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Liquid metal fabrication of ultrathin Ga₂O₃ and GaN layers for integrated optics

Panteha Pedram^{*a}, Ali Zavabeti^b, Nitu Syed^b, Amine Slassi^c, Chung Kim Nguyen^b, Benjamin Fornacciarì^a, Anne Lamirand^a, Jules Galipaud^d, Arrigo Calzolari^c, Andreas Boes^{b,e,f}, Torben Daeneke^b, Sébastien Cuffe^a, Arnan Mitchell^b, Christelle Monat^a

^aInstitut des Nanotechnologies de Lyon, UMR CNRS, Ecole Centrale de Lyon, Université de Lyon, 36 Av. Guy de Collongue, Écully, France; ^bRMIT University, 124 La Trobe St, Melbourne VIC 3000, Australia; ^cCNR-NANO Istituto Nanoscienze, Modena I-41125, Italy; ^dLaboratory of Tribology and System Dynamics, Ecole Centrale de Lyon, Université de Lyon, 36 Av. Guy de Collongue, Écully, France; ^eInstitute for Photonics and Advanced Sensing, the University of Adelaide, Adelaide, SA 5005, Australia; ^fSchool of Electrical and Mechanical Engineering, the University of Adelaide, Adelaide, SA 5005, Australia

*panteha.pedram@ec-lyon.fr

ABSTRACT

We report on the synthesis of 2D GaN materials by the so-called liquid metal chemistry and tuning of their composition between oxide and nitride materials. This technique promises easier integration of 2D materials onto photonic devices compared to traditional “top-down” and “bottom-up” methods. Our fabrication method is carried out via a two-step liquid metal-based printing method followed by a microwave plasma-enhanced nitridation reaction. The synthesis of GaN relies on plasma-treated liquid metal-derived two-dimensional (2D) sheets that were squeeze-transferred onto desired substrates. We characterized the composition and optical properties of the resulting nm-thick GaN films using AFM, XPS, and ellipsometry measurements. Finally, the optical indices measured by ellipsometry are compared with theoretical results obtained by density functional theory (DFT). Our results represent a first step toward integrating 2D materials and semiconductors into electronics and optical devices.

Keywords: 2D materials, liquid metal, Gallium oxide (Ga₂O₃), Gallium nitride (GaN), refractive index

1. INTRODUCTION

The development of silicon photonics in the last two decades has led to the realization of a wide range of compact optical devices that can manipulate or transfer light across a chip. However, when it comes to light emission or nonlinear optical properties, silicon is intrinsically limited, calling for the use of other materials with complementary properties. Since the isolation of graphene in 2004, several kinds of monolayer materials have emerged, offering new properties that can advantageously complement silicon photonics. In this context, various 2D materials have been integrated into a scalable and silicon-compatible platform, as they could provide a pathway toward realizing optoelectronic devices with enhanced functionalities [1].

In particular, gallium oxide (Ga₂O₃) and gallium nitride (GaN) stand out due to their interesting optoelectronic material properties. Indeed, wide-band-gap 2D Ga₂O₃ films of liquid gallium alloys have found unique applications in electronics [2], photonics [3], sensing [4], resistive switching [5], and bio-inspired artificial technologies [6]. Many different liquid phase methods have been shown for synthesizing Ga₂O₃ based on hydrothermal, microwave, and other solvothermal approaches [7-15]. However, such synthesis techniques generally require an extended processing time of up to several days. On the other hand, GaN has attracted attention because of the advent of ultraviolet, blue, and green light-emitting diodes [16], lasers [17-18], photodetectors [19], and solar cells [20]. These properties are due to its wide bulk bandgap (3.4 eV), which facilitates the efficient emission of blue and near UV light [21]. In addition, the high carrier mobility and chemical robustness, together with the piezoelectric and pyroelectric properties of GaN, have enabled a number of other critical applications, such as the design of optoelectronic quantum wells, a variety of field effect transistors, energy

harvesting systems, sensors, and photocatalysts. [22-26]. However, traditional fabrication techniques, such as mechanical exfoliation, cannot be applied to GaN due to its wurtzite structure which features covalent bonds in all three dimensions [27]. On the other hand, epitaxial methods are efficient but costly; whereas lower cost CVD methods do not allow thin films of several unit-cell thicknesses to be achieved due to inherent nucleation.

It has recently been shown that 2D materials can be fabricated via the method of ‘liquid metal chemistry’ (LMC) [28], which promises the integration of 2D materials onto photonic devices with more ease than traditional methods. This method is a two-step process consisting of a liquid metal-based printing method followed by a microwave plasma-enhanced nitridation reaction [29]. The term liquid metals covers metals and metal alloys present in liquid form below approximately 350°C, which includes several post-transition elements, group 12 elements, and their alloys [30]. Resorting to gallium enables the synthesis of ultrathin Ga₂O₃ nanosheets through the self-limiting oxide layer which forms on the surface of liquid gallium at room temperature [31] in a few seconds. Subsequently, ultrathin GaN can be synthesized using a plasma-assisted nitridation reaction on the deposited Ga₂O₃ nanosheets.

