UNIVERSITI PUTRA MALAYSIA

BLACK PHOSPHORUS/POLYDIMETHYLSILOXANE COATED MICROFIBER SATURABLE ABSORBER FOR ULTRASHORT PULSE FIBER LASER

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By

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Chair : Prof. Mohd Adzir bin Mahdi, PhD
Faculty : Engineering

Fiber lasers capable of generating ultrashort pulses have diverse applications in micromachining, medical science, and telecommunications. Ultrashort pulses generated from fiber lasers can be achieved through passive mode-locking *via* a saturable absorber (SA). Several black phosphorus (BP) fiber-based saturable absorbers have been reported with low damage thresholds, however, have poor repeatability for mass production due to uncontrollable deposition techniques. This research work focuses on a new technique to generate ultrashort pulses using optimized adiabatic tapered fiber saturable absorber with different dimensions of black phosphorus; vary from bulk to quantum dots.

The experimental work was initiated with the optimization of bulk BP polymer composite based saturable absorbers of different taper profiles in erbium-doped fiber ring laser cavity. The BP polymer composite was prepared by dispersing BP powders in tetrahydrofuran solvent then adding in polydimethylsiloxane (PDMS) to form composite. The sonicated BP was observed in chunky pieces within micrometers, proven by the scanning electron microscope morphological characterization. The characteristics of BP, PDMS, and BP/PDMS composite were analyzed through Raman spectroscopy, field emission scanning electron microscope, and ultraviolet-visible-near infrared absorption spectroscopy methods. A constant amount of BP polymer composite was deposited within the waist area of tapered fiber through spin coating method with optimized parameters; 4000 rpm for 6 minutes. The up/down transition and waist length were optimized with a waist diameter of 10 μm was chosen as the constant parameter. Based on the experimental findings, the optimum taper profile was 30 mm of up/down transition length and 1 mm of waist length, which resulted in transmission loss of 3.34 dB, saturation fluence of 21.56 $\mu J/cm^2$, and modulation depth of 3.14%. The fabricated BP/PDMS SA was integrated into a

ring cavity erbium-doped fiber laser (EDFL). By adjusting the polarization states of the circulating light in the cavity, the EDFL produced an optical spectrum with Kelly sidebands to prove the generation of conventional soliton (CS). The output pulse had a central wavelength of 1560.18 nm, 3-dB spectral width of 5.92 nm, pulse duration of 724 fs, repetition rate of 6.53 MHz, and time bandwidth product (TBP) of 0.53 showing the SA capable of generating ultrashort pulses.

Next, black phosphorus layers (BPLs) and black phosphorus quantum dots (BPQDs) were synthesized from bulk BP through liquid phase exfoliation method. The findings from high resolution transmission electron microscopy and atomic force microscopy suggested BPLs were in diameter range from 69 – 362 nm and thickness range of 42 – 62 nm. Whereas BPQDs displayed a lateral size of 2.92 ± 0.37 nm as analyzed from the TEM image. Then, BPLs/PMDS and BPQDs/PDMS composite SA were fabricated following the same procedure as implemented for BP/PDMS SA. These fabricated SAs were inserted into the same EDFL cavity to study their pulse performance characteristics. The transmission loss of BPLs/PDMS coated microfiber was 2.57 dB whereas BPQDs/PDMS composite coated microfiber was 2.12 dB at 1550 nm, respectively. The nonlinear absorption of BPLs/PDMS composite coated microfiber possesses saturation fluence of 9.22 $\mu J/cm^2$, modulation depth of 3.5%, and two-photon absorption (TPA) of 5.3 x 10^{-3} $cm^2/\mu J$ while the BPQDs/PDMS coated microfiber exhibited saturation fluence of 18.47 $\mu J/cm^2$, modulation depth of 2.12%, and TPA coefficient of 9.1 x 10^{-3} $cm^2/\mu J$. The nonlinear optical absorption response shows that due to different size of BPs, both SAs showed different TPA characteristics that caused CS to noise-like pulse (NLP) operation at different pump powers. In the CS operation, BPLs/PDMS composite SA generated 837 fs which was better in pulse duration compared to 1.11 ps of BPQDs/PDMS composite SA. Besides that, BPLs-SA had the lowest TBP value (0.39) among other structures BP-SAs which indicated the pulse was slightly chirped. However, in the NLP operation, BPQDs-SA showed shortest pulse generation at 152 fs due to strong TPA characteristic. Overall, the results validate the reliability of the proposed method to understand the effect of different BP/PDMS structures on microfiber based SAs for ultrashort pulse generation.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

