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A Hybrid Random Laser using Dye with Self-Organised GaN Nanorods

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Abstract

Self-organized GaN nanorods with population densities ranging between 0.1 and 0.5 μm^{-2} and average heights in the range 1.7 to 16.5 µm, prepared by metal-organic chemical vapor deposition, were used as scattering centres for random lasing by incorporating Rhodamine 6G liquid dye solutions as the gain media. A lasing threshold as low as 11.3 J/cm^2 was obtained from samples with nanorod density above 0.3 μm^{-2}. The threshold depended on the nanorod density and diameter, but not the nanorod height. Lasing emission was observed at multiple angles, a clear indication of random lasing.

Keywords: light confinement, nanotechnology, wide-bandgap semiconductor, random laser

1. Introduction

Lasing in GaN material was first observed at low temperatures from bulk GaN in 1971, with a threshold of 0.3 MW cm^{-2} [1]. Room-temperature lasing was achieved in GaN thin films in the early 1990s, with reports of a threshold of 0.8 MW cm^{-2} [2]–[4]. Evidence of spectral narrowing was later observed in GaN nanowires at a threshold of 700 nJ cm^{-2} [5]. In all these cases, lasing was from Fabry-Perot cavities, where the reflective cavity provides the feedback to support lasing. Random lasing eliminates the requirement for a physical cavity [6]. This opens the possibility of direct probing of analytes, biosensing applications [7] and laser-based sensing [8]. To achieve random lasing in GaN, emission must come from a collection of GaN nanostructures whereby multiple photon random walks within the nanostructures experience sufficient gain before exiting the medium. This remains a challenging problem especially in achieving a low lasing threshold. In addition to challenges with other fabrication methods, metal organic chemical vapor deposition (MOCVD) growth technology offers a more practical, controlled approach for making these nanostructures with higher yield. The purpose of this paper is to observe if GaN nanostructures that are prepared by MOCVD techniques (equivalent to a commercial setup in LED manufacturing companies for example) are effective scatterers for random lasing.

Rhodamine 6G (R6G) dye solution has been utilised as the gain medium for observing random lasing [9]–[13]. These random lasers are ideal for study because they can be optimized by independently controlling the properties of the scatterers or the gain.

Noginov *et al*. demonstrated spectral narrowing of the luminescence with the addition of scatterers to R6G dye solutions [9]. A decreased lasing threshold was observed when the scatterer density was increased in the rhodamine dye solution. Observations of bichromatic emission from random R6G dye lasers were attributed to emission from both dye monomers and dimers [10]. Later work combining dyes in a single medium also produced multiple output wavelengths [12]. Random lasers have been classified according to whether they exhibit incoherent feedback or coherent feedback (in which interference effects are important) [11]. Considering incoherent feedback lasers, colloidal suspensions of nanoparticles in R6G solutions typically exhibit lower random lasing thresholds when operating in the diffusive scattering regime compared to the weakly scattering regime [13].

The work here uses GaN nanorods deposited on a substrate as the scatterers and R6G dye solution as the gain medium. We observe random lasing from self-organized GaN nanorods prepared by MOCVD, with the nanorod density varied from 0.1 to 0.5 μm^{-2}. Since the scattering properties are critical for random lasers, understanding the role of GaN nanorods as scatterers in reducing the lasing threshold offers insights on the most suitable scattering parameters for GaN random lasers.

