



UNIVERSITI PUTRA MALAYSIA

***FEMTOSECOND MODE-LOCKED FIBER LASERS INCORPORATING
GRAPHENE-BASED SATURABLE ABSORBERS***

LAU KUEN YAO

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**FEMTOSECOND MODE-LOCKED FIBER LASERS INCORPORATING
GRAPHENE-BASED SATURABLE ABSORBERS**

By

LAU KUEN YAO

**Thesis Submitted to the School of Graduate Studies,
Universiti Putra Malaysia, in Fulfilment of the
Requirement for the Degree of Doctor of Philosophy**

November 2017

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Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment
of the requirement for the degree of Doctor of Philosophy

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LAU KUEN YAO

November 2017

Chairman : Mohd. Adzir b. Mahdi, PhD
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The advancement of mode-locked fiber laser (MLFL) in femtosecond range has wide applications especially in biomedical sciences. For instance, femtosecond lasers are employed for cancer treatment at cellular level. MLFL is achieved by mode-locking regime with an appropriate mode-locker. Over the past few years, researchers have shown substantial interest in fabricating efficient saturable absorbers (SAs) as passive mode-locker due to its minimal weight, mechanically stable and highly nonlinear properties. Two-dimensional materials are popular for SA fabrication due to broadband and almost wavelength independent absorption. However, fabrication techniques to incorporate two-dimensional material in SAs involve tedious procedures especially wet chemicals. This research work focuses on the generation of femtosecond pulses utilizing two different types of sandwich-structured SA; graphene-polymethyl-methacrylate (PMMA) thin-film and graphene nanoplatelet (GNP) powder. The graphene/PMMA-SA is made through simple transfer procedure of the thin-film on a fiber ferrule. On the other hand, the GNP-SA is realized by mechanical exfoliation technique of GNP powder on a fiber ferrule. The optical characterization shows that graphene/PMMA-SA possesses larger modulation depth and lower insertion loss as compared to GNP-SA, thus leading to stronger saturable absorption for pulse shaping mechanism. The main aspect of the study is to validate the fabrication techniques through enhanced laser architectures in erbium-doped fiber laser (EDFL); single- and dual-lasing output. The cavity optimization is carried out for both lasing operation in order to achieve stable mode-locking operation in femtosecond range. For the optimized setup, the EDFL with graphene/PMMA saturable absorber is able to generate around 700 fs at approximately 1556 nm wavelength range. For dual-lasing MLFL generation, its main challenge is to have a balance net cavity gain at different wavelengths. For erbium materials, the interested lasing wavelengths are about 1530 nm and 1560 nm within its emission range. In order to minimize the complexity of configuration,

most of the optical components are shared between these two lasing wavelengths. This research work proposes laser architectures that utilize common gain medium and SA. Two red/blue wavelength division multiplexers are employed to provide a mechanism to split/combine these two wavelength ranges. Based on the proposed laser architectures, lasing directions are also investigated; unidirectional and bidirectional. The optimized pulse width of 730 fs and 870 fs are obtained at 1530 nm and 1560 nm, respectively. These findings are achieved with the bidirectional MLFL architecture incorporating graphene/PMMA-SA. The achievement of this research work solves the limitation of optical pulses measured in picosecond range from previous works, while new architectures of dual-lasing mode-locked EDFLs are successfully designed and executed.



