Patterning Graphene Films by H₂O-Based Magnetic-Assisted UV Photolysis

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ABSTRACT: Properly cutting graphene into certain high-quality micro-/nanoscale structures in a cost-effective way has a critical role. Here, we report a novel approach to pattern graphene films by H₂O-based magnetic-assisted ultraviolet (UV) photolysis under irradiation at 184.9 nm. By virtue of the paramagnetic characteristic, the photo-dissociated hydroxyl [OH(X₂Π)] radicals are magnetized and have their oxidation capability highly enhanced through converting into an accelerated directional motion. Meanwhile, the precursor of H₂O(X̃1A₁) molecules distributes uniformly thanks to its weak diamagnetic characteristic, and there exists no unstable diamagnetic intermediate to cause lateral oxidation. Possessing these unique traits, the H₂O-based magnetic-assisted UV photolysis has the capability of making graphene microscale patterns with the linewidth down to 8.5 μm under a copper grid shadow mask. Furthermore, it is feasible to pattern graphene films into 40 nm-wide ribbons under ZnO nanowires and realize hybrid graphene/ZnO nanoribbon field-effect transistors with a hole mobility up to 7200 cm²·V⁻¹·s⁻¹. The X-ray photoelectron spectroscopy and time-of-flight secondary ion mass spectrometry analyses reveal that OH(X₂Π) radicals act as a strong oxidant and that another product of H(12S) adsorbs weakly on graphene.

KEYWORDS: chemical vapor deposition, H₂O-based magnetic-assisted UV photolysis, graphene films, mass spectrometry, high-quality micro-/nanoscale structures

INTRODUCTION

In addition to the breakthrough of growing large-area high-quality monolayer and multilayer graphene films by chemical vapor deposition (CVD), it is crucial to precisely cut them into certain micro-/nanoscale patterns in order to propel their fundamental studies and versatile applications. Nowadays, conventional photolithography and electron-beam lithography, combined with reactive ion etching (RIE), have remained the mainstream techniques, even though they are costly and inevitably incur resist contamination for atomic-layer-thick two-dimensional material. To surmount these shortcomings, Yong et al. attempted to use hard shadow masks as a substitute for lithography to pattern graphene films by oxygen plasma RIE procedure. However, such obtained graphene pattern had its quality degraded due to severe lateral oxidation. Direct laser writing, another frequently used approach in the semiconductor industry, has been attempted to pattern graphene films despite its prominent problems such as low precision, debris contamination, and substrate damage. Ever since the discovery of graphene, it has always been underway to seek feasible approaches to realize high-quality graphene patterning in an efficient and economical way.

Using oxygen molecules \([O_2(X^3Σ_g^-)]\) as the precursor, magnetic-assisted ultraviolet (UV) ozonation was proposed and tested to be feasible for patterning graphene microstructures under a shadow mask upon irradiation of a xenon excimer lamp \((λ = 172 \text{ nm})\). In this novel etching approach, the photo-dissociated paramagnetic oxygen radicals \([O(1^3P)]\), moving directionally in the inhomogeneous vertical magnetic field after magnetization, play a critical role in the highly enhanced
oxidation capability. As a contrast, the main product of oxygen radicals produced in the conventional oxygen plasma RIE process moves randomly in an applied electric bias due to its electrically neutral characteristic and thus induces severe lateral oxidation.\textsuperscript{16−18} The magnetic-assisted UV ozonation, however, has two major drawbacks that restrict the minimum linewidth and its practical applications. First, it appears tricky to precisely control the concentration of O(3P) radicals since the paramagnetic O\textsubscript{2}(X\Sigma\textsubscript{g}−) molecules incline to gather around the magnetic poles instead of distributing uniformly in the reaction chamber. Second, the photo-synthesized unstable intermediate of ozone sublimes arbitrarily and therefore can impair the quality of graphene patterning by lateral oxidation.\textsuperscript{14,15} Water [H\textsubscript{2}O(X\bar{1}A\textsubscript{1})], attractive for its potential application in the synthetic chemical fuel production of hydrogen,\textsuperscript{19,20} can surmount these obstacles and bring out marvelous opportunities when combined with the UV irradiation and magnetic field thanks to the following three traits. First, H\textsubscript{2}O(X\bar{1}A\textsubscript{1}) molecules, a weak diamagnetic substance, distribute uniformly in the circumstance of an inhomogeneous magnetic field. Second, under the vacuum UV irradiations, H\textsubscript{2}O(X\bar{1}A\textsubscript{1}) molecules photo-dissociate into paramagnetic hydrogen [H(1\Sigma\text{S})] and hydroxyl [OH(X\Sigma\Pi)] radicals without any intermediate product that can arouse lateral oxidation.\textsuperscript{21−25} Third, the paramagnetic OH(X\Sigma\Pi) radicals have a highly enhanced oxidation capability compared with O(3P) and can enhance the etching efficiency in principle.\textsuperscript{10,26}