2. Principles of random lasing

Random lasers are different to conventional lasers due to their multi-directional lasing emission. However, the threshold behavior [14], photon statistics [15]–[17] and relaxation oscillations [18] are similar to conventional lasers. Hence, the standard rate equations can be used to describe the lasing process in a random laser:

$$\frac{dN_1(t)}{dt} = P(t) - \frac{\beta q(t) N_1(t)}{\tau} - \frac{N_1(t)}{\tau}$$

(1)

$$\frac{dq(t)}{dt} = \frac{\beta N_1(t)}{\tau}[q(t)+1] - \frac{q(t)}{\tau_c}$$

where $N_1(t)$ is the number of excited molecules, *P(t)* is the pump rate, *q(t)* the number of photons in laser modes, τ is the spontaneous emission lifetime of the dye, and β is the β-factor for the random laser, the fraction of spontaneous emission that seeds the laser modes [19].The mean value of the cavity decay time, τ_c, can be derived from solving the diffusion equation [20], which gives the photon diffusion coefficient, $D(\lambda)$:

$$\tau_c = \frac{L^2}{8D(\lambda)} \qquad (2)$$

where *L* is the sample size (for optical pumping, this is the illumination area).

The critical size for a sample to achieve lasing is: $L_{cr} = \sqrt{l_g l_t/3}$ where l_g is the gain length (path length when the intensity is amplified exponentially) and l_t is the transport mean free path (the average distance when the scattered light direction is randomized) [21]. The critical size for the sample is a distinctive property of random lasers, due to the interaction between gain and scattering. From equations (1) and (2) it is evident that the threshold and laser process can be controlled by modifying $D(\lambda)$ and hence the transport mean free path, l_t (λ), and L – indicating the importance of scattering. The scattering mean free path is inversely proportional to the scattering cross section, σ_S, of the scatterers. The two conditions for achieving maximum σ_S are: (1) the size of the scatterers is of the order of the wavelength; and (2) the refractive index contrast is high [20]–[22]. In this paper, σ_S is considered when taking into account the filling fraction, FF, calculated as: $FF\ (= \frac{total\ integrated\ rod\ area}{full\ sample\ area})$.

3. Experimental Methods

GaN nanorods were grown on (0001) sapphire substrates in an Aixtron close-coupled showerhead MOCVD system. Details of the growth procedure were discussed in earlier work [22]. Briefly, the growth was initiated with a 10 s initial stage at 1,050 °C where trimethylgallium (13.65 mmol/min) and NH_3 (25 sccm) were introduced. Growth continued for a further 5 mins with a reduced NH_3 flow of 15 sccm with the injection of SiH_4 flow at 200 nmol/min to promote vertical growth. The nanorods were then cooled down under pure nitrogen ambient to prevent from decomposing. The morphology of the nanorods was investigated by field emission scanning electron microscope (FESEM; Nova NanoSEM 450, FEI, Japan). The images were examined further with ImageJ software.

For the lasing experiments, 1 µL of methanol: ethylene glycol (5:1) Rh6G solution (1 x 10^{-3} M) was added to the nanorod samples using a micropipette. The Rh6G dye solution was contained on the substrate by capillarity [23], even when positioned vertically [24].

The random lasers were excited using an Nd:YAG pulsed laser source (532 nm wavelength, 1 Hz repetition rate and 4 ns pulsewidth). The excitation energy was varied by rotating a half-wave plate before a fixed polarizer. A focusing lens (*f* = 20 cm) was used to focus the beam onto the sample that was placed vertically on a holder. The pump spot size was ~ 45 μm on the sample, and the pump beam was directed at 45º to the normal to the substrate surface. Each sample was exposed to the pump light for less than 10 minutes to avoid photo-bleaching of the dye. The fluorescence lifetime of Rh6G in methanol is 3 - 4 ns [25], comparable with the pulse width of the Nd:YAG laser source. The emitted radiation was collected into a multimode optical fiber connected to a fiber-coupled spectrometer (Ocean Optics, USB2000+UV-VS-ES, ~ 1 nm resolution). All measurements were performed at room temperature.

