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Robust micropatterns on graphene oxide films based on the modification of fluorescence lifetime for multimode optical recording

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ABSTRACT

Here we report on the fine modification of fluorescence lifetime of graphene oxide (GO) film by introducing a localized writing process with a well-controlled reduction degree. The modification results from the elimination of long-lived sp^3 functional groups and/or the conversion of that to short-lived sp^2 confined clusters. By optimizing the parameters of writing process, four-lifetime distributions with narrow width have been determined. The distinguishable lifetime distributions allow to create versatile and complex micropatterns on the GO film, which are robust with film-thickness- and reading-power-independent features. These features enable a multimode optical recording on GO film for high-capacity information storage based on the contrast of fluorescence lifetime. Our results present that lifetime modification of GO film can provide a new platform for the fabrication of micro-photonic information devices.

1. Introduction

Graphene has attracted considerable interest from the viewpoint of fundamental physics to the promising applications in various areas since its emergence [1–3]. To date, graphene has been used as the supercapacitors [4], transparent conductive electrodes in flexible electronic and optical devices [5,6]. Applications of graphene in electronics and optoelectronics require scalable production as well as versatile micropatterns in device architecture [7]. In response to these two challenges, the direct laser writing of graphene oxide (GO) films has become one of the most powerful approaches, benefiting from its facile processing abilities, high spatial resolution, and maskless feature [8,9]. By introducing a localized photoreduction process with a well-controlled reduction degree, direct laser writing provides a new landmark for the fabrication of graphene-based flexible electronics and optoelectronics. Currently, all-reduced GO (RGO) field effect transistor [8], microcircuits [9] and microelectrodes on flexible substrates [10], as well as three dimensional GO-RGO stacked-layered structure [11], have been fabricated by direct laser writing approach.

Beyond the manipulation of electrical conductivity, the optical properties of GO can also be modified by photoreduction, which provides unexpected applications in optical data storage [12]. Gu group has developed multimode optical recording and full-colour three-dimensional images in GO-dispersed polymers based on the hologram-encoded refractive-index modulation by focused femtosecond laser beam [13,14]. This method enables an enhanced information security and capacity (290 GB per disc [12]). To further improve the storage capacity, the manipulation of other intrinsic optical properties by photoreduction, such as fluorescence emission, is highly desired. Interestingly, experimental investigations have demonstrated that the fluorescence intensity can also be controlled by changing the reduction degree through laser reduction [15,16]. By enhancing the fluorescence intensity, Senyuk and co-workers have fabricated three-dimensional micropatterns in colloidal GO [17], which can be used in photonics and display technology. Our group has also demonstrated versatile micropatterns on single and multilayer GO films based on the fluorescence quenching effect through direct laser writing [18]. However, GO suffers from heterogeneous fluorescence intensity due to the non-uniform film thickness or the varied number of GO layers, which significantly hinders its optical applications. On the other

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hand, the fluorescence intensity is strongly dependent on the power of reading laser, which requires refined control of laser power to determine the same reading results.

In this work, we report the multimode optical recording on the GO film through the modification of local fluorescence lifetime by direct laser writing. Comparing to fluorescence intensity, fluorescence lifetime used for optical recording appears significant advantages, which is homogeneous under different film thicknesses and maintains the lifetime under varied reading powers. The fluorescence lifetime could be modified by changing the power as well as duration of writing laser. In our previous work [19], the manipulation of lifetime by changing the irradiation duration from 0–0.5 s has been performed. However, it's time-consuming and lifetime resolution is relative low. Here, we improved the manipulation of lifetime by varying the irradiation power with short duration (0.01 s), and the continuous tuning of fluorescence lifetime in the region of 1.05 ns–0.34 ns has been achieved, which could be used for direct and fine micropatterns and create a multimode optical recording on GO film. Under each writing power, the lifetime exhibits a narrow distribution. By optimizing writing powers, four distinguished lifetime distributions have been determined, offering a new route for multimode optical recording on GO film. The robust and scalable multimode optical recording, with film-thickness- and reading-power-independent features, enables an enhanced information capacity and promising applications in optical data storage.

