

Zinc oxide nanoparticles based passive saturable absorber for pulse generation in fiber laser

Nurul Alina Afifi Norizan^{*1}, Fauzan Ahmad², Muhammad Quisar Lokman³,
Sulaiman Wadi Harun⁴

^{1,2,3}Malaysia-Japan International Institute of Technology
Universiti Teknologi Malaysia, 54100 Kuala Lumpur, Malaysia

⁴Department of Electrical Engineering, Faculty of Engineering
University of Malaya, 50603 Kuala Lumpur, Malaysia

*Corresponding author, e-mail: alinaafifi@gmail.com

Abstract

A stable passive Q-switched pulsed generation in Erbium doped fiber laser by Zinc Oxide nanoparticles embedded in polyvinyl alcohol (ZnONP-PVA)-based saturable absorber is demonstrated in this paper. The surface morphology and thickness profile of the fabricated film were observed using FESEM and 3D measuring laser microscope with the measured thickness of 12 μm . Meanwhile the its optical properties is characterized using Raman spectroscopy. The developed ZnONP-PVA film, has modulation depth of 7.8% and intensity saturation of 88.97 MW/cm². The threshold input pump power to generate Q-switched pulse is at 45.4 mW and can be tuned until 92.4 mW before the pulse diminished. The operating wavelength of generated pulse is at 1535 nm with 3 dB bandwidth approximately of 2 nm with exclusion of parasitic continuous wave lasing. As the input pump power was tuned from threshold to maximum value, the recorded pulse train of repetition rate is tunable from 73.53 kHz to 103.10 kHz while the pulse width decreases from 6.8 μs to 4.8 μs . The calculated maximum output power and pulse energy at maximum input pump power was 5.14 mW and 49.85 nJ, respectively. The measured signal to noise ratio was 56 dB indicated that the generated pulse by ZnO NP based passive saturable absorber was stable.

Keywords: fiber laser, pulse, zinc oxide nanoparticles

Copyright © 2019 Universitas Ahmad Dahlan. All rights reserved.

1. Introduction

Pulsed laser technology have been received much attention in various fields including medical and optoelectronics field, due to their advantages [1]. Q-switching is one of the techniques in achieving pulsed laser generation. This technique helps to modulate the intracavity losses and thus form pulses [2]. Switching happens when the gain saturates after achieved a maximum energy which then switched from high states to lows states in form of short and intense pulse. In actively Q-switched fiber lasers, cavity loss is modulated by an active control element whereas in passively Q-switched fiber lasers, cavity loss is modulated by saturable absorber (SA).

Saturable absorber is commonly used by researchers nowadays to generate pulse laser. The study in using nanomaterial based passive SA was evolving from carbon nanotubes [3], graphene [4], transition metal dichalcogenides (TMDs), black phosphorus (BP) [5] and topological insulators (TIs) [6, 7] and most recently Zinc oxide (ZnO) [8-11]. These materials are promising in the world of fiber laser. However some materials have drawbacks to it such as Semiconductor saturable absorber mirror (SESAM) has low damage threshold, limited operation wave band and narrow spectrum response as well as complex and expensive fabrication [12, 13]. Meanwhile graphene has the absence of band gap, low absorption coefficient and weak optical absorption. carbon nanotubes based SA have to tolerate with lower damage threshold, lack reliability and less stable. For TI, it suffers indirect band gap in insulating bulk state meanwhile TMD has a thickness dependent bang gap [14]. Therefore, based on the downsides of the previous materials as SA, this paper will demonstrate a stable passive Q-switched pulsed generation from Erbium doped fiber laser by ZnONP embedded in PVA-based SA.

Zinc Oxide (ZnO) is a group II-IV compound semiconductor, with its covalence on the borderline between ionic and covalent semiconductors. ZnO has a hexagonal wurtzite structure

