Resonant random laser emission from graphene quantum dots doped dye solution

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Abstract
In this letter, we report on the fabrication of dye doped graphene quantum dots (GQDs) random lasers with resonant feedback that are optically pumped with nanosecond pulses. GQDs, synthesized by the pyrolysis of citric acid, are used as the scattering elements in an ethylene glycol solution of rhodamine B dye. It is experimentally demonstrated that using GQDs instead of titania nanoparticles not only results in the multimode random lasing emission, but also increases the operating lifetime, significantly. We observe discrete lasing modes with subnanometer linewidths at the pump fluences above the threshold. Furthermore, the dependences of the random lasing emission characteristics on the pumping position and concentration of GQDs are investigated, experimentally.

Keywords: random laser, resonant feedback, graphene quantum dot, pyrolysis method

1. Introduction

Random laser (RL) is a research topic of ongoing relevance and importance, especially with regards to the development of new low cost and mirrorless laser technologies [1]. Advances in RLs have led to an emerging desire for developing high precision remote sensors and detectors, lighting devices and imaging systems that are cheaper and simpler [1]. RL was first observed by Lawandy et al in the colloidal solutions of rhodamine 640 perchlorate dye and TiO₂ particles [2]. They demonstrated that light multi-scattering in a random amplifying medium can result in an intensive and narrow-band lasing emission, when the pump energy approaches a threshold. Later in 1999, a multi-mode random lasing emission was reported in ZnO powder [3]. Cao et al explained the multi-mode RL emission based on the recurrent light multi-scattering and the formation of closed loop paths as the random optical cavities [3]. Considering two different optical feedback mechanisms in a disordered amplifying medium, RLs are divided into two categories: RLs with non-resonant feedback and RLs with resonant feedback [4]. In the former category, the light interference effects and phase information are ignored due to the diffusive propagation of light. Hence, the random lasing frequency is mainly determined by the gain line-shape of the active medium. One recent theory suggested that the singled-peak and relatively broad emission spectrum of the RLs with non-resonant feedback is resulted from the overlap of many oscillating lasing modes [5]. On the contrary, in the latter case, the resonant nature of the feedback mechanism is because of the constructive interference and light localization in a strongly scattering medium. As a result, the mode frequencies of the RL emission spectrum are determined from the resonant frequencies of the random cavities.

Recent developments on graphene-based RLs (GRLs) have highlighted the role of graphene in realizing a new class of RLs that are suitable for designing high performance optoelectronic and nanoelectronic devices [6–10]. Graphene, a two dimensional honeycomb lattice of sp² hybridized carbon atoms, is well-known for its fascinating properties that are tunable through chemical doping and applying external...
magnetic or electric fields. In a recent study on GRLs, a highly porous vertical-graphene-nanowalls network was used to multi-scatter the emission light from perovskite nanocrystals and then provide the essential optical feedback for realizing an ultra-low threshold RL [7]. In 2017, an electrically pumped Dirac point induced RL based on a heterostructure graphene/graphene quantum dot (GQD)/graphene device sandwiched between a p-Si/SiO₂ substrate and a polymethylmethacrylate layer was demonstrated which had an ultra-low threshold current density [8]. Also, graphene structures have been used in RLs for tuning and enhancing the RL performance. H Fujiwara et al used graphene flakes to switch and tune a ZnO nanoparticle-based RL, by external white light illumination [9]. In another similar attempt in 2012, a hybrid structure of graphene oxide nanofoils/ZnO nanorods was suggested to enhance the RL action from ZnO nanorods, by using the graphene surface plasmons [10]. The common signature of the RL action in all of these researches is the nonlinear change of the emission intensity and linewidth versus the external pump as well as the appearance of subnanometer spikes in the emission spectrum.

