



UNIVERSITI PUTRA MALAYSIA

***MICROFIBER-BASED SATURABLE ABSORBER INCORPORATING
GRAPHENE POLYMER NANOCOMPOSITES FOR FEMTOSECOND
PULSE GENERATION***

NG ENG KHOON

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PULSE GENERATION**

By

NG ENG KHOON

**Thesis Submitted to the School of Graduate Studies,
Universiti Putra Malaysia, in Fulfilment of the
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Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Master of Science

MICROFIBER-BASED SATURABLE ABSORBER INCORPORATING GRAPHENE POLYMER NANOCOMPOSITES FOR FEMTOSECOND PULSE GENERATION

By

NG ENG KHOON

November 2017

Chairman : Mohd. Adzir b. Mahdi, PhD
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Since the advent of pulse laser, the duration of shortest pulse has rapidly changed from the nanosecond (10^{-9} s) to the femtosecond (10^{-12} s) regime. Ultrashort light pulses can be generated using mode-locking techniques which contain either an active element or a nonlinear passive element in a laser resonator. The current commercialized ultrashort laser technologies are typically based on semiconductor saturable absorber mirror (SESAM) which requires complex fabrication method and fine tuning between the fiber pigtail and SESAM to generate mode-locking. A number of fiber-based saturable absorber papers have been reported to overcome this problem. In line with this advancement, this research work focuses on microfiber-based saturable absorber incorporating graphene composite as a nonlinear component in different operating wavelength regions of laser cavity.

The first process in the experimental work is to fabricate a saturable absorber that is able to generate femtosecond pulse in a ring cavity. The graphene nanoparticles are prepared through liquid phase exfoliation method. Then, the nanoparticles are synthesized with PDMS to produce a graphene composite. Finally, the graphene composite is coated on a prepared microfiber through dip coating method. The prepared microfiber has waist diameter of 10 μm , waist length of 0.5 mm, and total length of 60.5 mm. The quality of coating on the microfiber is characterized through Raman spectroscopy, field effect scanning electron microscope and energy dispersive X-ray spectroscopy. The fabricated saturable absorber has transmission loss of less than 4.6 dB and modulation depth of 9.6%.

In this research, a ring-configuration erbium-doped fiber laser (EDFL) setup is employed to generate optical pulses with the assistance of the fabricated inline graphene composite saturable absorber. This saturable absorber initiates ultrashort pulse signal with observation of multiple Kelly's sidebands, output pulse train with constant round trip time and pulse width within femtosecond range. The generation of optical pulses is performed in two wavelength ranges; C-band and L-band. For each band, the dispersion is optimized to ensure that the fiber laser produces soliton pulses. The soliton pulse is observed with the presence of Kelly's sidebands at the laser output. For C-band, the fabricated saturable absorber is placed in a ring cavity with the employment of 5 m HP980 erbium-doped fiber (EDF). The mode-locked operation is observed at 33.54 mW pump power. The output pulse has a central wavelength of 1557.05 nm with 3 dB spectral width of 5.92 nm. The generated soliton pulse has pulse duration of 631 fs, repetition rate of 9.65 MHz and time bandwidth product of 0.46. For L-band fiber laser, the same saturable absorber is utilized with 17 m long LIEKKI EDF. The mode-locked threshold pump power is obtained at 39.6 mW. The output laser is generated at 1599.56 nm with 3 dB spectral width of 5.773 nm. Stable mode-locked pulse with pulse duration of 568 fs, repetition rate of 5.76 MHz, and time bandwidth product of 0.38.

In conclusion, the fabricated graphene composite microfiber has been proven to function capably as saturable absorber in C-band and L-band. This shows that it can operate in wide operating wavelength range. The quality of optical pulse in the range of femtosecond indicates its ability to generate ultrashort pulses with strong saturation absorption. The time bandwidth product above 0.315 denotes the operation is close to ideal Fourier transform limited pulse. Overall, the results validate the reliability of the proposed method to produce microfiber-based saturable absorber.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia
sebagai memenuhi keperluan untuk ijazah Master Sains

**MIKROFIBER-BERASASKAN PENYERAP BOLEH TEPU GABUNGAN
NANOKOMPOSIT POLIMER GRAPHENE UNTUK PENGHASILAN
GENERASI DENYUTAN FEMTOSAAT**

