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Coexistence of negative photoconductivity and hysteresis in semiconducting graphene

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Solution-processed graphene quantum dots (GQDs) possess a moderate bandgap, which makes them a promising candidate for optoelectronics devices. However, negative photoconductivity (NPC) and hysteresis that happen in the photoelectric conversion process could be harmful to performance of the GQDs-based devices. So far, their origins and relations have remained elusive. Here, we investigate experimentally the origins of the NPC and hysteresis in GQDs. By comparing the hysteresis and photoconductance of GQDs under different relative humidity conditions, we are able to demonstrate that NPC and hysteresis coexist in GQDs and both are attributed to the carrier trapping effect of surface adsorbed moisture. We also demonstrate that GQDs could exhibit positive photoconductivity with three-order-of-magnitude reduction of hysteresis after a drying process and a subsequent encapsulation. Considering the pervasive moisture adsorption, our results may pave the way for a commercialization of semiconducting graphene-based and diverse solution-based optoelectronic devices. © 2016 Author(s). All article content, except where otherwise noted, is licensed under a Creative Commons Attribution (CC BY) license (http://creativecommons.org/licenses/by/4.0/). [http://dx.doi.org/10.1063/1.4948313]

I. INTRODUCTION

Graphene is a monolayer of carbon atoms densely packed in a honeycomb crystal lattice. Since its first experimental realization in 2004,¹,² graphene has delivered profound applications because of its extraordinary optical and electronic properties.³–¹⁵ Unlike a semiconductor, graphene cannot be logically switched between on and off due to the lack of bandgap,¹⁶–¹⁸ which inhibits its applications in electronic and optoelectronic devices. Thereby, endowing graphene semiconducting properties with a moderate bandgap is highly desired. Although it has been demonstrated that the bandgap of graphene could be opened by shaping the graphene into nanoribbons¹⁹–²² or bonding oxygen to the graphene sheet (namely, graphene oxide, GO),²³ these approaches could bring with new problems. For example, the electronic properties are extremely sensitive to the geometrical parameters of the graphene ribbons, which require an extreme precision in cutting the graphene.¹⁷,²⁵,²¹ In addition, electronic properties of GO are strongly affected by the attached oxygen-containing functional groups, which undermines its reliability.¹⁷ Recently, zero-dimensional graphene quantum dots (GQDs) have been shown to exhibit a moderate bandgap due to the quantum size effect.²⁴–²⁶ Moreover, solution-processed GQDs are highly uniform and controllable in size and morphology,
and compatible to arbitrary substrates, which are competitive for practical applications in flexible electronics.27–29

Recently, a significant hysteresis effect accompanied with a weak negative photoconductivity (NPC) was observed in the current-voltage (I-V) curves of GQDs device at ambient environment.30 Hysteresis is defined as the difference in electric currents between the forward and backward voltage sweeps at zero bias voltage and has been shown to cause an erratic performance of the devices.31,32 NPC refers to the phenomenon that the total current under light irradiation is smaller than the dark current,33 which is unfavorable for traditional photoelectric conversion-based devices, such as, solar cells, etc. To the best of our knowledge, however, limited literature has reported the relation between hysteresis and NPC of materials, even for GQDs. Preliminary study has revealed a possible scenario for the hysteresis in GQDs, which is ascribed to the carrier trapping effect originating from highly electronegative surface adsorbates.34 NPC in GQDs has not been given a serious consideration,30 even though the origin of NPC in monolayer or few-layer graphene has been extensively studied in recent years.35–46 Hence, clarifying the origins of GQDs’ NPC and the relation with hysteresis could have a practical significance for improving photoelectric conversion performance of the semiconducting graphene-based photoelectric devices.

In this paper, we report the experimental observation of the coexistence (simultaneous appearance and absence) of NPC and hysteresis in GQDs. We demonstrate that the carrier trapping effect caused by surface adsorbed moisture should be responsible for our observations. By drying GQDs followed by an overlayer encapsulation, both NPC and hysteresis in GQDs can be effectively depressed as a result of the reduced relative humidity, leading to the positive photoconductivity (PPC). Due to the ubiquity of moisture, synchronously suppressing the NPC and hysteresis could enable high-performance of semiconducting graphene-based and solution-based optoelectronic devices.

II. METHODOLOGY

A. Fabrication of the GQDs devices

Details of materials, synthesis and characterization of the GQDs, as well as associated references are available in the Supplementary Material.49 The SiO2 substrate was cleaned by sequential ultrasonic cleaning process using acetone, methanol, and de-ionized water for 5 minutes, respectively. First, 180-nm thick Au electrodes were deposited on the two ends of a smooth SiO2 substrate by electron beam evaporation (EB3 3kW e-beam source, HHV Ltd.) at the vacuum degree of 5x10^-6 mbar, forming a channel with width and length of 10 mm and 1 mm, respectively, as shown in Fig. S6.49 Then an appropriate amount of GQDs aqueous solution (about 2 mL) was dispersed uniformly via an ultrasonic processing and dropped into the channel to fully cover the channel. After dried at 70°C for 30 min, the residual solvent was evaporated. This device was referred as SiO2-Au-GQDs. The average thickness of the GQDs layer (about 30 nm) was estimated by a step profiler (VEECO, DEKTAK 150).

