Graphene slurry based passive Q-switcher in erbium doped fiber laser

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ABSTRACT

In this work, a Graphene slurry based passive Q-switcher fabricated from Graphene-Polylactic acid (PLA) filament which is used for 3D printing. To produce the Graphene slurry, the diameter of the filament was reduced and Tetrahydrofuran (THF) was used to dissolve the PLA. The Graphene-THF suspension was drop cast to the end of a fiber ferrule and the THF then evaporated to develop Graphene slurry based SA which is integrated in fiber laser cavity. At threshold input pump power of 30.45 mW, a Q-switched Erbium-doped fiber laser (EDFL) can be observed with the wavelength centered at 1531.01 nm and this remained stable up to a pump power of 179.5 mW. As the pump power was increased gradually, an increase in the repetition rates was recorded from 42 kHz to 125 kHz, while the pulse width was reduced to 2.58 µs from 6.74 µs. The Q-switched laser yielded a maximum pulse energy and peak power of 11.68 nJ and 4.16 mW, respectively. The proposed Graphene slurry based saturable absorber also produced a signal-to-noise ratio of 44 dB indicating a stable Q-switched pulsed laser.

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1. INTRODUCTION

With the rapid growth of industrial nations in todays' world, technological advantages of ultrafast fiber lasers seemed attractive as an option not only in comprehensive industrials, but also in scientific applications. Since it is first reported in 2009 [1], Graphene has been put to practical and effective use of as saturable absorber in generating fiber lasers, may it be in Q-switching or mode-locking regime [2-5]. This is contributed by its own inherent properties of having ultra-broadband resonated nonlinear optical response as well as ultrafast relaxation time among many other appealing qualities of a good saturable absorber [6].

Q-switching operation in pulsed laser generation using Graphene can be realized by several synthesization approaches including liquid phase exfoliation (LPE), chemical vapour deposition (CVD), reduced Graphene Oxide (rGO), micro-mechanical cleavage [7] and by electrochemical exfoliation technique [8]. Some of these techniques are rather complicated and expensive to implement and others' physical morphology cannot be put into controlled settings. Zhao et al. [9] reported on the fabrication of Graphene-based SA using the optical deposition method using Graphene/Dimethylformamide (DMF) solution. The procedures was further explained and varied by Martinez et al. [1]. Reduced Graphene Oxide

was proven to have higher modulation depth and lower non-saturable losses at the expend of a larger scale and higher cost of production as compared to simply using Graphene Oxide. The results parameters obtained from the two Graphene based SAs were also quite similar as the rGO does not shows much of laser performance improvements [10].

The production of thin film based SAs Graphene thin film is produced by Zhang et al. [11] using chemical vapor deposition (CVD) method where a gaseous carbon source is decomposed and the film is grown from the substrate [12]. As reported by [11], the process of developing the Graphene thin film is found to be intricate and extensive. In addition, the integration of the SA in the laser cavity is dependent on the approaches mentioned earlier and it includes free space alignment, sandwiching, and evanescent field interactions among others.

Recently, blackmagic3d (www.blackmagic3d.com) has introduces the conductive Graphene-Polylactic acid (PLA) filament with diameter of 1.75 mm. 3D printing technology since its first invention in the 1980s have been commonly used by companies for the production of conceptual prototypes upon producing the products in mass quantities [13-14]. The printer works by depositing a material such as plastic or metal layer by layer to produce 3 dimensional objects, following the instruction set from the digital designs from computer aided design files (CAD). Graphene is often used as the additive in such material application due to the enhancement it can provide for the mechanical flexibility and robustness of the material used [15]. Though the optical properties are often looked upon in determining the suitable saturable absorber of a pulsed laser, the thermal resilience of a material is as important in order for the pulsed laser to achieve its maximum possible performance. This is because a temperature too high can damage the saturable absorber before it manage to achieve full saturable absorption during pulsed laser generation. As mentioned before, Graphene is superior in terms of its optical properties such as its zero energy band gap, ultrawide spectral range, and ultrafast recovery time as compared to other conventional materials with a further advantage of having high thermal conductivity [16].