4. Results and Discussion

The morphologies of all samples are shown in the electron micrographs in Figure 1. The nanorods are randomly distributed on the substrates, with the highest nanorod density in sample 1 and 2 and the lowest in sample 5 (Figure 1(a), (b) and (e), respectively). This was independent of the nanorod size. The average nanorod diameter was about 410 nm for sample 1 (Figure 1(a)), followed by sample 2 with diameter of about 450 nm (Figure 1(b)). Sample 3 had the largest diameter of about 990 nm while samples 4 and 5 had rod diameters of 570 and 740 nm, respectively. The rod height was similar for samples 2 and 5 (~ 1.8 µm), increased in samples 1 and 4 (2.4 µm and 4.7 µm) and highest in sample 3 (16.5 µm). The dimensional parameters listed in Table 1 were obtained from analysing electron micrographs using Image J software. For random lasing, the important parameters are the scattering efficiency (effectively the cross-sectional area and refractive index contrast of the scatterers) and the density of scatterers, (number of rods involved in lasing, considering the pumped area and FF). The calculated FF and the number of rods within the pump excitation area for each sample are listed in Table 1.

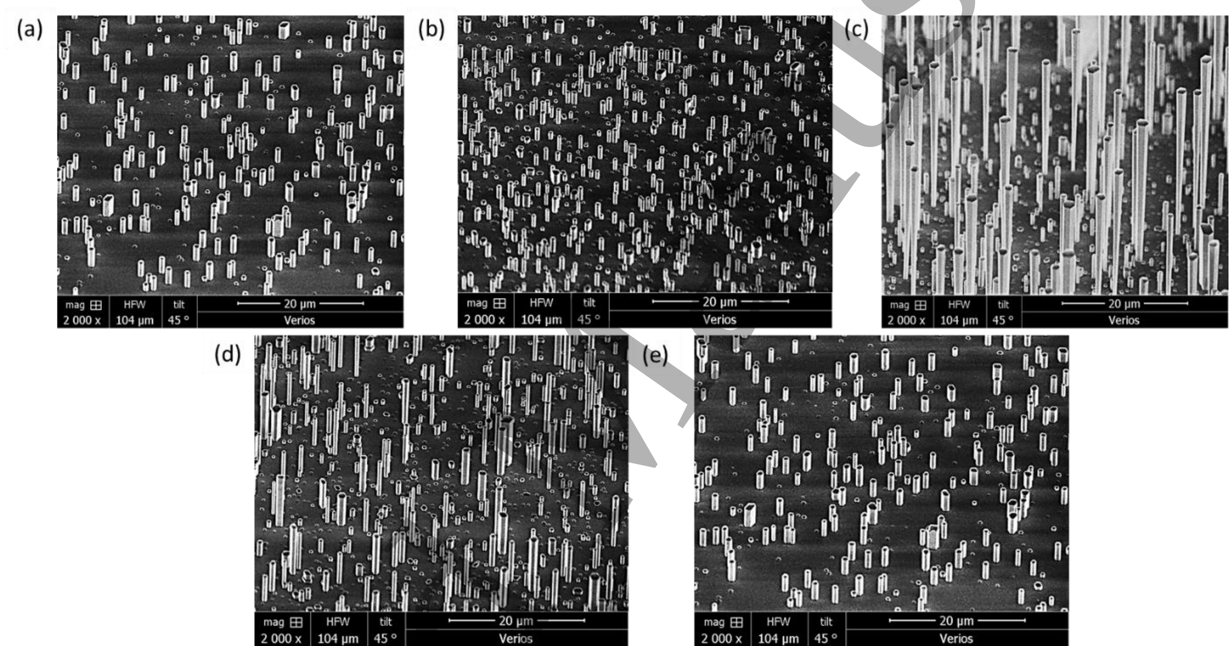

Figure 1: Figure 1(a) - (e): FESEM images of self-organized GaN nanorods with different nanorod densities and heights for samples 1,2,3,4 and 5, respectively at 45 degrees with the scale bar at 20 µm. The diameters of the nanorods range between 410 nm to 990 nm and the heights range between 1.7 µm to 16.5 µm.