B. I-V measurements of the GQDs devices

Two-probe measurements were conducted to obtain the current-voltage (I-V) curves of the devices using a semiconductor characterization system (Keithley 4200) coupled with a probe station (Cascade summit 11000 M) at room temperature. One Au electrode was biased and another one was grounded. The sweep rate was set as 0.1 V s^-1. A 375-nm UV laser (OBIS 375 nm) was used as the UV light source, coupled with a fiber with a spot diameter about 300 μm. And the power intensity was estimated at about 224.4 mW cm^-2.

III. RESULTS

A. Simultaneous appearance of hysteresis and NPC in the GQDs

The GQDs samples used in the present study were synthesized via sequential reduction of GO and dialysis process.47,48 Details are given in Supporting Information. Morphology, optical and
FIG. 1. (a) Schematic illustration of the device structure, (b) $I-V$ curves and (c) photocurrent of the SiO$_2$-Au-GQDs device (relative humidity, RH = 30%). Insets include an enlarged part of the hysteresis (inset in Fig. 1(b)) and photoconductance ($G$) (inset in Fig. 1(c)) of the device.

Semiconducting properties of as-synthesized GQDs are shown in Figs. S1-S5. The planar GQDs device structure is schematically shown in Fig. 1(a) (see Fig. S6 for an optical image), in which two Au electrodes are used to form the central channel and control the bias voltage. $I-V$ curves of the GQDs device were measured by sweeping the voltage forward and backward between -5 V and 5 V, and the measured results are plotted in Fig. 1(b). For the as-prepared SiO$_2$-Au-GQDs device at ambient atmosphere with a relative humidity (RH) of 30%, both the hysteresis and negative photoconductivity (NPC, $I_{UV} < I_{Dark}$) are observed. Herein, $I_{UV}$ refers to the total current when the sample is irradiated by ultraviolet light, and $I_{Dark}$ represents the dark current. Hysteresis is calculated based on the difference in electric currents ($I_{UV}$ or $I_{Dark}$) between forward and backward voltage sweeps at zero bias voltage, as shown in the inset of Fig. 1(b). Since the hysteresis in $I_{UV}$ and $I_{Dark}$ is nearly identical, only the hysteresis in $I_{Dark}$ is chosen to be discussed. The photocurrent...
\[ I_{op} = I_{UV} - I_{Dark} \] is defined as the difference between \( I_{UV} \) and \( I_{Dark} \), as plotted in Fig. 1(c). The photoconductance \( G = \frac{\partial I_{op}}{\partial V} \) is the derivative of the \( I_{op} \) to the bias voltage. Shown in the inset in Fig. 1(c) is the calculated photoconductance near the zero bias. Photoconductance discussed in this work refers to the average photoconductance of each sample, where the homogeneity of photoconductance of each sample could not change positive or negative characteristic of the photoconductance.

**B. Coexistence of hysteresis and NPC in the GQDs**

To elucidate the origins of NPC and hysteresis, we conduct a series of measurements in which the relative humidity is controlled. A SiO\(_2\)-Au-GQDs device was first dried at 70 °C for 30 min, and then reintroduced into the ambient atmosphere with different relative humidity RH = 30% and 70% for 10 min. The hysteresis and photoconductance measured for these cases are plotted in Fig. 2. It is seen from Fig. 2(a) and 2(d) that upon a drying treatment NPC disappears accompanied with three-order-of-magnitude reduction of hysteresis compared with that in the case of RH = 30% shown in Fig. 1(b) and 1(c). The disappearance of NPC and the dramatic reduction in hysteresis (namely, about 99.9%) confirm that adsorbed moisture is responsible for the coexistence of NPC and hysteresis of GQDs. When the SiO\(_2\)-Au-GQDs device was dried again at 70 °C for 30 min, followed with the reintroduction of the ambient atmosphere with RH = 30% for 10 min, NPC was observed to reappear accompanied with a noticeable hysteresis, as shown in Fig. 2(b) and 2(e). When the ambient atmosphere relative humidity is increased to RH = 70%, NPC and hysteresis become much stronger, as shown in Fig. 2(c) and 2(f). The above results imply that the surface adsorbed moisture could result in hysteresis and NPC of GQDs and their co-existence characteristics (simultaneous appearance and absence), as intuitively presented in the sample 1 to sample 4 of Fig. 3 and summarized in Table SI.49

