PASSIVELY Q-SWITCHED FIBER LASER EMPLOYING GRAPHENE AND CARBON NANOTUBES SATURABLE ABSORBER

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

Choose an item.

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OCTOBER 2022

DEDICATION

This thesis is dedicated to my parents, who taught me that the best kind of knowledge to have is that which is learned for its own sake. It is also dedicated to my wife one step at a time.

ACKNOWLEDGEMENT

Foremost, I would like to impart my gratefulness to Allah Almighty for his guidance and blessing along the bumpy path in completing this research.

In preparing this thesis, I was in contact with many people, researchers and academicians. They have contributed towards my understanding and thoughts. Particularly, I wish to express my sincere appreciation to my main supervisor, Assoc.Prof. Ir. Ts. Dr. Azura Binti Hamzah, for the encouragement, guidance and friendship throughout the process of completing this research. I am also very thankful to my co-supervisor Dr. Husni Hani Jameela Binti Sapingi for her advice and motivation. Without their continued support and interest, this thesis would not have been completed.

Universiti Teknologi Malaysia (UTM) and Unversiti Malaya also deserve special thanks for their assistance throughout this academic exploration as well as for allowing me to make use of their advanced laboratories facilities. My appreciation also goes to the members of the ODESY Ikohza in UTM. Not forgetting my family members especially my father, A.S. Mahroof, mother, A.M.Ajjimma and my wife M.A. Thilsath Banu for their continuous and unparalleled love, prayers, help and support.

I would like to express my gratitude and appreciation to my working place, Institute of Technology, University of Moratuwa, Sri Lanka for providing study leave and their continue support to pursue my higher studies (MPhil). Specially the director, deputy registrar, head of my division and other colleagues. Finally, I humbly extend my thanks to one and all, who directly or indirectly, lent their hands in this regard. My fellow postgraduate student should also be recognised for their support. My sincere appreciation also extends to all my colleagues and others who have provided assistance at various occasions.

ABSTRACT

Pulse laser has charmed photonic properties due to its high photon energy, broad absorption bandwidth and easy thermal management. These advantages allow it to be widely used in various applications especially in the area of electronic devices, scientific research, industrial, military and medical treatment. Q-switched fiber laser which has the ability to generate an energetic short pulse from a laser by modulating the intra-cavity losses and Q factor of the laser resonator. This Q-switched technique is practical for obtaining high energy and peak power with solid-state bulk lasers from microsecond to nanosecond pulses. These criteria make the research on Q-switched fiber laser is highly demanding. There are numerous techniques to generate the Qswitched fiber laser such as semiconductor saturable absorption mirror (SESAM), nonlinear polarization rotation (NPR), nonlinear optical loop mirror (NOLM), and nonlinear amplifying loop mirror (NALM). These techniques have drawbacks in terms of complex design and high fabrication cost. For instance, SESAM needs additional optical components such as lens, mirror or U-bench. To overcome these drawbacks, passively Q-switched erbium doped fiber laser (EDFL) employing carbon based saturable absorber (SA) is proposed in view of its advantages of ultrafast recovery time, simple fabrication process and easy to incorporate in the cavity. In this research, the passively Q-switched EDFL was generated by using graphene and carbon nanotubes (single-walled carbon nanotube (SWCNT) and multi-walled carbon nanotube (MWCNT)) based SA. The fabrication of graphene and CNT SAs was done by using dip coating and drop casting method. Polyvinyl alcohol (PVA) was used as the host polymer due to its excellent film-forming, very high flexibility and watersolubility. SWCNT SA solution was combined with PVA solution with the ratio of 1:1, 2:3 and 3:2, whereas MWCNT SA solution was mixed with PVA solution with the ratio of 1:1 and 2:3. Next, SAs were characterized for optical and physical spectroscopic characterization. For optical spectroscopic characterization, SAs were characterized by using Raman characterization and surface morphology, and for physical characterization was done by observing the thickness of the SA materials. The performance of Q-switched EDFL was analyzed in the same ring cavity configuration by using graphene, SWCNT and MWCNT based SA. The properties of passively Q-switched EDFL namely the output spectrum, repetition rate, pulse width, pulse energy, output power, pulse train, and signal to noise ratio were analyzed. When the performance of the three SAs was compared, the SWCNT 2:3 ratio SA had the best pulse width of 3.31µs, repetition rate of 134.4 kHz, and SNR value of 52 dB. The SWCNT with the ratio of 3:2 attained the highest pulse energy of 29.98 nJ. The findings of this research provide significant steps toward the development of future research in the advanced pulse laser technology especially in the application of industrial, military and medical area.