Table 1: The average rod diameter, height, rod density, FF, number of rods under illumination and the threshold for observing random lasing for all samples.

Sample	Av. rod density (μm^{-2})	Av. rod diameter (µm)	Av. rod length (µm)	Filling fraction, FF	Number of rods /illuminated area	Laser Threshold (J/cm^2)
1	0.51	0.41	2.4	0.067	816	40
2	0.46	0.45	1.7	0.073	736	15.6
3	0.11	0.99	16.5	0.085	176	15.6
4	0.31	0.57	4.7	0.079	496	11.3
5	0.10	0.74	1.8	0.043	160	-

Random lasing measurements were performed with all samples. Figure 2 (a) shows the setup of the lasing measurements. Since different optical cavities formed by the multiple scattering events induce different output emission directions, measurement of lasing at threshold was done for sample 1 from different collection angles as shown in Figure 2. This provides indirect evidence for random lasing. Conventional lasers do not show multidirectional emission, and a clamping of the sidelight fluorescence from non-oscillating modes is generally observed for conventional lasers. Results in Figure 2(b) show that the lasing emission obtained for sample 1 is multi-directional and no clamping of side emission was observed. No shift in wavelength is observed with angle. Considering the form of the emission spectra, we attribute the lasing emission results to intensity-based feedback rather than field-based feedback [11], [24], [26]. The emission intensity as a function of collection angle, plotted in Figure 2(c), is consistent with random lasing, and suggests that the scattering is random, arising from different scattering paths.

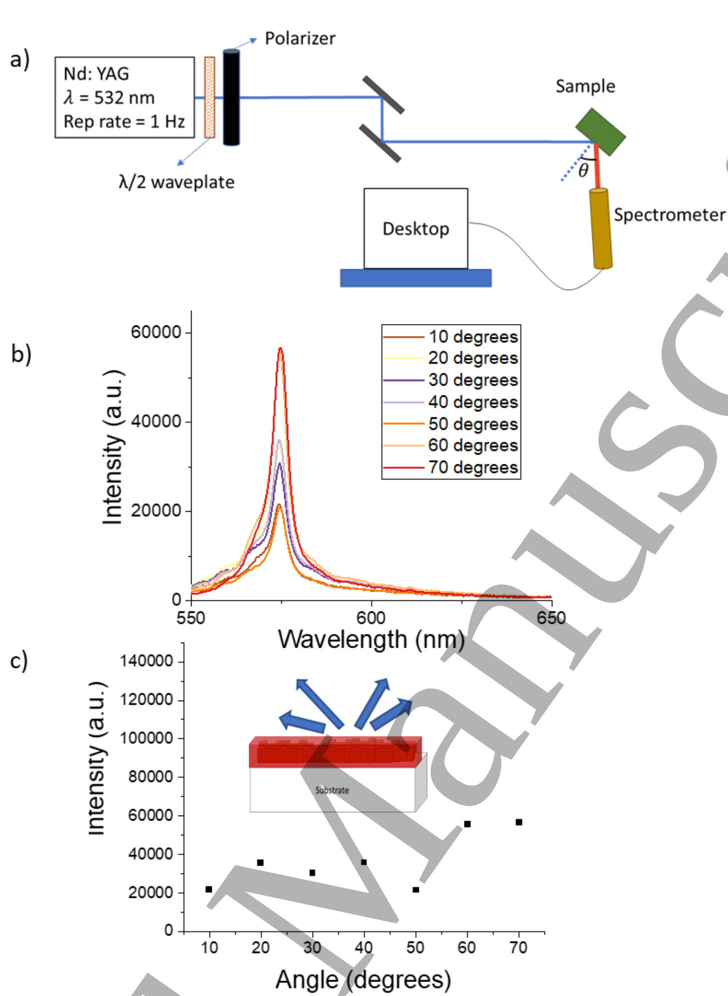

Figure 2 (a) Random lasing measurement setup, (b) and (c) room temperature lasing emission measured at different collection angles from sample 1. The emission collection angle θ is defined with respect to the normal to the substrate. For (c) the intensity is plotted against the angle to further clarify the randomness of the emission when measured at different angles. The insert in (c) illustrates this point.