By

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November 2017

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Sejak separuh abad lalu, laser telah dicipta untuk menjana tempoh denyut nadi terpanjang berubah dengan pesat dari rejim nanosaat (10^{-9} s) ke femtosaat (10^{-12} s). Aplikasi ini mengamalkan penggunaan penyerap boleh tepu (SA) di dalam kaviti laser. Denyutan cahaya ultrapendek boleh dijana menggunakan teknik selakan mod yang mengandungi unsur aktif atau elemen pasif tidak linear dalam resonator laser. Teknologi laser ultrapendek belakangan ini menggunakan kanca penyerap boleh tepu semikonduktor (SESAM). Alat ini memerlukan cara fabrikasi yang rumit untuk menjana rejim selakan mod. Pelbagai kerja penyelidikan telah dipapar untuk mengatasi masalah ini. Selanjutnya, kerja penyelidikan ini memberi tumpuan kepada mikrofiber berasaskan penyerap boleh tepu yang menggabungkan komposit graphene sebagai komponen tidak linear dalam kawasan kaviti laser yang beroperasi dalam jarak gelombang yang berlainan.

Penemuan pertama dalam kerja penyelidikan ini adalah untuk mencipta penyerap boleh tepu yang dapat menghasilkan denyutan femtosaat dalam kaviti laser berpusingan. Nanozarah Graphene disediakan dengan campuran antara polimer dan graphene melalui kaedah fasa pengelupasan cecair. Kemudian, graphene polimer dihasilkan melalui gabungan nanozarah graphene dan polimer (PDMS). Akhirnya, graphene polimer disalut pada mikrofiber dengan menggunakan teknik rendaman. Mikrofiber yang digunakan menunjukkan ukur lilit sebanyak $10\ \mu\text{m}$, panjang ukur lilit sebanyak $0.5\ \text{mm}$ dan jumlah panjang ialah $60.5\ \text{mm}$. Kualiti salutan di mikrofiber diuji dengan FESEM, Spektroskopi Raman dan Spektroskopi X-ray. Hasil salutan penyerap boleh tepu mempunyai kehilangan penghantaran kurang daripada $4.6\ \text{dB}$ dan kedalaman modulasi sebanyak 9.6 peratus.

Dalam penyelidikan ini, konfigurasi kaviti membulat laser gentian terdop erbium (EDFL) yang dipraktikkan dengan penyerap boleh tepu berunsur graphene menghasilkan selakan mod. Permulaan isyarat denyutan ultrapendek disaksikan dengan pemerhatian beberapa jalur-sisi Kelly, penghasilan denyut-pawai dengan pemalar masa pusingan dan lebar denyut dalam julat femtosaat. Generasi denyutan optik dijalankan dalam dua julat panjang gelombang iaitu Jalur-C dan Jalur-L. Penyerakan kaviti laser dioptimumkan untuk menjamin penghasilan denyutan soliton untuk setiap jalur. Denyutan soliton dicerap dengan kehadiran jalur sisi Kelly di pusat penghasilan gentian laser. Untuk Jalur-C, penyerap boleh tepu diletakan dalam kaviti membulat dengan penggunaan 5 m HP980 gentian terdop erbium (EDF). Operasi selakan mod didapati pada 33.54 mW kuasa pam. Laser ini menghasilkan panjang gelombang pada 1557.05 nm dan lebar jalur spectrum 3-dB sebanyak 5.92 nm. Rentetan itu, lebar optiks denyutan, kadar pengulangan dan produk masa lebar jalur dicapai pada 631 fs, 9.65 MHz dan 0.46 masing-masing. Pada kontrasnya, penyerap tepu yang sama digunakan untuk menjana selakan mod dengan mempraktikkan 17 m LIEKKI gentian terdop erbium. Operasi selakan mod diperhatikan pada 39.6 mW. Penghasilan laser didapati oleh panjang gelombang pada 1599.56 nm dengan lebar jalur spektrum 3-dB sebanyak 5.773 nm. Denyutan selakan mod yang stabil telah dibuktikan dengan lebar optiks denyutan sebanyak 568 fs, pencapaian kadar pengulangan pada 5.76 MHz dan produk lebar jalur dengan keputusan 0.38.

Natijahnya, penghasilan mikrofiber komposit graphene telah terbukti untuk berfungsi sebagai penyerap boleh tepu di Jalur-C dan Jalur-L. Pencapaian ini membuktikan potensi penyerap tepu ini untuk beroperasi dalam panjang gelombang yang lebar. Kualiti denyutan optiks dalam julat femtosaat menunjukkan keupayaannya untuk menghasilkan denyutan ultrapendek yang mempunyai serapan tepu yang kuat. Masa produk jalur lebar yang lebih dari 0.315 menunjukkan operasi ini hampir sama dengan denyutan terhad transformasi Fourier yang unggul. Ringkasannya, keputusan kerja penyelidikan ini menunjukkan kestabilan kaedah menghasilkan penyerap tepu menggunakan mikrofiber.