that exhibits partial polar. ZnO has unique characteristics such as large excitation binding energy of 60 meV and wide band gap of 3.37 eV at room temperature [15]. Advantages associated with wider bandgap include higher breakdown voltages, lower noise generation, ability to sustain large electric fields, high power operation and high temperature properties [16]. These advantages made ZnO are widely used mainly in optoelectronic devices. ZnO has high third-order nonlinear coefficient and when detailed third-order nonlinearity measurement was performed using single-beam z-scan technique, ZnO exhibits two photon resonance at the band edge and exciton energy level accounts for the nonlinear absorption whereas the free carrier effect induced the optical nonlinearity in ZnO [17]. Viswanath et al. [18] reported the highest value of third order nonlinear susceptibility for ZnO-PVA nanocomposite film was 19.006×10^{-5} esu. Meanwhile the optical properties reported by Ahmad et. al [8-10] of the developed ZnO film has saturation intensity of 0.016 MW/cm^2 , modulation depth of 3.5% and broadband absorption ranges from 1000 nm to 1600 nm. They developed the ZnO film by mixing ZnO powder with silane and ethanol. Other than that, they also reported the thickness of the ZnO film was 0.15 mm and for Q-switched Erbium-doped fiber laser (EDFL) the pulse generated operates at 1561 nm, repetition rate ranges from 41.7–77.2 kHz, shortest pulse width of 6 μs and signal to noise ratio of 56 dB. The thickness of ZnO film reported by them is quite thick. Aziz et. al [11] reported that the thickness of ZnO film measured was 50 μm . They synthesized ZnO powder by mixing zinc nitrate with hexamethylenetetramine in de-ionized water. Later they mixed the synthesized ZnO powder with polyvinyl alcohol (PVA) to develop ZnO film. They reported pulse generated operates at 1560.4 nm, repetition rate ranges from 11.8–77.9 kHz, shortest pulse width of 7 μs and signal to noise ratio of 62 dB. However, they did not report the characterization of the synthesized ZnO powder to be used as main material. In this work, we demonstrated ZnONP based passive SA by using commercially available ZnO nanopowder as the starting material and PVA as a binder. The characterization and output performances of the developed ZnONP based SA is reported in this paper.

2. Material Preparation and Characterization

The preparation of ZnONP based SA is illustrated in Figure 1. The polyvinyl alcohol (PVA) as the host polymer was fabricated by adding 1 g of PVA powder (Sigma Aldrich, 40000 Mw) into 120 ml of DI water which then magnetically stirred at the temperature of 145°C until completely dissolves. The PVA suspension is used as a binder in this experiment because of its low optical absorption and high flexibility. Fabrication of ZnO based passive SA is done by adding 10 mg of ZnO NP [Alfa Aesar, 99.99% trace metal basis, 81.37 Mw] into 5 ml of the dissolved PVA suspension. Mixture of ZnONP-PVA was firstly ultrasonicated for 3 hours and then was centrifuged at 2500 rpm for 5 minutes to separate the undissolved ZnONP with the dissolved ZnONP in PVA. Lastly, the ZnONP-PVA solution was decanted into petri dish and was left to dry for 3 days at an ambient temperature in order for it to develop into ZnONP-PVA film to be used as passive SA. A small area of the film which was about $1 \text{ mm}^2 \times 1 \text{ mm}^2$ was cut to be attached on the surface of fiber ferule.

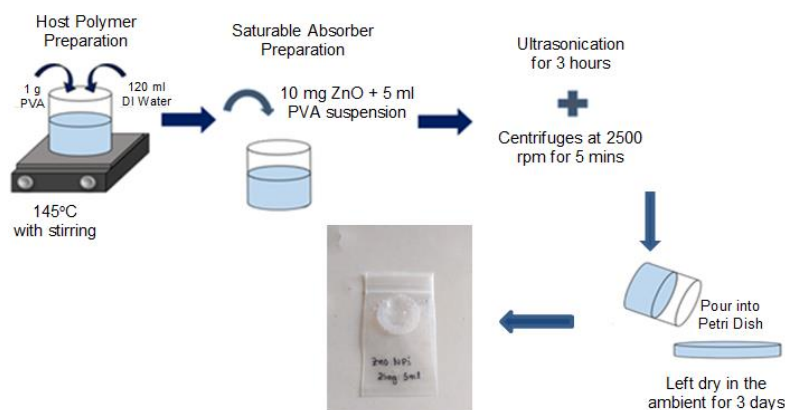


Figure 1. Preparation of ZnONP based SA

The surface morphology of the fabricated ZnONP-PVA film was characterized using focused ion beam scanning electron microscopy (FIB-SEM) [Helios Nanolab G3 UC, FEI]. Figure 2 shows the Field Emission Scanning Electron Microscopy (FESEM) of the ZnONP. The captured image clearly shows the evenly distributed ZnONP with average particle size of 40–100 nm.