Significant narrowing of the emission spectral width from 30 to 5 nm with increased pump power was observed in all samples except sample 5, for which a threshold was not apparent. This is most likely due to the low nanorod density and FF in sample 5, as this would increase the photon mean free path between scatterers, resulting in insufficient feedback to support lasing [27]. The dependence of output intensity and spectral width with pump energy for samples 1 to 4 are plotted in Figure 3. The spectral width of the emission from the dye narrows by a factor of 6 at threshold. The lowest threshold energy density is observed from sample 4 at about 11 J/cm^2. Samples 2 and 3 share the same threshold density of 16 J/cm^2 and sample 1 has the highest threshold of 40 J/cm^2.

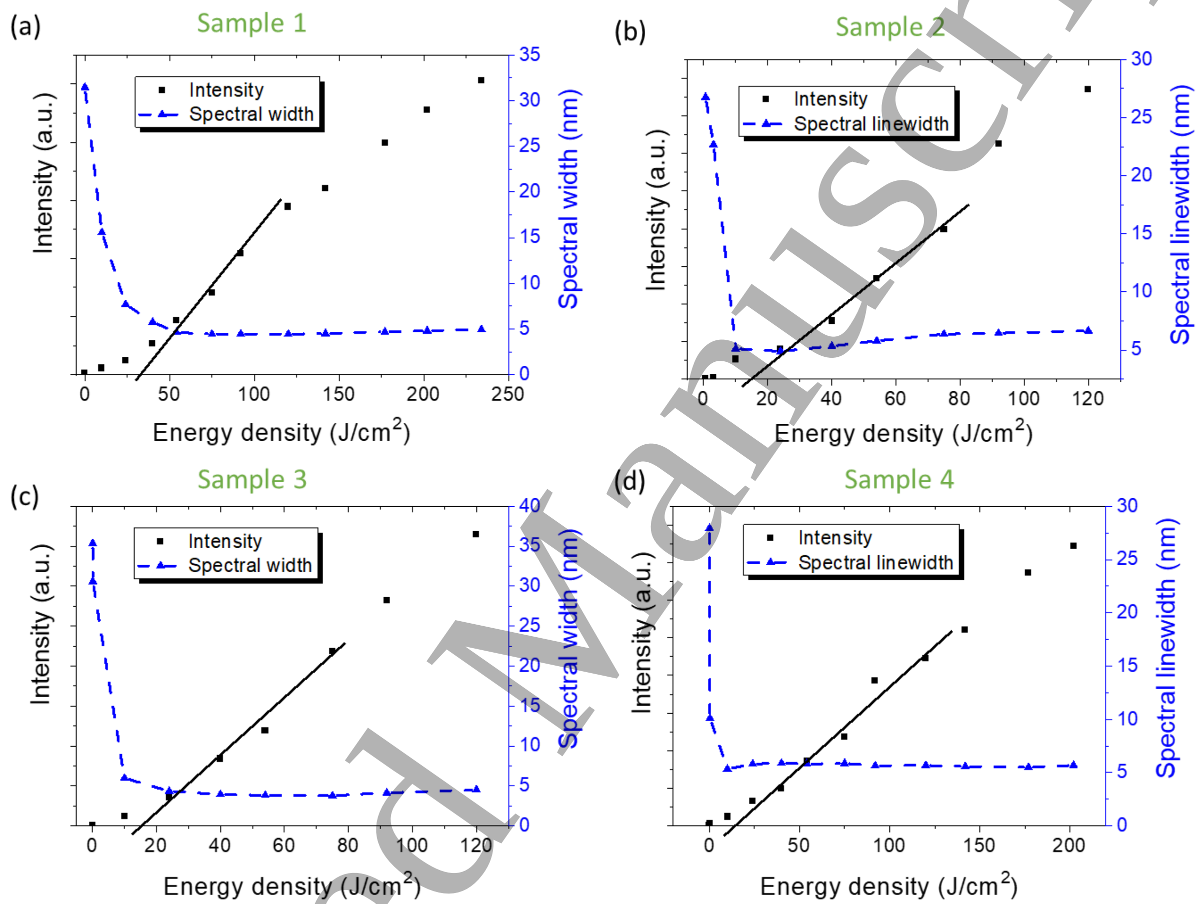

Figure 3: Random lasing intensity and spectral linewidth plotted vs pump energy for samples 1, 2, 3 and 4, respectively. The reduced linewidth corresponding with an increase in lasing intensity indicates the lasing threshold. The dotted lines represent the spectral width and the solid lines are fitting lines to estimate the lasing threshold. Sample 4 achieved the lowest threshold (11.3 J/cm^2) and sample 1 exhibited the highest threshold (40 J/cm^2). A constant pump excitation spot size was used throughout.

Considering the lasing thresholds and properties listed in Table 1, we observe the threshold condition is not greatly influenced by different rod heights. Sample 3, with an average rod height of 17 μm, has the same threshold as sample 2 that has an average rod height of just 1.7 μm. However, samples 2, 3 and 4 do share a similar FF. For sample