ABSTRAK

Laser nadi memiliki ciri-ciri fotonik yang terpesona kerana nilai tenaga fotonnya yang tinggi, tahap penyerapan jalur lebar yang luas dan pengurusan haba yang mudah. Kelebihan ini membolehkan ia digunakan secara meluas dalam pelbagai aplikasi terutamanya dalam bidang peranti elektronik, penyelidikan saintifik, industri, ketenteraan dan rawatan perubatan. Laser gentian suis-Q mempunyai kemampuan untuk menjana nadi pendek bertenaga daripada laser dengan memodulasikan kehilangan intra-rongga dan faktor Q bagi resonator laser. Teknik suis-Q ini adalah praktikal untuk mendapatkan tenaga tinggi dan kuasa puncak dengan laser pukal berpepejal daripada denyutan mikrosaat ke nanosaat. Kriteria ini menjadikan penyelidikan tentang laser gentian suis-Q mendapat perhatian. Terdapat pelbagai teknik untuk menjana laser gentian suis-Q seperti cermin serapan boleh tepu semikonduktor (SESAM), putaran polarisasi tak linear (NPR), cermin gelung optik tak linear (NOLM) dan cermin gelung penguat tak linear (NALM). Teknik ini mempunyai kelemahan dari segi reka bentuk yang rumit dan kos fabrikasi yang tinggi, Sebagai contoh, SESAM memerlukan komponen optik tambahan seperti kanta, cermin atau bangku-U. Untuk mengatasi kelemahan ini, laser gentian berasaskan erbium (EDFL) suis-Q bersifat pasif menggunakan penyerap boleh tepu berasaskan karbon (SA) dicadangkan memandangkan kelebihan kadar pemulihan ultra singkat, proses fabrikasi yang ringkas dan mudah untuk disatukan dengan rongga. Dalam penyelidikan ini, EDFL suis-Q bersifat pasif dijana dengan menggunakan grafin dan karbon nanotiub (Single-walled Carbon Nanotube (SWCNT) dan Multi-walled Carbon Nanotube, (MWCNT)) berasaskan SA. Pembuatan grafin dan CNT SA difabrikasi dengan menggunakan kaedah salutan celup dan tuangan titisan. Polivinil alkohol (PVA) digunakan sebagai polimer perumah kerana pembentukan filem yang sangat baik, fleksibiliti yang sangat tinggi dan keterlarutan air. Larutan SWCNT SA digabungkan dengan larutan PVA dengan nisbah 1:1, 2:3 dan 3:2 manakala larutan MWCNT SA dicampur dengan larutan PVA dengan nisbah 1:1 dan 2:3. Seterusnya, SA telah dicirikan dengan pencirian spektroskopi optik dan fizikal. Untuk pencirian spektroskopi optik, SA telah dicirikan dengan menggunakan pencirian Raman dan morfologi permukaan, dan untuk pencirian fizikal dilaksanakan dengan ukuran ketebalan bahan SA. Prestasi EDFL suis-Q telah dianalisis dalam konfigurasi rongga berbentuk cincin yang sama dengan menggunakan grafin, SWCNT dan MWCNT berasaskan SA. Sifat-sifat EDFL suis-Q pasif iaitu spektrum keluaran, kadar pengulangan, lebar nadi, tenaga nadi, kuasa output, nadi train dan nisbah isyarat kepada hingar (SNR) telah dianalisis. Apabila prestasi ketiga-tiga jenis SA dibandingkan, nisbah SWCNT-PVA 2:3 SA mempunyai lebar nadi terbaik 3.31µs, kadar pengulangan 134.4 kHz, dan nilai SNR 52 dB. SWCNT-PVA dengan nisbah 3:2 mencapai tenaga nadi tertinggi sebanyak 29.98 nJ. Hasil penyelidikan ini diharapkan dapat menjadi pemangkin ke arah pembangunan penyelidikan masa depan dalam teknologi laser nadi termaju terutamanya untuk sektor perindustrian, ketenteraan dan perubatan.

