METAL BASED PASSIVE SATURABLE ABSORBER EMBEDDED IN CHITIN HOST POLYMER

NIK NUR ANIS AWADAH BINTI NIK ZAIN

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DEDICATION

This thesis is dedicated to my husband, mom, dad and siblings who has been my biggest supporter throughout this journey.

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ABSTRACT

Pulse fiber laser is used in many applications such as those in high-speed communication, optical imaging, laser machining and fiber optic sensing. This is due to its simple setup, compact geometry, zero alignment configuration and cost-saving nature. Passive Q-switching is a technique to produce pulse fiber laser. Saturable absorbers play important roles in passive Q-switching technique. Research works on materials for saturable absorbers (SAs) have been conducted for decades. Saturable absorbers began with dyes or coloured glasses. Currently, Two-Dimensional (2D) materials are employed as the SA for the Q-switching. Metal-based SAs like gold, silver and copper are reported to exhibit excellent optical properties. However, reports on copper-based SA are still scarce despite their ultrafast response time, broad operation spectrum, and large third-order nonlinearity. Previously, researchers employed physical vapor deposition and chemical reduction that had drawbacks like using expensive methods, generating low volume of materials, and having issues such as toxicity, high cost, and impurities with reducing agents. Therefore, this study used three types of metal which were copper, aluminium, and brass. Three-dimensional filament was used to simplify the fabrication process of these metals. To do this, bio-compatible host polymer, materials characterisation, and the implementation in pulse fiber laser within a 1.5 μm region were employed. The three-dimensional metal filament was combined with metal: chitin as the bio compatible host polymer with a ratio of 1:4, 1:1.5 and 1:1. Regarding the performance of the SAs, the lowest threshold pump power for the passive Q-switching technique was obtained by copper-chitin 1:1 with 38.7mW. Meanwhile, the highest repetition rate and the lowest pulse width was by brass-chitin 1:4 with 165.60 kHz and 3.46 μs respectively. Besides, the highest signal to noise ratio (SNR) was 80.18 dB by copper-chitin 1:1. Finally, the SAs utilised were stable to produce pulse lasers through passive Q-switching, with the lowest SNR value of 64 dB.

ABSTRAK

Laser gentian nadi telah digunakan dalam banyak aplikasi seperti komunikasi berkelajuan tinggi, pengimejan optik, pemesinan laser dan penderiaan gentian optik. Ini disebabkan oleh ciri-cirinya, yang mempunyai persediaan mudah, geometri padat, konfigurasi penjajaran sifar dan sifat penjimatan kos. Penukaran Q pasif adalah salah satu teknik untuk menghasilkan laser gentian nadi. Penyerap tepu memainkan peranan penting dalam teknik penukaran Q pasif. Penyelidikan mengenai bahan untuk penyerap tepu (SA) telah diteruskan sejak beberapa dekad yang lalu. Penyerap tepu ini telah bermula daripada pewarna atau cermin mata berwarna, dan pada masa ini, bahan Dua-Dimensi (2D) telah digunakan sebagai SA untuk pensuisan Q. SA berasaskan logam seperti emas, perak dan tembaga telah dilaporkan mempamerkan sifat optik yang sangat baik. Tidak seperti rakan logamnya, laporan mengenai SA berasaskan tembaga masih kurang walaupun masa tindak balasnya yang sangat pantas, spektrum operasi yang luas dan ketaklinearan tertib ketiga yang besar. Daripada kerja yang dilaporkan kebanyakan penyelidik, menggunakan pemendapan wap fizikal dan pengurangan kimia yang mempunyai kelemahan kaedah yang mahal, menghasilkan isipadu bahan yang rendah dan mempunyai masalah dengan agen pengurangan seperti ketoksikan, kos tinggi dan kekotoran. Oleh itu, kajian ini menggunakan tiga jenis logam iaitu, tembaga, aluminium dan loyang, tertumpu kepada fabrikasinya dengan menggunakan filamen tiga dimensi untuk memudahkan proses fabrikasi, bersama-sama dengan menggunakan polimer perumah serasi bio, pencirian bahan, dan pelaksanaannya. dalam laser gentian nadi dalam kawasan 1.5 μm. Filamen logam tiga dimensi telah digabungkan dengan logam: kitin sebagai polimer perumah serasi bio dengan nisbah 1:4, 1:1.5 dan 1:1. Membandingkan prestasi SA, kuasa pam ambang terendah untuk teknik penukaran Q pasif diperolehi oleh kitin tembaga 1:1 dengan 38.7mW. Sementara itu, kadar ulangan tertinggi dan lebar nadi terendah adalah oleh loyang-kitin 1:4 dengan 165.60 kHz dan 3.46 μs, masing-masing. Dari sudut pandangan lain, nisbah isyarat kepada hingar (SNR) tertinggi ialah 80.18 dB oleh tembaga-kitin 1:1. Akhirnya, SA yang digunakan dalam kajian ini terbukti stabil untuk menghasilkan laser nadi melalui pensuisan Q pasif, dengan nilai SNR terendah 64 dB.