2. Experimental section

2.1. Sample preparation

The GO water dispersion with the concentration of 2 mg/mL was synthesized by the modified Hummers' method [20]. The multilayer GO films were prepared by spin-coating the dispersion onto the cleaned glass coverslip at 500 rmp with 12 s and then 2000 rmp with 60 s. The process was repeated for 5 times. The final sample was dried at room temperature for 24 h to remove the remaining solvent.

2.2. Optical setup

The experiments, including fluorescence intensity and lifetime measurements as well as laser direct writing, were performed by using a home-built scanning confocal microscope [21,22]. The details have been presented in supporting information (SI). Particularly, a 405 nm continuum-wave (CW) laser was used to directly write micropatterns on GO film, in view of its high spatial resolution and effective reduction on GO film. The lateral dimension of focal spot by an oil immersion objective (Nikon, 100×, 1.3 NA) was ~500 nm. The duration time for each spot was set as 10 ms with the laser power in the region of 10 μW to 10 mW. The GO sample was placed on a motorized three-dimensional PZT stage. The micropatterns were achieved by moving the GO sample with respect to the focused laser beam in a programmable and controlled way. The step size during the micropatterning was set as 200 nm, the speed of laser writing was kept constant at 20 μm/s. The micropatterns based on the lifetime imaging were reading by a pulsed laser (PicoQuant, LDH-D-C-635) with the wavelength of 635 nm and the pulse width about 60 ps. One should emphasize that the pulsed laser used here with the power of 0.5–1.5 μW and duration time of 10 ms showed negligible reduction on GO film.

3. Results and discussion

Benefiting from its solution-processing compatibility, GO dispersion can be used to prepare large scale film through facile approaches, such as spin-coating method. However, these approaches usually result in the non-uniform film thickness, mainly due to the large lateral scale of GO flakes (0.5–3 μm [20]) and random stacked processing. Fig. 1(a) presents a typical optical image of a GO film prepared by spin-coating method. Note that the film is inhomogeneous, comprised of some thick GO flakes on the surface. These flakes could well be the aggregation of monolayer GO in the dispersion, due to π-π stacking [23,24]. According to the atomic force microscopy (AFM), the film thickness is varied from 2.0 nm to 32 nm, with many folded wrinkles on the surface, as

