

# Direct Chemical Vapor Deposition of Large-Area Carbon Thin Films on Gallium Nitride for Transparent Electrodes: A First Attempt

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**Abstract**—Direct formation of large-area carbon thin films on gallium nitride by chemical vapor deposition without metallic catalysts is demonstrated. A high flow of ammonia is used to stabilize the surface of the GaN (0001)/sapphire substrate during the deposition at 950°C. Various characterization methods verify that the synthesized thin films are largely  $sp^2$  bonded, macroscopically uniform, and electrically conducting. The carbon thin films possess optical transparencies comparable to that of exfoliated graphene. This paper offers a viable route toward the use of carbon-based materials for future transparent electrodes in III-nitride optoelectronics, such as GaN-based light emitting diodes and laser diodes.

**Index Terms**—Chemical vapor deposition, GaN, graphene, transparent electrodes.

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## I. INTRODUCTION

GALLIUM nitride is a direct and wide bandgap (3.4 eV) semiconductor used in optoelectronic devices that emit in the green, blue, and ultraviolet ranges. Typical applications include high-brightness light-emitting diodes (LEDs) [1] and laser diodes [2]. Reproducible large-scale production of transparent conducting thin films on GaN is crucial to improve the current injection and light extraction efficiency in such devices. Extremely thin metal films can be somewhat transparent, but it is difficult to reduce the thickness to atomic level while maintaining continuity. Indium tin oxide (ITO), a doped n-type semiconductor, is the current industrial standard due to its high transparency and conductivity [3], [4]. Nevertheless, ITO is relatively expensive and is unstable in acidic and basic environments [4].

Graphene, one or few layers of C atoms in graphite, have been widely explored as viable ITO substitutes [5], [6]. Graphene is cost effective and highly stable while also possessing outstanding optical, electrical, and mechanical properties. Samsung has demonstrated production of 30-in stacked graphene layers by chemical vapor deposition (CVD) with properties superior to ITO [5]. Nevertheless, there have been few attempts to employ graphene as transparent electrodes in GaN-based optoelectronics, and those that have been considered are either wet-transferred CVD graphene [7]–[9] or chemically converted graphene suspensions [10]. The transfer process bound to metal-catalyzed graphene [5], [7]–[9], [11] is complicated and often results in a nonideal interface between the graphene and GaN due to residues, oxides, and wrinkles. The chemically converted graphene also involves solution-based processes [10]. Evidently, there is a substantial need to develop a reproducible and semiconductor-industry compatible technique for the synthesis of graphene directly on GaN.

Recently, we have pioneered the so-called noncatalytic CVD of graphene. For the first time, large-area continuous graphene thin films with controlled thickness are grown by CVD without any metal catalyst on  $\text{Si}_3\text{N}_4$  and  $\text{HfO}_2$  [12], [13]. We have predicted that the mechanism and technique can be generalized for graphene production over any nonmetallic substrates that can withstand  $\sim 1000^\circ\text{C}$  (e.g.,  $\text{SiO}_2$ ) [13]–[15]. In this paper, we demonstrate CVD-grown large-area carbon

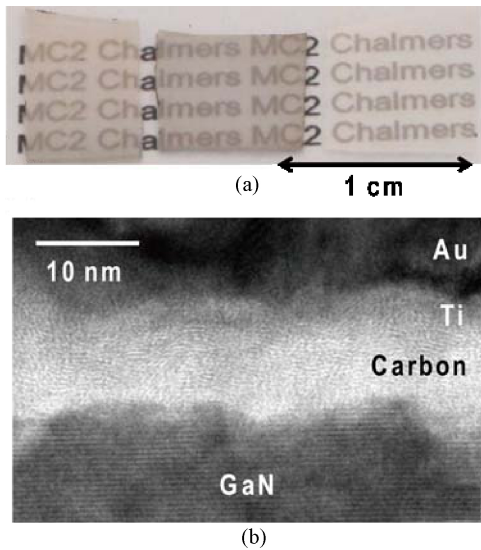


Fig. 1. (a) Carbon thin films on GaN/sapphire (left two) and the bare substrate (right). (b) High-resolution cross-section TEM micrograph of a carbon/GaN sample, where the carbon thin film is intentionally grown thicker for easy detection of the graphitic layers. The Au/Ti is deposited as part of the TEM specimen preparation procedure.

thin films directly on GaN. The transfer-free carbon thin films are macroscopically uniform, transparent, and conducting, as confirmed by optical and transmission electron microscopies (TEM), Raman, transmission, and Fourier transform infrared (FTIR) spectroscopies, as well as electron transport measurements. For this first attempt to deposit continuous carbon thin films on GaN, however, the material quality is still inferior to that of standard graphene, mainly due to difficulties in keeping the GaN intact at high temperatures, as well as the noncatalytic nature of the CVD. Nevertheless, the results indicate that there is much room for continued improvement following our proof-of-principle experiments, hinting at a promising future of the as-developed technology in GaN-based optoelectronics.