Here we report fast and low-cost synthesis of 2D GaN nanosheets that can be deposited directly onto flat substrates and silicon photonic devices such as optical waveguides. This technology paves the way towards the realization of hybrid photonic circuits that locally exploit 2D materials so as to adjust or enhance the optical properties of mature silicon photonic chips.

2. FABRICATION

The synthesis based on LMC relies on a two-step process utilizing the squeeze-printing of 2D Ga₂O₃, which is then converted into GaN using a plasma-assisted nitridation. In this work, as shown in Figure 1, a tiny droplet (~1 mm in radius) of liquid gallium (m.p. 29.8 °C) was placed on a SiO₂-terminated, atomically flat silicon wafer. A second Si wafer was firmly pressed onto the droplet, effectively squeezing the droplet. After the squeezing process, the top substrate was lifted and separated vertically. Upon removal of the top substrate, a large and continuous film of ultrathin gallium oxide was revealed. The Ga₂O₃ sheet remained attached to the SiO₂ surface due to strong Van der Waals forces.

Subsequently, oxide sheets are converted into nitrides using a microwave-activated nitrogen/hydrogen plasma reaction. The synthesis was carried out in a microwave plasma-enhanced chemical vapor deposition reactor at a substrate temperature of ~320 °C using a plasma consisting of N₂ (99.5%) and H₂ (0.5%). The pressure in the plasma chamber was 1 Torr. The plasma was generated with a RF power of 150 W, the concentration of the H₂/N₂ gas mixture was controlled by setting flow rate of the gases to 200 sccm/1 sccm, respectively, measured by a mass flow controller (MFC). During the transformation to GaN, the samples were exposed to the plasma for 10 minutes.

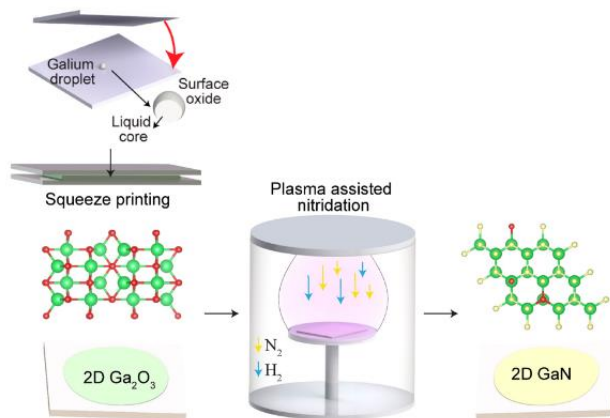


Figure 1. Synthesis process of large-area ultrathin GaN nanosheet. Schematic illustration of the squeeze-printing technique for depositing oxide skins onto substrates. The liquid metal droplet is squeezed between two Si substrates allowing the transfer of the layer. Then the synthesized 2D Ga₂O₃ is converted to 2D GaN utilizing a microwave-activated nitrogen/hydrogen plasma in a plasma reactor.

3. CHARACTERIZATION

3.1 X-ray photoelectron spectroscopy (XPS)

To ascertain that a quantitative conversion of Ga_2O_3 to GaN occurred, X-ray photoelectron spectroscopy (XPS) was utilized and relevant spectra were compared before (Fig. 2d) and after the nitridation process (Fig 2a-c). XPS analysis was conducted on samples grown on SiO_2/Si substrates using a Thermo Scientific K-alpha XPS spectrometer equipped with a monochromatic Al K- α source ($h\nu = \sim 1486.7$ eV). The analyzer was operated with a pass energy of 50 eV to record the core-level spectra with 300 μm spot size and 50 scans number for each sample. All the samples are adjusted with the C-C bond of carbon at 284.68 eV. Figure 2a shows the XPS spectra of a doublet in the Ga2p region centered at ~ 1118.19 eV and ~ 1144.8 eV corresponding to 2p $_{3/2}$ and 2p $_{1/2}$, respectively, in good agreement with expected values for GaN [32, 33]. The observed N1s peak at binding energy ~ 398.1 eV also corresponds to the N1s peak of GaN [33] (Figure 2b). To explore whether the degree of oxygen substitution in the developed 2D GaN samples was reduced compared to Ga_2O_3 , we compared the O1s for GaN and Ga_2O_3 . For GaN, in Figure 2c, the O1s peak centered at ~ 532.2 eV is in good agreement with the expected oxygen peak of the SiO_2 substrate [34]. In Figure 2d, we can see a double peak for O1s, the peak at ~ 530.9 eV can be assigned to the O-Ga-O bonds in gallium oxide, and the O1s peak at binding energy ~ 532.2 eV can be ascribed to the Si-O bonding from SiO_2 substrate. The absence of peak at ~ 530.93 eV for (c) confirms the effectiveness of the nitridation process for converting Ga_2O_3 into GaN.