5, with FF of 0.043, lasing was not observed. This shows the importance of FF in achieving lasing, that is determined by the total effective cross-sectional area of nanorods within the illuminated area. Figure 4 compares the lasing threshold with the number of rods within the pumped area as well as FF. Sample 4 has almost 500 rods in the pumped area and shows the lowest threshold. Increasing the number of nanorods to about 800 (sample 1) results in an increase in lasing threshold, indicating that the optimal density of scatterers (and scattering efficiency) is important in reducing lasing threshold. Note that for sample 1, FF is slightly lower (0.067) compared to samples 2,3 and 4 and may also be an added factor to an increase in threshold. Lasing in this case is achieved when the photon scattering mean free path is much smaller than the dimensions of the system, i.e., when the photons are undergoing diffusion [28]–[30]. Thus, the transport mean free path, l_t, is an important factor. The number of scatterers as well has how dense the scatterers are influences the transport mean free path of the photons responsible for random lasing and hence modifies the threshold required for a particular random laser. Thus, the scattering efficiency and the pump spot size together govern the random laser modes that are supported, as observed by Bahoura *et al* [31].

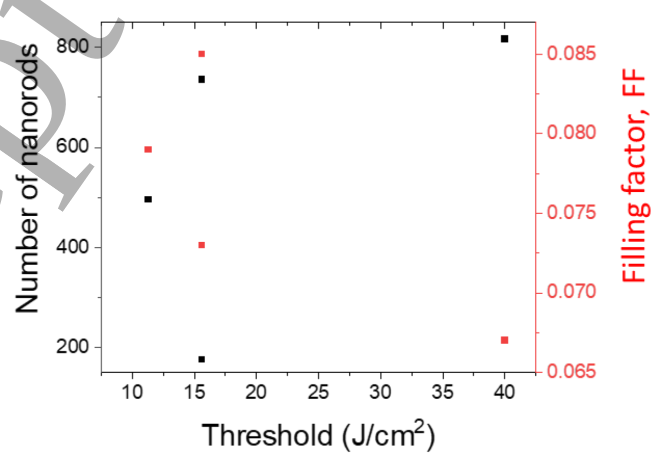

Figure 4: The number of rods involved in achieving the threshold of each sample.

