

GRAPHENE BASED PASSIVE Q-SWITCHER IN ERBIUM DOPED FIBER
LASER

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A thesis submitted in fulfilment of the
requirements for the award of the degree of
Master of Philosophy

Malaysia-Japan International Institute of Technology
Universiti Teknologi Malaysia

DECEMBER 2021

DEDICATION

To my family and friends who have been with me throughout this journey.

ACKNOWLEDGEMENT

First and foremost, utmost praises to Allah the Most Gracious for His countless guidance and blessings that enables me to pull through and complete this research.

I would like to sincerely express my gratitude to my supervisor, Dr Fauzan Ahmad for his patience, guidance, and knowledge that had kept me encouraged me in completing this research. Without his total support and understanding, I would not have been able to complete this thesis.

Besides that, I would like to thank the Optical Devices and System (ODESY) iKohza for providing a conducive workspace as well as experimental support. I am also indebted to my mentor, Dr. Siti Nur Fatin Zuikafly for her tremendous help and support that made the completion of this research possible. Not to say the least, my friends and colleagues that had lent a hand throughout the process.

Lastly, deepest appreciation for my parents, Haliza binti Salleh and Jasni bin Abdullah, whom without their emotional support and encouragement, this journey would not have even started. To my sisters and friends, your support throughout this challenging journey, whether directly or indirectly, is very much appreciated.

ABSTRACT

Graphene is known as the material of wonder for its extraordinary properties that can be utilized for the technology advancement in various fields. In this work, graphene based saturable absorber (SA) was used for pulsed laser generation. The SA was integrated within an erbium-doped fiber laser (EDFL) ring cavity for passive Q-switching, generating pulsed laser. Passively Q-switched EDFL has been achieved by using various materials as SAs, with the current trend focuses on the 2-Dimensional (2D) materials. Graphene was the most favourable for this research due to its low-cost and simple fabrication compared to the complex fabrication process, narrow wavelength range, and unstability in ambient temperature of other 2D materials. Two types of graphene were used as the starting material for SA fabrication in this work: electrochemical exfoliated (ECE) graphene (GrE) and graphene filament (GrF). The two graphene were combined with host polymers with the graphene : polymer ratios of 1 : 4, 2 : 3 and 2.5 : 2.5 to develop free standing films for easy integration within laser ring cavity. The host polymers used were polyvinyl alcohol (PVA) and chitin in which GrE used both to develop GrE-PVA SA and GrE-chitin SA respectively, while GrF was combined with chitin to develop GrF-chitin SA. The absence of PVA as host polymer for GrF-based SA was due to the usage of tetrahydrofuran (THF) in the preparation of the filament that reacted poorly with the PVA, inhibiting the formation of a free-standing SA film. Chitin is an alternative host polymer to produce a more environmental-friendly SA. Comparing the performance of the SAs, the lowest threshold pump power for the Q-switching activity was 16.7 mW, obtained by GrF-chitin 2.5 : 2.5 while the highest repetition rate and lowest pulse width were obtained by GrF-chitin 2 : 3 at 121.4 kHz and 3.32 μ s respectively. On the other hand, the highest signal-to-noise ratio (SNR) was 88.88 dB which was generated by GrE-PVA 2 : 3. Ultimately, the SAs used in this work is deemed stable for the use of pulsed laser generation by passive Q-switching as the lowest SNR value has also been considerably high at 64.64 dB.