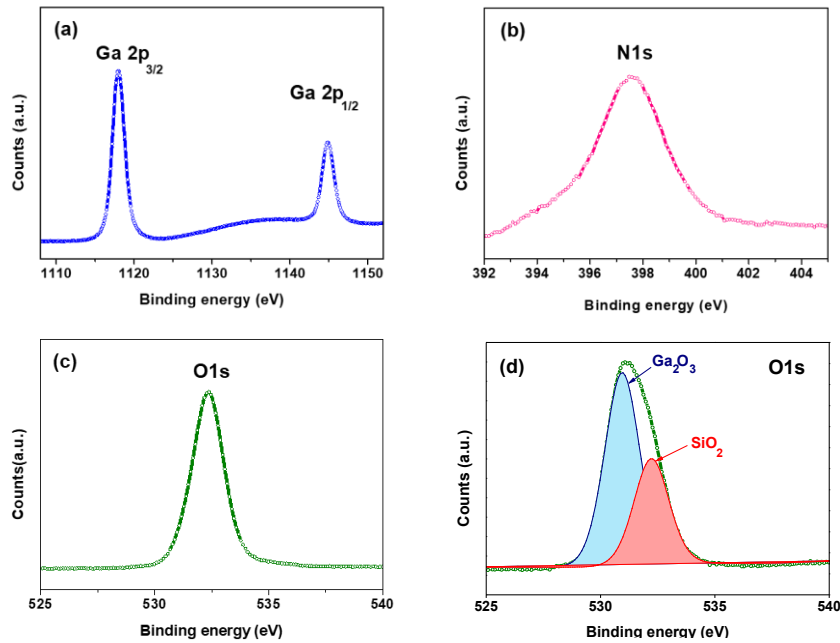


Figure 2. Material characterization of 2D GaN and Ga_2O_3 , after and before the nitridation process. (a, b and c) XPS results of the 2D GaN for the spectral regions of interest, (a) Ga 2p (b) N 1s (c) O1s (d) XPS result for O1s of the 2D Ga_2O_3 .

3.2 Atomic Force Microscopy (AFM)

In order to explore the consistency of the synthesized ultrathin GaN sheets, atomic force microscopy (AFM) was conducted on the deposited layer. Figure 3 shows the results from the measurement. The GaN was found to have formed a continuous and atomically flat nanosheet, approximately 3 ± 0.2 nm in thickness. In general, the ultrathin sheets featured minimal cracks and holes, and consistent thickness across large distances, on the order of ~ 100 μm (i.e., compatible with the typical size of integrated optical devices).

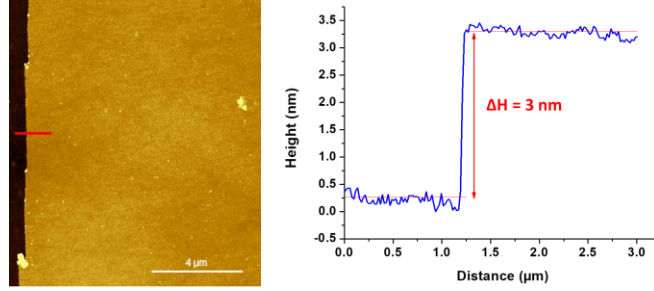


Figure 3. Morphology and structural characterization of 2D GaN. AFM topography image of a GaN sample taken at the edge of a nanosheet; the height profile was measured along the red line.

3.3 Spectroscopic Ellipsometry (SE)

Understanding Ga_2O_3 and GaN refractive index is essential to designing and analyzing gallium-based optoelectronic devices. Although the optical properties of Ga_2O_3 thin films have been investigated [35–38], there remains a lack of experimental measurement of the properties of ultrathin Ga_2O_3 sheets. Similarly, for GaN, available data focuses on either bulk or thin layers and is not suited for the study of ultrathin GaN sheets [39–43]. Therefore, further studies and more accurate measurements are needed to assess the properties of both materials in their 2D form.

Spectroscopic ellipsometry (SE) is a non-destructive characterization technique which can be performed on arbitrary materials in order to determine thin film layer thicknesses and/or optical constants. It has been used to study Ga_2O_3 [44–48] and GaN [49–53] bulk optical properties. In this report, the Ga_2O_3 and GaN ultrathin films were measured using a SE covering wavelengths from 260 to 2100 nm at 70° of angle incidence to determine the refractive index (n) and extinction coefficient (k) as a function of wavelength.