PENYERAP TEPU GENTIAN MIKRO TERSALUT FOSFORUS HITAM/POLIDIMETILSILOKSANA UNTUK LASER GENTIAN DENYUT ULTRA PANTAS

Oleh

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Laser gentian yang mampu menghasilkan denyut ultra pantas mempunyai pelbagai aplikasi seperti pemesinan mikro, sains perubatan, dan telekomunikasi. Denyutan ultra pantas dari laser gentian boleh dicapai melalui penguncian mod pasif dengan penyerap tepu. Beberapa penyerap tepu berasaskan gentian fosforus hitam (BP) telah dilaporkan dengan ambang kerosakan yang rendah dan teknik pemendapan yang tidak terkawal menyebabkan kebolehulangan untuk pengeluaran besar-besaran yang rendah. Kerja penyelidikan ini menumpukan pada teknik baharu untuk menghasilkan denyutan ultra pantas menggunakan penyerap tepu gentian tirus adiabatik yang telah dioptimumkan dengan dimensi BP yang berbeza; dari pukal kepada titik kuantum.

Eksperimen dimulakan dengan pengoptimuman penyerap tepu berdasarkan komposit polimer BP bersaiz pukal dengan profil tirus yang berbeza dalam laser gentian terdop-erbium berskema cincin. Komposit polimer BP disediakan dengan mengsonikasi BP pukal dalam pelarut tetrahidrofuran, kemudian ditambahkan polidimetilsiloksana (PDMS) ke dalam larutan. BP yang telah disonikasi mempunyai potongan kecil dalam skala mikrometer, dibuktikan dengan perincian morfologi mikroskop elektron pengimbas. Ciri-ciri komposit BP, PDMS dan BP/PDMS dianalisis menggunakan spektroskopi Raman, mikroskop elektron pengimbas pelepasan medan, dan kaedah spektroskopi penyerapan ultra violet-sinar tampak dan dekat infra merah. Komposit polimer BP yang berjisim sama didepositkan di kawasan pinggang gentian tirus melalui lapisan putaran pada 4000 putaran seminit untuk 6 minit. Peralihan naik/turun dan panjang pinggang telah dioptimumkan dengan diameter pinggang 10 μm dipilih sebagai parameter tetap. Dari penemuan eksperimen, profil tirus terbaik adalah 30 mm panjang peralihan ke atas/bawah dan 1 mm panjang pinggang, menghasilkan 3.34 dB kehilangan transmisi, 21.56 µJ/cm^2 kelancaran tepu, dan 3.14% kedalaman modulasi. Penyerap tepu BP/PDMS telah diintegrasi dalam laser gentian terdop-erbium berskema cincin. Dengan pelarasan polarisasi cahaya yang beredar dalam rongga, spektrum optik dengan jalur sisi Kelly membuktikan penghasilan soliton konvensional (CS). Denyut keluaran mempunyai panjang gelombang yang berpusat pada 1560.18 nm, 5.92 nm spektrum jalur lebar 3-dB, 724 fs jangka masa denyut, 6.53 MHz kadar pengulangan denyut, dan 0.53 jalur lebar masa (TBP).