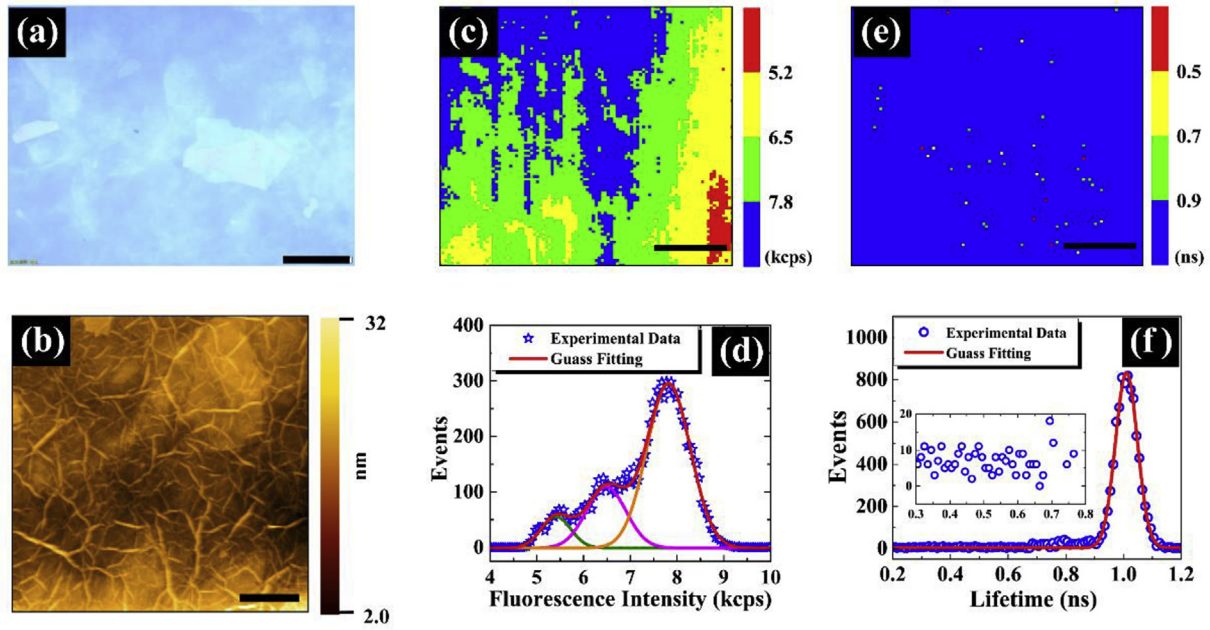

Fig. 1. (a), (b) Optical image and atomic force microscopy image of the prepared GO sample, respectively. The scale bars are 20 μm and 10 μm, respectively. (c), (d) Fluorescence imaging and its intensity distribution. (e), (f) Lifetime imaging and its distribution for the same area. Scale bar in (c) and (e): 10 μm. (A colour version of this figure can be viewed online.)

shown in Fig. 1(b). The surface roughness of the glass coverslip has also been characterized by AFM, which exhibits mean height of 1.5 nm and average roughness of 0.6 nm, as shown in Fig. S2. Thus, we suggest that the wrinkles and non-uniform thickness are originating from the randomly stacking and overlapping of GO themselves during sample preparation, rather than the surface roughness of the glass coverslip. The non-uniform thickness will result in the heterogeneous fluorescence intensity, as displayed in Fig. 1(c). Generally, the thicker the film, the stronger the fluorescence intensity. Fig. 1(d) presents the statistical result of fluorescence intensity for each pixel in image, which shows a broad distribution. Three main peaks have been determined with the full width at half maximum (FWHM) about 1.0 kcps, revealing that the investigated GO film may have three major thicknesses. The non-uniform thickness and heterogeneous fluorescence intensity will hinder its further applications in high quality electronic and photonic devices.

To improve the optical performance, the fluorescence lifetime here is used to characterize and pattern the GO film. As one of the most important optical properties, the fluorescence lifetime is identified by the intrinsic nature of chromophores [25]. As reported by Shang and Galande [26,27], the origin of GO's fluorescence can be classified into two kinds of chromophores: the graphene-like confined sp^2 clusters and the sp^3 structures consisting of oxygen-containing functional groups. Considering fluorescence intensity strongly depending on the amount of sp^3 and sp^2 structures, it significantly relates to the film thickness. On the contrast, the lifetime of GO film is only determined by the relative proportion of chromophores, neither the film thickness nor the fluorescence intensity [28,29]. Fig. 1(e) shows the lifetime imaging of the same area as Fig. 1(c), which appears almost uniform values. The distribution of lifetime has been shown in Fig. 1(f). Only one sharp peak with the centre value of 1.05 ns and FWHM of 0.10 ns is obtained. Although a few pixels with the lifetime beyond the main distribution (as the non-blue colour presented in Fig. 1(e) and the inset of Fig. 1(f)), overwhelming majority are in the region of 0.9 ns−1.1 ns. The values beyond this region are primarily stemming from the amorphous structure of GO [16].

To write and visualize the micropatterns based on the contrast of lifetime, the facile operation with the precise modification of GO's fluorescence lifetime is highly desired. As reported in the previous work [30], the lifetime of GO film can be directly varied by laser writing. The lifetimes for the sp^2 clusters and sp^3 functional groups are 0.14 ns and 1.37 ns, respectively. The overall lifetime is adopted by the relative proportion of the two kinds of chromophores [30]. During laser writing, the sp^3 functional groups will be eliminated or reduced to sp^2 clusters, thus changing their proportion. Consequently, the lifetime can be modified by varying the reduction degree of GO, which is readily controlled by the power of writing laser. The higher power the writing laser, the smaller the fluorescence lifetime of GO. Fig. 2(a) presents the time-resolved fluorescence decay traces of GO film after writing at different powers, which are 0 μW, 30 μW, 75 μW, and 1 mW for P_0, P_1, P_2, and P_3, respectively. Note that the trace under P_0 is the fluorescence decay of the original GO film without any writing. The decay traces from P_0 to P_3 become close to the instrument response function, indicating substantial decrease of lifetime as the increase of writing power. Moreover, the lifetimes as the function of writing power are shown in Fig. 2(b), which can be fitted by the single exponential function. The well-fitted function strongly suggests that the precise modification of GO's lifetime can be achieved by optimizing the writing power. Fig. 2(c) shows the lifetime imaging after writing at the power of 75 μW (P_2), proving that the lifetimes of the writing area are relatively uniform. Furthermore, four distinguished lifetime distributions have been obtained by writing the GO film with the power from P_0 to P_3, as shown in Fig. 2(d). The central values of them are 1.00 ns, 0.84 ns, 0.60 ns, and 0.34 ns with FWHM of 0.10 ns, 0.06 ns, 0.06 ns, and 0.05 ns, respectively. Note that FWHM becomes narrower with the increasing of the writing power, indicating that the lifetime of RGO is more homogenous than that of GO. Moreover, we also correlated the structural changes and lifetime variation during laser writing by Raman spectra. As shown in Fig. S3, the I_D/I_G ratio of GO film increased from 0.69 to 1.23 after writing at powers from P_0 to P_3, hinting that the GO has been gradually reduced [31,32]. These results further support our concept that the fine manipulation of fluorescence lifetime can be