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

**LASER GENTIAN PENGUNCIAN MOD DALAM MENJANA FEMTO-
SAAT BERINTEGRASIKAN PENYERAP TEPU BERUNSUR GRAPHENE**

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November 2017

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Kemajuan laser gentian penguncian mod (MLFL) dalam rangkaian femto-saat menjadi topik yang hangat terutamanya dalam aplikasi sains bio-perubatan. Contohnya, laser gentian ini diamalkan dalam rawatan kanser pada tahap sel dengan kepersisan tinggi. MLFL dicapai oleh rejim penguncian mod yang menggabungkan penyerap tepu (SA) yang bersesuaian. Semenjak beberapa tahun belakangan ini, para penyelidik telah menunjukkan minat dalam menghasilkan SA pasif yang mudah diserap dengan ciri-ciri ringan, stabil and sifat tidak linear yang tinggi untuk mencapai operasi penguncian mod. Walau bagaimanapun, teknik fabrikasi untuk mengamalkan bahan dua dimensi dalam SA melibatkan langkah-langkah rumit terutamanya penggunaan bahan kimia basah. Kajian ini bertumpu pada penajaan MLFL dengan membentuk dua SA berstruktur sandwic yang mempunyai seni bina yang tidak rumit. SA ini menggabungkan bahan graphene terdiri daripada filem nipis yang berstruktur lapisan graphene berpusat di atas polimetil-methacrylate (PMMA) dan serbuk graphene nanoplatelet (GNP). SA berunsur graphene/PMMA dibentuk melalui prosedur pemindahan filem tipis pada ferrule serat. Serentak itu, SA berunsur GNP direalisasikan dengan teknik pengelasan mekanikal serbuk GNP pada ferrule serat. Keputusan pencirian SA menunjukkan bahawa graphene/PMMA-SA mempunyai kehilangan sisipan yang lebih rendah dan kedalaman modulasi yang lebih tinggi berbanding dengan GNP-SA. Rentetan itu, ciri ini menyebabkan penyerapan yang lebih kuat untuk menjana denyutan laser. Aspek utama kajian ini adalah untuk megesahkan teknik fabrikasi melalui seni bina laser yang dipertingkat dalam laser serat erbium-doped (EDFL) melalui pengeluaran tunggal dan dwi-lasing. Pengoptimuman kedua-dua rongga laser dijalankan untuk mencapai operasi pengendapan mod stabil dalam julat femto-saat. Rongga EDFL yang dioptimumkan dengan penyerap graphene/PMMA dapat menjana 700 fs pada panjang gelombang dalam lingkungan 1556 nm. Cabaran utama untuk generasi dwi-lasing MLFL merupakan pencapaianan

keseimbangan keuntungan rongga bersih pada panjang gelombang yang berlainan. Panjang gelombang yang merit untuk bahan erbium merupakan 1530 nm dan 1560 nm. Banyak komponen optik dalam rongga dwi-lasing MLFL dikongsi pada kedua-dua gelombang panjang ini untuk meringankan kerumitan konfigurasi. Kerja penyelidikan ini mencadangkan seni bina laser yang menggunakan medium keuntungan biasa dan SA pada laluan perkongsian bahan optik. Dua multiplexer bahagian gelombang merah/biru digunakan untuk membelah dan menggabungkan dua julat panjang gelombang ini. Berdasarkan seni bina laser yang dicadangkan, arahan lasing juga disiasat sama ada satu arah ataupun dwi-arah. Lebar optik denyutan optimum diperolehi dengan pencapaian 730 fs dan 870 fs pada 1530 nm dan 1560 nm masing-masing. Penemuan ini dicapai dengan seni bina MLFL dua hala menggunakan graphene/PMMA-SA. Natijahnya, pencapaian kajian ini menyelesaikan batasan optik denyutan yang hanya menghasilkan piko-saat daripada kerja-kerja lain manakala seni bina rongga dwi-MLFL telah direka dan dilaksanakan dengan sempurna.

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I certify that a Thesis Examination Committee has met on 30 November 2017 to conduct the final examination of Lau Kuen Yao on his thesis entitled "Femtosecond Mode-Locked Fiber Lasers Incorporating Graphene-based Saturable Absorbers" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Doctor of Philosophy.

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TABLE OF CONTENTS

| | |
|------------------------------|-------------|
| ABSTRACT | Page |
| <i>ABSTRAK</i> | i |
| ACKNOWLEDGEMENTS | iii |
| APPROVAL | v |
| DECLARATION | vi |
| LIST OF FIGURES | viii |
| LIST OF TABLES | xiii |
| LIST OF ABBREVIATIONS | xvi |
| | xvii |