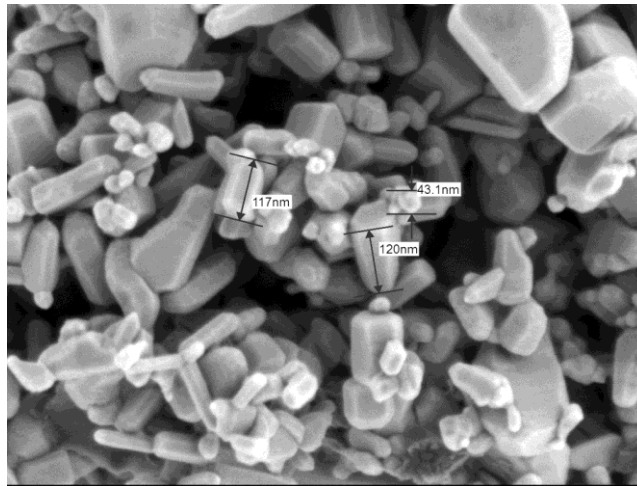


Figure 2. FESEM image of ZnONP

The thickness of the fabricated film was measured using 3D measuring laser microscope (Olympus, LEXT OLS4100). Figure 3 shows the thickness measurement of the ZnONP-PVA film. In order to obtain precise measurement, the film was attached to double sided tape to fix the film position. The thickness of the film was measured by measuring the height of the smooth surface (ZnONP-PVA film) from the rough surface (double sided tape). The thickness of the film measured was 12 μm .

Figure 4 shows the absorption spectrum for ZnONP-PVA with PVA as the reference. There are a total of 8 sets of zone centre optical phonons which consist of transverse and longitudinal polar modes A1L, A1T, E1L, E1T and low and high frequency modes E2L, E2H. The high frequency phonon E2H which is the main dominant peak is known as Raman active optical phonon that shows the characteristic of wurtzite hexagonal ZnO phase [19]. Other peaks are also usually observed [20-21]. The as-grown ZnO structures of wurtzite hexagonal phase with good crystallinity is demonstrated at the relatively higher and sharp peak of E2H which is at 437 cm^{-1} . Based on the Raman spectrum of ZnONP-PVA film, the E2H peak obtained is at 435 cm^{-1} . Compared to the theoretical, the shifting of E2H peak is due to the defects and internal strength as it is mixed with PVA.

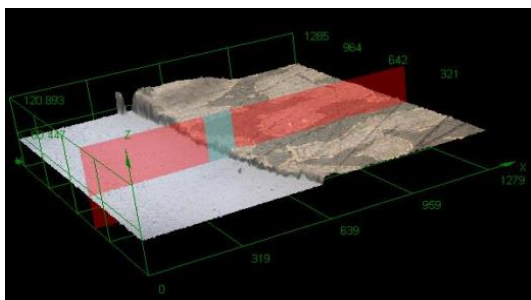


Figure 3. Thickness of ZnONP-PVA film

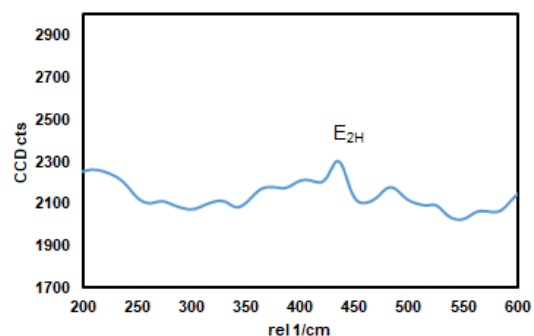


Figure 4. Raman spectrum of ZnONP-PVA film

The modulation depth and intensity saturation of ZnONP-PVA were obtained using twin detector measurement. Figure 5 shows the setup of twin balanced detector for the measurement of the nonlinear response. It consists of a self-made mode lock seed with a pulse width of 3.41 ps and repetition rate of 1 MHz. The mode-locked laser was amplified using Erbium doped fiber amplifier. 50% of the light was channeled to the fiber ferrule with deposition of ZnONP-PVA SA while the other 50% was channeled to the fiber ferrule without deposition of SA. The data were collected as the attenuator input was constantly reduced. By using optical power meter, the output power with and without ZnONP-PVA SA were measured. The curve was fitted using transmission fitting equation:

$$\text{Transmission Fitting, } T(I) = 1 - \left[\frac{\alpha_s}{1 + \frac{I}{I_{sat}}} + \alpha_{ns} \right] \quad (1)$$

where α_s is the modulation depth, I is the input intensity, I_{sat} is the saturation current and α_{ns} is the non-saturable absorption.