Seterusnya, lapisan fosforus hitam (BPLs) dan titik kuantum fosforus hitam (BPQDs) telah disintesis dari BP pukal melalui kaedah pengelupasan fasa cecair. Penemuan dari mikroskop elektron transmisi beresolusi tinggi dan mikroskopi daya atom mencadangkan BPLs berada dalam julat diameter 69-362 nm dan julat ketebalan 42-62 nm. Manakala BPQDs menunjukkan ukuran lateral 2.92 ± 0.37 nm seperti yang telah dianalisis dari gambar TEM. Kemudian, penyerap tepu BPLs/PMDS dan BPQDs/PDMS komposit telah difabrikasi berdasarkan prosedur yang sama seperti yang dilaksanakan untuk penyerap tepu BP/PDMS. Penyerap tepu yang telah difabrikasi dimasukkan ke dalam rongga EDFL yang sama untuk mengkaji ciri-ciri prestasi denyut mereka. Kehilangan transmisi mikrofiber bersalut BPLs/PDMS dan BPQDs/PDMS adalah masing-masing 2.57 dB dan 2.12 dB pada 1550 nm. Penyerapan tidak linier mikrofiber bersalut komposit BPLs/PDMS mempunyai kelancaran tepu 9.22 µJ/cm^2, kedalaman modulasi 3.5%, dan pekali penyerapan dua foton (TPA) 5.3x10^{-3} cm^2/µJ manakala mikrofiber bersalut BPQDs/PDMS mempamerkan kelancaran tepu 18.47 µJ/cm^2, kedalaman modulasi 2.12%, dan pekali TPA 9.1 x 10^{-3} cm^2/µJ. Tindak balas penyerapan optik tidak linier pada kedua-dua penyerap tepu menunjukkan ciri-ciri TPA yang berbeza disebabkan oleh dimensi BP yang berbeza. Hal ini menyebabkan operasi dari CS kepada NLP pada daya pam yang berbeza. Dalam operasi CS, penyerap tepu komposit BPLs/PDMS menghasilkan 837 fs durasi denyutan, yakni lebih baik berbanding 1.11 ps penyerap tepu komposit BPQDs/PDMS. Selain itu, penyerap tepu BPLs mempunyai nilai TBP terendah (0.41) berbanding struktur penyerap tepu BP yang lain, sebagai indikasi bahawa denyut sedikit terherot. Namun begitu, dalam operasi NLP, penyerap tepu BPQDs/PDMS telah menunjukkan penjanaan denyut terpendek pada 152 fs kerana ciri TPA yang kuat. Secara keseluruhan, dapatan mengesahkan kebolehpercayaan kaedah yang dicadangkan untuk memahami pengaruh struktur BP yang berbeza berdasarkan penyerap tepu mikrofiber untuk penjanaan denyut ultra pantas.

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This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfillment of the requirement for the degree of Doctor of Philosophy. The members of the Supervisory Committee were as follows:

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LIST OF ABBREVIATIONS

AFM	Atomic Force Microscopy
ASE	Amplified Spontaneous Emission
BP	Black Phosphorus
BPLs	Black Phosphorus Layers
BPQDs	Black Phosphorus Quantum Dots
CNT	Carbon Nanotube
CS	Conventional Soliton
CVD	Chemical Vapor Deposition
CW	Continuous Wave
DMS	Dispersion-Managed Soliton
DS	Dissipative Soliton
EDX	Energy Dispersive X-ray Spectroscopy
FESEM	Field Emission Scanning Electron Microscope
FWHM	Full Width at Half Maximum
FWM	Four-Wave Mixing
GVD	Group Velocity Dispersion
HRTEM	High Resolution Transmission Electron Microscopy
ISO	Isolator
LD	Laser Diode
LPE	Liquid Phase Exfoliation
NALM	Nonlinear Amplifying Loop Mirror
NOLM	Nonlinear Optical Loop Mirror
NLO	Nonlinear Optical

NLP	Noise Like Pulse
NLSE	Nonlinear Schrodinger Equation
NPR	Nonlinear Polarization Rotation
OC	Optical Coupler
OPM	Optical Power Meter
OSA	Optical Spectrum Analyzer
PC	Polarization Controller
PCF	Photonic Crystal Fiber
PDI	Polarization-Dependent Isolator
PDL	Polarization Dependent Loss
PDMS	Polydimethylsiloxane
PL	Photoluminescence Spectroscopy
RBW	Resolution Bandwidth
RSAE	Reverse Saturable Absorption
SA	Saturable Absorber
SAE	Saturable Absorption Effect
SESAM	Semiconductor Saturable Absorber Mirror
SMFs	Single Mode Fibers
SNR	Signal to Noise Ratios
SPM	Self-Phase Modulation
TBP	Time Bandwidth Product
TEM	Transmission Electron Microscopy
THF	Tetrahydrofuran
TIR	Total Internal Reflection

TIs	Topological Insulators
TMDCs	Transition Metal Dichalcogenides
TPA	Two Photon Absorption
UV-Vis-NIR	Ultraviolet-Visible-near-IR
VBW	Video Bandwidth
VOA	Variable Optical Attenuator
WDM	Wavelength Division Multiplexer
XPM	Cross-Phase Modulation

CHAPTER 1

INTRODUCTION

1.1 Overview

Ultrashort pulse lasers have been making continuous technological advancements in diverse fields, such as medical, science and industrial applications. To name a few, applications for ultrashort pulse lasers include ophthalmology [1], bio-imaging [2], advanced micro-machining [3], quantum information processing [4], molecular spectroscopy [5], and nonlinear microscopy [6]. Some of the commercially available ultrafast lasers are titanium-sapphire, diode-pumped, and fiber lasers [7]. Out of those, fiber laser is considered as one of the most promising lasers that utilize rare-earth-doped fibers as optical gain medium. Besides, such laser is compact, flexible, and reliable, as well as exhibits excellent heat dissipation mechanism for the generation of ultrashort pulses.