CHAPTER

| | |
|--|-----------|
| 1 INTRODUCTION | 1 |
| 1.1 Overview | 1 |
| 1.2 Problem statements | 2 |
| 1.3 Research objectives | 3 |
| 1.4 Research scope | 4 |
| 1.5 Thesis organization | 4 |
| 2 LITERATURE REVIEW | 6 |
| 2.1 Overview | 6 |
| 2.2 Mode-locking | 6 |
| 2.2.1 Active mode-locking | 7 |
| 2.2.2 Passive mode-Locking | 8 |
| 2.2.3 Saturable absorber | 9 |
| 2.2.4 Graphene | 10 |
| 2.3 Nonlinear Schrodinger Equation | 11 |
| 2.4 Dynamics of pulse evolution in mode-locked lasers | 15 |
| 2.4.1 Dispersion | 15 |
| 2.4.2 Group velocity dispersion | 16 |
| 2.4.3 Kerr nonlinearity | 18 |
| 2.4.4 Soliton formation | 19 |
| 2.5 Time bandwidth product | 20 |
| 2.6 Recent development in graphene-based saturable absorbers | 21 |
| 2.7 Summary | 32 |
| 3 MATERIAL AND OPTICAL CHARACTERIZATION OF GRAPHENE-BASED SATURABLE ABSORBERS | 33 |
| 3.1 Introduction | 33 |
| 3.2 Material characterization | 33 |

| | | |
|----------|--|-----------|
| 3.2.1 | Graphene/PMMA thin film | 33 |
| 3.2.2 | Graphene nanoplatelet powder | 36 |
| 3.3 | Fabrication of saturable absorbers | 39 |
| 3.4 | Material characterization of graphene materials on fiber core | 41 |
| 3.4.1 | SEM image | 42 |
| 3.4.2 | Energy dispersive x-ray spectrum | 45 |
| 3.4.3 | Raman spectrum | 46 |
| 3.5 | Optical characterization of graphene materials n fiber core | 47 |
| 3.5.1 | Transmission loss | 47 |
| 3.5.2 | Nonlinear saturable absorbance properties | 48 |
| 3.6 | Summary | 51 |
| 4 | SINGLE-LASING MODE-LOCKED ULTRASHORT FIBER LASER | 52 |
| 4.1 | Introduction | 52 |
| 4.2 | Single-lasing ML-EDFL cavity | 52 |
| 4.3 | Net dispersion optimization using cut-back method | 53 |
| 4.3.1 | Net GVD calculation | 53 |
| 4.3.2 | Optical spectrum | 54 |
| 4.3.3 | Pulse trains | 58 |
| 4.3.4 | Pulse duration | 60 |
| 4.3.5 | Pulse stability | 61 |
| 4.3.6 | Power development | 63 |
| 4.3.7 | Time bandwidth product | 65 |
| 4.3.8 | Summary | 66 |
| 4.4 | Investigation of ML-EDFL performance using different coupling ratio with graphene/PMMA-SA and GNP-sA | 68 |
| 4.4.1 | Optical spectrum | 68 |
| 4.4.2 | Pulse trains | 72 |
| 4.4.3 | Pulse duration | 74 |
| 4.4.4 | Pulse stability | 76 |
| 4.4.5 | Power development | 78 |
| 4.4.6 | Time bandwidth product | 81 |
| 4.4.7 | Summary | 82 |
| 5 | DUAL-LASING MODE-LOCKED ULTRASHORT FIBER LASER | 84 |
| 5.1 | Introduction | 84 |
| 5.2 | Research motivation for dual-lasing EDFL | 84 |
| 5.3 | Unidirectional signal propagation splitting two mode-locked lasers in dual-lasing ML-EDFL regime | 85 |
| 5.3.1 | Optical spectrum | 85 |
| 5.3.2 | Pulse duration | 87 |
| 5.3.3 | Pulse trains | 87 |
| 5.3.4 | Pulse stability | 88 |
| 5.3.5 | Power development | 89 |

| | | |
|----------|---|-----|
| 5.4 | Bidirectional signal propagation splitting two mode-locked lasers in dual-lasing ML-EDFL regime | 90 |
| 5.4.1 | Optical spectrum | 91 |
| 5.4.2 | Pulse duration | 92 |
| 5.4.3 | Pulse trains | 93 |
| 5.4.4 | Pulse stability | 94 |
| 5.4.5 | Power development | 95 |
| 5.5 | Summary | 97 |
| 6 | CONCLUSION AND RECOMMENDATION FOR FUTURE WORK | 98 |
| 6.1 | Conclusion | 98 |
| 6.2 | Main contribution of research | 100 |
| 6.3 | Recommendations for future work | 100 |
| | REFERENCES | 102 |
| | BIODATA OF STUDENT | 113 |
| | LIST OF PUBLICATIONS | 114 |