In this work, we report on the patterning of a graphene film by H\textsubscript{2}O-based magnetic-assisted UV photolysis under irradiation at 184.9 nm. In an inhomogeneous vertical magnetic field, the photo-dissociated paramagnetic OH(X\Sigma\Pi) radicals are magnetized and then have an accelerated directional motion toward graphene.\textsuperscript{18,19−23} As a consequence, the directional-motion-
induced oxidation enhancement makes it feasible to realize high-resolution micro-/nanoscale graphene patterning. The etching mechanism of graphene by H2O-based magnetic-assisted UV photolysis is explored through X-ray photoelectron spectroscopy (XPS) and time-of-flight secondary ion mass spectrometry (TOF-SIMS) measurements.

**RESULTS AND DISCUSSION**

Figure 1 shows the schematic configuration of H2O-based magnetic-assisted UV photolysis designed for patterning graphene films, using a home-designed vacuum machine equipped with a low-pressure mercury (Hg) lamp ($\lambda = 184.9$ nm, $P = 200$ W). The deionized water contained in a self-designed stainless-steel bottle provides gaseous H2O molecules when connected to the evacuated photochemical reaction chamber. A cuboid-shaped neodymium–iron–boron (NdFeB) permanent magnet put underneath the sample is used to provide the inhomogeneous vertical magnetic field ($B_z = 0.57$ T, $V_B = 160$ T·m$^{-1}$).

The H2O-based magnetic-assisted UV photolysis proceeds as illustrated in the right part of its zoom-in view (Figure 1). The ground-state H2O($X^1A_g$) molecules are excited to the first excited electronic state of H2O($A^2B_1$) upon UV irradiation and then dissociate primarily into paramagnetic H($1^1S$) and OH($X^2\Pi$) radicals on a time scale of femtosecond as described in eq 1.

$$\text{H}_2\text{O}(X^1A_g) + h\nu (120 < \lambda \leq 186.4 \text{ nm}) \rightarrow \text{H}_2\text{O}(A^2B_1)$$
$$\rightarrow \text{H}(1^1S) + \text{OH}(X^2\Pi)$$  

(1)

The available energy in this direct photodissociation is mainly converted into the translational motion of H($1^1S$) and OH($X^2\Pi$) radicals. In the presence of the inhomogeneous vertical magnetic field, the paramagnetic H($1^1S$) and OH($X^2\Pi$) radicals are both magnetized and then accelerate toward graphene surface with the magnetic force denoted by cyan arrows in the right part of Figure 1. Specifically speaking, each of the two kinds of radicals has an attractive magnetic force $F_z$ on the order of $10^{-22}$ N according to the formula $F_z = g_\text{f} \mu_\text{eff} V B_z$. Herein, the Landé $g_\text{f}$-factor takes the value of 2 and 4/3 for H($1^1S$) and OH($X^2\Pi$), respectively; the effective magnetic moment $\mu_\text{eff}$ of H($1^1S$) and OH($X^2\Pi$) equals to $1 \mu_\text{B}$ ($\mu_\text{B}$ is the Bohr magneton) and $2 \mu_\text{B}$, respectively.

The H2O-based magnetic-assisted UV photolysis can be used to pattern graphene films into high-quality microscale structures with the aid of a copper shadow mask. As shown in Figure 2a, the mask is composed of orthogonal ribs with a period of 62 μm. The high-resolution scanning electron microscopy (SEM) image in Figure 2b (biased at 5 kV) shows that the ribs have a step structure. In detail, the central flat region is ~7.8 μm wide and ~6.6 μm thick, and each edge has a width ranging from 1.3 to 1.8 μm with a thickness of ~3.8 μm. When treated by H2O-based magnetic-assisted UV photolysis (working distance, i.e., distance between the lamp and graphene: 32 mm; duration: 8 min × 3 times), the polycrystalline graphene can be patterned into 8.5 μm-wide grid microstructures (Figure 2c,d). The pattern has a profile conformal to the shadow mask but with its linewidth falling between the central and total width of the bar. Besides, some filamentary residues exist in the blank area around the graphene edges due to the presence of ripples (see the Supporting Information, Figure S1).