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LIST OF ABBREVIATIONS

2D	-	Two Dimensional
3D	-	Three Dimensional
AFM	-	Atomic Force Microscope
BP	-	Black Phosphorus
CNT	-	Carbon NanoTube
CVA	-	Chemical Vapour Deposition
CW	-	Continuous Wave
DI	-	Di-Ionized
EDFA	-	Erbium Doped Fiber Amplifier
EDX	-	Energy Dispersive X-ray analysis
FBG	-	Fiber Bragg Gratings
FESEM	-	Field Emission Scanning Electron Microscope
FRA	-	Fiber Raman Amplifier
GPS		Global Positioning System
KHz	-	Kilo Hertz
LASER	-	Light Amplification by Stimulated Emission of Radiation
LD	-	Laser Diode
LED	-	Light Emitting Diode
LiDAR	-	Light Deduction and Ranging
MHz	-	Mega Hertz
MMF	-	Multi-Mode Fiber
MWCNT	-	Multi-Wall Carbon NanoTube
NALM	-	Nonlinear Amplifying Loop Mirror
NPR	-	Nonlinear polarization rotation
NOLM	-	Nonlinear Optical Loop Mirror
OFCS	-	Optical Fiber Communication System
OPM	-	Optical Power Meter
OSA	-	Optical Spectrum Analyser
OSC	-	Oscilloscope
PC	-	Polarisation Controller
PMF	-	Polarization-Maintaining Fiber

PMMA	-	Polymethyl Methacrylate
PVA	-	Polyvinyl Alcohol
PW	-	Pulsed Wave
RBM	-	Radial Breathing Modes
RFSA		Radio Frequency Spectrum Analyzer
SA	-	Saturable Absorber
SDS	-	Sodium Dodecyl Sulphate
SEM	-	Scanning Electron Microscope
SESAM	-	Semiconductor saturable absorption mirror
SHL	-	Super Hybrid Lens
SMF	-	Single Mode Fiber
SNR	-	Signal to Noise Ratio
SOA	-	Semiconductor Optical Amplifier
SWCNT	-	Single-Wall Carbon NanoTube
TDFA	-	Thulium Doped Fiber Amplifier
TIs	-	Topological Insulators
TMDs	-	Transition Metal Di-chalcogenides
UTM	-	Universiti Teknologi Malaysia
WDM	-	Wavelength Division Multiplexer
YDF	-	Ytterbium Doped Fiber

LIST OF SYMBOLS

μs	-	Micro second
ns	-	Nano second
ps	-	Pico second
fs	-	Femto second
$\mathbf{f}_{\mathbf{o}}$	-	Resonant frequency
3	-	Energy stored
μJ	-	Micro Joule
L	-	Cavity Length
nJ	-	Nano Joule
E_1	-	Ground state
E_2	-	Excited state
E ₃	-	High level
Ep	-	Pulse energy
Fr		Repetition rate
h	-	Planck's constant
Er^{3+}	-	Erbium ion
Q	-	Q-factor
Po	-	Output Power
Δ_t	-	Pulse width
n	-	Refractive index
d_t	-	Diameter of tube
D	-	Disordered or defect region
G	-	Graphite region
2D or G-	-	Second-order Raman scattering from D-band variation
Tm^{3+}	-	Thulium
V	-	Frequency
Vg	-	Frequency separation between mode
dB	-	Decibel

