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Two-beam-laser interference mediated reduction, patterning and nanostructuring of graphene oxide for the production of a flexible humidity sensing device

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ABSTRACT

Two-beam-laser interference was used for the simultaneous reduction, patterning and nanostructuring of graphene oxide on flexible polyethylene terephthalate substrates for the production of a high performance humidity sensing device. Hierarchical graphene nanostructures were formed after laser interference treatment of graphene oxide, which holds great promise for gaseous molecular adsorption, and thereby significantly increases their sensing performance. By tuning the laser power, the content of oxygen functional groups, could be changed within a certain range, which contributes not only controllable conductivity but also tunable response/recovery time of the humidity sensor due to the interaction between water molecules and oxygen functional groups on the graphene oxide sheets. The laser interference processing of graphene oxide films is a mask-free, surfactant-free and large-area approach to the production of hierarchical graphene micro-nanostructures, and thus shows great potential for fabrication of future graphene-based microdevices.

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1. Introduction

Recently we have witnessed the increasing interest in graphene due to its outstanding electrical, mechanical, thermal and chemical properties [1–7]. Typically, electron transport through graphene is highly sensitive to adsorbed molecules because of its unique single-layer structure that makes every carbon atom a surface atom. Therefore, graphene has been generally considered as a promising sensing material for various gases, for instance, toxic aromatic compounds [8–14]. Notably, graphene single layer was reported to be effective for detecting gaseous species down to the single molecular level [8]. However, it is well known that mechanically derived graphene suffers from serious problems in scalable and large-area preparation. As an alternative choice, chemical exfoliated graphene oxide (GO) was adopted for sensing application. In the case of GO sensors, the oxygen groups impart graphene sheets solution-processing compatibility, and thus contribute to formation of continuous films on all kinds of substrates. Nevertheless, GO is electrically insulating, its conductivity could be partly restored only if certain reduction processes were performed to remove the oxygen groups.

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Despite the fact that chemical or thermal reduction does not cure the material to pure graphene, reduced graphene oxide (RGO) which possesses both conductivity and chemically active defect sites is also proved to be a promising candidate for gas sensing. To date, RGO has been successfully used for detecting a wide range of gas molecules including NO₂, NH₃, H₂O, CO, Cl₂ and H₂ [15–19].

Generally, the gas-sensing mechanism of RGO is ascribed to the adsorption/desorption of gaseous molecules on the surface of RGO, which act as electron donors or acceptors and thus change the resistance [13,18]. As compared with graphene single layer, multilayered RGO exposes relatively low surface area to the sensing gas, which significantly debases their sensing performance. To make the inside RGO layer visible to the sensing gas, it is highly desired to assemble the RGO film into hierarchical nanostructures. For instance, Ji et al. reported a layer-by layer film of RGO and ionic liquids for gas sensing [20]. With the help of nonvolatile ionic liquids, layered nanostructures were successfully formed by in situ reduction and subsequent electronic assembly. The controlled spacing between graphene layers would provide 2D cavities, thereby promote the molecular adsorption significantly. However, up to date, despite the rapid progress in highly sensitive RGO sensors, surfactant-free fabrication and patterning of RGO nanostructures for high performance sensing device remains a challenging task.

We present here the fabrication of humidity sensing device on flexible substrate by two-beam-laser interference (TBLI) reduction and patterning of GO. Interestingly, hierarchical nanostructures can be introduced during the TBLI reduction process, which contributes to guest molecular adsorption and further enhances their sensing performance. By tuning the laser reduction degree, oxygen functional groups could be removed in a controlled fashion, thus the conductivity of the RGO film could also be changed within a certain range. As a result, the nanostructured RGO sensor shows not only excellent sensitivity to humidity at room temperature, but also fast and tunable response/recovery time, demonstrating the unique advantages of TBLI fabrication of RGO sensing devices.

2. Experimental

2.1. TBLI reduction of GO films

GO was produced by Hummers' method from natural graphite (Aldrich, <150 μ m). The GO films were prepared by spin-coating GO solution on PET substrate at 1000 rpm for 20 s, dried at 60 °C for 1 h and cooled to room temperature. Then the sample was exposed by two beams which were split from the UV laser to reduce and produce nanostructures. A frequency-tripled, Q-switched, single-mode Nd:YAG laser (Spectra-physics) with about 10 ns pulse width was used for laser interference. 0.1–0.3 W laser power measured before the spectroscope. The exposure time was 10 s.