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I certify that a Thesis Examination Committee has met on 22 November 2017 to conduct the final examination of Ng Eng Khoon on his thesis entitled "Microfiber-Based Saturable Absorber Incorporating Graphene Polymer Nanocomposites for Femtosecond Pulse Generation" 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 Master of Science.

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LIST OF ABBREVIATIONS

ASE	Amplified spontaneous emission
Bi_2Te_3	Bismuth telluride
BP	Black phosphorus
CNT	Carbon nanotube
CVD	Chemical vapor deposition
CW	Continuous wave
EDF	Erbium-doped fiber
EDFL	Erbium-doped fiber laser
EDX	Energy dispersive X-ray spectroscopy
FESEM	Field emission scanning electron microscope
FWHM	Full width at half maximum
GVD	Group velocity dispersion
ISO	Isolator
LD	Laser diode
NPR	Nonlinear polarization rotation
OSA	Optical spectrum analyzer
OPM	Optical power meter
OC	Optical coupler
PC	Polarization controller
PCF	Photonic crystal fiber
PDL	Polarization dependent loss
PDMS	Polydimethylsiloxane
PEO	Polyethylene oxide

PER	Peak-to-pedestal extinction ratio
PET	Polyethylene terephthalate
PMMA	Polymethyl-methacrylate
PVA	Polyvinyl alcohol
rGO	Reduced graphene oxide
RF	Radio frequency
SA	Saturable absorber
SESAM	Semiconductor saturable absorber mirror
TBP	Time bandwidth product
TIs	Topological insulators
THF	Tetrahydrofuran
TMDCs	Transition metal dichalcogenides
VOA	Variable optical attenuator
WDM	Wavelength division multiplexer
2D	Two-dimensional



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CHAPTER 1

INTRODUCTION

1.1 Overview

Fiber lasers consist of optical fibers as gain medium in which their core is doped with rare earth elements such as erbium, ytterbium and thulium. In those early years, ultrashort fiber lasers have been in the spotlight owing to their potential applications in manufacturing, micromachining, biomedical imaging, supercontinuum generation and optical metrology. Generally, ultrashort pulses are light pulses with pulse duration of picoseconds or femtoseconds. The generation of ultrashort pulses via active and passive mode locking technique has fascinated researchers to explore this field due to their various potentials as described earlier.

In pulsed fiber lasers, there are many methods to generate ultrashort pulses at a different wavelength with different pulse energies and durations. One of the well-known methods is to fabricate an optical saturable absorber (SA) as one of the elements in a laser cavity [1]. The function of SA is to allow extreme light intensity to propagate at reduced light absorption. In addition, ultrashort pulses can also be generated by using nonlinear polarization rotation technique (NPR) [2]. When a high intensity light propagates inside the non-polarization maintaining fiber, the nonlinearity takes place which causes self-phase modulation and birefringent effect. Therefore, readjustment of polarization rotation becomes essential and it is very hard to achieve experimentally. A more stable NPR-based fiber laser can be realized by using Faraday rotators with combination of polarization maintaining fiber in a laser cavity [3]. Optical modulators can also be utilized to generate ultrashort pulses by manipulating the propagation properties of oscillating light. The two common types of optical modulators are acousto-optic and electro-optic types. This type of active mode-locking can generate optical pulse of 533 ps with repetition rate of 80 MHz [4].

In this research work, we focus on studying the generation of ultrashort pulses using a passive SA in a laser cavity. There are various techniques to fabricate SA such as sandwich-type [5], microfiber [6], hollow-core photonic crystal fiber (PCF) [7], D-shaped fiber [8], and semiconductor saturable absorber mirror (SESAM) [9]. These techniques incorporate nanomaterials that have required optical properties for mode-locking such as two-dimensional materials (graphene) [10], carbon nanotube [6], topological insulators [11] and transition metal dichalcogenides [12]. Based on the reported techniques, graphene-polymer based-microfiber SA is chosen as our focused device due to its simple fabrication, low cost and wide broadband wavelength.