## II. MATERIAL GROWTH

The substrates used herein are unintentionally doped GaN (0001) Lumilog templates (3.5- $\mu\text{m}$ -thick) grown on sapphire by metalorganic chemical vapor deposition. After standard organic rinsing, the GaN/sapphire template is loaded into a Black Magic CVD System (AIXTRON, Ltd., Swavesey, Cambridge, U.K.). Typically, the carbon thin films are grown at 950 °C at 750 mbar for 5 min in a flow of 160 sccm  $\text{C}_2\text{H}_2$  and 1000 sccm  $\text{NH}_3$ . It is known that GaN can dissociate at high temperatures. Thus, both the deposition temperature and time have been reduced compared to our earlier work [13]–[15]. We also opt to use  $\text{C}_2\text{H}_2$  as a precursor due to its propensity to thermally decompose at lower temperatures compared to  $\text{CH}_4$ . Finally and most importantly, an overpressure of  $\text{NH}_3$  is used in order to protect the GaN surface from dissociating at the high deposition temperature [16], [17]. It can compensate the loss of N from GaN during growth, and also release  $\text{H}_2$  that is generally needed for carbon CVD. We stress that  $\text{N}_2$  atmosphere cannot be used for this purpose. Its ultrastrong  $\text{N} \equiv \text{N}$  triple bond (946 kJ/mol) renders that  $\text{N}_2$  is rather inert. Fig. 1(a), from left to right, shows the GaN/sapphire template with

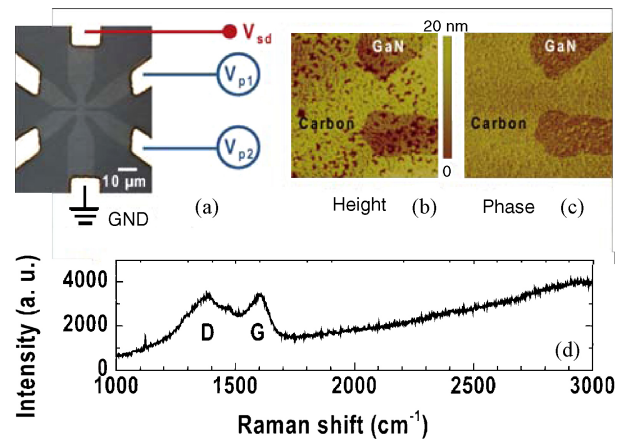


Fig. 2. (a) Optical image of a device fabricated on the middle sample of Fig. 1(a) together with the four-probe measurement circuit. The active area is 4  $\mu\text{m} \times 4 \mu\text{m}$  large. (b), (c) 8  $\mu\text{m} \times 8 \mu\text{m}$  AFM height and phase images of the central region of (a). (d) Raman spectra (514 nm, 0.5 mW) of the as-grown carbon thin film on GaN.

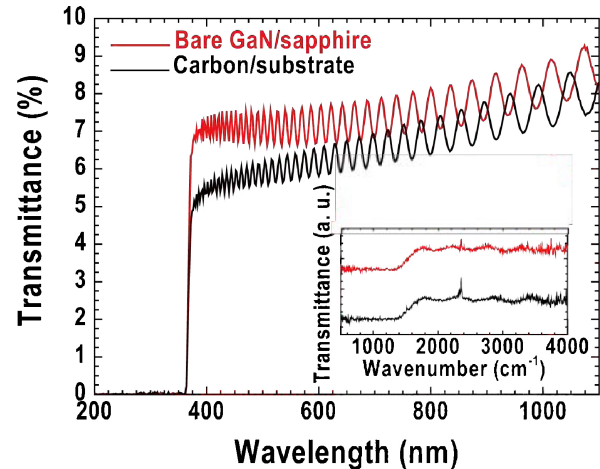


Fig. 3. UV-VIS-NIR transmittance of the carbon thin film and the bare GaN/sapphire. The carbon thin film absorbs  $\sim 2\%$  of the incident light. Inset: FTIR spectra of the same samples, which have been shifted along the ordinate for clarity.