LIST OF FIGURES

| Figure | Page |
|--|------|
| 1.1 The scope of the research. (SESAM – semiconductor saturable absorber mirror; TI – topological insulator; TMD – transition metal dichalcogenides; BP – black phosphorus; GVD – group velocity dispersion; OCR – output coupling ratio). | 4 |
| 2.1 Schematic diagram of actively mode-locking scheme | 7 |
| 2.2 Schematic diagram of passive mode-locking operation using SA. | 8 |
| 2.3 (a) Absorption of photogenerated carriers in graphene, (b) the absorption (Fermi-Dirac distribution) and recombination (equilibrium distribution) of electron-hole pair under low excitation intensity, (c) the filling of the band-edges blocking further absorption under high excitation intensity. | 11 |
| 3.1 (a) Trivial transfer Graphene TM , (b) Schematic diagram and (c) 3-D profiler image of GPTF (PMMA and graphene layer) laying on polymer substrate. | 34 |
| 3.2 Raman spectrum of GPTF. | 35 |
| 3.3 Raman spectrum of the graphene layer with etched PMMA. | 36 |
| 3.4 (a) UV-VIS spectrum, (b) Raman spectrum, (c) XRD, (d) TGA, (e) FESEM image and (f) TEM image of GNP powder. | 38 |
| 3.5 GPTF transfer process from polymer substrate to filter paper. | 39 |
| 3.6 Fabrication procedures of graphene/PMMA-SA. | 40 |
| 3.7 Fabrication procedures of GNP-SA. | 41 |
| 3.8 SEM image of transferred GPTF on fiber ferrules with magnification size of (a) 750, (b) 5,000, and (c) 20,000. | 43 |
| 3.9 SEM image of GNP powder on fiber ferrules with magnification size of (a) 750, (b) 5,000, and (c) 20,000. | 44 |
| 3.10 EDX spectra of (a) GPTF and (b) GNP powder on fiber core region of the fiber ferrule. | 45 |
| 3.11 Raman spectra of (a) GPTF and (b) GNP powder on fiber core region of the fiber ferrule. | 46 |

| | | |
|------|---|----|
| 3.12 | Transmission loss of (a) graphene/PMMA-SAs and (b) GNP-SAs across C-band wavelength region. | 48 |
| 3.13 | Nonlinear saturable absorbance of SA measurement setup. | 49 |
| 3.14 | Nonlinear saturable absorbance properties of (a) graphene/PMMA-SAs and (b) GNP-SAs. | 50 |
| 4.1 | Experimental setup of single-lasing ML-EDFL. | 53 |
| 4.2 | Spectral bandwidth optimization with different SMF length. | 55 |
| 4.3 | Optical spectrum of MLFL with additional SMF length of (a) 3 m, (b) 2 m, and (c) 0.5 m. | 57 |
| 4.4 | Fundamental pulse repetition rate measurement with SMF length variation. | 58 |
| 4.5 | (a) Fundamental and (b) multiple pulse trains of ML-EDFL with 2 m SMF added at the pump power of 80 mW and 100 mW, respectively. | 59 |
| 4.6 | Pulse duration measurement with sech^2 fitted profile using different additional SMF length. | 60 |
| 4.7 | Autocorrelation traces for ML-EDFL with additional SMF length of (a) 0.5 m and (b) 2 m. | 61 |
| 4.8 | (a) PER measurement using different additional SMF length, and (b) frequency spectrum of ML-EDFL deployed with additional 2 m SMF length. | 62 |
| 4.9 | (a) Output power and (b) Pulse energy against pump power variation using different additional SMF length. | 64 |
| 4.10 | Central wavelength and spectral bandwidth measurement of ML-EDFL using graphene/PMMA-SA with different coupling ratios. | 68 |
| 4.11 | Optical spectrum of ML-EDFL generated by graphene/PMMA-SA with OCR of (a) 30 %, (b) 60 % and (c) 80 %. | 70 |
| 4.12 | Central wavelength and spectral bandwidth measurement of ML-EDFL using graphene/PMMA-SA with different coupling ratios. | 71 |
| 4.13 | Optical spectrum of ML-EDFL generated by GNP-SA with OCR of (a) 10 %, (b) 30 %, (c) 50 %, and (d) 60 %. | 72 |
| 4.14 | Pulse repetition rate measurement of ML-EDFL using (a) graphene/PMMA-SA and (b) GNP-SA with different OCR. | 73 |