Passive mode-locking technique is used to generate femtosecond pulses in ultrafast fiber laser system. This technique is usually realized with either free-space optical alignment or optical fiber laser cavities by employing either artificial or real saturable absorber (SA). Examples of artificial SA are nonlinear polarization rotation (NPR), nonlinear optical loop mirror (NOLM), and nonlinear amplifying loop mirror (NALM) in figure-of-eight configurations. Artificial SA usually deploys changing polarization state with a polarizer, such as the combination of a polarization-dependent isolator (PDI) and polarization controller (PC) for an NPR-based mode-locked fiber laser. However, artificial SA has the disadvantage of low environmental stability. The perturbation of light polarization state could experience a complex transient process before formation of soliton molecules, 23 times longer than carbon nanotubes (CNTs)-based mode-locked laser [8]. In addition, NPR-based SA shows stronger fluctuation of pumping strength, i.e., Q-switched instabilities are a typical phenomenon with longer nascent time generated before the formation of mode-locked laser, as compared to material-based SAs [9].

On the other hand, real SAs rely on material nonlinear saturable absorption properties to initiate and stabilize short pulses in various regimes. In particular, saturable absorption process depends on the imaginary part of the third-order nonlinearity of a material, whereas the real part indicates its nonlinear refractive index [10]. In addition, there are several properties that determine the quality of a material-based SA, such as direct bandgap tunability [11], nonlinear saturable absorption characteristics [12], and recovery time of a material that determines its power density for saturation [13], to name only a few. At the present time, the SA can be fabricated from different integration schemes by incorporating materials of saturable-absorption properties, such as III-V compound semiconductors [14], CNTs [15], graphene [16], topological insulators (TIs) [17], transition metal dichalcogenides (TMDCs) [18], black phosphorus (BP) [19], MXene [20], antimonene [21], and bismuthene [22]. The major advantage of these materials is their relatively higher environmental stability than artificial SA.

In this research work, I focused on investigating different BP structures-based SA for generation of ultrashort pulses. BP was chosen due to its tunable bandgap, easy preparation for exfoliation into different structures, direct bandgap regardless thickness, and structure dependent saturable and reverse saturable absorption effect which are regarded remarkably important in ultrafast laser applications.

1.2 Problem Statement and Motivation

Up to date, BP has been reported as layers or quantum dots embedded on fiber-based functional SA devices that capable to generate ultrashort pulses. However, there is no reported study investigating different BP structures-SA in fixed fiber laser configuration towards their pulse performance.

Besides that, there are weaknesses in commonly reported deposition techniques, which are based on optical deposition, sandwiching, and wrapping method to transfer the mono or few-layer BP material onto fiber-based devices. For optical deposition method, it often requires precise control of the light output power to deposit material on the fiber surface. The uniformity of material deposition is still questionable in this method. Because of that, optimization of light output power is required for each different material in solution. Besides this technique, the sandwiching method is often reported in depositing material or thin film between two fiber ferrules platform. The drawback of this method is uncontrollable thickness of material deposited on the core where several trials and adjustment of materials thickness were needed to produce short pulses. Whereas for thin film placement, it required high technical skill and practice to place the tiny film on the core without any folding. Furthermore, this type of SA has low thermal damage threshold because the deposited materials interact with the highest intensity of light directly at the center of the fiber core. Next, the wrapping method is also performed on microfiber platform with material composite (thin film substrate). This method is complex and difficult to handle. In fact, there is possibility of material-wrapped gap in between microfiber. Hence, the pulse performance will be affected. Overall, all these methods are either difficult to control or scale up in production. In addition, optimized materials-based microfiber-SAs has not been reported from the perspective of microfiber dimension. The only related investigation was study on CNT based material with different waist diameter microfiber towards pulse performance [23].