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Appendix AList of equipment used throughout the research122

CHAPTER 1

INTRODUCTION

1.1 Research Background

Fiber laser technology is dominant in laser generation, especially in the fields of medicine, industrial processing as well as telecommunication. The first fiber laser was successfully experimented during 1960s(Dutta Majumdar and Manna, 2011). To reap the advantages such as stable giant pulse, high pulse energy, high peak power and low repetition rate, fiber lasers are now heavily used in various commercial and scientific instruments notably, surgery, spectroscopy, telecommunication and military instrument(Liu *et al.*, 2018).

There are two modes of fiber laser operation which are. continuous wave (CW) and pulse wave (PW). The laser beam with a continuous optical output is emitted from a CW laser, whereas, the laser beam is emitted in pulses (Liu *et al.*, 2021) in PW lasers. PW fiber lasers can either be implemented by Q-switching or mode-locking (Norizan *et al.*, 2018). The Q-switched fiber laser is a straignt forward, simple, compact and cost-effective technique to constrauct a pulsed laser(Norizan *et al.*, 2018) which in practice can be aceived by active and passive methods (Liu *et al.*, 2018).

The passive Q-switching is an effective technique to generate Q-switched pulsed fiber laser in a cavity, where a saturable absorber (SA) is to be used in addition(Norizan *et al.*, 2018). A general passively Q-switched ring cavity fiber laser experimental setup consists of a pump laser or laser diode (LD) pump, wavelength division multiplexer (WDM), gain medium, coupler, polarization controller (PC), isolator and SA. The core of the gain medium is doped with different types of dopants as a gain medium. Various types of gain media are being used to perform pulsed laser in the cavity, notably, erbium (Er³⁺), ytterbium (Yb³⁺), thulium (Tm³⁺), neodymium (Nd³⁺) dysprosium (Dy³⁺), gadolinium (Gd³⁺), samarium (Sm³⁺), promethium (Pm³⁺),

praseodymium (Pr^{3+}) and holmium (Ho^{3+})(Naji *et al.*, 2011). The most common dopant used as a gain medium is Er^{3+} . Many techniques have previously been utilized to analyse the performance of passively Q-switched EDF (1.55 µm) lasers for the telecommunication field, commercial industry, and medicine. Because Er^{3+} has a wavelength of 1550nm, EDFA is employed in the C-band (1530 - 1560 nm) and Lband (1570 - 1610 nm), where fiber loss is the lowest, and is supported by Wavelength Division Multiplexing (WDM) technology in the entire wavelength bands and it allows high bit rate transfer across long distance (Naji *et al.*, 2011).

The Q-switched pulsed laser regime was experimented by integrating different types of SAs. Passively Q-switched pulsed lasers use two types of SAs, namely, artificial SA and real SA. The artificial SA is a nonlinear switching-based fiber laser, in which the transmission or reflectivity of the fiber laser is determined by the nonlinear phase shift(Radzi *et al.*, 2020). Nonlinear polarization rotation (NPR), nonlinear optical loop mirror (NOLM) and nonlinear amplifying loop mirror (NALM) are some of the artificial SAs and they require continuous maintenance and complicated adjustment in the fiber laser cavity setup to create the absorption-emission mechanism (Radzi *et al.*, 2020). Moreover, these nonlinear SAs are less resistant to environmental changes (Chernysheva *et al.*, 2016). The NPR technique cannot achieve self-starting in Q-switching fiber lasers (Liu *et al.*, 2018). NPR is also susceptible to environmental factors like vibration and temperature, which limits its usefulness. On the other hand, to achieve sufficient nonlinear phase shifts in NOLM method, a long fiber is needed (Ahmed *et al.*, 2014).