Figure 4a shows the refractive index and extinction coefficient of Ga_2O_3 deposited on Si substrates extracted by fitting the SE measurements with a Tauc Lorentz model for a 2.78 ± 0.13 nm thick film. We obtain a refractive index of around 1.89 at 632 nm and 1.865 at 980 nm wavelength, which is close to that given in Ref. [67] for Ga_2O_3 films ($n=1.91$ at 980 nm). We repeated our measurement after transformation of Ga_2O_3 into GaN. Figure 4b displays the refractive index and extinction coefficient of GaN after the nitridation process. The extracted refractive index is 2.318 at 632 nm and 2.14 at 980 nm, showing that the nitridation process was effective in turning the oxide films into GaN with distinct optical properties. This value is only slightly lower than the values found in the literature for bulk GaN crystals ($n \sim 2.34$ at 632 nm), metalorganic chemical vapor deposited (MOCVD) GaN layers ($n \sim 2.33$ at 632 nm) on sapphire from Ref. [69] and GaN epilayers ($n \sim 2.42$ at 632 nm) from Ref. [71]. To confirm the accuracy of our measurement, we calculated the theoretical optical indices of Ga_2O_3 and GaN using density-functional theory (DFT). The relatively good agreement between the experimental optical indices and DFT results is also an indication of the structural quality of the ultrathin layer and the effectiveness of the nitridation process. We note that the relatively high index difference (~ 0.42) between the Ga_2O_3 and GaN thin films is relevant for adjusting the optical properties of silicon photonic devices on top of which this 2D material could be deposited.

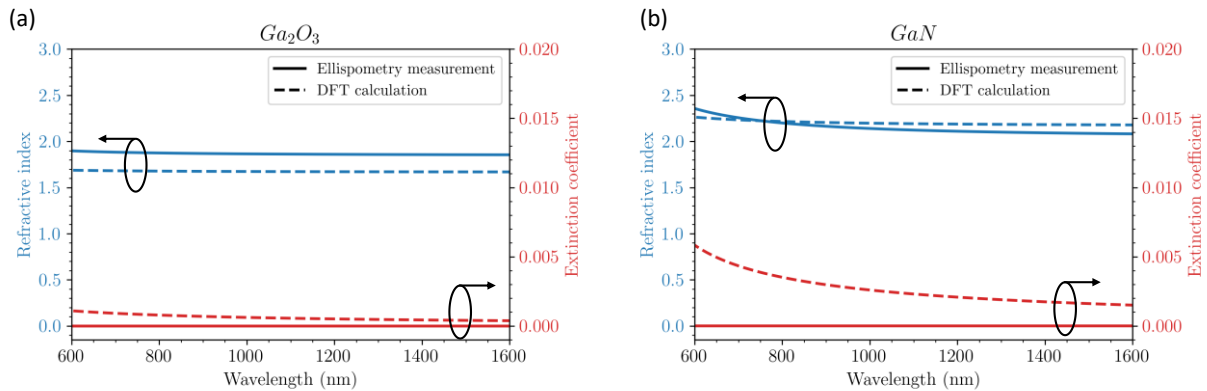


Figure 4: Optical properties of Ga_2O_3 and GaN ultrathin films, extracted from spectroscopic ellipsometry and DFT in the range 600–1600 nm. (a) Shows the refractive index n and extinction coefficient k of Ga_2O_3 as a function of wavelength, (b) shows the refractive index n and extinction coefficient k of GaN as a function of wavelength.

4. CONCLUSION

In conclusion, we reported the realization of ultrathin Ga₂O₃ and GaN films by liquid metal-based van der Waals squeeze printing technique followed by a plasma assisted nitridation process. A liquid metal-based reaction route that relies on the exfoliation of naturally occurring interfacial oxide of molten gallium, followed by the subsequent microwave plasma enhanced nitridation reaction, is reported to obtain ultrathin 2D GaN sheets. The use of a reactive N₂/H₂ plasma was found to induce the replacement of oxygen atoms with nitrogen. The proposed synthesis method is scalable and fast, providing access to large-area 2D GaN nanosheets. We characterized the composition and optical properties of the resulting nm-thick Ga₂O₃ and GaN films using XPS, AFM, and Ellipsometry measurements. DFT calculations of Ga₂O₃ and GaN were conducted to compare the optical indices obtained by Ellipsometry, and give further evidence of the nature of the resulting thin films. This work provides a pathway towards integrating 2D materials onto hybrid photonic chips so as to locally adjust or functionalize an otherwise passive optical circuit.

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