deposited carbon thin films using 158 and 160 sccm  $\text{C}_2\text{H}_2$ , and the bare substrate, respectively. The carbon thin films on the undersides of the samples have been removed by  $\text{O}_2$  plasma treatment (with the top surface protected by the photoresist). The thin films are macroscopically smooth and uniform as evidenced by optical microscope observation [examples of optical microscopy and atomic force microscopy (AFM) images of the middle sample in Fig. 1(a) can be found in Fig. 2(a) and (b), where the carbon thin film is already patterned], indicating that the growth strategy effectively preserves the GaN surface morphology. In contrast, GaN layers heated to 950 °C with no  $\text{NH}_3$  protection exhibit a severely damaged morphology (not shown). Notwithstanding, AFM measurement reveals tolerable nm-scale surface roughening [Fig. 2(b)].

## III. RESULTS AND DISCUSSION

To date, graphitization over nonmetallic surfaces is poorly understood. We propose a noncatalytic mechanism based on hydrocarbon pyrolysis and subsequent self-assembly of

graphene flakes [13]–[15]. At  $\sim 1000$  °C, the carbon precursor decomposes and the carbon atoms form nanoscale graphene flakes, similar to the widely used industrial process to produce large quantities of carbon black [18]–[20]. However, if a very hot and flat substrate is present, the graphene flakes can self-organize into textured thin films rather than porous lumps [13]–[15]. As the substrate plays no or very weak catalytic role, even if it is covered by carbon, the deposition still continues. Therefore, the thickness of carbon layers can be easily controlled by tuning the growth time and/or precursor concentration. The coatings maintain their metallic luster resembling oriented graphite even for tens of nm [13]–[15]. Indeed, in Fig. 1(a), the carbon thin films on the left and middle samples are about 2–3 nm and 4 nm, respectively (determined by AFM and TEM), rendering the middle sample more opaque than the left one. Here, we note that very high  $C_2H_2$  flow is used during growth to enhance the deposition speed and hence reduce the time for GaN at high temperatures. The partial pressure  $P_{C_2H_2}$  is  $\sim 100$  mbar, orders of magnitude higher than conventional recipes [13], [15], [21]. Therefore, merely 2 sccm change in  $C_2H_2$  already means  $\sim 1.3$  mbar change in  $P_{C_2H_2}$ . That is why the carbon film thickness is very sensitive to the change in  $C_2H_2$  flow under our experimental conditions. At this stage, however, despite the general controllability over film thickness, the extremely thin carbon layer on GaN (even if the substrate is stabilized by  $NH_3$ ) is not uniform, in contrast with those on inert substrates, e.g.,  $Si_3N_4$  [13] and  $SiO_2$  [14], [15]. This is similar to the case of Ni-catalyzed graphene [22], where the substrate has a high affinity to C and the graphene is nonuniform. In fact, in Fig. 1(a), the transport properties are consistent for all devices on the middle sample, which is not the case for the left sample. Epitaxial GaN grown along its  $c$ -axis has two faces (Ga-face and N-face). In this paper, our samples have the Ga-face polarity. As the carbon deposition is largely noncatalytic [13]–[15], we believe this polarity plays a minor role in the growth. However, we do not absolutely rule out any chemical interactions between the substrate and the carbon layer. Since N-face GaN is also important in optoelectronics, we will investigate the subtle influence of this polarity on the carbon CVD in the future.

Due to difficulties in wet etching of the GaN layer, the carbon thin films are not transferred to Cu grids for plan-view TEM investigations. Nevertheless, Fig. 1(b) depicts a high-resolution cross-sectional TEM image of a GaN/sapphire sample with a thicker [compared to the middle sample in Fig. 1(a)] carbon film to ease the identification of graphitic layers. The GaN epi-layer is seen to be somewhat roughened due to the high-temperature process. Rather homogeneous conformal carbon layers with some degrees of texture are observed, which accounts for the metallic luster of very thick films [13]–[15]. In Fig. 1(b), the thickness of the carbon layer is equivalent to  $\sim 20$  layers of graphene, assuming an ideal graphitic interplane distance of  $3.35$  Å. Further detailed TEM analysis can be found in Appendix A.