The quality of graphene patterns is further characterized by its lateral oxidation using micro-Raman spectroscopy. Figure 2e shows the defect (D) mode image scanned across a rectangular area which, for clarity, is superimposed on the same position of its optical image. The bright lines distributed along two edges certify lateral oxidation underneath the mask. The Raman spectral evolution scanned along the dashed arrow line across the bar is plotted in Figure 2f. The peak intensity ratios of D to G mode ($I_D/I_G$) and 2D to G mode($I_{2D}/I_G$) are plotted in Figure 2g. Compared with the pristine polycrystalline graphene (see the Supporting Information, Figure S1), we can conclude from Raman characterization that lateral oxidation occurs mainly within 2~3 μm with strong D peaks and that the central region appears slightly oxidized or mechanically damaged by the mask with a decreasing intensity ratio of $I_{2D}/I_G$ compared with pristine graphene (Figure S1). We think this degradation along edges mainly stems from the imperfect vertical magnetic field and the step structure of the mask, that is, its imperfect contact with graphene and the resultant gas diffusion and dissipation in the gap. As elaborately stated in the Supporting Information (Figure S2), the specific shadow mask can impose a great impact on the quality of graphene patterns.

The potential of patterning graphene film by H2O-based magnetic-assisted UV photolysis is explored by fabricating graphene nanostructures. Figure 3a shows the SEM topographic image of ZnO nanowires distributed on graphene film. (a) SEM image of ZnO nanowires distributed on graphene film. (b) High-resolution SEM image of a pure graphene nanoribbon attained from that covered by ZnO nanowire outlined in (a). (c) Optical image of a hybrid graphene/ZnO nanoribbon FET device, and (d) its drain–source current as a function of back-gate voltage measured in N2 atmosphere.

Figure 3. Characterization of graphene nanoribbons patterned under ZnO nanowire mask by H2O-based magnetic-assisted UV photolysis. (a) SEM image of ZnO nanowires distributed on graphene film. (b) High-resolution SEM image of a pure graphene nanoribbon attained from that covered by ZnO nanowire outlined in (a). (c) Optical image of a hybrid graphene/ZnO nanoribbon FET device, and (d) its drain–source current as a function of back-gate voltage measured in N2 atmosphere.
graphene nanoribbon, the ZnO nanowire (with a diameter of ~115 nm) is kept in situ throughout the electrical transport measurement with the curve plotted in Figure 3d. The entire graphene nanoribbon (length: ~8 μm, width estimated from statistics: ~80 nm) is calculated to have an average hole mobility μ_h of ~7200 cm^2·V^−1·s^−1 from the formula μ_h = \frac{1}{eC_g^\frac{\Delta\sigma}{\Delta V_g}}, where C_g = 1.15 × 10^−8 F·cm^−2 is the gate capacitance, and Δσ/ΔV_g is the slope of the conductivity σ varying with the gate voltage V_g near the Dirac point. The good hole mobility obtained at room temperature indicates that the graphene nanowire has a high quality. However, the graphene nanoribbon has a rather poor electron mobility of ~330 cm^2·V^−1·s^−1 as calculated from the relatively flat part close to the Dirac point at ~50 V. This reduction is mainly related to the electron scattering due to the adsorption of chemical functional groups. Herein, we neglect the impact of the remaining mask on the electrical conductivity of graphene since the ZnO nanowire mask has high resistivity (see Supporting Information, Figure S3). In the highly oxidative environment, the instability of the ZnO nanowire mask (Figure S4) and its passivation (Figure S5) are discussed in the Supporting Information.

The working mechanism of how graphene interacts with the highly accelerated H(1S)/OH(X2Π) radicals can be deduced from its varying status of surface chemical functional groups. For this purpose, a piece of graphite treated the same by H2O-based magnetic-assisted UV photolysis is used for XPS measurement. Compared with the pristine graphite (ref 14), a new peak of O 1s is appeared near the edge of the C 1s in the survey spectrum (Figure 4a). The O 1s peak situates at 532.5 eV corresponding to the O 1s peak of O = C, C − O chemical functional groups. Herein, the intensity of C=O group appears slightly stronger than that of C−O group, suggesting an oxidation process different from O(^3P) radicals.