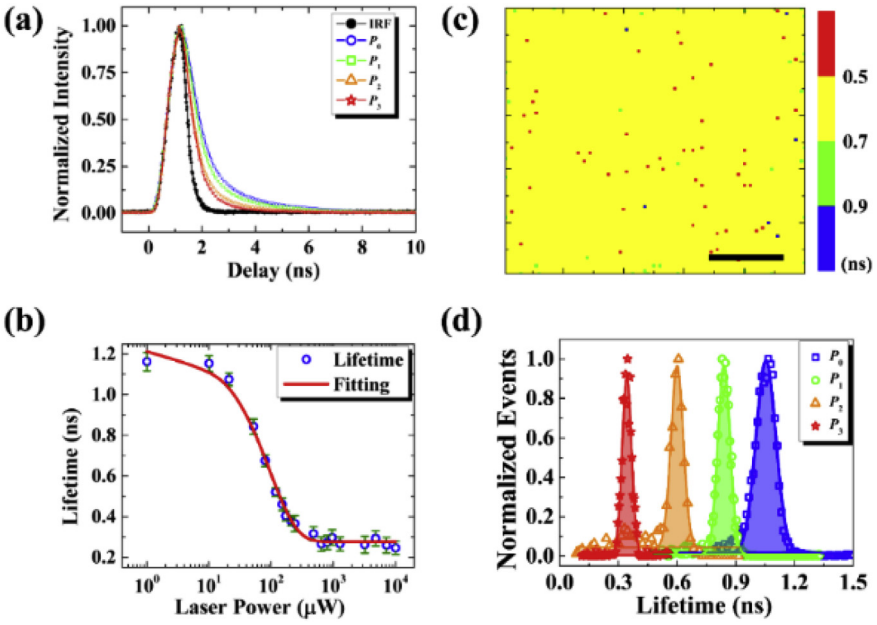

Fig. 2. (a) Time-resolved fluorescence decay traces for GO film after writing at powers of P_0 to P_3, which are 0 μW, 30 μW, 75 μW, and 1 mW, respectively. (b) Lifetime decay as a function of writing power. (c) Lifetime imaging of GO film after writing at the power of 75 μW (P_2). Scale bar: 10 μm. (d) Lifetime distributions of the GO film after writing at the powers of P_0 to P_3. (A colour version of this figure can be viewed online.)

achieved by controlling the reduction degree of GO through laser irradiation.