2.2. Humidity sensing devices

After nanosecond laser interference reduction of GO films, Au electrodes were deposited onto the substrates through a sha-

dow mask. The controlled humidity environments were achieved using saturated aqueous solutions of LiCl, CH₃COOK, MgCl₂, K₂CO₃, Mg(NO₃)₂, NaBr, NaCl, KCl and KNO₃ in a closed glass vessel at an ambient temperature of 25 °C which yielded approximately 11%, 23%, 33%, 43%, 54%, 59%, 75%, 85%, 95% relative humidity (RH), respectively. Performances of flexible humidity sensing devices were measured from a ZL5 intelligent LCR meter.

2.3. Characterization

X-ray photoelectron spectroscopy (XPS) was performed using an ESCALAB 250 spectrometer. Current–voltage curves of humidity devices were measured from a Keithley SCS 4200 semiconductor characterization system. Scanning electron microscope (SEM) experiments were performed on a Hitachi S-4800 electron microscope.

2.4. First principle study

The theoretical simulations are carried out with generalized gradient approximation of Perdew–Burke–Ernzerhof [21] implemented in Vienna ab initio simulation package code [22,23]. The electron-ion interaction is described by the frozen-core all-electron projector augmented wave method [24]. In periodic supercell, graphene sheet contains 32 carbon atoms and a vacuum layer of 20 Å is used to avoid any interaction with neighboring images. All calculations are done with cutoff energy of 500 eV for plane wave expansion and $3 \times 3 \times 1$ Monkhorst-Pack mesh grid for Brillouin zone sampling.

3. Results and discussion

3.1. TBLI reduction of GO

As a promising approach for large-area fabrication of periodic micronanostructures, multibeam interference lithography has been used for the production of 2D gratings, 3D photonic crystals, and flower-like arrays in a rapid and cheap manner [25-27]. On the other hand, laser direct writing has been proved capable for effective reduction of GO films, which shows great potential for fabrication and integration of graphene-based devices [28,29]. In this work, for the first time, TBLI was adopted for larger-area reduction and patterning of GO film towards a RGO sensing device. As shown in Fig. 1, when two laser beams irradiate on the GO film at same position, interference occurs. Generally, in the laser interference region, the laser intensity distribution is constant along the yaxis, and sinusoidal along x-axis. The highest laser intensity is calculated to be four times of each laser beam, and the minimum value is zero. So the GO film was proposed to be reduced with a similar distribution: that is oxygen groups were readily removed in the region of high laser intensity, and survive in the low intensity region. In fact, the interference period could be precisely controlled by changing the angle of two laser beams. However, in this work, the period is fixed at $2\,\mu m$, and we only change the laser power for comparison.



Fig. 1 - Illustration of TBLI reduction of GO film. The laser intensity distribution is calculated by Matlab.



Fig. 2 – SEM images of RGO film prepared by TBLI reduction under the laser powers of (a) 0.1 W, (b) 0.15 W, (c) 0.2 W and (d) 0.3 W, respectively. Insets are magnified images and theoretical calculation results of laser intensity distributions in each condition.

Fig. 2 shows SEM images of GO films reduced under different laser powers. Firstly, a laser beam of 0.1 W is separated into two beams and interfered on the surface of GO film. Here the peak power is calculated to be 0.2 W (inset of Fig. 2a). In this condition, the GO surface was slightly cut apart in the region of high laser intensity. In fact, the GO film shows unobvious morphology change after irradiation under the power bellow this value, so the threshold power of GO reduction is considered to be 0.2 W. Then we increase the laser power to 0.15 W (power before splitting into two beams), and the peak power is as high as 0.3 W. As shown in Fig. 2b, GO film was patterned into hierarchical nanostructures in this condition. It is worthy pointing out that the surface topology of RGO has strong dependence on the laser intensity distribution. In this condition, the region exposed to higher laser intensity is well reduced to RGO stripes of ~200 nm in width, which

could be clearly distinguished from other regions. Whereas in the zero intensity region, pristine GO was not reduced. Interestingly, in the transition region, GO was partially reduced and ablated due to the removal of both oxygen functional groups and some carbon species (Fig. 2b). Layered graphene nanostructure formed on both sides of the incisions. When we further increase the laser power to 0.2 and 0.3 W, as shown in Fig. 2c and d, the RGO region expands to \sim 600 and \sim 900 nm, respectively, and the pristine GO region disappeared at the same time. Additionally, the layered nanostructures in the transition region become disorder due to heat transfer induced further reduction. In order to distinguish the RGO region and the transition region clearly, TBLI (0.2 W) along the three directions deflected 60 degrees was performed on the GO film. The residual array of the transition region confirms our previous results. Besides the laser power,

the exposure time also shows similar influence on the reduction and patterning of GO films. Briefly, longer exposure time would result in deeper reduction of GO, and form similar morphology with that fabricated under higher laser power.