Fig. 2(d) shows the Raman spectra measured on the middle sample in Fig. 1(a). The G band centered at  $\sim 1600$   $cm^{-1}$

and the D band at  $\sim 1376$   $cm^{-1}$  are clearly seen, which are attributed to zone center phonons of  $E_{2g}$  symmetry and  $K$ -point phonons of  $A_{1g}$  symmetry, respectively [23]. They are typically observed in disordered graphitic structures [24]. There is no distinguishable 2-D band, typically found at  $2500$ – $2800$   $cm^{-1}$ , which is perhaps flooded by substrate luminescence, but more likely implies a defective carbon lattice, which may, at least in part, be related to the nonintact underlying GaN. The Raman analysis indicates that the as-grown thin films are likely to be at the transition stage from amorphous carbon to nanocrystalline graphitic carbon [24]. Fig. 3 shows the ultraviolet-visible-near-infrared (UV-VIS-NIR) transmission spectra, where the baseline (100% transmittance) is set to atmosphere (no sample in the beam). The interference fringes are due to the GaN films [25]. Similarly, the strong absorption edge is due to the GaN rather than the carbon thin films. The incident face of the carbon thin films is specular whilst the underside of the sapphire substrate is unpolished. Thus, more than 90% of the incident light is lost by scattering alone. However, useful data can be extracted by comparing the spectra of the bare GaN/sapphire and the carbon coated substrate [Fig. 1(a), middle]. At the absorption edge,  $\sim 2\%$  reduction in transmittance is extracted for the carbon thin films, which is comparable to the case of standard graphene [26]. Indeed, the porosity and grain boundaries of other nanocrystalline thin films have been empirically evidenced to give rise to much higher than expected optical transmission [27]. The insert presents the FTIR spectra of the same samples. There are no apparent changes in the spectra after the carbon deposition. This suggests that not only the carbon film is thin (C-related peaks are below the detection limit of the spectrometer), but also there is no bound water (that possibly exists in solution-processed graphene) at the interfaces ( $-OH$  group should show features at  $3000$ – $3700$   $cm^{-1}$  [28], [29]).

Finally, arrays of Hall-bar devices are fabricated by standard photolithography (see Appendix B). An optical micrograph of a typical device on the middle sample in Fig. 1(a) is shown in Fig. 2(a). According to our four-probe resistance measurements, the nominal sheet resistance  $R_s$  is  $2.3$   $k\Omega/\square$ . However, this is not the intrinsic  $R_s$  of the carbon thin films, because the GaN itself is unintentionally doped and thus conducting (see Appendix B). Our future goal is then to grow these thin films on high resistivity GaN substrates so that the sheet resistance of the carbon layer could be measured accurately. Currently, the electrical characterization data show that the thin films are conducting current but also more resistive than expected, implying that they might be carbon atoms connected primarily by  $sp^2$  bonds with a fraction of  $sp^3$  bonds, as discussed in Appendix B. Fig. 2(b) and (c) depicts the AFM height and phase images of the central part of the device, respectively. The nanopits in the carbon thin film in (b) are absent in (c). Here, the contrast in the phase image is primarily due to the difference in the chemical composition of the sample surface, which suggests that the roughness is mainly due to the high-temperature effect on GaN, and the carbon thin film grows conformally with no observable holes.

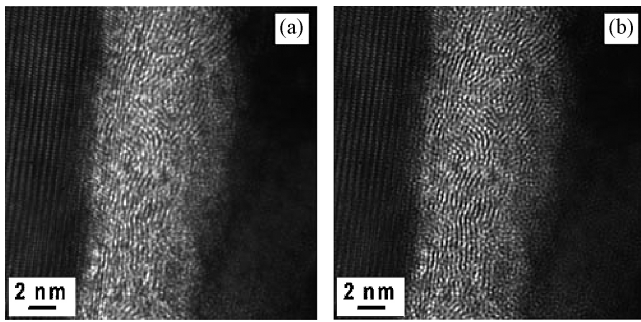


Fig. 4. Cross-sectional TEM micrographs of the same carbon thin film sample as shown in Fig. 1(b). (a) Raw data. (b) Amplified according to the description. In each image, the left section is the GaN.

#### IV. CONCLUSION

We elaborated on our abilities to deposit carbon thin films, for the first time, directly on GaN (0001)/sapphire substrates by CVD without the use of metallic catalysts. The as-grown thin films are scalable, continuous, uniform, and have a controllable thickness. The carbon coatings are electrically conducting and show optical transmission comparable to standard graphene. The transfer-free process favors the large-scale commercial application of such carbon thin films for future transparent electrodes to address the current crowding problem in optoelectronic devices. Further, detailed investigations of the carbon thin films on p-type GaN substrates and on functional device structures are underway, including the CVD growth and the electrical characterization, which will be published elsewhere. It is hoped that this paper will provide a basis for a generalized carbon CVD growth model on other semiconductors.