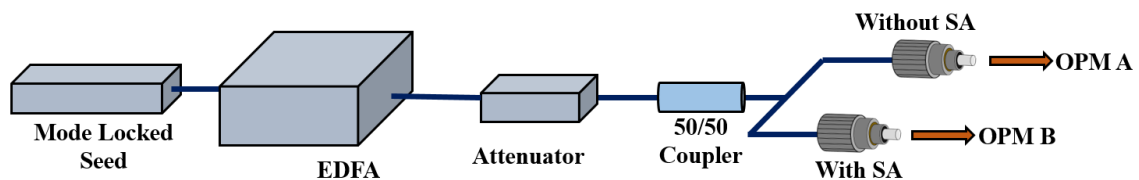


Figure 5. Twin balanced detector setup for nonlinear transmission analysis of ZnONP-PVA film

Figure 6 gives the curve fitting graph of transmissions of ZnONP-PVA at various input intensities. Based on the graph, the modulation depth obtained is 7.8% with non-saturable absorption of 3.8%. The transmission ratio started at 0.88 and the intensity saturation obtained is 88.97 MW/cm². The developed ZnO film reported by Ahmad et.al [8-10] has saturation intensity of 0.016 MW/cm², modulation depth of 3.5%. The results obtained suggested that the developed ZnONP-PVA film is suitable to act as SA for a stable Q-switched laser operation.

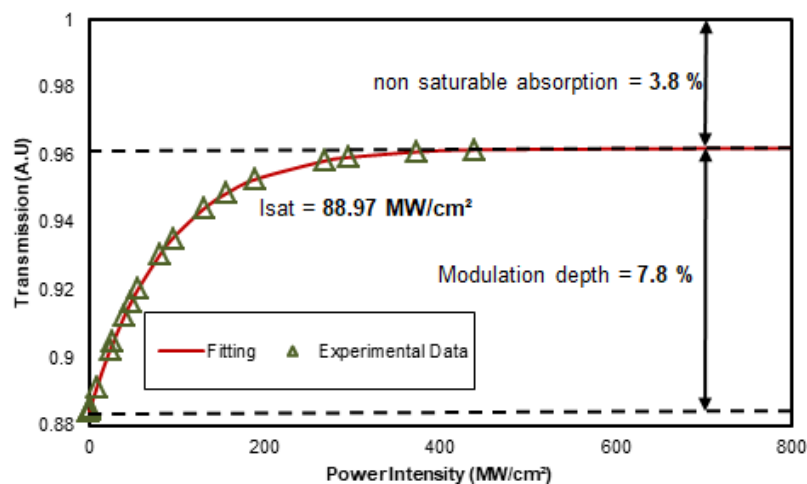


Figure 6. Modulation depth of ZnONP-PVA film

3. Results and Analysis

The input pump power for the lasing of the continuous wave was 18.3 mW. As the input pump power was increased, the Q-switched pulse started to be observed. More photons are

provided to the SA as the pump power increases, thus allowing the SA to saturate and emits light. The threshold input pump power to generate Q-switched pulse is at 45.4 mW and can be stably increased up to 92.4 mW pump power before the pulse diminished beyond the value. At maximum threshold pump power, the operating wavelength of the Q-switched pulse is approximately 1531.27 nm as shown in Figure 7. The spectrum also shows 3 dB spectral broadening of 2 nm.