ABSTRAK

Grafin terkenal sebagai bahan yang mengagumkan kerana sifatnya yang luar biasa yang dapat digunakan untuk kemajuan teknologi dalam pelbagai bidang. Dalam kajian ini, penyerap boleh tepu (SA) berasaskan grafin digunakan untuk penjanaan laser berdenyut. SA disepadukan ke dalam rongga gelang laser gentian berasaskan erbium (EDFL) untuk menghasilkan laser berdenyut melalui suis-Q pasif. Suis-Q pasif bagi EDFL telah dicapai dengan menggunakan pelbagai bahan sebagai SA, dimana tumpuan semasa adalah pada bahan berasaskan 2 dimensi (2D). Grafin adalah bahan 2D yang dipilih untuk kajian ini kerana cara pembuatannya yang ringkas dan berkos rendah berbanding bahan 2D lain yang mempunyai proses pembuatan yang rumit, julat panjang gelombang yang rendah, dan ketidakstabilan dalam suhu bilik. Dua jenis grafin digunakan sebagai bahan awal bagi pembuatan SA dalam kajian ini, iaitu grafin daripada pengelupasan elektrokimia (ECE) (GrE) dan filamen grafin (GrF). Grafin digabungkan bersama polimer perumah dengan nisbah grafin : polimer 1 : 4, 2 : 3, dan 2.5 : 2.5 untuk menghasilkan filem berdiri pegun bagi memudahkan disepadukan ke dalam rongga gelang laser. Polimer perumah yang digunakan adalah alkohol polivinil (PVA) dan chitin di mana kedua-duanya digunakan bersama GrE dan masing-masing menghasilkan GrE-PVA SA dan GrE-chitin SA manakala GrF digabungkan bersama chitin membentuk GrF-chitin SA. Ketidakerasian yang ditunjukkan oleh PVA terhadap tetrahidrofuran (THF) yang digunakan dalam penyediaan filamen menghalang pembentukan filem berdiri pegun berasaskan GrF dan PVA. Chitin ialah polimer perumah alternatif untuk menghasilkan SA yang lebih mesra alam sekitar. Sebagai perbandingan prestasi SA yang dihasilkan, ambang kuasa masukan terendah bagi operasi suis-Q adalah 16.7 mW, yang mana diperolehi oleh GrF-chitin 2.5 : 2.5 sementara kadar pengulangan tertinggi dan lebar nadi terpendek diperolehi GrF-chitin 2 : 3 pada 121.4 kHz dan 3.32 μ s. Selanjutnya, nisbah isyarat kepada hingar (SNR) tertinggi adalah 88.88 dB yang mana dihasilkan oleh GrE-PVA 2 : 3. Secara keseluruhannya, SA yang digunakan dalam kajian ini adalah stabil bagi penghasilan laser berdenyut melalui suis-Q pasif memandangkan nilai SNR terendah juga boleh dianggap sebagai nilai tinggi, pada 64.64 dB.

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LIST OF ABBREVIATIONS

0D	-	0-Dimensional
1D	-	1-Dimensional
2D	-	2-Dimensional
3D	-	3-Dimensional
AOP	-	Average Output Power
BP	-	Black Phosphorus
CNT	-	Carbon Nanotube
CSV	-	Comma Separated Values
CVD	-	Chemical Vapour Deposition
CW	-	Continuous Wave
dB	-	Decibel
dBm	-	Decibel per milliwatt
DI	-	Deionized
ECE	-	Electrochemical Exfoliation
EDF	-	Erbium-doped Fiber
EDFA	-	Erbium-doped Fiber Amplifier
EDFL	-	Erbium-doped Fiber Laser
FD NORM	-	Standard Frequency Domain
FESEM	-	Field Emission Scanning Electron Microscope
FWHM	-	Full Width at Half Maximum
GlcN	-	Glucosamine
GlcNAc	-	N-acetylglucosamine
GnP	-	Graphene Nanoplatelets
Gr	-	Graphene
GrE	-	Electrochemical exfoliated graphene
GrE-Chitin	-	Electrochemical exfoliated graphene-Chitin
GrE-PVA	-	Electrochemical exfoliated graphene-Polyvinyl Alcohol
GrF	-	Graphene Filament
GrF-Chitin	-	Graphene Filament-Chitin
GVD	-	Group Velocity Dispersion

LD	-	Laser Diode
LPE	-	Liquid Phase Exfoliation
MWCNT	-	Multiwalled Carbon Nanotube
NPR	-	Nonlinear Polarization Rotation
OPM	-	Optical Power Meter
OSA	-	Optical Spectrum Analyzer
OSC	-	Mixed domain oscilloscope
PDMS	-	Polydimethylsiloxane
PEO	-	Polyethylene Oxide
PLA	-	Polylactic Acid
PMMA	-	Polymethyl Methacrylate
PVA	-	Polyvinyl Alcohol
QD	-	Quantum Dot
RF	-	Radio Frequency
RFSA	-	Radio Frequency Spectrum Analyzer
SA	-	Saturable Absorber
SDS	-	Sodium Dodecyl Sulphate
SESAM	-	Semiconductor Saturable Absorber Mirror
SMMA	-	Styrene Methyl Methacrylate
SNR	-	Signal-to-Noise Ratio
THF	-	Tetrahydrofuran
TI	-	Topological Insulator
TMD	-	Transition Metal Dichalcogenide
WDM	-	Wavelength Division Multiplexer