| | | |
|------|---|----|
| 4.15 | Pulse duration measurement of ML-EDFL using (a) graphene/PMMA-SA and (b) GNP-SA with different OCR. | 75 |
| 4.16 | PER measurement of ML-EDFL of ML-EDFL using (a) graphene/PMMA-SA and (b) GNP-SA with different OCR | 77 |
| 4.17 | Power development of ML-EDFL of ML-EDFL using (a) graphene/PMMA-SA and (b) GNP-SA with different OCR | 79 |
| 4.18 | Pulse energy development of ML-EDFL of ML-EDFL using (a) graphene/PMMA-SA and (b) GNP-SA with different OCR | 80 |
| 5.1 | Experimental setup of MLFL-U. | 85 |
| 5.2 | Optical spectrum of MLFL-U using (a) graphene/PMMA-SA and (b) GNP-SA. | 86 |
| 5.3 | Autocorrelation trace of MLFL-U using (a) graphene/PMMA-SA and (b) GNP-SA. | 87 |
| 5.4 | Oscilloscope trace of MLFL-U using (a) graphene/PMMA-SA and (b) GNP-SA. | 88 |
| 5.5 | RF spectrum of MLFL-U using (a) graphene/PMMA-SA and (b) GNP-SA. | 89 |
| 5.6 | Development curve for average output power and pulse energy as a function of pump power for MLFL-U using (a) graphene/PMMA-SA and (b) GNP-SA. | 90 |
| 5.7 | Experimental setup of MLFL-B. | 91 |
| 5.8 | Optical spectrum of MLFL-B using (a) graphene/PMMA-SA and (b) GNP-SA. | 92 |
| 5.9 | Autocorrelation trace of MLFL-B using (a) graphene/PMMA-SA and (b) GNP-SA. | 93 |
| 5.10 | Oscilloscope trace of MLFL-B using (a) graphene/PMMA-SA and (b) GNP-SA. | 94 |
| 5.11 | RF spectrum of MLFL-B using (a) graphene/PMMA-SA and (b) GNP-SA. | 95 |
| 5.12 | Development curve for average output power and pulse energy as a function of pump power for MLFL-B using (a) graphene/PMMA-SA and (b) GNP-SA. | 96 |

LIST OF TABLES

| Table | | Page |
|-------|--|------|
| 2.1 | Summary of recent mode-locked fiber lasers using graphene-based SA. | 23 |
| 2.2 | Summary of previous works on mode-locked fiber lasers incorporating GNP-SA. | 27 |
| 2.3 | Summary of previous works on dual-lasing mode-locking operation. | 29 |
| 4.1 | GVD calculation tabulated for ml-EDFL with different SMF length. | 54 |
| 4.2 | Summary of power development and pulse energy evolution with the laser slope efficiencies. | 65 |
| 4.3 | TBP estimation for ML-EDFL with different fiber length. | 66 |
| 4.4 | Optimization of ML-EDFL with different SMF length. | 67 |
| 4.5 | TBP estimation for ML-EDFL of two graphene-SAs with different OCR. | 81 |
| 4.6 | Summary of ML-EDFL optimization using graphene/PMMA-SA with different OCR. | 82 |
| 4.7 | Summary of ML-EDFL optimization using GNP-SA with different OCR. | 83 |
| 5.1 | Comparison for average output power and pulse energy achievement with respective slope efficiencies measurement for MLFL-U and MLFL-B using graphene/PMMA-SA and GNP-SA. | 96 |