In this work, we introduce a new approach in preparing Graphene based saturable absorber for pulsed fiber laser generation by using conductive Graphene-Polylactic acid (PLA) filament as the starting material to develop a Graphene slurry based passive Q-switcher. Graphene as saturable absorber (SA) has been proven excellence for fabrication [9] as high potential medium [17] for generating nonlinear optical behavior. Possessing quite a few advantages over other conventional material for SA such as wavelength-independent saturable absorption characteristics [18], low saturation absorption threshold and large modulation depth [9], as well as ultrafast recovery time and wider operation spectral range [19]. Thus, in this paper, a simple way of using Graphene as SA is demonstrated and the resulting laser performance is reported.

2. RESEARCH METHOD

The saturable absorber is prepared by first extruding the Graphene-PLA filament through a 3D printer nozzle of diameter 0.4 mm at 210° C in order to reduce its diameter to 200μ m. This makes for an easier mixing process. The extruded filament is then weighed to about 25 mg and mixed with 1 ml of Tetrahydrofuran (THF) which helped to dissolve the PLA while ultrasonicated for 10 minutes to produce a Graphene-THF suspension. The SA is incorporated in the laser cavity by dropping the suspension onto one end of a fiber ferrule and let dry for a few seconds to leave the Graphene slurry behind as the THF evaporated.

Figure 1(a) shows the Graphene slurry attached to the end of fiber ferrule after THF evaporated at ambient temperature. Figure 1(b) is the FESEM image that shows the morphology of the Graphene slurry. Figure 2 is the Raman spectroscopy of the Graphene slurry with silicon substrate. The distinct peaks of Raman shift for D band, G band and 2D band are observed at 1348 cm⁻¹, 1582 cm⁻¹ and 2699 cm⁻¹, respectively. The Raman spectroscopy also revealed the Intensity of G peak and 2D peak which is at 389 and 287, respectively with the ratio of I(G)/I(2D) was about 1.35 indicating that the sample is a multi-layer Graphene with the number of Graphene layer (nGL) of around 25 layer [17] A peak is also observed at 1453 cm⁻¹ due to the glass slide.

The experimental setup of the Q-switched Erbium-doped fiber laser (EDFL) is as shown in Figure 3. The SA used was the newly fabricated Graphene slurry which is integrated by dropping the Graphene-THF suspension onto one end of a fiber ferrule and let dry before it is connected to the cavity with a fiber connector. Due to the Graphene slurry passive Q-switcher integration into the cavity, an insertion loss of 3 dB was measured. A 1m long Erbium-doped fiber (EDF) is used as the gain medium, connected to a 980/1550 nm wavelength division multiplexer (WDM), an isolator to ensure no backward propagation of light in the cavity, and a 95/5 coupler attached together in a ring-configured setup. The EDF's core and cladding diameter is 8 μ m and 125 μ m, respectively, with a characterized numerical aperture (NA) and

Erbium ion absorptions of 0.16 as well as 45 dB/m at 980 nm and 80 dB/m at 1530 nm, respectively. A laser source of 980 nm is used to pump the whole cavity via the WDM. The output coupler of 95/5 is used to tap out 5% of the oscillating light for output characterization through a 3 dB coupler for simultaneous observations on the optical spectrum analyzer (OSA)/oscilloscope (OSC) and optical power meter (OPM)/Radio frequency spectrum analyzer (RFSA). Important to note that the optical spectrum analyzer (OSA)(Yokogawa AQ6370B) used have a spectral resolution of 0.05 nm and a 460 kHz bandwidth photo-detector (Thor lab, Thorlab DET01CFC) is used to observe the pulse train on the oscilloscope (GW Instek). SMF-28 single-mode fiber made up the rest of the cavity and all the components used are polarization dependent such that they support any light polarization. There is no significant pulse jitter observed through oscilloscope during the experiment.