Due to these drawbacks, there have been plenty of ongoing research dedicated towards its replacement with a newer technique that has a wider operating range, specifically the real SA. Real SAs can also be defined as material-based SA fiber lasers, for instance, semiconductor saturable absorption mirror (SESAM) and nanomaterial SAs.

Initially, when using SESAM and nanomaterial SAa in Q-switching technique, there have been some drawbacks such as, complex fabrication, costlier experimental setup, narrow-banded operating bandwidth, low damage thresholds, short operation lifespan, long recovery time (Kalaiyan *et al.*, 2020), requires additional optical components, (Dong *et al.*, 2011) and low energetic pulse.

Due to these drawbacks in SESAM and some nanomaterials SAs, recently, many research have been focusing on passively Q-switched fiber lasers based on carbon-based SAs, notably, graphene and carbon nanotubes (CNTs). Graphene and CNTs have more advantages, notably, ultrafast recovery time, simple fabrication process, cheaper in the market, no need additional components, easy to install in the laser cavity, and operate at broad bandwidth. CNTs also have excellent mechanical and optical properties (Note and Transform, 2000), as well as graphene has outstanding electronic (Zuikafly *et al.*, 2018a) and optical properties (Liu *et al.*, 2012).

Different types of polymer materials can be used as a host to SA materials, notably, polymethyl methacrylate (PMMA), styrene methyl methacrylate (SMMA), polyvinyl alcohol (PVA) and polycarbonate. Polymer/carbon base SA nanocomposites have piqued the interest of several researchers. Polymer/carbon base nanocomposites can exhibit both the outstanding characteristics of carbon base SAs and the polymer's good processability.

A significant challenge exists in processing and engineering applications for polymeric/carbon base composites because carbon base SA present as bundles, aggregates, and ropes during composite fabrication, resulting in poor dispersion in the polymer matrix and many irregularities sites in the fabricated composite(Salimbeygi *et al.*, 2013). PMMA/CNT raised the glass transition temperature of PMMA in another investigation(Yanmaz *et al.*, 2021). One of the challenges of PVA is high solubility in water(Hassan *et al.*, 2005). On another hand, SMMA's poor moisture absorption complicates the formation of a free-standing polymer composite SA thin film(Lau and Hou, 2021). In this scenario, the important concerns in increasing polymer properties are the homogenous distribution of carbon-based SA in the polymer matrix and the interface contact between SA and the polymer. Improvements in polymer characteristics are not yet at the anticipated level because to the problems associated with spreading the carbon-based SA in the polymer matrix.

PVA is a semi-crystalline and highly biocompatible and non-toxic. As a result, PVA is a promising polymer with prospective applications in different application notably, pharmaceutical and medical devices. PVA can be a good candidate for the matrix of carbon-based SAs because of its water solubility and high processability, and because the excellent dispersion of carbon-base SAs results in an aqueous solution (Salimbeygi *et al.*, 2013). PVA possesses outstanding film forming, adhesion, flexibility, oxygen barrier, and emulsifying capabilities(Huda *et al.*, 2020).

Correspondingly, a number of studies had been published related to passively Q-switched fiber laser. Overall, due to its unprecedented level of utility for many commercial and advanced scientific applications, the passively Q-switched fiber laser is always very efficient to produce pulse lasers.

1.2 Problem Statement

The laser output pulse parameters of passively Q-switched fibre lasers solely depend on the optical properties of saturable absorber materials being used, except for the resonant frequency of the laser, which depends on the laser gain medium, basically the doping ions. Nonlinear optical absorption property, and the modulation depth of the materials are the two key properties being capitalised in SA application. Different SA materials (SESAM, TMD, nanomaterials) with different host material (PVA, PMMA, SMMA) were used together with various gain media (EDF, TDF, YDF) in a passively Q-switched fiber laser ring cavity individually, in the past.