LIST OF SYMBOLS

Bi_2Se_3	-	Bismuth selenide
Bi_2Te_3	-	Bismuth telluride
BP	-	Black phosphorus
c	-	Velocity of light in vacuum
CaF_2	-	Calcium fluoride
E_c	-	Conduction band
E_p	-	Pulse energy
E_v	-	Valence band
Er^{3+}	-	Erbium ion
$E(k)$	-	Energy dispersion relation at K-point
h	-	Planck constant
I	-	Incident light
InGaAs	-	Indium gallium arsenide
k	-	Wavevectors in horizontal and vertical axes
K-point	-	Point at the middle of an edge in Brillouin zone
M-point	-	Point at the center of an edge in Brillouin zone (saddle point)
MoS_2	-	Molybdenum disulphide
MoSe_2	-	Molybdenum diselenide
MoTe_2	-	Molybdenum ditelluride
MXene	-	Transition metal carbides and nitrides
Na^+	-	Sodium ion
NaOH	-	Sodium hydroxide
Ni	-	Nickel
NiS_2	-	Nickel disulfide
PtS_2	-	Platinum disulfide
R_r	-	Repetition rate
ReSe_2	-	Rhenium diselenide
Sb_2Te_3	-	Antimony telluride
SnS_2	-	Stannic sulfide
SiC	-	Silicon carbide

TiSe ₂	-	Titanium diselenide
ν_0	-	Operating frequency
ν_F	-	Fermi velocity
WS ₂	-	Tungsten disulphide
WSe ₂	-	Tungsten diselenide
α	-	Length between the adjacent points of the hexagonal structure in reciprocal lattice
τ_d	-	Pulse width
γ_{th}	-	Threshold gain coefficient
σ -bond	-	Bond between valence electrons in graphene
π	-	Pi (3.142)
Γ -point	-	Gamma-point (origin) in Brillouin zone

CHAPTER 1

INTRODUCTION

1.1 Research Background

The foundation for laser technology development was dated to the early 1990s, specifically 1917, by none other than Albert Einstein. In the publication titled “The Quantum Theory of Radiation”, Einstein had proposed the theory of “stimulated emission” when he was investigating the light interaction with matter. He had come out with the hypothesis that light consist of individual “energy packages” based on his take on the quantum hypothesis by Planck (Bertolotti, 2015).

Only after 40 years was the “stimulated emission” theory put into practice by Charles Townes. Townes had carried out experiments on microwaves that led to the device construction that was able to generate and amplify the microwaves. Crediting to Einstein’s theory, Townes’ discovery was named “Maser”, which is an acronym for “microwave amplification by stimulated emission of radiation”. Several years later, Townes began exploring the prospect of extending the maser theory to a higher frequency and had a discussion with Gordon Gould on using optical pumping for this purpose (Hecht, 2010). After their discussion, both Townes and Gould work independently to find the solution, which leads to the invention of laser, which is an acronym for “light amplification by stimulated emission of radiation”, by Gould (Hecht, 2005). Since then, many researchers had taken interest in developing laser devices while figuring out the suitable active medium to excite its population inversion. Despite a hypothetical statement that ruby is not compatible with lasers, Theodore Maiman had investigated the material and proven the statement wrong. His discovery of ruby laser in 1960 was assembled from a flash lamp, a chromium-doped ruby, and a metal sleeve (Hecht, 2005). Though his discovery was not given much attention during his time, the significance of his discovery that was rooted back to the principle proposed by Einstein had become clear throughout the years.

After the demonstration of ruby laser by Maiman, Elias Snitzer proposed the use of glasses doped with rare earths in laser devices, resulting in the first demonstration of fiber laser. Later in 1964, he teamed up with Charles Koester to further develop his idea into the invention of the first fiber amplifier by imitating Maiman's ruby laser design with fiber instead of ruby (Hecht, 2010). Twenty years later, the concept of fiber laser by Snitzer had inspired David Payne to dope the fiber core with rare earth element. From one of his extensive experiments, Payne had reported that erbium doped fiber had produced gain near the minimum attenuation region of optical fiber (Mears et al., 1987). Furthermore, the pump wavelength of 980 nm and 1480 nm had been found to provide good erbium amplification for a practical amplifier design, by Payne's research group and Snitzer respectively (Laming et al., 1989; Snitzer et al., 1988). The broad bandwidth and low crosstalk of erbium amplifier is advantageous for wavelength-division multiplexing (Taga et al., 1990). The advancement in rare-earth doped optical fibers technologies had a big contribution on the fiber laser construction, in which alignment-free operation at different operating wavelengths are possible.