There are, in general, a number of types of papers that have investigated the use of semiconductor QDs (SQDs) as the gain or scattering media in RLs [11–14]. However, the preparation method of SQDs consists of rather complex and sophisticated procedures. On the other hand, there is a demand for developing lower toxic and more photostable QDs due to the increasing advances in related bioimaging and biosensing applications. Hence, QDs, the new and emerging carbon-based QDs, have received considerable attention because of their unique properties and widespread potential applications [15, 16]. They are more photostable, biocompatible and environmentally friendly than conventional SQDs such as CdSe, CdS, CdTe and PbS [17]. Because of their outstanding properties, they can be regarded as a promising alternative of SQDs in many applications such as light emitting diodes, solar cells, bioimaging, biosensing and photocatalysis [17]. While developing GQD-based RLs seems interesting from a practical point of view, using GQDs in RLs has rarely been investigated. Up to our knowledge, there is only one report on RLs fabricated based on GQDs [18]. In ref [18], lasing emission with RL characteristics was reported from a Fabry–Perot cavity containing a mixture of GQDs and TiO₂ nanoparticles as the gain and scattering media, respectively. They also compared GQDs, fabricated by laser ablation method, with the same functionalized carbon dots (C-dots). Regardless of the sophisticated, boring and expensive preparation method, an improved lasing performance and a fivefold enhancement of the optical gain of GQDs over the same functionalized C-dots were demonstrated.

In this letter, GQDs are synthesized by a simple and inexpensive method based on direct pyrolysis of citric acid. It is worth mentioning that using the pyrolysis method provides us with a much better and facile control over the size of the carbon-based products [19]. In addition, the large mass production of the synthesized GQDs, their fast solubility in water and high stability in air at room temperature are other advantages of this method that encourage us to use it for the synthesis of GQDs. Furthermore, the ease of fabrication of GQDs in the pyrolysis method is very attractive from a practical point of view. Very recently, it was shown that one can use the GQDs-functionalized three-dimensional ordered mesoporous ZnO for acetone detection, due to their special structural properties as well as high photostability, low toxicity, bright luminescence and biocompatibility [20]. Also, a similar study on diagnosis of exhalate acetone in diabetes Mellitus was reported, based on the water solution of the synthesized GQDs [21]. Herein, we report on the fabrication of an RL with resonant feedback by adding GQDs into the dye solution. In our proposed structure, GQDs act as the scattering medium in order to provide the optical feedback. Rhodamine B (RhB) dye is also used as the gain medium to provide the optical amplification. We then perform the structural characterization analysis of the synthesized GQDs based on the transmission electron microscope (TEM), dynamic light scattering (DLS), UV–Vis absorption and photoluminescence (PL) measurements. It is demonstrated that the proposed GQDs-dye based RL exhibits a much longer operating lifetimes in comparison with a TiO₂-dye based RL. Furthermore, we observe that discrete resonant lasing modes with sub-nanometer linewidths appear at pump fluences above the threshold. In addition, the dependences of the RL emission spectrum on the excitation position and the concentration of GQDs are studied and showed to be well-consistent with the theory of RLs with resonant feedback.

2. Experimental method

2.1. GQDs synthesis

GQDs were synthesized by the pyrolysis of citric acid [19]. Citric acid was purchased from Merck (monohydrate, ACS, ISO, Reag). In a similar method to [19], we transferred 5.000 g citric acid into a beaker. Then, it was placed on a hot plate and heated to 203 °C at the air temperature. Citric acid melted at the temperature of 136 °C and turned into pale yellow at 161 °C. After 45 min, the beaker contained a dense orange liquid of GQDs. It should be noted that the dense orange liquid of GQDs was very similar to honey in both colour and viscosity. Figure 1(a) exhibits two camera photographs of the synthesized orange liquid of GQDs. However, it hardened and solidified soon after it cooled down. Figure 1(b) depicts the TEM image of the synthesized GQDs. Scale bar is 44.8 nm and we estimate the diameter of GQDs to be 5.93 nm. In order to obtain the size distribution of the synthesized GQDs, we performed a DLS measurement. The DLS size histogram, presented in figure 1(c), reveals the average hydrodynamic diameter of 6.04 nm for GQDs and distribution of their size in the range 5.5–6.5 nm.

The normalized absorbance and PL spectra of the synthesized GQDs are depicted in figure 1(d). The absorbance spectrum of GQDs shows a peak centred at 332 nm and a tail extended to visible region. The sharp fall in the UV–Vis spectrum suggests the absorption edge of 375 nm that corresponds to the optical band gap of approximately 3.3 eV for the synthesized GQDs. The normalized PL spectrum, shown in figure 1(d), was recorded under a 360 nm excitation source. It is inferred...
from figure 1(d) that GQDs mostly emit light at 475 nm under UV excitation. In addition, the normalized absorbance and PL emission spectra (532 nm excitation wavelength) of RhB pristine solution are shown in figure 1(e). It is observed that the RhB absorption and PL spectra are centred at 553 nm and 630 nm, respectively. Thus GQDs are transparent for wavelengths within the emission spectrum of RhB solution and they are suitable for light scattering in RhB based RLs without any light absorption.