Two-dimensional (2D) graphene and one-dimensional (1D) single-wall carbon nanotube (SWCNT) and multi-wall carbon nano tube (MWCNT) are very common and deeply researched SAs in the past, having each one of them researched individually and independently in different all fiber laser configurations, reporting the output laser pulse behaviours. However, the performances and comparative analysis of these three SAs can logically be done only if these three are researched deploying exactly the same laser experimental configuration, where such experimental analysis using Q-switched erbium doped fibre laser have not been analysed yet. Here, in this research, 2D nanomaterial graphene, and 1D nano material SWCNT and MWCNT are fabricated into SAs, having the same host material of PVA and used together with erbium doped fibre (1.7 m) gain medium in an exactly the same experimental configuration and environment, generating laser pulse stream in a passively Q-switched erbium doped fibre laser researching for output parameters such as pulse width, repetition rate, pulse energy, output power, and SNR. Finally, the output parameters of all three SAs are subject to comparative analysis.

1.3 Objective

The main aim of this research work is to develop a compact and high performing passively Q-switched erbium doped fiber laser (EDFL) using carbon-based SAs. To achieve the target of the research, the following objectives have been outlined:

- (a) To fabricate graphene and carbon nanotube saturable absorber.
- (b) To characterize graphene and carbon nanotube saturable absorber by physical and optical properties.
- (c) To analyze the performance of pulse energy, output power, signal to noise ratio, repetition rate and pulse width of Q-switched fiber laser by employing graphene and carbon nanotubes SAs.

1.4 Scope of Study

This research is designed to focus on the following scopes:

- (a) The research study is based on ring cavity fiber lasers.
- (b) The gain medium used to generate pulse laser is EDFA.

- (c) The Q-switched fiber laser is based on passively Q-switched fiber laser. Carbon based SAs, namely, Graphene, SWCNT and MWCNT are employed in the ring cavity.
- (d) The host material used in this experiment to fabricate SAs is PVA.
- (e) The fabrication of SAs is via dip coating and drop casting method.
- (f) The SAs are characterized by optical (Raman Characterisation and surface morphology) and physical spectroscopy characterization (3D image and thickness measurement).
- (g) The comparative analysis of performance for passively Q-switched EDFL is in terms of pulse energy, output power, signal to noise ratio, repetition rate and pulse width.

1.5 Significance of study

Q-switched fiber laser techniques contribute towards the development in various fields due to its unique properties such as having high photon energy, broad absorption bandwidth and easy thermal management. Q-switching is of much interest to experts in multiple fields including medicine, material processing, LiDAR, telecommunication, environmental detection, laser acceleration (Fauziah *et al.*, 2017), photoacoustic imaging (Xian *et al.*, 2019), military and spectroscopy.

Different gain media are utilized for various applications, namely, 1-micron is used for material processing, 1.55-micron is used for telecommunication purpose and 2-micron is used for medical purpose. One of the main contributions of Q-switching fiber laser is devoted towards the medical field, namely tattoo removal, pigmentation removal and other skin related problems(Goh and Ho, 2015). Due to its low phase noise, this technology also advances radar, military and spectroscopy technology. Q-switched technique is widely used in cosmetic and data storage industry, due to its high peak power. Overall, the Q-switched fiber laser technique is significant for many applications that will turn up for future needs.

This research contributes to the advancement of fiber laser in telecommunication applications as 1.55-micron wavelength (EDF) has been used. It may be immediately employed in the advanced technology of fiber laser by selecting the best carbon-based SA material from 1D (SWCNT and MWCNT) and 2D (graphene) nanomaterial.

1.6 Flow of Research

Three work stages are executed to ensure all the research objectives are covered. The workflow shows the progress of the research to be completed throughout this project, notably, fabrication of SA materials, spectroscopy characterisation and comparative analysis of the performance of each SAs. The work stages and flow of the research are established and presented in Figure 1.1.