Technological advantages offered by pulsed laser sources made them the reliable alternatives for various applications, ranging from optical communications to industrial materials processing (Siegman, 1986). Common methods used in pulsed laser generation are Q-switching and mode-locking, where both can be realized through active or passive techniques though the latter is frequently preferred due to its simplicity and flexibility. For active technique, external modulators are needed to induce pulses when triggered by electrical signals (Kieu and Mansuripur, 2006). This resulted in a complicated and costly laser system with lack of reliability and controllability. On the other hand, the passive technique modulates the intracavity loss using a saturable absorber (SA) as opposed to an external modulator (Wang et al., 2011). This in turn, provide a simple and flexible laser system.

The remarkable quality of graphene in terms of mechanical strength, structural substantiality along with the electrical and chemical properties, has pushed graphene to the center of attention in various fields, especially photonics. Since the first report by Zhang et al. (2009), graphene has been widely used as SA in pulsed laser generation

(Yusoff et al., 2019; Zuikafly et al., 2019; Aziz et al., 2017; Mansoor et al., 2018; Hussin et al., 2017). It's astounding characteristics such as ultrafast carrier relaxation time and ultra-broadband operating wavelength with nonlinear optical response has made it an excellent SA material for pulsed laser generation (Bonaccorso et al., 2010).

1.2 Problem Statement

Various SAs have been introduced for passive Q-switching throughout the years. Among them are semiconductor saturable absorber mirror (SESAM), and 2D materials such as carbon nanotube (CNT), black phosphorus (BP), tungsten disulfide (WS_2) and graphene. SESAM is one of the earliest SAs used for pulsed laser generation, but it has a complex fabrication process and narrow wavelength range. This motivates the interest towards 2D materials as SA with remarkable saturable absorption properties. However, in practical environment, SAs based on 2D materials also have some drawbacks such as the range of the absorption wavelength of CNT SA depends on the tube diameter while BP SA is easily degraded as it is unstable in ambient atmosphere (Wang et al., 2019). As for graphene SA, although it has low modulation depth and large non-saturable losses, its zero-bandgap property, wide operating wavelength along with its simple and low-cost fabrication justified its relevance as a good SA in terms of performance and economic. The merits and demerits of some SA materials are summarised in Table 1.1.

Table 1.1 Pros and Cons of Some Saturable Absorber Materials (Wang et al., 2019)

Materials	Advantages	Disadvantages
SESAM	Mature technology Wide applications	Complex fabrication Narrow wavelength range High cost
CNT	Wide waveband absorption Low cost	Absorption wavelength range depends on tube diameter Difficult to disperse
BP	Direct and layer-sensitive bandgap	Unstable in ambient atmosphere
WS ₂	Layer-sensitive bandgap	Complex fabrication
Graphene	Zero bandgap Wide operating wavelength Simple fabrication Low cost	Low modulation depth Large non-saturable losses

The first mechanical exfoliation of graphite for graphene synthesis in 2004 had spurred various breakthroughs and discoveries in the conventional graphene synthesis methods (Vijayaraghavan, 2013). Mechanical exfoliation method was replaced with chemical exfoliation method such as chemical vapour deposition (CVD) despite the low cost of the former, to cater for large scale production and production of graphene flakes with the size of lower than tens of micrometers (Avouris and Dimitrakopoulos, 2012). Despite the possibility of large production of single-layer as well as few-layer graphene, the various variables involved in the CVD process such as different substrate choices and cooling rates may negatively affect the segregation of carbon (Yu et al., 2008; Bae et al., 2010). On the other hand, graphene synthesis by graphitization of hexagonal silicon carbide (SiC) crystals involving high temperature of approximately 1500 °C reported by Emtsev et al. (2009) produced irregular graphene layers with wrinkled surface and restricted mobility of graphene carriers (Farmer et al., 2009). Taken into consideration of the limitations of these synthesis methods, an electrochemical exfoliation method is used for graphene synthesis in this research. Using apparatus that are mostly available in the laboratory, this synthesis method can

be done in room temperature within the span of few hours. Other approach is by using a commercially available graphene filament. The fabrication of graphene saturable absorber from both approaches can be done at room temperature with relatively simple procedure.