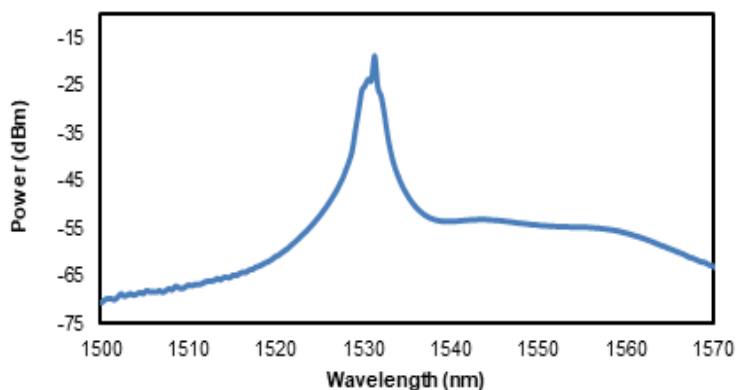


Figure 7. Optical spectrum at maximum input pump power of 92.4 mW

Using digital oscilloscope, pulse trains for different input pump power were obtained. The recorded pulse train of repetition rate is tunable stably from 73.53 kHz to 103.10 kHz when the input pump power was increased from threshold to maximum threshold pump power. Increasing repetition rate as the input pump power increases is one of the characteristics shown in Q-switched fiber laser [22]. With the output pulse trains were stable throughout the process of increasing input pump power, it is presumed that the fiber laser works in highly stable Q-switching regime [14]. Figure 8 (a) shows the pulse train of 103.10 kHz obtained at maximum threshold pump power with pulse separation of 10 μ s. Noting there is no observable amplitude modulations, which suggests that during Q-switching operation, self-mode locking is totally suppressed [23] and that the laser operated in regime of continuous wave Q-switched. The shortest pulse width obtained at maximum pump power is 4.8 μ s as displayed in Figure 8 (b).

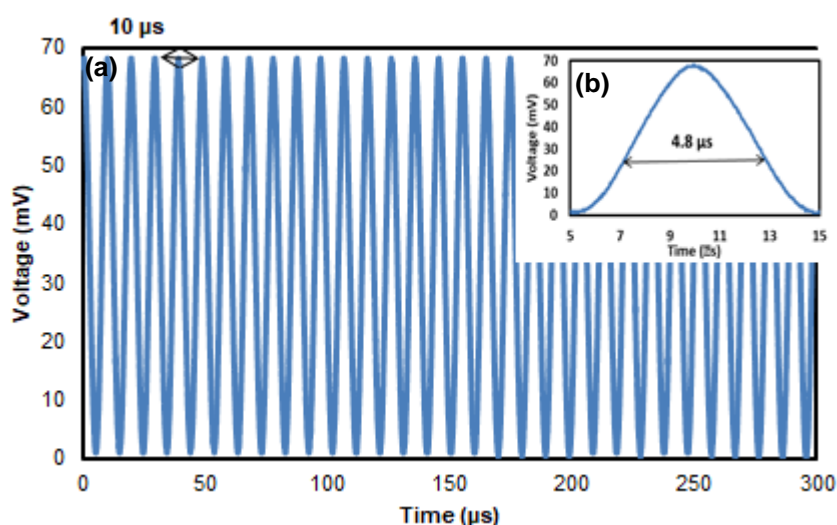


Figure 8. (a) Oscilloscope trace at maximum pump power of 92.4 mW (103.10 kHz) and (b) pulse width at FWHM

Referring to Figure 9, as the input pump power is varied from threshold to the maximum, the repetition rate increases from 73.53 kHz to 103.10 kHz, while the pulse width reduces from 6.8 μs to 4.8 μs , showing the typical trend of a Q-switched pulse [24]. This is due to the increasing in pump power which provides more photons to the SA. The SA saturates faster and emits more light. Thus, the repetition rate becomes faster and pulse width narrowed. The pulse width obtained in this paper is the shortest compared to the other reported ZnO NP based SA [8-11]. Figure 10 shows the average output power and pulse energy in a function of pump power. Both average output pump power and pulse energy increase as the input pump power increases from threshold to maximum. The calculated average output power and pulse energy was 5.18 mW and 49.85 nJ, respectively at maximum input pump power. The pulse energy obtained is quite high compared to other reported based SA Q-switched laser such as multi-walled carbon nanotubes (MWCNTs), topological insulator (TI) and graphene [8-11]. The measured signal to noise ratio (SNR) was 56 dB as shown in Figure 11. This indicates that the generated pulse by ZnONP based passive SA was stable at repetition rate of 103.10 kHz. Within 500 kHz span, up to 5th order harmonics of fundamental repetition rate were observed, indicated high stability of Q-switching operation. This value of SNR is comparable with other reported ZnO, Graphene, Bismuth Selenide based SA for Q-switched fiber laser [8-10, 25].