In summary, none of the mentioned techniques above offer an exact fair ground to compare pulse performance of different BP dimension incorporated onto the same SA. It is important to understand the differences to improve the techniques for scalable and repeatable fabrication.

1.3 Aim and Objective

I. To synthesize and characterize different BP structures via liquid phase exfoliation method,
II. To optimize spin coating parameters and microfiber dimensions of BP-based SA for shortest pulse duration,
III. To fabricate SA coated with varied BP structure based on optimized parameters as attained in Objective II, and
IV. To compare with other BP SA on ultrashort pulse laser performance.

1.4 Scope of Work

The scope of work in this research is outlined in Figure 1.1, where the highlighted subsets are the focus of this research.

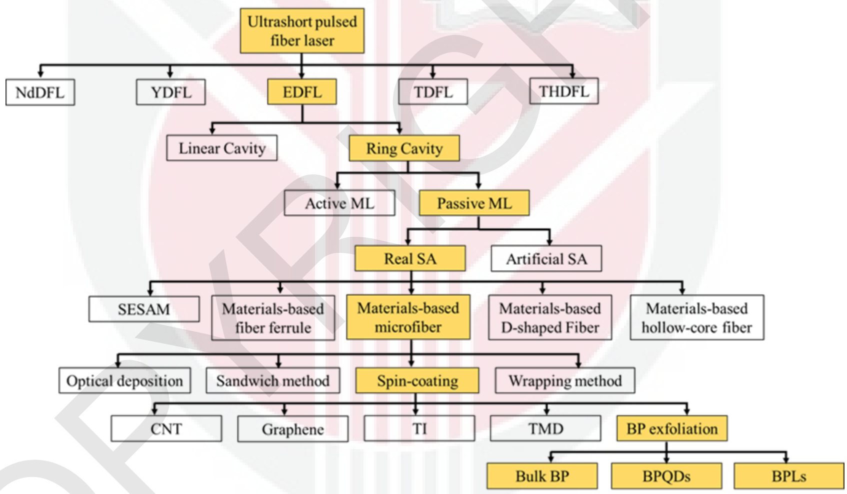

Figure 1.1: Scope of Work.

In this research, ring-type EDFL setup is selected for ultrashort pulse generation due to its well understood mechanism. Next, passive mode-locking technique based on real SAs is selected as it allows greater control of pulse performance against environmental perturbation as experienced by artificial SAs. For the real SAs, a microfiber is chosen as integrated structure due to its simplicity and strong evanescent field for light matter interaction at the tapered region. Before the microfiber is coated, optimization of microfiber dimensions and spin coating are investigated. In this work, BP material is mixed with polydimethylsiloxane (PDMS) before incorporated onto microfiber. Different structures of BP, black phosphorus layers (BPLs), and black phosphorus quantum dots (BPQDs) are synthesized and integrated as SA for better comparison in pulse generation.

Overall, the main focus of this work is to explore new and repeatable fabrication technique of BP-polymer microfiber SA that can provide a stable pulse laser source. The performance of mode-locking at 1.56 µm wavelength region will be discussed thoroughly in terms of spectral bandwidth, center wavelength, repetition rate, signal-to-noise ratio, pulse width, pulse energy and stability of the fiber laser system.

1.5 Organization of Thesis

The dissertation is organized as follows:

Chapter 1 consists of overview of ultrashort pulse laser, specifically on passively mode-locked fiber lasers. Issues with passive mode locking fiber laser are highlighted as well as the aim and objectives formed from those issues. The scope of work and thesis organization are also included in this chapter.

Chapter 2 presents the experimental and theoretical background of ultrashort pulse laser. This includes the mechanism of laser, effects of pulse propagation in optical fibers, soliton formation, and innovative fabrication techniques in the generation of mode-locked fiber lasers. In addition, optical properties of BP and its composite are also discussed. A critical review of reported BP-SAs passively mode-locked laser is also tabularized.

Chapter 3 demonstrates the experimental works of different dimension of BP-SA and few characterization tests are presented as supporting evidence for the generation of mode locking. This includes details of mode-locking performance from an optimized BP-SA.

Chapter 4 introduces the experimental synthesize of BP layers and BPQDs. Both BP structures are embedded on microfiber functions as SA for femtosecond pulse generations investigation. All the findings are discussed and analyzed.

Lastly, Chapter 5 concludes the main achievements of the research work. Recommendations for possible future investigation are suggested.

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