flush out the air after placing the sample in the middle of tube. Then, the temperature was ramped up from RT to the nitridation temperatures, i.e. 800 °C, 850 °C, 1000 °C and 1100 °C in N_2 ambient. N_2 was stopped and NH_3 was introduced into the furnace after reaching the required temperature. Finally, the furnace was switched off and a flow of NH_3 was immediately stopped upon reaching the required nitridation time. The sample was cooled down in N_2 ambient. The details of nitridation setup and procedure can also be found in Ref. [43].

The surface morphology, elemental composition, crystallinity and elemental bonding properties of the nitridated structures were characterized using field-emission scanning electron microscopy (FESEM, JEOL), energy dispersive X-ray spectroscopy (EDX), X-ray diffractometer (XRD, PAN analytical Empyrean) and Fourier transform infrared spectroscopy (FTIR, Perkin Elmer), respectively.

3. Results and discussion

The chemical reactions that took place in the ECD process were reported in our previous work [44]. Since H_2O was mixed together with $Ga(NO_3)_3$ and NH_4NO_3, it created the existence of O element which contributed to the formation of GaON and Ga_2O_3. The complete chemical reactions were summarized as the following [44,45]:

$$NH_4NO_3 \rightarrow NH^{4+} + NO^{3-} \quad (1)$$

$$NH^{4+} + NO^{3-} \rightarrow NH_3 + HNO_3 \quad (2)$$

$$Ga(NO_3)_3{\cdot}H_2O \rightarrow Ga^{3+} + 3NO^{3-} + H_2O \quad (3)$$

$$Ga(NO_3)_3 + NH_3 \rightarrow GaON + HNO_3 + H_2 + 2NO_2 + O_2 \quad (4)$$

$$Ga^{3+} + 2H_2O \rightarrow GaOOH + 3H^+ \quad (5)$$

$$2GaOOH \rightarrow Ga_2O_3 + H_2O \quad (6)$$

The NH^{4+} ions generated from the ionization of NH_4NO_3 donates their most acidic proton to produce NH_3 and HNO_3. Meanwhile, the hydrolysis of water molecules and generated Ga^{3+} ions from the ionization of $Ga(NO_3)_3{\cdot}H_2O$ produced GaOOH and hence, the intermediate reaction results to the formation of Ga_2O_3. GaON was formed due to the reaction of NH_3 and excessive $Ga(NO_3)_3$. These continuous reactions lead to the formation of thin film which contains both Ga-based structures.

In this study, NH_3 was used as N source for the nitridation of the grown Ga-based structure, i.e. GaON and Ga_2O_3. The chemical reactions involved during the nitridation of Ga-based structures were summarized as the following:

$$2GaON + NH_3 \rightarrow 2GaN + HNO_2 + H_2 \quad (7)$$

$$2NH_3 \rightarrow N_2 + 3H_2 \quad (8)$$

$$Ga_2O_3 + 2H_2 \rightarrow Ga_2O + 2H_2O \quad (9)$$

$$Ga_2O + 2NH_3 \rightarrow 2GaN + 2H_2 + H_2O \quad (10)$$

A complete transformation to GaN structure was achieved through the ammoniation of GaON with NH_3 gas and deoxidization of Ga_2O_3 as illustrated by Eq. (7) and Eq. (9), respectively. NH_3 was also decomposed to H_2 at high temperature which promoted the deoxidization of Ga_2O_3 structure to form gaseous Ga_2O which then reacted with NH_3 to form GaN structure as illustrated by Eq. (10).

First, we discuss the effect of nitridation temperature on the as-deposited ECD structures. Here, the nitridation time was fixed at 20 min. The top-view FESEM image of the as-deposited ECD structure is shown in Fig. 1**a**. The observed cracking and grain-like structure were proposed to be resulted from the stacking nature of polycrystalline MLG used in this study [44]. Fig. 1**b-e** show the grown GaN nanostructures after the nitridation at temperatures of 800, 850, 1000 and 1100 °C, respectively. As shown in Fig. 1**b** and **c**, the grown structures show nanowire-like structures at temperatures of 800 and 850 °C. Here, it can be understood that the densities increase [46] but their diameters decrease with the increase of temperatures. The dissociative absorption of NH_3 on Ga-based compound surface will reduce the surface energy

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