It was reported that hydrogen atoms incline to form dimers on graphene through weak physical adsorption and migration or chemically bond with graphene to form graphane, or even pattern graphene by anisotropic etching at a high temperature. The exact role of photo-dissociated H(1S) radicals in graphene patterning can be tracked by TOF-SIMS measurement of pristine graphite and that treated by H2O-based magnetic-assisted UV photolysis. In order to exclude influence from the atmospheric hydrogen, deuterium (D2O) instead of H2O was used as the precursor in the magnetic-assisted UV photolysis. In this case, the gaseous D2O(X^1A1) molecules photo-dissociate into D(^1S) and OD(X^2Π) radicals under irradiation at 184.9 nm as described in eq 2.

\[ D_2O(X^1A_1) \rightarrow (120 < \lambda \leq 186.4 \text{ nm}) \rightarrow D_2O(\bar{A}^1B_2) \rightarrow D(^1S) + OD(X^2Π) \]  

The isotopic D(^1S) radicals, which are paramagnetic and supposed to play the same role as H(^1S) in the photochemical reaction, can be easily discerned when combined with graphene. Meanwhile, the paramagnetic OD(X^2Π) radicals act as a strong oxidant resembling OH(X^2Π) as confirmed in the Supporting Information (Figure S6).

Figure 5 compares the C_n− ion (n = 1, 2, 3, 4) calibrated negative mode spectra of pristine graphite and that treated by D2O-based magnetic-assisted UV photolysis. As observed, the secondary H−, H2−, C_n−, D−, and OD− ions exist in both cases. The H/D-related ion signals in Figure 5b indicate the existence of H2O-based magnetic-assisted UV photolysis. Since neither C2D− nor D2− can be detected, deuterium atoms are supposed to bond weakly with graphite rather than in the form of carbon-based materials or D2O/D2 molecules.

The OD− ions, existing in pristine graphite, increase about 8 times after treatment by D2O-based magnetic-assisted UV photolysis, much higher than the D− ions. This result reveals that...
The quality of nanoribbons fluctuates drastically as it is affected by the quality of the exact nanoscale graphene. For microscale graphene FET devices, it is unavoidably affected by the ripples. As a consequence, the selected best graphene/ZnO nanoribbon FET device in Figure 3 has a hole mobility far exceeding that of the best microscale one (Figure S2).

It is notable that the inhomogeneous vertical magnetic field is a prerequisite for patterning graphene films by H_{2}O-based magnetic-assisted UV photolysis. When no magnetic field is supplied, graphene keeps almost intact even though the other photochemical parameters used are the same (see the Supporting Information, Figure S7). This phenomenon reveals that the etching capability of randomly moving OH(X^2Π) radicals is drastically reduced compared with the accelerated directional ones.

Using the same shadow masks in Figures 2 and S2, we have patterned graphene microstructures and FET devices by the conventional oxygen plasma RIE technique. The graphene patterns are severely damaged due to lateral oxidation of randomly moving oxygen radicals (see the Supporting Information, Figure S8). When no bias is applied to control the directionality of positive ions in an oxygen plasma machine, graphene pattern cannot form due to the random motion of all reactant molecules, including oxygen radicals and oxygen ions O_2^- (see the Supporting Information, Figure S9). Compared with the conventional oxygen plasma RIE, the H_{2}O-based magnetic-assisted UV photolysis manifests an outstanding directional etching trait. Furthermore, H_{2}O-based magnetic-assisted UV photolysis, a combination of the vacuum UV discharge lamp and permanent magnet with H_{2}O molecules as the oxidant precursor, manifests to be more cost-effective compared with oxygen plasma RIE etching technique for its future industrial applications.

As clarified, the low-pressure mercury lamp instead of the xenon excimer lamp is used as the UV source to pattern graphene micro-/nanoscale structures in the H_{2}O-based magnetic-assisted UV photolysis. The experimental results indicate that graphene patterns fabricated under UV irradiation of the xenon excimer lamp are inferior in quality due to the limitation of the previous technique (see the Supporting Information, Figure S10). Interestingly, we have recently circumvented this limitation and realized high-quality graphene patterns with the etching efficiency improved by a few orders. This topic will be discussed in detail elsewhere.

Different shadow masks have been used to pattern graphene films in this work. Although convenient and well demonstrating the etching directionality of H_{2}O-based magnetic-assisted UV photolysis, the strategy exhibits some limitations as follows. First, shadow masks may damage graphene from the mechanical pressing and scratching due to their direct contact. Second, not all shapes (e.g., a ring) and complex circuit diagrams can be transferred onto a shadow mask. In this case, multiple shadow masks should be carefully designed to avoid freestanding parts and aligned with each other to realize graphene patterns by multiple etching. Third, fabrication of shadow masks with the linewidth down to 10 μm appears difficult. To eliminate these limitations, it is significant to explore the feasibility of combining H_{2}O-based magnetic-assisted UV photolysis with conventional photolithography and e-beam lithography techniques.

