Enhancement of ultrafast photoluminescence from deformed graphene studied by optical localization microscopy

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Abstract

By using localization techniques, we demonstrated that the morphology of a 2D material in three dimensions can be optically obtained with nanometer precision in z-axis. This technique provides a convenient method to study the correlation between the optical properties and the morphology of 2D materials for the same area. We utilized optical localization microscopy to directly study the correlation between the ultrafast photoluminescence and the morphology of graphene. We observed enhancement of the ultrafast photoluminescence from the deformed graphene. In comparison to the planar graphene, the enhancement factor of ultrafast photoluminescence could be up to several times at the highly curved region. We found that the intensity of photoluminescence from the uniaxially rippled graphene depends on the polarization of excitation light. Furthermore, Raman spectroscopy was used to measure the strain distribution. Pump–probe measurements were conducted to reveal the carrier dynamics. From the experimental results, two mechanisms were confirmed to mainly account for the enhancement of ultrafast photoluminescence from the deformed graphene. One is the deformation-induced strain increases the absorption of graphene. The other is the prolonged carrier relaxation time in the curved graphene.

1. Introduction

There has been increasing interest in strain engineering on 2D materials to control their physical properties [1, 2]. Strain engineering could lead to deformation of 2D materials in three dimensions, and it is important to visualize the morphology. Scanning probe microscopy such as atomic force microscopy and scanning tunneling microscopy is capable of visualizing the morphology with nanometer resolution. However, these techniques would be challenging to image a suspended 2D material in air/vacuum because the probe could vibrate the suspended 2D material in the near field. Electron microscopes also enable to achieve images with nanometer resolution but they acquire the projection of the three-dimensional (3D) objects into 2D images. Compared with the aforementioned method, typical far-field optical microscopy is a convenient method to achieve 3D optical images and has the advantage of directly investigating the correlation between the optical properties and the morphology in the same area of the sample. But the resolution is on the order of 1 μm, restricted by the Abbe diffraction limit.

In this work, we demonstrated to utilize optical localization techniques to optically obtain the 3D morphology of a 2D material with nanometer precision in z-axis. Compared with scanning probe microscopy and electron microscopy, far-field optical microscopy provides a direct and convenient way to study the correlation between the optical properties and the morphology in the same area. We utilized the optical localization microscopy to study the optical properties of the representative 2D material, graphene under strain.
In optical domain of strained graphene, most of investigations focused on Raman scatterings [3–16]. A few reports indicated that the optical absorption response of graphene can be modified by strain [17–21]. But photoluminescence, which is also important for fundamental science and applications in optical domain, has not yet been investigated for strained graphene. Pristine graphene is a semimetallic material with zero bandgap. No observable photoluminescence upon continuous-wave excitation was found in pristine graphene because carriers could fully relax through carrier–carrier scatterings and carrier–phonon scatterings before the relatively slow process of radiative recombination. However upon femtosecond–laser excitation, the energy of electrons and holes subsequently spreads in the conduction and valence bands through carrier–carrier scatterings. And the transient temperature of hot carriers can be up to a few thousand degrees Kelvin [22]. Because radiative recombination process occurs during this process, significant photoluminescence across the entire visible range was found in graphene in recent years [22–29]. Since the process of photon emission lasts only a few hundred femtoseconds [22–24], it is also called ultrafast photoluminescence. In this report, we mapped the ultrafast photoluminescence from deformed graphene in three dimensions. And we directly studied the correlation between the ultrafast photoluminescence and the 3D deformation of graphene for the same area. We found the ultrafast photoluminescence was enhanced due to the deformation–induced strain. The enhancement is attributed to the increase of absorption and the prolonged carrier relaxation time. The finding of photoluminescence enhancement should help to increase the efficiency of light emission in graphene-based ultrafast optoelectronic devices [25, 26].

2. Results and discussions

2.1. 3D morphology of 2D materials obtained by optical localization techniques

We first designed an experimental setup to use a tip with a micron-size apex for deforming a suspended graphene, and observed the corresponding ultrafast photoluminescence by imaging technique. Figure 1(a) shows the schematic of the experimental setup. The graphene monolayer was transferred onto a Ni mesh. Detailed processes can be found elsewhere [30]. Figure 1(b) reveals the optical reflection image of the graphene on the Ni mesh. Because the Ni mesh exhibits periodic square holes with sizes of 50 microns, graphene monolayer is suspended in the square region. As shown in figure 1(a), we could thus use a glass tip to contact and deform the suspended graphene within the square region. Figure 1(c) shows the scanning electron microscope (SEM) image of the glass tip. The size of the tip apex is ≈2 microns. Detailed process of preparing a glass tip can be found in the supplementary material, which is available online at stacks.iop.org/NJP/22/013001/mmedia. Femtosecond laser pulses, centered at 800 nm, were used to excite the suspended graphene. A typical spectrum of the photoluminescence from graphene is shown in figure 1(d). It exhibits the characteristics of thermal emission that the intensity of photoluminescence decreases with decreasing wavelength. The ultrafast photoluminescence was collected by a photomultiplier tube (PMT) as shown in the schematic in figure 1(a). By scanning the laser spot in x–y plane and the objective lens in z-axis, the ultrafast photoluminescence of the suspended graphene was mapped in three dimensions.