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LIST OF ABBREVIATIONS

0D	-	0-Dimensional
1D	-	1-Dimensional
2D	-	2-Dimensional
AgNPs	-	Silver Nanoparticles
AuNP	-	Gold Nanoparticles
BP	-	Black Phosphorus
CB	-	Conduction Band
CNTs	-	Carbon Nanotubes
CuNWs	-	Copper Nanowires
CW	-	Continuous Wave
EDF	-	Erbium Doped Fiber
EDFA	-	Erbium Doped Fiber Amplifier
EDFL	-	Erbium Doped Fiber Laser
EDS	-	Energy Dispersive Spectroscopy
FESEM	-	Field Emission Scanning Electron Microscope
FWHM	-	Full Width at Half Maximum
GNS	-	Gold-Nanosphere
IR	-	Infrared
LSPR	-	Localized Surface Plasmon resonance
PDMS	-	Polydimethylsiloxane
PE	-	Pulse Energy
PEO	-	Polyethylene Oxide
PLA	-	Polylactic Acid
PMMA	-	Polymethyl Methacrylate
Pp	-	Peak Power
PVA	-	Polyvinyl Alcohol
PVP	-	Poly Vinyl Pyrrolidone
RFSA	-	Radio Frequency Spectrum Analyzer
Rr	-	Repetition rate
SA	-	Saturable Absorber

SEM	-	Scanning Electron Microscope
SESAMs	-	Semiconductor Saturable Absorber Mirrors
SNR	-	Signal to Noise Ratio
SPR	-	Surface Plasmon Resonance
THF	-	Tetrahydrofuran
TIs	-	Topological Insulators
TMDCs	-	Transition Metal Dischalgenides
UV	-	Ultraviolet
VB	-	Valence Band
WDM	-	Wavelength Division Multiplexing

LIST OF SYMBOLS

A_{op}	-	Average Output Power
Ag	-	Silver
$AgNO_3$	-	Silver Nitrate
Al	-	Aluminium
Au	-	Gold
AZO	-	Aluminium Zinc Oxide
CO_2	-	Carbon dioxide
Cu	-	Copper
CuO	-	Copper Oxide
E_p	-	Pulse Energy
Er^{3+}	-	Erbium ion
Fe_2O_3	-	Iron (III) Oxide
Gr-Ag	-	Graphene Silver
$HAuCl_4$	-	Gold (III) chloride
He	-	Helium
NaOH	-	Sodium Hydroxide
Nd: glass	-	Neodymium-Doped Glass
Nd: YAG	-	Neodymium-Doped Yttrium Aluminium Garnet
Ne	-	Neon
P_E	-	Pulse Energy
P_E	-	Pulse Energy
P_o	-	Average Output Power
P_p	-	Peak Power
Zn	-	Zinc
ZnO	-	Zinc Oxide
α_s	-	Modulation Depth
λ	-	Wavelength
τ_p	-	Pulse Width