The first stage is concerned with the investigation on the current research and technologies related to SA in Q-switched fiber lasers. This includes the fundamental theory, SA Q-switched techniques, SA material selection, problem occurrences and methods of SA fabrication. Graphene and CNTs are selected as SAs in this research work. The fabrication of SAs is also included in the first stage. There are few types of SA fabrications to generate Q-switch. The dip coating and drop casting fabrication method are used to fabricate graphene and CNT SAs.

The second stage is focused on the spectroscopic characterization of the SA materials such as optical and physical characterization. Under optical characterization, Raman characterization and surface morphology characterization are done for the SA materials. Raman spectroscopy and FESEM spectroscopy are the equipment to find out the surface morphology, Raman spectrum, purity, and architecture of graphene and CNTs. Physical characterization is done by observing the 3D image, thickness histogram and thickness of the material by using 3D laser measuring microscope.

The third stage is concerned with the generation of Q-switched fiber laser employing different carbon-based SAs. Each SA is inserted in the fiber laser cavity and the performance of pulse width, repetition rate, signal to noise ratio, pulse train, pulse energy and output power will be analyzed here. The optimal coating material will be identified by evaluating the performance of graphene, SWCNT, and MWCNT SA materials from a same experimental configuration of Q-switched erbium doped fiber laser.



Figure 1.1 Flowchart of research methodology

1.7 Thesis Outline

This research work consists of five chapters. The related information is mentioned in each chapter. **Chapter-1** briefs an overview of the research background on fiber laser and Q-switching techniques. And then, the study background and literature review are used to identify the problems statement. This is followed by the objectives to achieve the targets. Then the scopes, significance of the research and flow of research are presented accordingly. Finally, the thesis outline of the research is clearly stated in the first chapter.

Chapter-2 discusses the literature review based on previous research paper, journals and articles concerning the Q-switched fiber laser, particularly on passively Q-switching techniques. Here, the possible areas of improvement for the Q-switched fiber laser are identified. The various topics related to Q-switched fiber laser are discussed in this chapter. The structure of optical fibers, types and operation of fiber laser, and the principal of optical amplification and lasers are discussed here. Then the technique of fiber laser, gain medium and respective wavelength are briefly discussed here. This chapter also covers the pulse parameter, including pulse energy, output power, pulse width, repetition rate, and SNR value. Different types of SAs, and SAs (graphene, SWCNT and MWCNT) used in this research are also detailed in this chapter. The use of the Q-switching approach for various wavelengths is then discussed. The SA mechanism and Q-switching process are then briefly explained. Afterwards, different fabrication method and spectroscopy characterization of SAs are briefly discussed. After then, the equipment utilized for SA characterization are described. Finally, a critical literature review on previous research on Q-switched fiber laser is described based on a table.

Chapter-3 explains the research methodology and the experimental methods. The configuration of the experimental setup, fabrication method, notably, dip coating and drop casting method, and spectroscopy characterization, namely surface morphology using FESEM spectroscopy, thickness measurement using 3D laser measuring microscope and Raman characterisation using Raman spectroscopy of SAs are explained in this chapter with brief description. **Chapter-4** covers the result analysis of the research. First of all, the output image of raman characterization, thickness measurement and surface morphology for all the SAs are listed here. And then, the experiment results are analyzed and verified in terms of output power (mW), pulse train, pulse width (μ s), repetition rate (kHz), signal to noise ratio (SNR) and pulse energy (nJ). Each SA's specs tropic characterisation, including its surface morphology, Raman characterization, and thickness measurement, is examined, and discussed. Based on the experiment results, the best coated SA material among graphene, SWCNT and MWCNT will be selected. Finally, the result of each SA is compared with some benchmark papers.

Chapter-5 concludes the main target of the research study. The achievement of each objective and contribution of the study are mentioned in this chapter. At the end, some recommendations related to the research are presented as an extension of the existing research study by employing different gain medium or double pumping scheme.

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