**C. Hysteresis and photoconductance (G) of the GQDs**

In order to avoid the moisture-induced NPC and hysteresis and obtain a stable device performance, the SiO\(_2\)-Au-GQDs device was dried again at 70 °C for 30 min, and followed by an immediate deposition of a 30-nm-thick SiO\(_2\) overlayer by electron beam evaporation. In this case, instead

![Fig. 2.](image-url)
FIG. 3. Sample numbers mean the SiO$_2$-Au-GQDs device (1) in an environment with relative humidity RH = 30%, (2) under drying condition with relative humidity RH = 0%, (3) under drying followed with a reintroduction of low relative humidity RH = 30%, (4) under drying followed with a reintroduction of high relative humidity RH = 70%, and (5) under drying followed with overlayer encapsulation and reintroduction of relative humidity RH = 30%, respectively. Moreover, due to the very similar values of hysteresis in $I_{\text{UV}}$ and $I_{\text{Dark}}$ for each sample as listed in Table SI, only the hysteresis in $I_{\text{Dark}}$ is plotted. And only the average photoconductance for each sample is plotted, considering that the homogeneity of photoconductance of each sample could not change positive or negative characteristic of the photoconductance, as calculated and summarized in Table SI.

of the NPC, we observed a PPC accompanied with a greatly suppressed hysteresis even when the device was reintroduced into the ambient atmosphere (RH = 30%), as shown in the sample 5 of Fig. 3 and supported by Fig. S8 and Table SI. The performance of the device with a SiO$_2$ overlayer encapsulation is comparable to that of the dried cases shown in Fig. 2(a) and 2(d), demonstrating that the overlayer encapsulation processing is a simple while effective strategy to concurrently avoid hysteresis and NPC and obtain PPC of the GQDs devices.

D. Schematic diagram of the carrier transfer

The possible process of electronegative surface adsorbed moisture induced the coexistence of hysteresis and NPC in GQDs is proposed in Fig. 4. For GQDs at ambient atmosphere and in dark, the adsorbed moisture molecules could be ionized by trapping/capturing free electrons from...
GQDs due to their strong electronegativity. Accordingly, holes would accumulate in GQDs, resulting in a large conductivity (p-type conductance) and dark current ($I_{\text{Dark}}$) of GQDs, as well as the hysteresis even in the case of zero bias voltage. When GQDs are illuminated with the UV light, photo-generated electrons and holes are separated. The photo-generated electrons could recombine with the holes in GQDs which have been accumulated in the dark step, while the photo-generated holes could migrate to GQD surface and neutralize the electrons that have been trapped by the surface adsorbed water molecules in the dark step. Ultimately the holes accumulated in photo-excited GQDs were reduced, leading to the decrease in conductivity and total current of GQDs. Therefore, $I_{\text{UV}}$ is always smaller than $I_{\text{Dark}}$, and consequently the photoconductance ($G \equiv \partial I_{\text{op}}/\partial V$, the slope of the $I_{\text{op}}$ to the bias voltage) would be a negative value. When the surface moisture adsorption is eliminated, NPC could disappear and PPC could emerge along with a greatly reduced hysteresis. In short, the adsorbed moisture molecule gives rise to not only the hysteresis but also the NPC in the GQDs.

Due to the generality of moisture, the surface adsorbed moisture induced a coexistence of NPC and hysteresis may have its universality in various photoelectric conversion devices. For example, moisture adsorption induced NPC in stannic oxide (SnO$_2$)\textsuperscript{50} and zinc selenide (ZnSe),\textsuperscript{51} as well as moisture adsorption induced hysteresis in carbon nanotube (CNT),\textsuperscript{52,53} molybdenum disulfide (MoS$_2$),\textsuperscript{31,54} zinc oxide (ZnO)\textsuperscript{55} have been demonstrated in recent years. Like we have done for GQDs device, diverse solution-based materials could be encapsulated to possess stable PPC, which enable their practical applications in optoelectronics, even in flexible transparent optoelectronic devices.\textsuperscript{27–29,56} Moreover, a shorter-wavelength excitation may generate an enhanced photoresponse based on the absorption spectrum of the GQDs (Figs. S4 and S5),\textsuperscript{49} though the 375-nm UV light source could reasonably well endow GQDs PPC.

IV. CONCLUSIONS

In summary, the coexistence of NPC and hysteresis in GQDs was experimentally investigated. We compared the hysteresis and photoconductance of GQDs under different relative humidity conditions, and revealed that both the NPC and hysteresis in GQDs are induced by carrier trapping effect of surface adsorbed water molecules. Such a mechanism in turn suggests that the NPC and hysteresis in GQDs could be eliminated via drying and overlayer encapsulation process, which is also verified by our experiments. Owing to the universal moisture adsorption, our study may make semiconducting graphene-based materials and diversified solution-processed materials more promising for applications in optoelectronic devices.

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