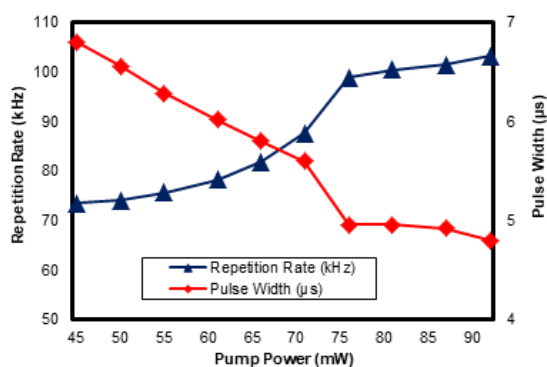


Figure 9. Repetition rate and pulse width in a function of pump power

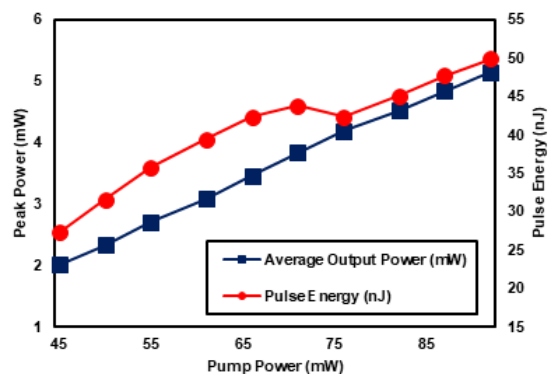


Figure 10. Average output power and pulse energy in a function of pump power

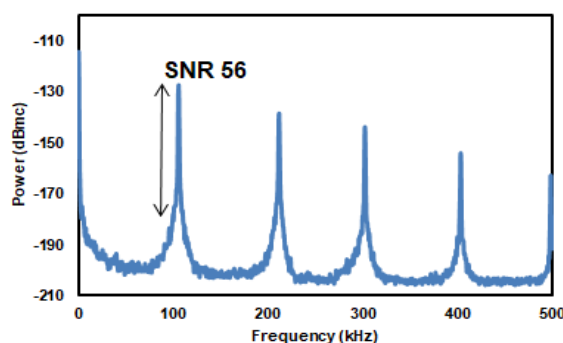


Figure 11. RF spectrum at maximum pump power of 92.4 mW

4. Conclusion

We have successfully demonstrated Q-switched Erbium-doped fiber laser (EDFL) by utilizing Zinc oxide nanoparticles embedded in polyvinyl alcohol (PVA) based passive saturable absorber. The output performances of the generated pulse was illustrated, tabulated and discussed in this paper. The proposed laser is capable of generating Q-switched laser at centre wavelength of 1531.27 nm. Starting from the threshold of 45.4 mW, at the maximum pump power of 92.4 mW, maximum pulse repetition rate and pulse energy 103.10 kHz and 49.85 nJ respectively, as well as shortest pulse width of 4.8 μs has been demonstrated.