Figure 2(a) shows the 3D image of the ultrafast photoluminescence under the situation that the tip apex was close to but did not contact the graphene. The colors indicate the intensity of the ultrafast photoluminescence in 3D space. Although the suspended graphene is supposed to be one-atomic-layer thin in air, it appears like a few microns along z-axis in the 3D images due to the effects of optical diffraction limit. But the colors, corresponding to high intensity such as red and yellow colors, could still roughly reveal the morphology of the suspended graphene. Subsequently, we controlled the tip to approach the suspended graphene in z direction, as illustrated in the schematic in figure 1(a). By mapping the same range of figure 2(a), the 3D image of the ultrafast photoluminescence was obtained as shown in figure 2(b). A hot spot in white color, indicating strong optical intensity, was clearly observed. The intensity of this spot consistently increased when the tip constantly moved toward the suspended graphene until the graphene was broken.

Although we observed the enhancement of photoluminescence at the deformed spot where the glass tip poked from figures 2(a) and (b), the optical images could not clearly sketch the morphology of the suspended graphene. We followed the concept of localization microscopy, which could be used to achieve nanometer resolution in fluorescence microscopy [31] to process the 3D optical images. An important principle of the localization microscopy is that the size of a luminescent object should be on the nanometer scale. Although the point light source will spread into a micron-scaled spot through the microscopic system, the nanometer object can still be located with nanometer precision from the optical image. In our work, graphene is the only luminescent origin and it is atomically thin in z-axis. Similar data processing of localization technique can thus be applied to locate graphene in z-axis. Figures 2(c) and (d) show the 3D morphology images, which are processed from figures 2(a) and (b) respectively following the description in the supplementary material.
Figure 1. (a) The schematic of the experimental setup. (b) The optical reflection image of the graphene on the Ni mesh. (c) The SEM image of the glass tip. (d) A typical spectrum of ultrafast photoluminescence from the graphene.

Figure 2. The 3D images of the ultrafast photoluminescence (a) without and (b) with a glass tip poking the suspended graphene. The red arrow in (b) indicates the enhancement of photoluminescence at the tip location. The 3D morphology of the suspended graphene in (c) and (d) are obtained from (a) and (b) respectively after localization processing.
Figure 2(c) reveals the 3D morphology of the suspended graphene for the area of $8 \times 6.5 \mu m$ when the glass tip is close but does not contact graphene. After the glass tip pokes the suspended graphene upward as shown in figure 2(d), part of the graphene covers the glass tip, and the morphology of graphene reveals the shape of the glass tip. According to figure 2(d), the graphene covers the glass tip around 200 nm in height (z-axis). And the diameter of the tip apex is $\sim 2 \mu m$, which agrees with the SEM image in figure 1(c). It is interesting to notice a cave-like feature at the center of the glass tip in the SEM image. The optical image in figure 2(d) also shows that the height of the graphene at the apex center drops $\sim 100$ nm, implying that the apex shape of the glass tip can be resolved. The agreements between the SEM image and the morphology of graphene processed from the localization technique confirms that the precision to locate the morphology of graphene in z-axis in figure 2(d) is below 100 nm.

2.2. Mechanisms of photoluminescence enhancement in deformed graphene

The correlation between the intensity of the ultrafast photoluminescence (indicated by colors) and the morphology of the graphene can be directly observed in figure 2(d). It shows significant enhancement in intensity around the region where the tip pokes the suspended graphene. It was reported that a gold tip could increase the interaction between graphene and light via the plasmonic effect and reflection [32]. However, these effects should be minimized since we used a transparent glass tip. The 4% reflectivity from the glass may slightly increase the absorption of graphene and enhance the intensity of ultrafast photoluminescence. But it could not account for the significant enhancement. The contact of the glass could also slightly change the Fermi-energy of the graphene. But the doping effect should decrease the intensity of photoluminescence [25], which also cannot explain the enhancement. A possible mechanism is that the deformation-induced strain, due to the contact of the tip, modifies the optical absorption of graphene [17–21]. In figure 2(d), we noticed that the ultrafast photoluminescence enhances at the highly curved region. As a result, the intensity of the ultrafast photoluminescence could be relevant to the strain through the absorption modulation.