The precise modification of lifetime allows for direct and fine micropatterns on GO film. As shown in Fig. 3(a) and Fig. S4 in the SI, many desired micropatterns based on the modification of lifetime have been created readily. More importantly, the four distinguished lifetimes offer a mechanism for multimode optical recording [14]. As shown in Fig. 3(b), the GO film has been written with the power of P_0 to P_3 at desired areas, which are squares with the lateral of 5 μm (25 × 25 pixels with the step size of 200 nm). The five areas without writing (P_0) remain their original intensity (22–28 kcps). While the intensities of six areas (with the colour of light green) written at the power of P_3 are sharply decreased, the central values of them are only 2–6 kcps. Because of the thermal diffusion originated from the CW reduction laser [33,34], the fluorescence intensities for these areas are gradually decay at the edge. This thermal diffusion also results in the edge effect in their lifetime imaging, as presented in Fig. 3(c). This shortcoming can be solved by alternating the CW laser to femtosecond laser with low repetition rate and low power [35]. To extract the correct information from the lifetime imaging, we calculated the mean values of each area by using the centre square with the lateral of 3 μm (15 × 15 pixels). The processing result has been presented in Fig. 3(d), of which the four discretized values (colours) can be applicable for recording multimode information [36]. That is, each area contains two bits of information. Consequently, the information can be encoded by the two binary sets. This approach provides a new route to improve the capacity of optical recording, which can overcome the capacity limit by combing a long lifetime chromophore with a precise modification on its lifetime.

Along with the modification of lifetime, GO's fluorescence intensity is also modified, as shown in Fig. 3(b). The result appears that the fluorescence intensity can be used as the parameter to record information as well [17]. However, this approach is greatly sensitive to the reading power, which will lead to the information inaccurate or even unreadable, when the reading power is changed or even fluctuation (See Fig. S5 for the reading results under three adjacent powers). On the contrary, optical recording based on the contrast of lifetime is not only independent on the film thickness, but also insensitive to the reading power. Fig. 4 presents the lifetime imaging of the same patterns under reading power of 1.0 μW, 0.5 μW, and 1.5 μW, respectively. The pattern of "Panda" can be clearly visualized on the lifetime imaging, as shown in Fig. 4(a), (d) and (g), respectively. In contrast to fluorescence intensities (Fig. S5), there are no significant differences for fluorescence lifetimes under three reading powers, and two similar distributions of lifetimes can be clearly identified, as presented in Fig. 4(b), (e) and (h). The central values for the encoding areas are 0.58 ns, 0.60 ns, and 0.60 ns, respectively; while the central values for the original areas are 1.05 ns, 1.03 ns, and 1.05 ns, respectively. By using an optimized threshold value of 0.80 ns to distinguish the encoding and uncoding areas, the difference among three conditions can be further declined, as shown in Fig. 4(c), (f) and (i), respectively. The robust micropatterns based on the contrast of lifetime with film-thickness- and reading-power-independent features enable a high information fidelity and security.

To further explore the promise of this technique in the information storage, we characterize the stability and reliability of multimode optical recording after multiple laser reading. Four encoding areas were selected, which were irradiated by the 405 nm CW laser with the power of P_0 to P_3 and duration of 10 ms, respectively. The pulsed laser with the wavelength of 635 nm and the power of 1 μW was still used as the reading laser. The lifetime was reading per 2 seconds with integration time of 0.1 s for each read. Fig. 5 presents fluorescence lifetime trajectories and the corresponding statistics. It can be found that the lifetimes for each area have a narrow distribution, which is completely enclosed in the lifetime distribution after writing with the corresponding laser powers, as illustrated in Fig. 2(d). Negligible change in the fluorescence lifetime of each area after 300 cycles reading indicates that the multimode optical recording based on the variation of fluorescence lifetime is stability and reliability. This result further explore that our technique has promising applications in the information storage and related optical applications.