Fig. 3 shows the high-resolution SEM images of RGO nanostructures fabricated by nanosecond laser of 0.15 W. Notably, the parallel beam cut the flat GO film into stripe structures after the reduction and partly ablation. It could be clearly observed from the image that on both sides of the incisions, GO was separated into single layers and rolled up from the edge, which could also be distinguished from the frizzy GO region. Possible reason to the formation of this layered nanostructures would be the ultrafast removal of interlayer water and oxygen-containing gaseous species during nanosecond laser irradiation. This layered nanostructures which is formed in a mask-free, surfactant-free and large-are manner, would significantly increase the surface area of RGO to the environment, and thus hold great promise for guest molecules adsorption and desorption.

3.2. Tunable oxygen content

To quantitatively evaluate the reduction degree of RGO under different laser power, C1s XPS was used to measure the surface oxygen content. As shown in Fig. 4a, the three peaks at 284.6, 286.6 and 288.5 eV are attributed to C-C (nonoxygenated ring carbon), C-O (hydroxyl and epoxy carbon), and C=O (carbonyl), respectively [24]. Notably, the content of oxygen atoms in pristine GO is as high as 46.5%, the content of carbon not bonded to oxygen is only 32%. After reduction, for instance 0.15 W laser, C-C percentage increases to 68%; C-O percentage decreases to 23%, indicating the successful removal of oxygen groups. Increase of laser power would lead to the further reduction of the GO film. Fig. 4b shows the dependence of C-C, C-O and C=O contents of RGO samples on the reduced laser power. The tendency is very clear, that is with the increase of laser power, C-C percentage increases significantly; whereas the C-O and C=O percentages decrease. Survey X-ray photoelectron spectra of the RGO samples indicate that oxygen content could be tuned in the range of 46.5–5.6% by changing the reduction laser power, giving the possibility for controlling the density of oxygen functional groups, as well as the conductivity. In our work, RGO



Fig. 4 – (a) C1s XPS spectra of pristine GO and RGO prepared by TBLI with different laser powers, (b) dependence of C–C, C–O C=O and O atom percentage of the RGO films on the laser powers.

reduced by nanosecond laser with an output power of 0.3 W gives the lowest oxygen content of 5.6%, meaning that a majority of oxygen-containing groups are removed. In this



Fig. 3 - High-resolution SEM image of RGO hierarchical nanostructures prepared by TBLI with the laser power of 0.15 W.

case, we can thus infer that almost the whole GO film was reduced due to the heat transfer effect, however, this thermal effect also partly destroyed the layered nanostructures as observed in the SEM image (Fig. 2d). As the laser power could be tuned continuously, the laser mediated reduction of GO direct gives the promise for precise control of the oxygen-containing functional groups in the resultant RGO films.

3.3. Sensing performance

With the help of TBLI reduction and nanostructuring, RGO humidity sensing device were fabricated on flexible PET substrates (inset of Fig. 5a). As shown in Fig. 5a, the current-voltage curves of RGO reduced under different laser powers exhibit linear dependence, confirming their good ohmic contact and tunable conductivity, generally, the higher the laser power, the smaller the resistance. The results of absorption/ desorption resistance measurements as a function of % RH at room temperature are shown in Fig. 5b. It can be seen that the resistance for GO almost keep the same in the whole % RH range, showing no humidity sensitive properties due to its insulating properties. After TBLI reduction with the laser power of 0.15 W, the resistance of the devices decreased by more than two orders of magnitude with an increase in RH over the range of 11-95% RH. Moreover, as can be seen, the curves presented good linearity and small hysteresis of \sim 6%, showing its good sensitive properties [30,31]. When



Fig. 5 – (a) I–V curves of RGO sensor prepared by TBLI with laser power of 0.15 W, 0.2 W and 0.3 W at room temperature. The inset is a photograph of a typical RGO humidity sensor. (b) Humidity hysteresis characteristic of the GO and RGO sensors.

the laser power was tuned to 0.2 and 0.3 W, the range of the resistance variety become smaller and the linearity of the resistance versus % RH slightly deteriorated over a wide humidity range. The decrease of impedance change was related to their relatively higher conductivity and the deterioration of hierarchical nanostructures as compared with the device fabricated by 0.15 W laser.