Figure 1. (a) Graphene slurry at the end of fiber ferrule, (b) FESEM image of Graphene slurry



Figure 2. Raman spectroscopy of Graphene slurry



Figure 3. Integration of Graphene slurry based SA in fiber laser in ring cavity; OSA-optical spectrum analyzer, RFSA-Radio frequency spectrum analyzer, OSC-oscilloscope, OPM-optical power meter

3. RESULTS AND ANALYSIS

Figure 4 shows a typical Optical Spectrum Analyzer (OSA) with and without the Graphene slurry based SA. As observed, the operating wavelength was shifted from longer wavelength at 1565.77 nm without Graphene slurry based SA with output power of -24.4 dBm to 1531.27 nm at -40.02 dBm, respectively when the Graphene slurry based SA integrated in the laser cavity. The OSA trace without Graphene slurry based SA also contain several peaks compare to the OSA trace with the SA and this shows that the proposed SA could suppressed the ambiguous peaks during pulse oscillation. Spectral broadening is also observed with 3 dB bandwidth of around 2 nm. The observed shifted central wavelength and spectral broadening is due to induced losses and intensity dependent of the fabricated SA, which passes light once the SA is fully saturated.



Figure 4. OSA trace with and without Graphene slurry based SA

Figure 5 shows pulse train of the generated pulse with the respective pulse width at input pump power of (a) 30 .4 mW (b) 100 mW and (c) 179.4 mW. The pulse trains shows a consistent peak from the threshold input pump power of 30.4 mW to the maximum input pump power of 179.4 mW. At 100 mW, a stable pulse train with a pulse width of $3.78 \ \mu s$ is also observed. No distinct fluctuations of the generated pulse are observed during the laser generation. With the uniform recorded pulse shape as shown in the figure, it is safe to say that there is no self-mode locking of the pulse and no pulse jitter effects can be detected.



Figure 5. Pulse train and pulse width at input pump power, (a) 30 .4 mW, (b) 100 mW, and (c) 179.4 mW

Figure 6 shows the relation of generated repetition rate and pulse width with the function of input pump power. As the input pump power tuned from 30.4 mW to 179.4 mW, the repetition rate increases from 42 kHz to 125 kHz. The maximum input power and the range of repetition rates that could be operated by the Graphene slurry is higher than other reported works [20-23]. Meanwhile, the temporal pulse width is reduced from 6.7 µs to the shortest pulse width of 2.58 µs. The shortest pulse width generated is 2.58 µs, shorter than the one reported by Aziz et al. and Yap et al. [22, 23] and comparable to other works [3, 9, 19]. The input pump power is proportional to the generated repetition rates and inversely proportional to the generated pulse width. This behavior is a typical characteristic of Q-switching operation, where the pulse repetition rate changes with the pump power [23, 24] and to reduce the pulse width, higher repetition rates should be attained. The minimum input pump power of 30.4 mW required to generate Q-switched pulse is lower than other reported works by Popa et al. [3] with 74 mW, Cao et al. [19] with 33 mW, Sobon et al. [10] with 120 mW, Muhammad et al. [25] with 65.29 mW, and Ahmad et al. [26] with 39 mW. The input threshold pump power could be control by different weight ratio of the reduced diameter of Graphene-PLA in THF. The working range of the input pump power is larger than reported by other works [22, 24].

Figure 7 depicts the relation of the of the instantaneous peak power and pulse energy with increasing pump power. From Figure 7, it can be seen that the instantaneous peak power increase from 0.7 mW to 4.16 mW and the same increasing trend is also observed for calculated pulse energy that increases from 5 nJ to 11.6 nJ. The calculated instantaneous peak power shows low fluctuations between pump power increment and the same goes to the calculated pulse energy. A signal-to-noise ratio of 44 dB is recorded using an Radio frequency spectrum analyser (RFSA) at the repetition rate of 87.8 kHz indicting that the pulse produces is stable, as shown in Figure 8. During the experimental works, we observed that the SNR for each repetition rate is producing SNR at the same value and that indicates the generated pulse is stable with low fluctuations. The recorded SNR is comparable with the Graphene polymer composites based SA [3].