1.2 Problem Statement and Motivation

Current state-of-the-art femtosecond fiber lasers are still based on SESAMs for achieving mode-locking [9, 13-14]. The fabrication of SESAMs involves complex techniques and sophisticated equipment that results in higher cost [15-16]. Furthermore, the high dispersion band of SESAM prevents the generation of shortest possible optical pulse length. In addition, fine alignment between the fiber pigtail and the SESAM is required for achieving successful mode-locking. A special mount is also required to hold the fiber and the SESAM together in place. Moreover, regular maintenances are required to optimize and realign the fiber and SESAM. Therefore, an alternative method is to employ a microfiber-based SA embedded with nanomaterials. This type of SA is based on the light interaction from the evanescent field. In this case, it does not need any complicated fabrication techniques and alignment to achieve mode locking. Therefore, it is possibly to become a mature SA device as an alternative replacement to SESAMs.

1.3 Aim and Objective

- I. To design and fabricate a microfiber by incorporating graphene polymer composites.
- II. To study the characteristics of graphene polymer-based microfiber to function as SA.
- III. To investigate the pulsed laser performances of fabricated graphene polymer-based microfiber SA in mode-locked erbium-doped fiber laser (EDFL).

1.4 Scope of Work

The scope of work in the research is summarized in Figure 1.1. Generally, this research looks into the design and development of a microfiber-based SA for generation of ultrashort pulses. The microfiber is picked due to the evanescent wave property especially for lights that travel at the waist diameter. In this work, graphene is chosen as the nonlinear material. In order to attach this graphene on optical fiber surface, a polymer matrix is required. In this research work, graphene is mixed with polydimethylsiloxane (PDMS) polymer. The PDMS is selected owing to its refractive index of ~ 1.4 which is suitable for light waveguide in optical fibers. The graphene polymer-based microfiber will be optimized first based on optical and material characterization results in order to achieve mode locking. Overall, the main focus of this work is to explore a new fabrication technique of graphene-polymer microfiber SA that is capable to produce ultrashort pulses. For generation of ultrashort pulses in laser cavities, erbium-doped fiber (EDF) has been chosen due to its maturity in fiber laser research domain. Two different wavelength regimes

namely as C-band and L-band are investigated in this research work. The performance of the mode locking will be discussed in terms of spectral bandwidth, center wavelength, repetition rate, radio frequency spectrum, pulse width, pulse energy and stability of the fiber laser system.

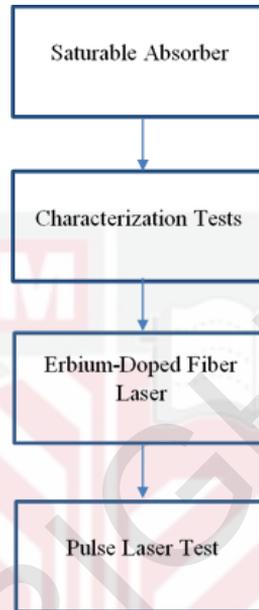


Figure 1.1: Scope of Work.

1.5 Organization of thesis

The organization of this thesis is explained as below:

Chapter 1 consists of the introduction and overview of mode-locked laser, especially fiber-based passive mode locker. The issues with passive mode locking fiber laser is highlighted as well as with the aim and objectives that are formed from those issues. The scope of work and thesis organization are also included in this chapter.

Chapter 2 introduces the fiber laser techniques in producing femtosecond laser source. This includes thorough discussion on related innovative fabrication techniques in the generation of mode-locked fiber lasers. Moreover, principle of graphene absorption is included together with microfiber adiabatic criterion for better understanding of fabrication SA.

Chapter 3 contains the methodology used in this work and few characterization tests are presented as a supporting evidence of generation mode locking. This includes details of fabrication process in getting a graphene polymer-based microfiber.

Chapter 4 provides the laser setups and investigation on mode locking performance. Two different wavelength regimes; C-band and L-band will be studied. All the findings are discussed and analyzed in this chapter.

Lastly, in chapter 5, the conclusion of research work with all the important results are highlighted. Recommendations for future work are also suggested at the end of this chapter.



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LIST OF PUBLICATIONS

- K. Y. Lau, **E. K. Ng**, M. H. Abu Bakar, A. F. Abas, M. T. Alresheedi, Z. Yusoff, M. A. Mahdi, "Low threshold L-band mode-locked ultrafast fiber laser assisted by microfiber-based carbon nanotube saturable absorber," *Optics Communications*, 413 (2018), 249-254.
- K. Y. Lau, **E. K. Ng**, M. H. Abu Bakar, A. F. Abas, M. T. Alresheedi, Z. Yusoff, M. A. Mahdi, "Low threshold linear cavity mode-locked fiber laser using microfiber-based carbon nanotube saturable absorber," *Optics & Laser Technology*, 102 (2018), 240-246.