#### APPENDIX A TEM OBSERVATION

Fig. 4 shows the high-resolution TEM images obtained on the same sample as shown in Fig. 1(b). In order to improve the visibility of the graphitic layers of our carbon thin films, the spatial frequencies from  $2.44 \text{ nm}^{-1}$  to  $3.44 \text{ nm}^{-1}$  in the micrograph [Fig. 4(a)] are amplified with the following method. First, the entire micrograph is transformed by fast Fourier transform (FFT). Then, a ring mask with 5 pixel feathering and inner and outer radii of  $2.44 \text{ nm}^{-1}$  and  $3.44 \text{ nm}^{-1}$  is applied using the Digital Micrograph Software. The thus-masked FFT is then transformed back by the inverse transformation function. This yields a micrograph with areas that exhibit the corresponding characteristic spatial frequencies. Finally, the amplification in the original micrograph is established by adding the filtered image to the original micrograph. This improves the visibility of the carbon c-axis lattice fringes [see Fig. 4(b)], while also allowing for spatial information as to where these spatial frequencies are present with respect to the whole Au/Ti/C/GaN structure.

#### APPENDIX B ELECTRON TRANSPORT MEASUREMENTS

Hall-bar devices are fabricated by photolithography. The first exposure and the subsequent lift-off define the metallic contacts to the carbon coatings (45 nm Au/5 nm Cr, no

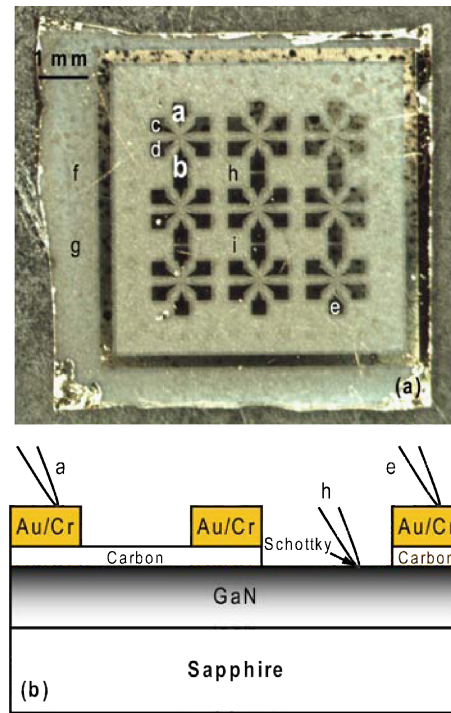


Fig. 5. (a) Stereo microscope image of the device arrays fabricated in the middle sample of Fig. 1(a). The letters indicate the contact points during the measurement. (b) Schematic of the cross-section of the carbon thin film devices on the GaN/sapphire substrate. a, e, and h highlight the three contact points measured with metallic probes.

annealing). The second exposure together with an  $\text{O}_2$  plasma etch (50 W, 1 min, 250 mTorr) patterns the carbon. Fig. 5(a) shows a stereo microscope image of the middle sample in Fig. 1(a), where a–g indicate the contact points during the electrical measurements on a Karl Suss PM5 probe station connected to a Keithley 4200-SCS parameter analyzer.

Fig. 6(a) shows the  $I$ – $V$  curves of a typical device [see Fig. 2(a)] fabricated in the sample shown in Fig. 5(a). Two-terminal measurement [a↔b in Fig. 5(a)] shows nearly linear behavior. Four-terminal measurement [a–d in Fig. 5(a)] eliminates the contact resistances, giving the sheet resistance  $R_s$  of  $2.3 \text{ k}\Omega/\square$ . However, while measuring across the whole sample [a↔e in Fig. 5(a)], we get a current over  $\pm 4 \text{ V}$  span, as plotted in Fig. 6(b). This indicates that all the devices are electrically connected to each other. We exclude this conductance as coming from the deposited carbon. As discussed previously, the carbon layer grown on this sample is  $\sim 4 \text{ nm}$  thick, equivalent to almost ten graphene layers, and should be completely burned out by  $\text{O}_2$  plasma etching under our conditions. We note that if carbon is yet present, the conductance can be detected simply by directly probing the surface [such as f↔g in Fig. 5(a), where the carbon at the sample edge has been protected by the resist. Notice the slightly darker color induced by the presence of carbon at the sample edge compared with the etched central region of the chip]. Indeed, there is a finite conductance between f and g. However, when probing between h and i [Fig. 5(a)], there is no measurable conductance, proving the thoroughness of the  $\text{O}_2$  plasma etch. Therefore, the conductance observed in Fig. 6(b) can only come from the unintentionally doped GaN.