LIST OF ABBREVIATIONS

| | |
|-------|---|
| AC | Autocorrelation |
| AFM | Atomic force microscope |
| ASE | Amplified spontaneous emission |
| BP | Black phosphorus |
| CNT | Carbon nanotube |
| CVD | Chemical vapour deposition |
| cw | continuous wave |
| DMF | Dimethyl-formamide |
| EDF | Erbium-doped fiber |
| EDFL | Erbium-doped fiber laser |
| EDX | Energy dispersive X-ray |
| FESEM | Field effect scanning electron microscope |
| FWHM | Full-width at half-maximum |
| FWM | Four-wave mixing |
| GDD | Group delay dispersion |
| GNP | Graphene nanoplatelet |
| GO | Graphene oxide |
| GPTF | Graphene/PMMA thin film |
| GVD | Group velocity dispersion |
| LD | Laser diode |
| MD | Modulation depth |
| ML | Mode-locking |
| MLFL | Mode-locked fiber laser |

| | |
|-------|-----------------------------------|
| MOPA | Master oscillator power amplifier |
| MWCNT | Multi-walled carbon nanotube |
| NLSE | Nonlinear Schrodinger equation |
| OC | Optical coupler |
| OCR | Output coupling ratio |
| OPM | Optical power meter |
| OSA | Optical spectrum analyzer |
| PC | Polarization controller |
| PCF | Photonics crystal fiber |
| PD | Photodetector |
| PDMS | Poly-dimethyl siloxane |
| PER | Peak-to-pedestal extinction ratio |
| PMMA | Poly-methylmethacrylate |
| PVA | Polyvinyl-alcohol |
| SMMA | Styrene-methylmethacrylate |
| SPM | Self-phase modulation |
| SSFM | Split-step Fourier method |
| SWCNT | Single-walled carbon nanotube |
| TDFL | Thulium-doped fiber laser |
| TEM | Transmission electron microscope |
| TGA | Thermogravimetric analysis |
| TI | Topological insulator |
| TMD | Transition metal dichalcogenides |
| TOD | Third order dispersion |

| | |
|--------|---------------------------------|
| UV-VIS | Ultraviolet-visible |
| VOA | Variable optical attenuator |
| WDM | Wavelength division multiplexer |
| XRD | X-ray diffraction |
| YDFL | Ytterbium-doped fiber laser |



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CHAPTER 1

INTRODUCTION

1.1 Overview

Ultrashort pulse lasers in the order of few femtoseconds attract substantial research efforts in modern times due to their vast applications. For instance, micro-processing, biomedical diagnoses, optics communication, optical metrology, and optical signal processing are routinely applied with femtosecond lasers [1, 2]. In medical field, three-dimensional image of human retina, blood vessels and skin epidermal layers are operated with femtosecond laser sources [3]. In molecular science, the gaseous atoms and molecules are probed and manipulated by the femtosecond laser-induced optical frequency combs [4]. In astrophysics, the spectrometers are accurately calibrated by the femtosecond lasers, which results in the Doppler Shift measurement of stellar objects within 1cm/s error deviation [5]. In high energy application, femtosecond lasers are generally used for timing synchronization in large-scale accelerator to generate coherent X-ray pulses in free-electron laser systems [6].

Ultrashort pulse lasers performance is highly attractive at 1.5 μm region due to broad gain bandwidth depending on the composition of fiber core and on the inversion level. In this wavelength range, erbium-doped fiber (EDF) has emerged as a strong candidate for gain medium owing to its large gain bandwidth of typically tens of nanometers. Erbium ions provide signal amplification around 1550 nm wavelength which creates substantial research contribution for arrays of pulsed laser applications.

Femtosecond pulses are generated from passively mode-locked (ML) regime employing appropriate saturable absorbers (SAs) in fiber laser cavities. The first semiconductor-based SA was demonstrated with semiconductor saturable absorber mirror (SESAM) by Keller et al. in 1992 for mode-locking operation [7]. SESAM shows excellent performance in terms of its possibility for defect engineering and micro-fabrication growth [8]. However, complex fabrication and packaging of cost-ineffective SESAM such as post-growth processing in ion implantation reduces the device response time [9, 10]. In addition, SESAM has limited operating wavelength range or narrow tuning range of few tens nm, which is not suitable for the broadband tunable pulse generation [11]. The limitation of SESAM has encouraged the findings of carbon nanotube (CNT) SA as an alternative. CNT shows superior properties such as sub-picosecond recovery time, mechanically and environmentally robust, low saturation intensity, non-costly, and ease of integration into optical system [10]. Nevertheless, the strict requirement for diameter and chirality control of CNT-SA for energy bandgap design makes CNT

typically less competent and thus encourages the efforts of discovering other new materials as SA for fiber laser system [9, 12]. For instance, stringent requirement of concentration and thickness control of single-walled carbon nanotube (SWCNT) is required to produce stable mode-locked pulse operation as reported in [13]. In addition, multi-walled carbon nanotube (MWCNT) generates broader optical pulse width than the SWCNT due to larger nonlinear saturated absorption as demonstrated in [14].