For graphene SA fabrication, graphene was homogenized with host polymer for ease of integration within the laser cavity. Saturable absorber materials ranging from topological insulators to metal nanoparticles had widely used synthetic polymers such as polydimethylsiloxane (PDMS), polyvinyl alcohol (PVA), polyethylene oxide (PEO), and polymethyl methacrylate (PMMA) for that purpose (Aziz et al., 2017; Ng et al., 2020; Nady et al., 2018; Zhang et al., 2018). However, with the increasing environmental awareness, biodegradable and compostable alternatives in various aspects are passionately explored. Hence, chitin biopolymer is included in this work as an alternative to the conventional host polymer, namely PVA. The Q-switching laser performance of the fabricated graphene SAs in 1.5 μm region is reported, evaluated and compared in terms of repetition rate, pulse width, peak power, pulse energy and signal-to-noise ratio (SNR).

1.3 Objectives

The general purpose of this research is to demonstrate the pulse train of the Q-switching operation in erbium-doped fiber laser (EDFL) ring cavity using graphene based passive SA. Hence, the objectives of this research are:

1. To fabricate an environmental-friendly graphene-based SA using chitin biopolymer.
2. To investigate the physical and optical characteristic of graphene-based SA.
3. To generate and characterize pulsed laser in the 1.5 μm region in terms of repetition rate, pulse width, pulse energy, peak power, and signal-to-noise ratio.

1.4 Research Scope

The research covers the fabrication process of graphene SAs, including graphene synthesis by electrochemical exfoliation. Detailed process for electrochemical exfoliation from the preparation of the sodium dodecyl sulphate (SDS) solution to the centrifugation of the graphene suspension was demonstrated. Besides electrochemical exfoliated graphene, graphene filament was also used to develop the saturable absorbers along with the host polymers of PVA and chitin. The preparation of PVA solution was also done. The surface morphology and thickness of the fabricated saturable absorbers were then identified by using field emission scanning electron microscope (FESEM) and 3D measuring laser microscope respectively. The surface morphology of the SAs was observed to identify the molecular structure of the SAs and validate the even dispersion of graphene in the polymer matrices. After that, the performance of the SAs in passive Q-switching laser generation in the 1.5 μm region is observed in terms of repetition rate, pulse width, peak power, pulse energy and SNR. Reliable Q-switched pulse is of high repetition rate and low pulse width in the range of kHz and μs respectively. Meanwhile, laser generated with SNR value greater than 30 dB is often depicted as possessing high laser stability. Performance comparisons were done on graphene SAs with different starting materials and also between those of different host polymers.

1.5 Significance of Study

This research contributes to the advancement of the pulsed laser generation in the 1.5 μm region which is prominent for telecommunication field. Application such as LIDAR, remote sensing, ranging, and 3D imaging may benefit from this advancement (Agrawal and Ganotra, 2020). The significance of the research includes the detailed demonstration of graphene synthesis through electrochemical exfoliation. Also, the use of graphene filament as the base material for graphene saturable absorber fabrication. Besides that, chitin biopolymer was also incorporated as an ecological-friendly alternative to the conventional synthetic polymer, namely PVA. The performance of the graphene saturable absorbers with both host polymers were also

analysed in the passive Q-switching operation. This research demonstrated the application of a relatively new and more biological-friendly approach in the production of graphene saturable absorber that will contribute to the progress of fiber laser in the telecommunication field.

1.6 Overview

The thesis is organized in five chapters in which the first chapter introduced the background and history of laser, as well as the motivation and objectives of the research. The rest is as follows:

Chapter 2 started off with the background of EDFL and Q-switching along with its key parameters. Along with the working principle of saturable absorber, the host polymer for its fabrication is also discussed as well as the integration method. Then, graphene and its properties are thoroughly reviewed as well as its application in pulsed laser generation. Not only that, synthesis method of graphene is also discussed.

Chapter 3 compiles the methodology in realizing the objectives of this research. The fabrication process of the graphene SAs from preparation to the end-product is documented. Then, the characterization in terms of surface morphology and thickness is reported. After that, the fiber laser cavity used in this research is introduced along with its components followed by the equipment used during the experiment. The data collection and representation are also explained.

Chapter 4 discusses the performance of the Q-switching operation within the EDFL cavity realized by the fabricated graphene SAs.

Chapter 5 concludes the research along with suggestion for improvement in the future works extended from this study.

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