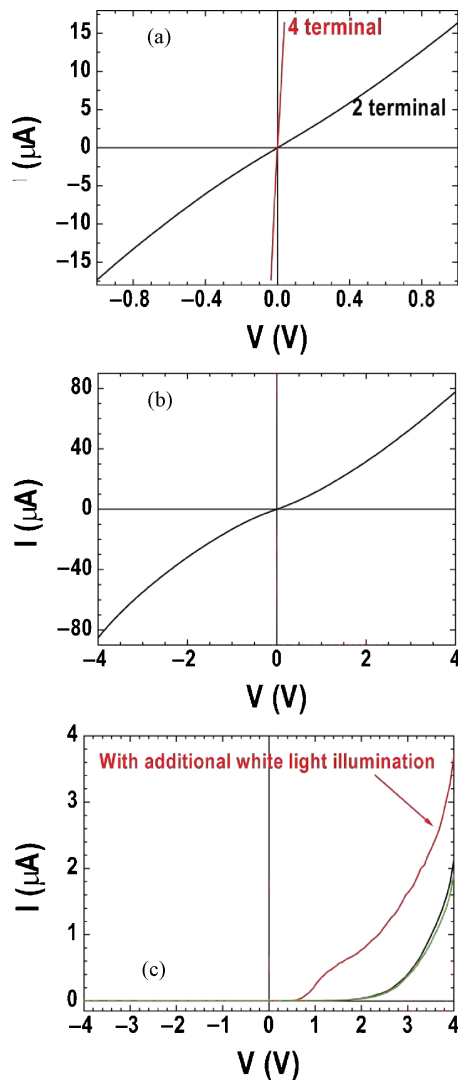


Fig. 6. (a) Resistance measurements of the upper left device shown in Fig. 5(a). The lower two figures show  $I$ - $V$  curves measured (b) between a and e and (c) between a and h in Fig. 5(a), respectively.

We now show that the carbon has electrical contacts to the underlying GaN. Fig. 5(b) illustrates our proposed model. In the device process, carbon is patterned after metallization. Therefore, beneath the Au/Cr pads, carbon is present. Note that thermal annealing is typically used to form ohmic contacts between metals and n-GaN [30] or p-GaN [31]. In our case, the Au/Cr layer is neither in direct contact to the GaN nor high-temperature annealed. Thus, the current between a and e [Fig. 6(b)] means that the carbon must possess electrical contacts to GaN (and to Au/Cr). In fact, as mentioned previously, probing between h and i shows no current. Moreover, Hall bars are also fabricated on a separate GaN/sapphire substrate that has not undergone any carbon deposition, where no conductance is observed between Au/Cr electrodes. This is due to the presence of two Schottky barriers at the interfaces between the metal probes and the GaN (equivalent to two face-to-face Schottky diodes). Nevertheless, measurement between a and h has only one Schottky barrier in the current path and should therefore show a rectification behavior. Indeed, in Fig. 6(c), we obtain a

clear current rectification resembling those of Schottky diodes [32] [ $a \leftrightarrow h$  in Fig. 5(a)].

Interestingly, a photoconductivity effect is observed in this rectification. In Fig. 6(c), the black curve is the measured  $I$ - $V$  property under ordinary room illumination; the red curve is under additional white light illumination; and the green curve is the case when the sample is returned to standard illumination again. At the present stage, the exact origin of this photocurrent is unclear (UV light is normally required to induce photoconductivity in GaN). Photocurrents have been reported in graphene [33], but it is unlikely that the observed nonnegligible photoconductivity is solely due to that. Doping in GaN, in some cases, can produce a significant photoluminescence in the visible light range [34]. Therefore, the GaN might have a broadened spectral response through the activation of these unintentionally doped sites.

These data confirm that the directly grown carbon thin films indeed conduct current, and their presence enhances the current injection between Au/Cr and GaN (better contacts), indicating a promising future. However, the conductance is smaller than values extracted from high-quality graphene layers with equivalent thickness. The contacts with the GaN are also not ideally ohmic. Some  $sp^3$  bonds (as in diamond) are probably responsible for the unexpected high resistivity and high transparency. Further investigations are necessary to reveal the mechanism.

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