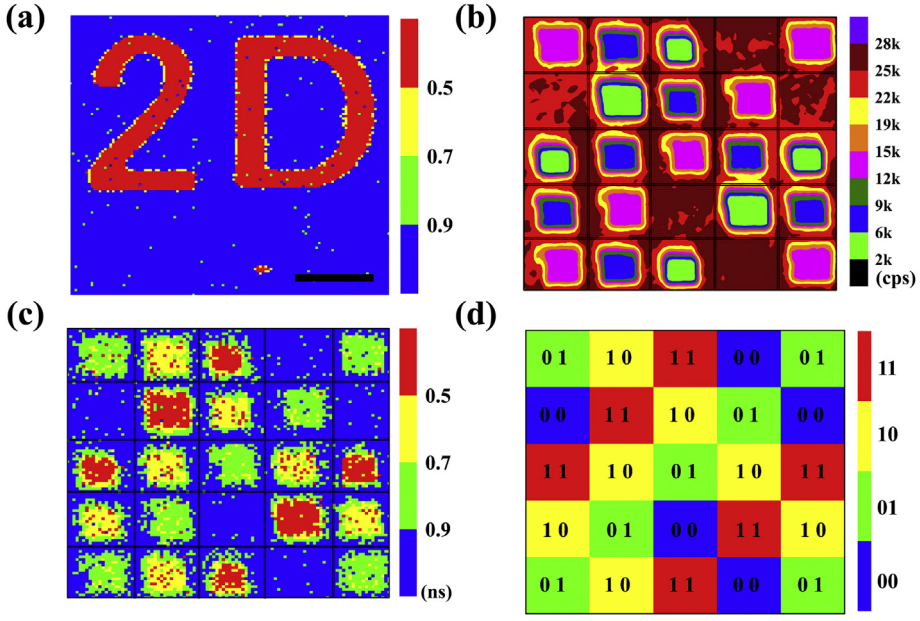

Fig. 3. Micropatterns and multimode optical recording on GO films based on the modification of lifetime. (a) Micropattern of the alphabets ("2D") on the film, written at the power of 1 mW and duration of 10 ms. Scale bar: 10 μm. (b), (c) Fluorescence intensity and lifetime imaging of the encoding information, written at the powers of P_0 to P_3. (D) The processing lifetime imaging by calculating the mean values and the corresponding encoded two binary data. (A colour version of this figure can be viewed online.)