The response and recovery curves for these devices are shown in Fig. 6. Generally, the time taken by a sensor to achieve 90% of the total impedance change is defined as the response or recovery time. For the device of 0.15 W, the response time was only 2 s when increasing from 11% to 95% RH, and it reached a plateau quickly, indicating the short response time. However, when decreasing from 95% to 11% RH, the recovery time was more than 100 s (Fig. 6a). For the device fabricated by the laser of 0.2 W, the response time was about 3s, and the recovery time was about 10s (Fig. 6b). When we further increase the laser power to 0.3 W, the response time become 50 s, and the recovery time was about 3 s (Fig. 6c). Based on the above results, it is reasonable to get the conclusion that the response and recovery time of the RGO humidity sensor is very fast as compared with conventional semiconductor oxides sensor [32]. Moreover, by



Fig. 6 – Response and recovery properties of the RGO sensor. GO films were reduced under the laser power of (a) 0.15 W; (b) 0.2 W and (c) 0.3 W, respectively; (d) GO was also reduced at 350 °C under the protection of nitrogen for comparation. (e) Response/recover curve of RGO (0.2 W) sensing device for 5 cycles. The curves were measured between 11% and 95% RH at room temperature.



Fig. 7 – First-principle study of the interaction between single water molecule and graphene sheets, as well as epoxy or hydroxyl groups functionalized graphene.

changing the reduced laser power, both response and recover time of the RGO sensor could be tuned in a controlled fashion.

As a controlled experiment, thermally reduced GO film was also used for humidity sensing device (Fig. 6d). As a comparison, RGO film reduced at 350 °C under the protection of nitrogen has similar oxygen content and resistance with that of RGO reduced by 0.2 W laser interference. SEM image show that the surface of RGO after thermal reduction is very smooth. However, its resistance variability range is very small, about 1/5 of that of 0.2 W laser reduced RGO, and two order of magnitudes less than that of 0.15 W one, confirming the importance of nanostructures in the humidity sensing devices. In addition, as a typical example, sensing repeatability of this device is also evaluated. As shown in Fig. 6e, the resistance variability range is almost constant for 5 cycles, indicating the reliability of our RGO humidity sensor.

3.4. First-principle study

To get further insight into the mechanism of the tunable response/recovery property of our humidity sensors, first principle study was carried out to give an essential explanation (Fig. 7). Pristine graphene sheet with 32 carbon atoms and the same structure functionalized with epoxy or hydroxyl groups were used as calculation models. The interaction between a H₂O molecule and graphene is week Van der Waals force as demonstrated by our calculation, 0.044 eV/H₂O, see Fig. 7. However, for GO models which are functionalized with epoxy and hydroxyl groups, H₂O and oxygen radical would bind together with the form of hydrogen bond which is much stronger than Van der Waals forces. In the present calculations, the binding energy between H₂O and epoxy is 0.201 eV/H₂O and the one for hydroxyl can reach 0.259 eV/ H₂O. So the oxygen functional groups on GO would hold significantly larger ability to absorb H₂O molecules than pure graphene. In our work, the device fabricated by 0.15 W laser exhibit relatively large amount of oxygen functional groups, thus it shows fast response time and slow recovery time due to the relatively strong interaction with water molecules and the large surface area derived from the hierarchical nanostructures. However, for the case of device fabricated by 0.3 W laser, the residual oxygen content is only \sim 5%, relatively weak interaction with water molecules contributes a slow response and very short recovery time on the contrary. Here, by changing the laser power, oxygen content could be tuned continuously, as shown in the XPS spectra (Fig. 4). Therefore, the response and recovery time for these humidity sensors could also be tuned within a certain range. It is worth pointing out that the presence of oxygen-containing groups on RGO would also promote the adsorption of other organic gaseous molecules such as ethanol, and thus these organic molecules would act as a disturbance. However, unlike water molecules, no charged ions would be produced when organic molecules were adsorbed onto the RGO film at room temperature, therefore, the influence by such gaseous organic compounds is neglectable.

4. Conclusions

TBLI was proved to be a novel approach to simultaneous reduction, patterning and nanostructuring of GO in a surfactant-free, mask-free and large-area manner. With the help of TBLI, RGOs with hierarchical nanostructures and tunable oxygen functional groups were used for fabrication of humidity sensing devices on flexible substrate of PET. The presence of nanostructures increases the surface area and thus significantly improves the sensing performance. The modulation of oxygen-group content gives the feasibility for controlling the adsorption/desorption behavior of water molecules, contributing the tunable response/recovery time. The TBLI assisted reduction and patterning of GO films shows great potential for the production of nanostructured graphenebased microdevices.

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.carbon.2011.12.011.

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