The optical conductivity of a uniaxially-strained graphene is expected to be associated with light polarization and the strain amplitude, according to

\[
\sigma_\parallel(\omega) \approx \sigma_0(\omega)[1 + a(1 + \nu)\varepsilon]\quad\text{and}\quad\sigma_\perp(\omega) \approx \sigma_0(\omega)[1 - a(1 + \nu)\varepsilon],
\]

where $\sigma_0(\omega)$ is the isotropic optical conductivity without strain. $a$ and $\nu$ are the numerical constant and the Poisson ratio. $\varepsilon$ is strain. The absorption coefficient $\alpha(\omega)$ follows the relation

\[
\alpha(\omega) \approx \sigma_0(\omega) \cos 2\phi_0, \tag{1}
\]

where $\phi_0$ is the angle between the polarization of the light and the strain direction. According to equation (1), the absorption is maximum when the polarization of light is perpendicular to the direction of uniaxial strain, and is minimum when the polarization is parallel to the direction of uniaxial strain.

Since the modulation of absorption should depend on the angle between the light polarization and the strain direction, we further investigated the ultrafast photoluminescence from rippled graphene with uniaxial strain. Figures 3(a)–(c) show the schematics of preparing uniaxially rippled graphene. First, a polydimethylsiloxane (PDMS) substrate was stretched along one direction. A 1-μm-thick poly(methyl methacrylate) (PMMA) thin film was used to transfer graphene onto the stretching PDMS. After the stretching force was released, graphene on the PMMA membrane (graphene/PMMA) formed into rippled structures. Details procedure can be found elsewhere [33]. The SEM image of figure 3(d) reveals the rippled graphene/PMMA on the PDMS substrate. The ripples are along the same direction that the stretching force applies. Figure 3(e) shows the 3D morphology of the rippled graphene obtained by processing the 3D photoluminescence mapping with the localization technique. We have also measured the rippled PMMA membrane without graphene, which was prepared by the same process in figures 3(a)–(c)), and detected no signals. This confirmed that graphene is the only luminescent material in the samples, which validated the process of localization microscopy in z-axis. The 3D morphology in figure 3(e) clearly shows the rippled structure of graphene, which is confirmed by the SEM images. The colors in figure 3(e) indicate that the intensity of the ultrafast photoluminescence is enhanced in the curved region. A planar graphene on PDMS, which was prepared without stretching force during the process of figures 3(a)–(c), was also measured for comparison. Under the same experimental condition for optical mapping, the 3D morphology of the planar graphene was obtained as shown in figure 3(f). Compared with figures 3(e) and (f), the photon counts are around 10 (indicated by blue color) in the planar region. But the photon counts can be up to 40–70 (indicated by green and red colors) at the highly curved region as shown in figure 3(e).

In order to confirm the enhancement of the ultrafast photoluminescence is associated with the deformation-induced strain through the absorption modulation, dependence of laser polarization was conducted on the uniaxially rippled graphene. We focused the laser spot on the top of a ripple where it exhibited enhanced ultrafast photoluminescence as shown in figure 3(e). The intensity of the photoluminescence was strongest when the polarization was along the $y$-axis and was the weakest when the polarization was along the $x$-axis.
Figure 4(a) shows the spectra of the ultrafast photoluminescence when the laser polarization is perpendicular and parallel to the direction of uniaxial strain (x-axis), respectively. The polarization dependence of the ultrafast photoluminescence agrees with the polarization dependence of the absorption described in equation (1). This supports the argument that high uniaxial strain exists at the highly curved region, which changes the absorption and thus changes the intensity of the ultrafast photoluminescence of graphene. We have also conducted Raman spectroscopy to confirm the strain distribution in the rippled graphene. It was reported that the Raman G band of graphene was broadened and redshifted under uniaxial strain [5]. In addition, the Raman 2D band of graphene was also linearly redshifted with strain [3, 5]. We found both the peak positions of G and 2D bands have redshift in strained region. The spectra of Raman G band around the top and the bottom of a graphene ripple are shown in figure 4(b). Around the top region of a ripple where it is highly curved, the Raman spectra of G band indeed exhibits features of redshift and broadening due to the strain effects. We measured the Raman spectra of G bands at different positions across a ripple of graphene. By fitting all the spectra, the peak values and full-width at half-maximum (FWHM) were obtained as shown in figures 4(c) and (d), respectively. At highly curved region at position ∼20 μm, the FWHM of G band could broaden to 20 cm⁻¹ from 10 cm⁻¹ and the peak value could have redshift up to 20 cm⁻¹ from ∼1590 cm⁻¹.