2.2. RL experiment

The gain medium was a solution of 8.770 mM RhB dye (sigma Aldrich) dissolved in ethylene glycol (Merck, 99.5% purity). It was prepared after 90 min stirring, by using a magnetic stirrer. To prepare the disordered gain medium composed of dye and GQDs, we added 2 cc solution of the gain medium into the beaker containing GQDs. Then, the solution of RhB dye and GQDs were mixed together under vigorous stirring for about 90 min. It is important to mention here that the stirring process was carried out under gentle heat, in order to prevent the mixture from hardening. The prepared mixed solution with the concentration of 11.90 M GQDs was signed as sample 1. For preparing the RL cell, the prepared solution was then transferred into a cuvette with dimensions of $44.30 \times 17.20 \times 0.05$ mm$^3$.

In RL experiments, we utilized an Nd-YAG pulsed laser that was operating at a pulse width of 10 ns, pulse repetition rate of 10 Hz and the wavelength of 532 nm as the excitation source. The pump light is passed through an aperture diaphragm and is focused on the RL cell by using a cylindrical lens with 15 cm focal length. The dimensions of the excitation stripe on the RL sample were controlled and adjusted by using an aperture diaphragm and were $10.30 \times 0.35$ mm$^2$. An optical fibre was used to collect the RL light, emitting from the sample during the pumping process. Then, the emitted light was guided and coupled to a spectrometer (Ocean Optics, 0.1 nm resolution). Finally, the RL emission spectrum was visualized and analysed, by using a computer.

3. Results and discussion

Figure 2 depicts the emission spectrum corresponding to the sample composed of 8.770 mM RhB and 11.90 M GQDs (sample 1), and that of neat dye solution, at a pump fluence of 114.84 mJ cm$^{-2}$. The broadband emission spectrum of the neat dye solution exhibits a broad PL spectrum with full width at half maximum (FWHM) and the central wavelength of 64 and 598 nm, respectively. On the other hand, several peaks with FWHM less than 1 nm appear in the emission spectrum of sample 1.

Figure 2 indicates that the presence of GQDs in dye solution results in the resonant and multi-mode RL emission. The appearance of these random lasing modes implies the excitation of resonant cavities that have formed inside the pumped region. One can interpret this observation by using the theory of RLs with resonant feedback [22]. Based on this theory, the light multi-scatters by GQDs and finally returns back to a scatterer by which it was scattered before. If the returned light interferes constructively, then a closed loop path is formed. Since the light is confined to this closed loop path and amplified, this closed loop can be regarded as a cavity. There are many such cavities, each with different losses and quality factors, distributed randomly inside the gain medium. The condition of light constructive interference determines the resonant frequencies of the random cavities. On the other hand, during pumping, the confined light is amplified by the gain medium via stimulated emission process. If the optical gain at some resonant frequencies of a special cavity or several cavities exceeds loss, then the lasing oscillation occurs at the corresponding resonant modes.

The lasing behaviour that we observed for sample 1 is further investigated in figure 3. The pump fluence varies from 5.83 mJ cm$^{-2}$ to 223.6 mJ cm$^{-2}$. The nonlinear variation
of the spectrally integrated output intensity versus the pump fluence is a firm proof that demonstrates the lasing action. As it is shown in figure 3(a), the RL threshold can be estimated as 70 mJ cm$^{-2}$. It is noteworthy to mention here that the strong mode competition between the excited resonant modes results in the lowering of the output intensity at high pump fluences above the threshold. It is then responsible for the observed fluctuations in figure 3(a).