Direct laser writing technique has two major shortcomings, that is, low precision and substrate damage when used to pattern atomic-thick graphene films (see the Supporting Information, Figure S11). In the etching process, a pulse laser with strong
peak intensity is a prerequisite to realize graphene patterning. If the UV discharge lamp is substituted by a vacuum UV laser, H2O-based magnetic-assisted UV photolysis can convert into a chemically assisted direct laser writing approach with the following advantages. First, the vacuum UV laser can be a continuous one with much lower optical power since the photo-dissociated directional OH radicals play a major role in the etching efficiency. Second, the writing precision can be improved since the etching originates from a much shorter wavelength of a vacuum UV laser. Third, the adoption of a continuous low power laser can eliminate mechanical damage to the substrate underneath. We think chemically assisted direct laser writing can be an attractive topic in the future.

**CONCLUSIONS**

In summary, we have realized the patterning of a graphene film by the novel approach of H2O-based magnetic-assisted UV photolysis. The photo-dissociated paramagnetic H(12S) and OH(XII) radicals are magnetized in the inhomogeneous vertical magnetic field ($B_z = 0.57$ T, $VB_z = 160$ T·m⁻¹) and then turn into accelerated and directional motion toward graphene. Using the copper hole grids as the shadow mask, we can obtain graphene microstructures with the linewidth down to 8.5 μm. The H2O-based magnetic-assisted UV photolysis has a capability of patterning 40 nm wide graphene nanoribbons under ZnO nanowires. The hybrid graphene/ZnO nanoribbon FET devices have the hole mobility up to 7200 cm²·V⁻¹·s⁻¹. The functionality of H(12S) and OH(XII) radicals in the graphene patterning has been ascertained. This work introduces a novel cost-effective approach of etching high-quality graphene micro-/nanoscale structures with high directionality, and it also opens up a new avenue of functionalizing and patterning the booming two-dimensional materials.

**METHODS**

**Strategy for Patterning Graphene Films by H2O-Based Magnetic-Assisted UV Photolysis.** Either high-quality single-crystal or polycrystalline CVD-grown monolayer graphene on copper foil was transferred onto the SiO2 (300 nm)/Si (p⁺) substrate taking the regularly used poly(methyl methacrylate)-mediated approach. A home-designed UV vacuum machine equipped with a low-pressure mercury lamp was used for graphene patterning. A 400-mesh copper grid (Gilder Grids AG400HS), a conventional sample support in tunneling electron microscopy, was put tightly on the graphene film as the shadow mask to pattern graphene microstructures. The chamber was first pumped to 2 × 10⁻³ Pa and then filled with gaseous H2O molecules to 200 Pa (if not specified otherwise). In the H2O-based magnetic-assisted UV photolysis, parameters including the working distance and UV irradiation were adjusted according to the specific micro/nano-structure of masks. Herein, a strategy of two to four cycles of UV exposure was taken to keep a high etching efficiency with the fixed initial H2O pressure in the closed chamber, as well as to avoid excessive temperature rise in the UV photolysis. In order to carry out TOF-SIMS measurement, a graphite film was treated for 120 s by D2O-based magnetic-assisted UV photolysis, with the anode power of 150 W. A time-of-flight secondary ion mass spectrometer system (IONTOF S-100) equipped with a Bi⁰ beam source was used to acquire the chemical information of pristine graphite and that treated by H2O-based magnetic-assisted UV photolysis under a bias of 30 keV. The scanning area was 100 μm × 100 μm with an acquisition time of 200 s. The software used for peak analysis was SurfaceLab 7.0. In order to reduce the influence from the atmospheric carbon, the samples freshly cleaved and those further treated by UV photolysis, were stored in situ in the low vacuum chamber (~5 Pa) before being transferred into the corresponding high-vacuum chamber for XPS and TOF-SIMS measurements.

**ASSOCIATED CONTENT**

**Supporting Information**

The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acsami.0c16005.

Influence of the shadow mask on graphene patterning, electrical transport property of a single ZnO nanowire, stabilization of ZnO nanowires treated by H2O-based magnetic-assisted UV photolysis, patterning graphene film by D2O-based magnetic-assisted UV photolysis, influence of an inhomogeneous vertical magnetic field on graphene patterning, patterning graphene film by oxygen plasma, patterning graphene film under a xenon excimer lamp, and patterning graphene film by direct laser writing technique (PDF).

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Notes

The authors declare the following competing financial interest(s): Shanghai Jiaotong University, along with the authors, has filed provisional patents on the technology and intellectual property reported here (patent application numbers CN 201810981805.7; title: Patternning graphene film by magnetic-assisted photochemical oxidation with a specific setup).

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