Figure 6. Pulse repetition rate and pulse width versus pump power

Figure 7. Peak power and pulse energy versus pump power



Figure 8. RFSA measurement of signal to noise ratio Graphene slurry based SA of 44 dB with 500 kHz span

4. CONCLUSION

We have successfully demonstrated a passively Q-switched Erbium doped fiber laser around 1.5 µm using Graphene slurry based passive saturable absorber. The Graphene slurry based saturable absorber was fabricated by dissolving commercial Graphene-PLA filament in THF, and then drop casted to the end of fiber ferrule and integrated in erbium-doped fiber laser (EDFL) by mating the deposited fiber ferrule with another clean fiber ferrule via FC connector. As the pump power is tuned over a wide range, from 30.45 mW to 179.5

mW, a stable passively Q-switched EDFL centered at 1531.27 nm is observed with tunable repetition rate in the range of 42 kHz to 125 kHz with the shortest pulse width of 2.58µs. The generated pulsed produced maximum pulse energy, maximum peak power and signal to noise ratio of 11.68 nJ 4.16 mW and 44 dB, respectively.

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REFERENCES

- Q. Bao, H. Zhang, Y. Wang, Z. Ni, Y. Yan, Z. X. Shen, K. P. Loh, and D. Y. Tang, "Atomic-Layer Graphene as a Saturable Absorber for Ultrafast Pulsed Lasers," *Adv. Funct. Mater.*, vol. 19, pp. 3077-308, 2009.
- [2] M. B. S. Sabran, M. A. Ismail, S. Azooz, H. Ahmad, A. Z. Zulkifli, and S. W. Harun. "Q-switching and mode-locking pulse generation with graphene oxide paper-based saturable absorber," *The Journal of Engineering*, vol. 2014, pp. 1-7, 2015.
- [3] D. Popa, Z. Sun, T. Hasan, F. Torrisi, F. Wang, and A. C. Ferrari, "Graphene Q-switched, tunablefiber laser," *Appl. Phys. Lett.*, vol. 98, pp. 073106, 2011.
- [4] H.H. Liu, K.K. Chow, S. Yamashita, S.Y. Set, "Carbon-nanotube-based passively Q-switched fiber laser for high energy pulse generation," *Optics & Laser Technology*, vol. 45, pp. 713–716, 2013.
- [5] H. Ahmad, S. N. Aidit, S. I. Ooi, > Rezayi, and Z. C. Tiu. "Passively Q-switched and mode-locked erbium doped fiber laser based on n-doped graphene saturable absorber," *Laser Physics*, vol. 27(10), pp. 105302, 2018.
- [6] M. K. Kavitha and J. Manu. "Graphene: a review of optical properties and photonic applications," Asian journal of Physics, Vol. 25(7), pp. 809-831, 2016.
- [7] Z. Sun, T. Hasan, and A.C. Ferrari, "Ultrafast lasers mode-locked by nanotubes and Graphene," *Physica* E, vol. 44, pp. 1082, 2012.
- [8] P. Yu, S. Lowe, G. Simon, and Y. Zhong, "Electrochemical exfoliation of graphite and production of functional Graphene," *Current Opinion In Colloid & Interface Science*, vol. 20(5-6), pp. 329-338, 2015.
- [9] N. Zhao, M. Liu, H. Liu, X. Zheng, Q. Ning and A. Luo, "Dual-wavelength rectangular pulse Yb-doped fiber laser using a microfiber-based Graphene saturable absorber," *Opt. Express*, vol. 22(9), pp. 10906, 2014.
- [10] G. Sobon et al., "Graphene Oxide vs. Reduced Graphene Oxide as saturable absorbers for Er-doped passively mode-locked fiber laser," *Optics Express*, vol. 20(17), pp. 19463, 2012.