Figure 3(b) displays the emission spectra corresponding to the sample 1 at some pump fluences before and after threshold. One can see a broadband PL spectrum at very low pump fluences such as 5.83 mJ cm$^{-2}$. Its corresponding FWHM and central wavelength are approximately 56 and 608 nm, respectively. By increasing the pump fluence, the amount of gain increases which results in the decrease of the linewidth of the emission spectrum. Furthermore, the light amplification enhances when the dwell time of the emission light in the disordered amplifying medium increases. Hence, we observe a relatively narrow component on top of the broadband PL spectrum at the pump fluence of 47.71 mJ cm$^{-2}$. Its FWHM and central wavelength are 11.6 and 611.3 nm, respectively. By further increasing the pump fluence, the penetration depth of the pump light as well as the amount of gain increase. At threshold pump fluence, the increased gain balances with the losses of some random cavities inside the gain medium. As a result, lasing oscillation occurs in the resonant frequencies of the cavities and some very narrow lasing modes appear in the emission spectrum. For example, the third spectrum in figure 3(b) which corresponds to the pump fluence of 92.09 mJ cm$^{-2}$, exhibits the simultaneous oscillation of five lasing modes with FWHM less than 1.3 nm. When the pump fluence is increased further, the resonant cavities with higher losses are also activated. This results in the excitation of more resonant lasing modes and the occurrence of mode competition effect.

In order to investigate the effects of randomness on the RL emission spectrum of sample 1, we change the pumping position on the sample and measure the corresponding emission spectra. The six emission spectra, shown in figure 4, are measured at the same pump fluence of 114.84 mJ cm$^{-2}$ and six different pumping positions. The obtained emission spectra from down to top are attributed to positions 1–6, respectively. They are shifted vertically for better comparison. One can observe that the number of lasing modes and their wavelengths are changed, when the pump light illuminates different positions on the sample. Furthermore, new lasing modes may oscillate and appear in the emission spectrum, by changing the pumping position on the sample. The dependence of the RL emission spectrum on the excitation position is well-explained by the theory of RLs with resonant feedback [22]. Since resonant cavities are distributed randomly inside the gain medium, different cavities are excited when different specific positions on the sample are illuminated in each pumping process. Considering the different quality factors and resonant modes of the cavities, the emission spectrum differs when different positions on the sample are pumped.

To understand the effects of GQDs concentration on RL emission, in figure 5 we present the RL emission spectra of samples 1, 2 and 3 composed of 8.770 mM RhB dye and different GQDs concentrations of 11.90, 16.65 and 21.41 M, respectively, at the constant pump fluence of 237.17 mJ cm$^{-2}$.

It should be noted that the excitation position at which the emission spectra of figure 6 are measured, is different from the six excitation positions that were mentioned in figure 4. It is inferred from figure 5 that the output intensity and the number of lasing modes increase, by increasing the concentration of GQDs. It is because multiple light scattering is increased by increasing the concentration of the scattering elements. As a result, the possibility of light trapping inside the gain medium is enhanced which implies the reduction of cavity losses. In fact, those cavities which have been inactive before in the case of lower scatterer concentrations can now be excited in higher scatterer concentrations. Hence, the number of random resonant cavities and consequently the lasing modes in the RL emission spectrum increase.

The RL threshold is then expected to decrease as the concentration of GQDs (the scattering elements) increases. Figure 6(a) depicts and compares the spectrally integrated output intensity versus the pump fluence for samples 1, 2 and 3. It is clearly observed that the RL threshold decreases, by increasing the concentration of GQDs. The approximate values of the RL threshold corresponding to samples 1, 2 and 3 are 70, 56 and 39 mJ cm$^{-2}$, respectively. Moreover, the emission spectra of samples 3, 2 and 1 corresponding to the pump fluences before and after each respective RL threshold are presented in figures 6(b)–(d), respectively.

In figures 6(b)–(d), one can see that the narrow peaks appear in the emission spectrum for pump fluences well above threshold.

One important question concerns the importance of using GQDs as the scattering centres instead of other types of scatterers that have been diversely investigated so far. To answer this question, we use TiO$_2$ nanoparticles with average size of 90 nm as the scattering medium in the solution of 8.770 mM RhB dye. TiO$_2$ nanoparticles that are among the most used scatterers in the majority of the investigated dye-based RL systems have a high refractive index of 2.6 and a large optical bandgap of 3.2 eV. As shown in figure 7(a), only one lasing peak is observed in the emission spectrum of TiO$_2$-dye based RL at the pump fluence of 92.1 mJ cm$^{-2}$. The corresponding central wavelength and linewidth are 609.3 nm and 6.7 nm, respectively. We perform some investigations on this RL system (the corresponding results are not presented here for brevity) and observe that although the intensity of the single emission peak increases with increasing the concentration of TiO$_2$ nanoparticles, the number of emission peaks in this case does not increases. However, we observe above that using GQDs as the scattering elements in an RL system can lead to the appearance of many emission peaks in the spectrum and resonant RL modes.