CHAPTER 1

INTRODUCTION

1.1 Research Background

The acronym "laser" stands for "light amplification by stimulated emission of radiation," and it is a technology that is commonly employed in our daily lives. Lasers have grown so widespread in recent years that it is hard to believe the first one constructed only 60 years ago (Maiman, 1960). While the science of light has not changed, laser technology has grown swiftly, and there is now a variety of laser kinds accessible that were unimaginable 60 years ago. The creation of short and ultra-short pulses in a fiber laser cavity is one of the most recent advances in laser technology.

Pulse fiber lasers produce exceptionally high peak power from brief pulses than continuous wave fiber lasers. This is useful for nonlinear optics, high-speed communications, optical imaging, material processing, medical treatment, range detection, fiber optic sensing, and laser machining (Sadeq et al., 2018; Ahmad et al., 2015). In comparison to continuous wave fiber lasers, pulsed fiber lasers have shown promise in various photonics sectors during the last decade. These fiber lasers use a particular optical fiber doped with rare earth elements, including ytterbium, erbium, and thulium, allowing light to flow through at 1 µm, 1.5 µm, and 2 µm wavelengths to emit a few tens of milliwatts of power (Ibrahim, 2017).

Erbium is the most often utilised gain medium in Erbium-Doped Fiber Laser (EDFL), highlighted as one of the potentials for growth in optoelectronic and optical domains such as wavelength division multiplexing (WDM) systems, fiber sensors, and fiber optics instrumentation. In the last decade, many researchers have been drawn to EDFL because of its capabilities in optical communication and fiber sensor applications (Kang et al.,2016). Due to its broad gain bandwidth centred at 1550 nm, erbium-doped fiber (EDF) is extensively utilised in Erbium-doped fiber amplifier

(EDFA) as an amplifier device in current optical communication systems. Furthermore, a growing number of researchers are focusing on pulsed laser production due to its ability to create pulses and increase laser output performance using a passive saturable absorber, which is not possible with a continuous wave (CW) laser.

Q-switching pulsed fiber lasers can be made using active or passive modulation methods. Active techniques are often sophisticated and costly, making them unsuitable for small or high-density systems (Ahmad *et al*., 2013). Meanwhile, passive techniques provide a viable alternative to actively modulated systems with a wide range of real-world applications. Because of its simplicity, compactness, and flexibility, the passive Q-switching approach will be the focus of this study. The use of a saturable absorber to absorb laser photons until it is saturated before releasing them, switching the energy level from maximum to minimum in the form of a brief and strong pulse, is known as passive Q-switching. The passively Q-switched approach has been demonstrated to be a beneficial way of generating dependable and steady optical pulses from a fiber laser (Ahmad *et al.*, 2015). Saturable absorber (SA) modulates cavity loss in passive Q-switched fiber lasers and switching occurs when the gain reaches its maximum energy and saturates. The saturable absorber absorbs light at low intensities (opaque) and transmits it at high intensities (transparent) (Ismail *et al*., 2016).