In regards to this, graphene has been reported by Bao et al. [15] to be a potential candidate as an SA that possesses ultrafast non-linear saturable absorption and gapless linear dispersion of Dirac electrons [16, 17, 18]. Under low electron excitation intensity, photons are absorbed and electrons are excited from lower Dirac cone to upper Dirac cone for a typical graphene Fermi-Dirac cone. If there is no further excitation to saturate the absorption process, the negatively-charge carriers will recombine with the hole in the valence band to form a balanced Fermi-Dirac distribution. With higher excitation intensity whereby more photons react with graphene atoms, the photo-generated carriers are tremendously excited, thus, filling the states near the edge of valence band and conduction band completely. In this case, no absorption can take place at the edges of conduction band and valence band since no two electrons can fill in the same state [19]. As a result, the saturable absorption status is achieved to support optical pulses generation.

1.2 Problem Statement and Motivation

SA-based mode-locked erbium-doped fiber laser (EDFL) is an attractive aspect due to its stability and simple structure. Based on recent publications, graphene is one of popular materials that have been utilized as the SA in mode-locked EDFLs. Among SA structures, the most common one is the sandwich-structured SA owing to its fabrication simplicity [9]. This SA requires a nanomaterial-based thin film to be placed between two fiber ferrules. The light interaction occurs within the fiber core which is layered by this thin film. Most graphene thin films compose of graphene/polymer composites. In this case, an appropriate solvent to disperse graphene powder is important to form the composite with polymer. On the other hand, some solvents utilized to disperse graphene powder such as ethanol and dimethyl-formamide do not dissolve in polymer easily. Furthermore the orientation of graphene layers in composites is uncontrollable. From reported works, the characteristics of graphene-based SA are highly dependent on the number of layers [20]. In general, the SA performance is deteriorated with higher number of graphene layers. Therefore, it is very important to control the graphene layer orientation for this thin-film based SA.

Since the discovery of graphene, the mechanical exfoliation technique using a scotch tape becomes famous. The technique can be utilized to imprint nanomaterial directly on a substrate. For sandwich-structured SA, this technique can be applied.

The advantage of this technique as compared to the thin-film, it does not require any polymer hosts and dissolving solvents. The utilization of graphene nanoplatelet (GNP) as sandwich-structured SA has been recently reported. However, the pulse width of optical pulses is only limited to picosecond range only. This is due to the low modulation depth of GNP-SA which reflects to the high number of stacking GNPs. Hence, the fabrication technique of imprinted GNP on a fiber ferrule can be further enhanced to achieve femtosecond pulses.

Additionally, previous researches on mode-locked fiber laser (MLFL) operated solely in single-lasing regime incorporating graphene-based SA, thus raising the opportunity to explore the potential of graphene-based SA in dual-lasing mode-locked fiber laser performance. There are only several recent works reported on dual-lasing pulsed laser using a SA. For instance, CNT-SA is extensively used to produce dual-lasing ML laser. However, CNT shows disadvantages in generating dual-lasing mode-locked laser because its tube diameter is absolutely significant in determining the laser operating wavelength [21]. Therefore, the limitation of CNT-SA is evaded by incorporating graphene-SA for the generation of dual-lasing pulsed laser. However, another conundrum that has yet to be solved is the long pulse width achieved in dual-lasing mode-locking at picosecond range incorporating graphene-based SA, which raises research gap in improving the pulse laser performance.

1.3 Aim and Objective

The main objective of this research is to generate optical pulses in femtosecond range using graphene-based SA through simple fabrication methods. The specific objectives are as follow:

- I. To fabricate sandwich-structured SA using graphene/PMMA through thin/film transferring process.
- II. To fabricate sandwich-structured SA using GNP powder through direct imprinting technique.
- III. To generate femtosecond pulses in the wavelength range of 1.55 μm using EDFL ring cavity architecture incorporating the fabricated graphene-based SAs.
- IV. To design and develop dual-lasing femtosecond pulsed EDFL utilizing the fabricated graphene-based SAs.