We further quantitatively estimated the changes of ultrafast photoluminescence due to the strain-induced changes of absorption. The fluence of photoluminescence [22]
where $\tau_{\text{em}}$ is the effective emission time for each laser excitation pulse, $E(h\nu)$ is the emissivity, and $T_{\text{em}}$ is the transient temperature of hot carriers. If we assume that the strain-induced change of absorption only affects $T_{\text{em}}$ in equation (2), the emission fluence should roughly follow the power law of 2.5 of the absorbed fluence [22]. In figure 4(a), the ratio of the emission fluences for both laser polarization is $\sim 3.2$. According to equation (1), the ratio of the absorbed fluence in figure 4(a) can be represented by $[1 + a(1 + \nu)\varepsilon]/[1 - a(1 + \nu)\varepsilon]$. By using $a = 4$ and $\nu = 0.16$ as used in previous report [21], the strain is estimated as $\sim 5\%$ at the measuring point in figure 4(a). We also compare the intensity of the ultrafast photoluminescence from the rippled graphene and the planar graphene. According to figures 3(e) and (f), the enhancement factor could be up to 5 at the highly curved region compared with the planar region. If absorption change is the only mechanism to contribute to the enhancement, it requires strain as large as 20%. We thus suspected that the strain-induced absorption modification should not be the only mechanism to account for the large enhancement we observed. Previous reports indicated that strain could modify the band structure of graphene [34], which should lead to changes of carrier relaxation time [35]. The effective emission time $\tau_{\text{em}}$ in equation (2) is associated with the cooling time of hot carriers since the photoluminescence results from transient thermal emission. Prolonged carrier relaxation (or cooling time) should also increase the effective emission time, leading to enhanced photoluminescence as reflected in equation (2). In order to investigate this effect, we utilized confocal pump–probe microscopy to measure the carrier relaxation of the planar graphene and the highly curved graphene. The FWHM of the optical spot on the graphene was $\sim 500$ nm. Detailed experimental setup can be found elsewhere [36].

The black dotted line in figure 5(a) shows the transient reflection changes of the planar graphene. We used two exponential decay functions to fit the temporal response as shown in blue line. Both the fitting components, the fast decay (in green dashed line) and the slow decay (in red dashed line), are also shown for reference. The procedure of fitting process is described in the supplementary material. The time constants of the fast decay and
slow decay were obtained as $150 \pm 100\,\text{fs}$ and $800 \pm 200\,\text{fs}$, respectively. These results quantitatively agree with previous reports of ultrafast carrier dynamics in graphene [37]. The fast decay is associated with the carrier–carrier scattering process. After excitation of femtosecond pulses, the energy of carriers is subsequently spread into the Fermi-distribution through the carrier–carrier scattering on the timescale of $\sim 100\,\text{fs}$. In our experimental conditions, the transient temperature of the Fermi-distribution is typically 2000–3000 K, enabling thermal emission in the visible range. Following, the temperature of hot carriers decreases through the carrier–phonon scattering process until it reaches equilibrium with the lattice temperature. This cooling process is related to the slow decay. The intensity of the photoluminescence due to the thermal emission temporarily decreases during the cooling dynamics. The effective emission time $\tau_{\text{em}}$ in equation (2) is thus associated with the relaxation time of the cooling process, and is on the order of a few hundred femtoseconds [22]. In contrast, the black dotted line in figure 5(b) shows the transient reflection changes of the highly curved graphene. The relaxation dynamics is apparently slower than that in figure 5(a). The time constants of the fast decay and the slow decay were obtained as $300 \pm 200\,\text{fs}$ and $1.7 \pm 0.3\,\text{ps}$, respectively. We have measured several spots of the curved graphene. Overall, the time constants of the slow decay are within the range of 1 and 2 ps. The inhomogeneity may result from different strain distribution. However, these results evidently reveal that the prolonged carrier relaxation time in the strained graphene, leading to prolonged $\tau_{\text{em}}$ in equation (2), also enhances the ultrafast photoluminescence in addition to the strain-induced increase of absorption.

3. Conclusions

We used the concept of localization microscopy to optically obtain the 3D morphology of graphene with z-precision below 100 nm. This technique can be easily extended to other 2D materials and provides a convenient way to directly study the correlation between the optical properties and the morphology in the same area. By using this technique, we investigated the correlation between the deformation morphology and the ultrafast photoluminescence of graphene. The intensity of the ultrafast photoluminescence of highly deformed graphene is enhanced several times. The enhancement is attributed to the increase of absorption and the prolonged carrier relaxation time.

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