Since there are many scattering nanoparticles that can provide multi-mode RL emission [23–26], the resonant feedback is not the only reason that makes the use of GQDs so prominent. Another important issue for practical applications is the photo-stability of the RL sample under long duration excitation. To measure the photo-stability of the TiO$_2$-dye
and GQDs-dye based RLs, we measure the maximum emitted intensity as a function of the laser shots. The measurements are carried out under subsequent pump pulses with repetition rate of 3 Hz and the pump fluence of 92.1 mJ cm\(^{-2}\) that is well above the lasing threshold of the sample 3. Figures 7(b) shows that the emission intensity of TiO\(_2\)-dye based RL undergoes a \(\sim 70\%\) reduction after \(\sim 700\) laser shots. Furthermore, the emission spectra corresponding to the first and 4500th shots are compared in figure 7(c).

Figures 7(d) and (e) show the results of the photo-stability measurements corresponding to the GQDs-dye based RL. Interestingly, we observe no fast reduction of the emission intensity for shot numbers up to \(\sim 3500\). Then, a sudden fall in the emission intensity occurs and the RL emission intensity undergoes a \(\sim 82\%\) reduction after the 4000th laser shot. As shown in figure 7(f), the number of lasing modes varies from shot to shot and the RL modes start to disappear after the 3725th shot. It then seems sound to claim that the GQDs-based resonant RL destroys when the random lasing modes disappear in the emission spectrum.

Although the whole mixture photo-stability is limited by dye not by scatterers, the TiO\(_2\)-assisted photodegradation of dyes is a well-studied subject [27]. The fast exponential decay of the shot-dependent variation of the emission intensity corresponding to the TiO\(_2\)-dye based RL was believed to be caused by a photocatalytic process as a consequence of the two-photon absorption in TiO\(_2\) under visible light illumination [27, 28]. In addition the nanoparticle precipitation and the chemical bonding with the cuvette walls are two other reasons that cause the short-time observations of the TiO\(_2\)-dye based RL properties [28]. Due to the high viscosity of GQDs, they do not precipitate or, at most, they precipitate very slowly at the bottom.
Figure 6. (a) Plots of the spectrally integrated output intensity versus the pump fluence corresponding to the samples 1, 2 and 3. (b), (c) and (d) Emission spectra of samples 3, 2 and 1, respectively at three different pump fluences.

Figure 7. (a) Emission spectra of 0.05 M TiO$_2$ dispersed in 8.770 mM RhB and 21.41 M GQDs doped 8.770 mM RhB (sample 3) at the pump fluence 92.1 mJ cm$^{-2}$, (b) Emitted intensity versus the shot number corresponding to the TiO$_2$-dye based RL, (c) Emission spectra of the TiO$_2$-dye based RL measured at the first and 4500th shots, (d) Emitted intensity versus the shot number corresponding to the GQDs-dye based RL, (e) Emission spectra of the GQDs-dye based RL measured at the first and 4500th shots, and (f) The number of excited modes as a function of the shot number corresponding to the GQDs-dye based RL. The pump fluence is 92.1 mJ/cm$^2$.

of the cuvette. In addition, undoped GQDs show two-photon absorption in the near infrared region, not visible region [29]. This can be another reason for the enhanced photo-stability of the GQDs-dye based RL over TiO$_2$-dye based RL. However, we believe that finding a true reason for this observation needs further experimental investigations on GQDs and is far from the scope of this paper. Since the stability of the lasing modes in each laser shots is very desirable for practical applications, the investigations on stabilizing the lasing modes of the proposed GQDs-dye based RL is of great importance.

4. Conclusion

In summary, we fabricated a dye doped GQDs RL with resonant feedback. It was experimentally demonstrated that using GQDs instead of TiO$_2$ nanoparticles not only increases the RL operating lifetime, but also provides the strong light multi-scattering necessary for the formation of random resonant cavities inside the gain medium and the appearance of discrete lasing modes with sub-nanometer linewidths in the emission spectrum. Furthermore, the dependences of the RL emission
characteristics on the excitation position and the concentration of GQDs were investigated. It was shown that the scattering of light by GQDs provides the necessary optical feedback for realizing random lasing emission. Since using GQDs in RLs is promising for the development of advanced biosensing and bioimaging systems, this work may promote advances in future applications of RLs, specially sensing and imaging applications.

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