1.4 Research Scope

Figure 1.1 illustrates the scope of research that will be studied in this doctoral work which focuses on the generation of passively mode-locked fiber lasers with ring-cavity at femtosecond range pulse width incorporating graphene-based saturable absorbers. The topics related to the main contribution of this work are investigated due to various advantages and several gaps that were filled and explored. The work is conducted at 1550 nm using EDF as active gain medium.

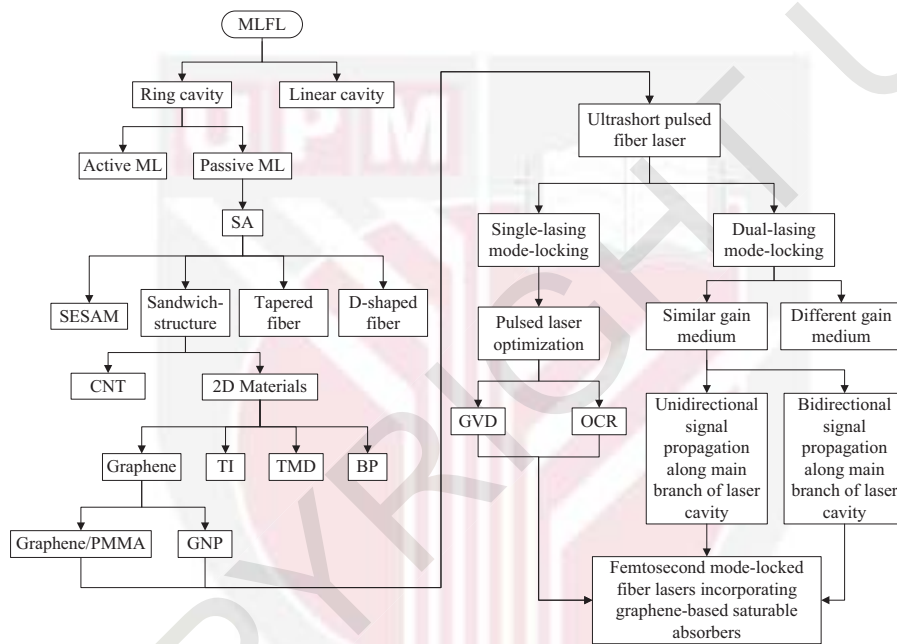


Figure 1.1: The scope of the research. (SESAM – semiconductor saturable absorber mirror; TI – topological insulator; TMD – transition metal dichalcogenide; BP – black phosphorus; GVD – group velocity dispersion; OCR – output coupling ratio).

1.5 Thesis Organization

This thesis consists of six chapters. The first chapter (Chapter 1) is devoted to the introduction of thesis. In this chapter, the application of ML laser to produce ultrashort pulses, problems and objectives of the research work are presented. The literature review is then elaborated in Chapter 2. The graphene, SA, ML scheme are discussed here. In addition, the pulse evolution of soliton-based ML

mechanisms is explained based on Nonlinear Schrodinger Equation, which is solved by using Split Step Fourier Method.

Chapter 3 presents the material characterization of graphene/PMMA thin film and GNP powder which are used to fabricate SA. The SA fabrication procedures are elaborated as well. The SA fabrication is repeated to ensure its repeatability which is then optically characterized in terms of transmission loss and nonlinear saturable absorption performance. Chapter 4 and Chapter 5 describe the ML laser performance by introducing the SA in an EDFL cavity. In Chapter 4, dispersion and output laser coupling ratio are varied and studied in order to study their effects on the optical spectrum and pulse duration. Chapter 5 is related to the dual-lasing ML operation whereby the graphene-based SA is employed to generate simultaneous lasers at approximately 1530 nm and 1560 nm.

In the last chapter (Chapter 6), the overall observation and findings drawn based on the experimental results discussed in the previous chapters is concluded. This is thereafter continued by the discussions on the achievements and problems met by the proposed works as well as recommendations for improvement that can be practically implemented in future studies.

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