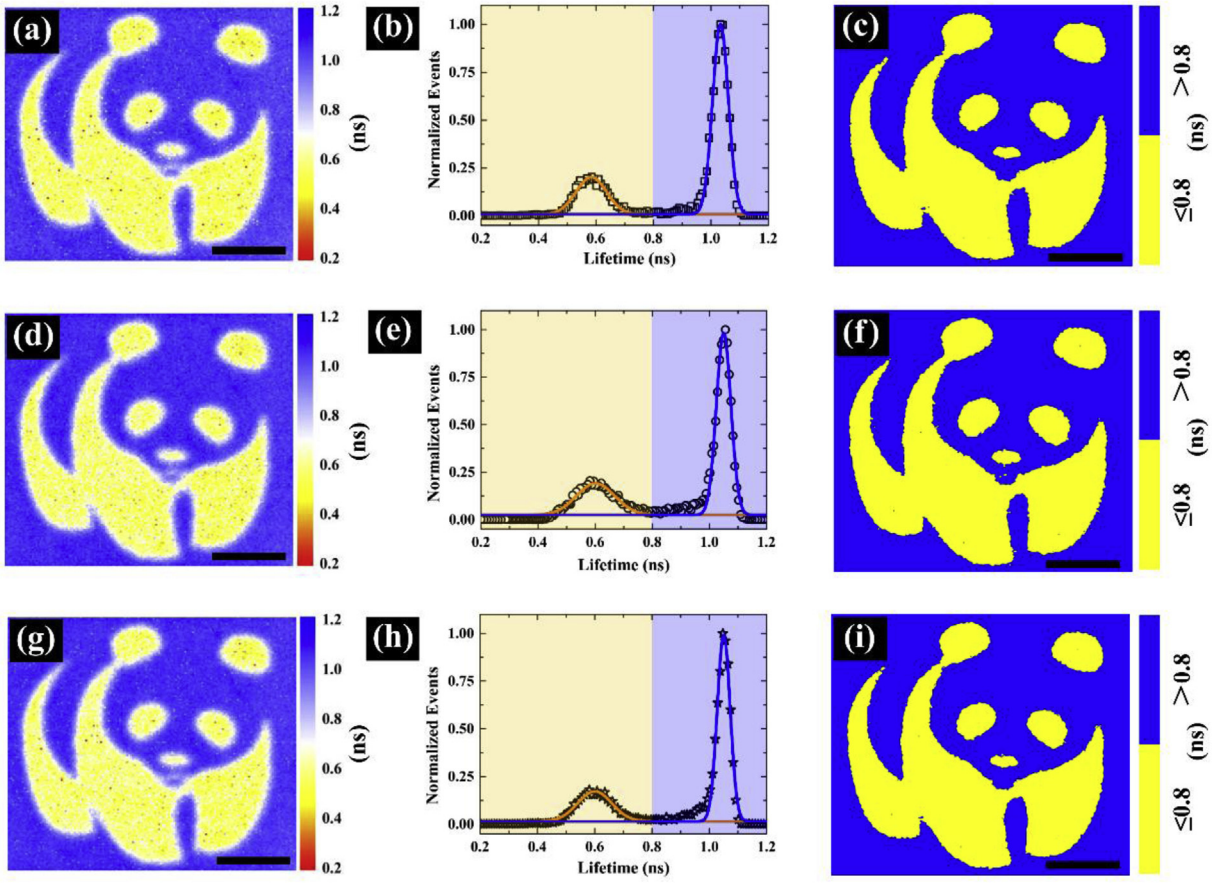

Fig. 4. (a), (d), (g) Lifetime imaging of the encoding micropatterns reading by a 635 nm picosecond laser at powers of 1.0 μW, 0.5 μW and 1.5 μW, respectively. (b), (e), (h) The corresponding lifetime distributions. (c), (f), (i) The corresponding imaging after processing with the threshold value of 0.80 ns. The encoding areas are written by 405 nm CW laser at the power of 75 μW and duration of 10 ms. Scale bar, 10 μm. (A colour version of this figure can be viewed online.)

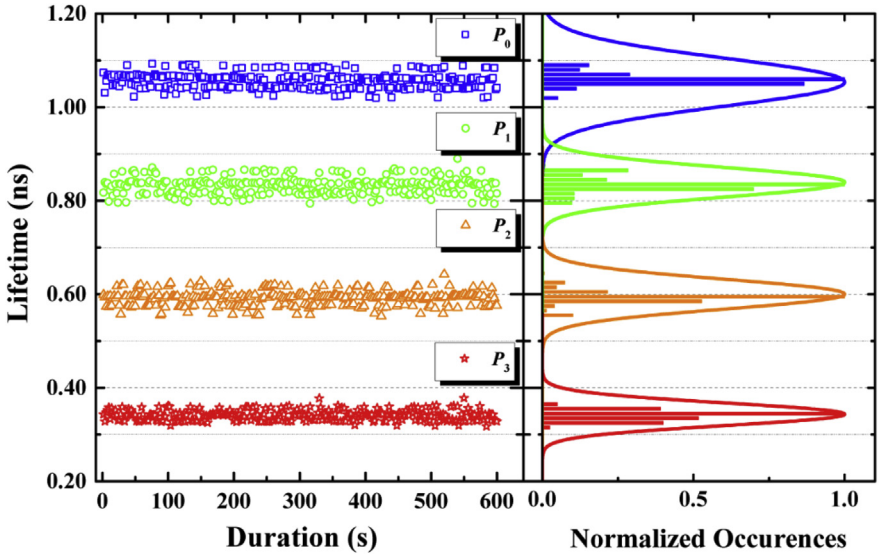

Fig. 5. Fluorescence lifetime trajectories and the corresponding statistics of four encoding areas, which were irradiated by the 405 nm CW laser with the power of P_0 to P_3 and duration of 10 ms, respectively. The Gauss distributions are derived from lifetime distributions of GO film after writing at the power of P_0 to P_3. (A colour version of this figure can be viewed online.)

4. Conclusion

In summary, four-lifetime distributions of GO film with narrow width were successfully achieved by varying the power of writing laser. The distinguishable lifetimes have been used for versatile micropatterning and multimode optical recording. These

micropatterns on GO film based on the modification of lifetime are robust, possessing film-thickness- and reading-power-independent characteristic. The temporal evolution of fluorescence lifetime upon multiple laser reading has been explored by monitoring the lifetime variations of encoding areas during reading process. Negligible change in lifetime of encoding areas after 300 cycles reading indicates the stability and reliability of multimode optical recording based on fluorescence lifetime, as well as the promising applications of our technique in information storage and related optical devices. Although the thermal diffusion effect reduces the spatial resolution, it can be solved by alternating the CW laser to femtosecond laser with low repetition rate and low power. These features enable a mechanism for multimode optical recording on GO film for high-capacity information technologies, and flexible micro-photonic devices.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.carbon.2018.10.046.

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