References

- [1] Xia F, Wang H, Jia Y. Rediscovering black phosphorus as an anisotropic layered material for optoelectronics and electronics. *Nature communications*. 2014; 5: 4458.
- [2] Yu J, et al. 1 J/pulse Q-switched 2 μm solid-state laser. *Optics Letters*. 2006; 31(4): 462-464.
- [3] Zhou DP, Wei L, Dong B, Liu WK. Tunable passively Q-switched erbium-doped fiber laser with carbon nanotubes as a saturable absorber. *IEEE Photonics Technology Letters*. 2010; 22(1): 9-11.
- [4] D Popa D, Sun Z, Hasan T, Torrisi F, Wang F, Ferrari AC. Graphene Q-switched, tunable fiber laser. *Applied Physics Letters*. 2011; 98(7): 1-3.
- [5] Woodward RI, Kelleher EJR. 2D saturable absorbers for fibre lasers. *Applied Sciences*. 2015; 5(4): 1440-56.
- [6] Lin YH, et al. Using n-and p-type Bi_2Te_3 topological insulator nanoparticles to enable controlled femtosecond mode-locking of fiber lasers. *ACS Photonics*. 2015. 2(4): 481-490.
- [7] Lin YH, et al. Soliton compression of the erbium-doped fiber laser weakly started mode-locking by nanoscale p-type Bi_2Te_3 topological insulator particles. *Laser Physics Letters*. 2014; 11(5): 055107.
- [8] Ahmad H, Salim MAM, Ismail MF, Harun SW. Q-switched ytterbium-doped fiber laser with zinc oxide based saturable absorber. *Laser Physics*. 2016; 26(11): 115107.
- [9] Ahmad H, Lee CSJ, Ismail MA, Ali ZA, Reduan SA, Ruslan NE, Ismail MF, Harun SW. Zinc oxide (ZnO) nanoparticles as saturable absorber in passively Q-switched fiber laser. *Optics Communications*. 2016; 381: 72-76.
- [10] Ahmad H, Lee CSJ, Ismail MA, Ali ZA, Reduan SA, Ruslan NE, Harun SW. Tunable Q-switched fiber laser using zinc oxide nanoparticles as a saturable absorber. *Applied optics*. 2016; 55(16): 4277-81.
- [11] Aziz NA, Latiff AA, Lokman MQ, Hanafi E, Harun SW. Zinc oxide-based Q-switched erbium-doped fiber laser. *Chinese Physics Letters*. 2017; 34(4): 044202.
- [12] Rashid FAA, et al. Using a black phosphorus saturable absorber to generate dual wavelength in a Q-switched Ytterbium-doped fiber laser. *Laser Phys. Lett*. 2016; 13(8).
- [13] J Sotor, G Sobon, I Pasternak, K Krzempek G, Dudzik A, Krajewska W, Strupinski KM. Abramski, Dual wavelength fiber mode-locked laser based on graphene saturable absorber. *Proc. of SPIE* 8961, 89612A. 2014.
- [14] Ismail EI, Kadir NA, Latiff AA, Ahmad H, Harun SW. Black phosphorus crystal as a saturable absorber for both a Q-switched and mode-locked erbium-doped fiber laser. *RSC Advances*. 2016; 6(76): 72692-7. DOI: 10.1039/C6RA14008D.
- [15] Madathil AN, Vanaja KA, Jayaraj MK. *Synthesis of ZnO nanoparticles by hydrothermal method*. Proc of SPIE. 2007; 6639: 1-9.
- [16] Hudgins JL, Simin GS, Santi E, Khan MA. An assessment of wide bandgap semiconductors for power devices. *IEEE Transactions on Power Electronics*. 2003; 18(3): 907-914.
- [17] Lin JH, Chen YJ, Lin HY, Hsieh WF. Two-photon resonance assisted huge nonlinear refraction and absorption in ZnO thin films. *Journal of Applied Physics*. 2005; 97(3): 033526.
- [18] Viswanath V, Beenakumari C, Muneera CI. ZnO-PVA Nanocomposite Films For Low Threshold Optical Limiting Applications. *Light and its Interactions With Matter*. 2014; 1620: 604-610.
- [19] Khan A. Raman Spectroscopic Study of the ZnO Nanostructures. *J Pak Mater Soc*. 2010; 4(1): 5-9.
- [20] Xu CX, Sun XW, Dong ZL, Cui YP, Wang BP. Nanostructured singlecrystalline twin disks of zinc oxide. *Crystal Growth & Design*. 2007; 7(3): 541-4.
- [21] Khan A, Kordesch ME. Large-scale fabrication of metallic Zn nanowires by thermal evaporation. *Physica E: Low-dimensional Systems and Nanostructures*. 2006; 33(1): 88-91.
- [22] Yan P, et al. Passively mode-locked fiber laser by a cell-type WS₂ nanosheets saturable absorber. *Scientific reports*. 2015; 5: 12587.
- [23] Sun Z, Hasan T, Ferrari AC. Ultrafast lasers mode-locked by nanotubes and graphene. *Physica E*. 2012; 44(6): 1082-1091.
- [24] Xinju L. *Laser Technology*. 2nd Edition. Boca Raton: CRC. 2010.
- [25] Tiu ZC, Ahmad F, Tan SJ, Zarei A, Ahmad H, Harun SW. Multi-wavelength Q-switched erbium-doped fiber laser with photonic crystal fiber and multi-walled carbon nanotubes. *Journal of Modern Optics*. 2014; 61(14): 1133-1139.