The relationship between random lasing threshold and FF has been investigated in various gain media [32]–[34]. We observed that when the density of scatterers is too small, no lasing emission is observed (scattering too low). However, when the filling fraction is high, the sample needed more pump energy to generate random lasing emission. Low threshold lasing occurs when the pump and emission photons are effectively confined within the gain medium. Improving the sample design (by optimising FF and nanorod density to confine the light within the excitation volume) would therefore lower the random lasing threshold.

Finally, lasing from two different dye volumes was compared. Figure 5 shows the spectra obtained from sample 3 (rod height ~ 16.5 μm) with a higher (Figure 5(a)) and a lower (Figure 5(b)) volume of dye. Interestingly, the results show a lower lasing threshold with a lower volume of dye solution. For random lasing, this is attributed to the importance of the nanorods as scatterers to increase the amplification and feedback [35]–[37]. If the dye solution occupies the volume above the scatterers, as illustrated in the insets of Figure 5, then the light cannot be scattered as effectively, with longer mean free paths, and the feedback is reduced. This result again highlights the importance of an optimum density of scatterers for random lasing. The slight red shift of the emission peak by a few nanometers for the smaller volume of dye is observed in rhodamine dyes [38], since rhodamine molecular dimers typically produce red-shifted emission at higher excitation energy [39].

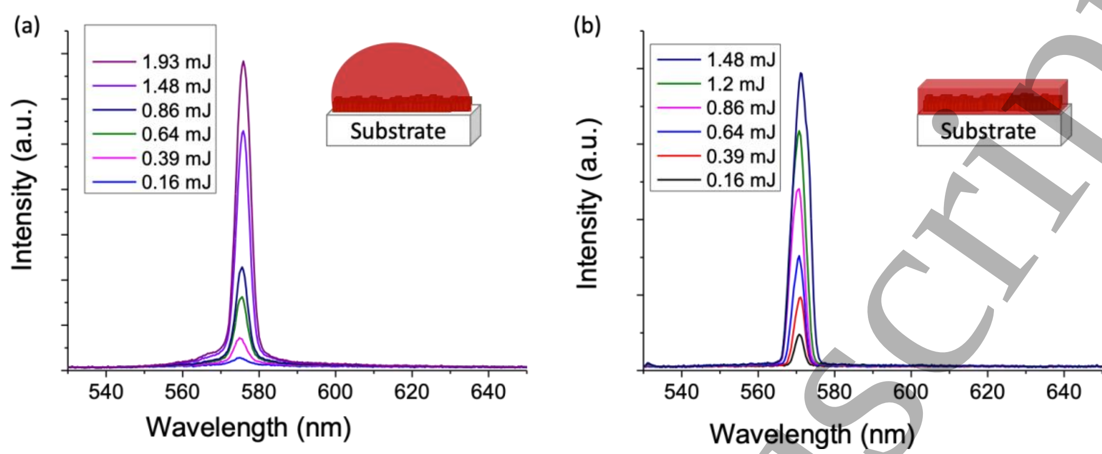

Figure 5: Comparison of sample 3 with (a) thicker dye and (b) thinner dye on GaN nanorods. Reducing the volume of dye reduces the lasing threshold due to reduced dimer formation and increased scattering feedback from the nanorods.

Conclusion

Random lasing from hybrid dye solution on-GaN nanorods reveals the dependence of the lasing threshold on nanorod density, rod size and filling fraction. The lowest pump threshold was observed for sample 4 at about 11.3 J/cm^2. The number of nanorods involved in effective random lasing was about 496 rods with a filling fraction of 0.079 and an average nanorod density of 3.1 x 10^7 rods/cm^2. Increasing the nanorod density further, resulted in an increase in the number of rods involved in scattering, which increased the lasing threshold. An increased lasing threshold was observed for excess volumes of dye, attributed to reduced scattering feedback from the nanorods and the contribution of dimer emission. The density of rods has been shown to affect lasing threshold therefore suggesting further investigation of the effect of scattering mean free path on the lasing threshold towards a more comprehensive understanding of the lasing behavior.

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