

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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November 2017

Chairman : Mohd. Adzir b. Mahdi, PhD Faculty : Engineering

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 sandwichstructured 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

By

LAU KUEN YAO

November 2017

Pengerusi : Mohd. Adzir b. Mahdi, PhD Fakulti : Kejuruteraan

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 langkahlangkah 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 pencapaian

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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ν

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 Philosphy. The members of the Supervisory Committee were as follows:

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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

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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
РММА	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
ТЕМ	Transmission electron microscope
TGA	Thermogravimetric analysis
TI	Topological insulator
TMD	Transition metal dichalcogenides
TOD	Third order dispersion

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



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, microprocessing, 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 scorch 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 ringcavity 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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