- [11] L. Q. Zhang, Z. Zhuo, J. X. Wang, and Y. Z. Wang, "Passively Q-switched Fiber Laser Based on Graphene Saturable Absorber," *Laser Physics*, vol. 22 (2), pp. 433–436, 2012.
- [12] R. Vajtai, Springer handbook of nanomaterials. 1st ed. Berlin HeidelBerg: Springer-Verlag; 2013.
- [13] S. Leigh, R. Bradley, C. Purssell, D. Billson and D. Hutchins, "A Simple, Low-Cost Conductive Composite Material for 3D Printing of Electronic Sensors," *PLoS ONE*, vol. 7(11), pp. 49365, 2012.
- [14] C. Schubert, M. van Langeveld and, L. Donoso, "Innovations in 3D printing: a 3D overview from optics to organs," *British Journal of Ophthalmology*, vol. 98(2), pp.159-161, 2013.
- [15] E. García-Tuñón et al., "Graphene Oxide: An All-in-One Processing Additive for 3D Printing," ACS Appl. Mater. Interfaces, vol. 9(38), pp. 32977–32989, 2017.
- [16] E. Pop, V. Varshney and A. K. Roy, "Thermal properties of graphene: Fundamentals and applications," *MRS Bull.*, vol. 37, pp. 1273, 2012.
- [17] H. Ahmad et al., "Highly Stable Graphene-Assisted Tunable Dual-Wavelength Erbium-Doped Fiber Laser". *Appl. Opt.*, vol. 52 (4), pp. 818, 2013.
- [18] P. L. Huang, W. L. Chen, T. W. Peng, C. Y. Su, C. Y. Yeh, W. H. Cheng. "Investigation of saturable and reverse saturable absorptions for graphene by Z-scan technique," *IEEE Photonics Technology Letters*, vol. 27(17), pp. 1791-1794, 2015.
- [19] Z. Wang, Y. Chen, C. Zhao, H. Zhang and S. Wen, "Switchable Dual-Wavelength Synchronously Q-Switched Erbium-Doped Fiber Laser Based on Graphene Saturable Absorber," *IEEE Photonics Journal*, vol. 4(3), pp. 869-87, 2012.
- [20] N. A. Awang, N. S. Aziz, A. N. Azmi, F. S. Hadi, and Z. Zakaria. "Experimental and numerical comparison Q-switched fiber laser generation using graphene as saturable absorber," *MATEC Web of Conferences*, vol. 150, pp. 01009, 2018.
- [21] W. J. Cao, H. Y. Wang, A. P. Luo, Z. C. Luo and W. C. Xu, "Graphene based, 50 nm wide-band tunable passively Q-switched fiber laser," *Laser Physics Letters*, vol. 9(1), pp. 54-58, 2012.
- [22] Y. K. Yap, N. M. Huang, S. W. Harun, H. Ahmad, "Graphene Oxide-Based Q-Switched Erbium-Doped Fiber Laser," Chin. Phys. Lett., 30(2), 024208, 2013.
- [23] N. A. Aziz, Z. Jusoh, M. Q. Loman, M. Yasin, E. Hanafi, and S. W. Harun. "Q-switched erbium-doped fiber laser with graphene oxide embedded in PMMA film," *Digest Journal of Nanomaterials and Biostructures*, vol. 12(2), pp. 325-330, 2017.

- [24] G. Sobon et al., "Linearly polarized, Q-switched Er-doped fiber laser based on reduced Graphene oxide saturable absorber," *Appl. Phys. Lett.*, vol. 101, pp. 241106, 2012.
- [25] F.D. Muhammad, M. Z. Zulkifli and H. Ahmad, "Graphene based Q-switched tunable S-band fiber laser incorporating arrayed waveguide gratings (AWG)," *Journal of Nonlinear Optical Physics & Materials*, vol. 23(1), pp. 1450004, 2014.
- [26] H. Ahmad, M. Z. Zulkifli, F. D. Muhammad, A. Z. Zulkifli, and S. W. Harun, "Tunable Graphene-based Q-switched erbium-doped fiber laser using fiber Bragg grating," *Journal of Modern Optics*, vol. 60(3), pp. 202-212, 2013.

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