Saturable absorber (SA) generally holds the property to absorb light with increasing light intensity which is shown by most materials at extremely high optical intensities. For pulsed laser generation, the design of the SA optimizes several crucial parameters, including linear absorption, absorption saturation intensity, modulation depth, damage threshold, wavelength independence and bandwidth, along with the ratio of saturable to non-saturable losses (Wang *et al*., 2008; Sun and Shi, 2013). The evolution of 1-,2- and 3-Dimensional material in material science has caused many materials to be used as SA to generate passively Q-switched fiber laser (Ismail *et al*., 2019). Initially, semiconductor saturable absorber mirrors (SESAMs) were the first materials used as an SA. However, due to the material's slow saturation recovery (approximately picoseconds), narrow operating band width, low damage threshold, and complicated and costly fabrication (Ma et al., 2019), researchers are investigating materials with a high damage threshold, good fiber compatibility, and low optical saturable intensity. (Keller, 2003; Dvoyrin, Mashinsky and Dianov, 2007; Kurkov, 2011; Kurkov *et al.*, 2010, Sholokhov *et al.*, 2011). 2-D materials such as graphene and transition metal dichalcogenide (TMDCs) have the simplicity of manufacture and inexpensive cost used as SA due to their contrasting characteristics from SESAMs (Woodward and Kelleher, 2015; Set *et al*., 2004; Novoselov *et al*., 2004; Geim, 2009). However, graphene typically has low optical absorption of 2.3% per layer, and TMDCs are progressively available in the visible region because of their substantial bandgaps (Ismail *et al*., 2019). Carbon nanotubes (CNTs) have also been used widely due to their advantages of simplicity, ultrafast recovery time, and cost-effective production. Due to its complex bandgap control that limits saturable absorption at specific wavelengths has limited CNT function as SA (Ahmad *et al.*, 2015). Black phosphorus (BP) has also been used as SA. Still, they are so vulnerable to high laser optical damage threshold and easily oxidized to oxygen and moisture environment (Ismail *et al*., 2019). Recently, metal nanomaterials have attracted many researchers to utilize as the next generation of SA due to their unique optical properties such as ultrafast response time, broad saturable absorption band, and large third-order nonlinearity. (Guo *et al*., 2015; *Wu et al*., 2015; Turner *et al.*, 2013; Varnavski *et al*., 2005).

This work investigates metal based passive SA using metal based three-dimensional (3D) filament in Polylactic acid (PLA) for 3D printing (Aluminium-PLA, brass-PLA, and copper-PLA). Polyvinyl alcohol (PVA) (Apandi *et al.*, 2017), polyethylene oxide (PEO) (Haris *et al*., 2016) and polymethyl methacrylate (PMMA) (Aziz *et al*., 2017) were commonly used as a host polymer to bind material and to develop passive SA. Tetrahydrofuran (THF) is proposed as a solvent to dissolve PLA in filament and extract the metal. However, THF cannot be mixed with the PVA, PEO and PMMA. Therefore, chitin synthesized from the exoskeletons of arthropods or the cell walls of fungi is proposed as a host polymer due to its advantages as a biocompatible, biodegradable, and non-toxic polymer (Elieh-Ali-Komi and Hamblin, 2016).

1.2 Problem Statement

For the fabrication of metallic nanoparticles, a variety of approaches are used, which are divided into two categories: bottom up methods and top down methods (Jamkhande *et al.,* 2019). The major difference between the two procedures is the nanoparticle preparation starting material. In top-down methods, bulk material is utilised as the beginning material, and particle size is reduced to nanoparticles using various physical, chemical, and mechanical processes, whereas in bottom-up methods, atoms or molecules are used as the starting material (Jamkhande *et al.*, 2019). In order to fabricate solid state material, bottom-up methods need to be used either by using physical vapor deposition or chemical vapor deposition. As reported study by Ahmad *et al.*, was using physical vapor deposition method to fabricate the gold pellets by heating it using Kenosistec E-Beam and a thermal evaporation chamber (Ahmad *et al.*, 2017). It also has been reported on fabrication of gold nanoparticles (AUNP) by using electron beam deposition where PVA thin film with thickness of 30 μm was coated with AUNPs using electron beam (E-beam) deposition (Ahmad *et al.*, 2019). Unfortunately, this approach has drawbacks of expensive methods and generates low volume of materials (Jamkhande *et al.*, 2019). Wu et al. (2015), had used the chemical reduction method which GNPs were made by reducing Chloroauric acid ($HAuCl_4$) solution with trisodium citrate in their experiment. Although chemical reduction is the simplest approach for fabricating metal nanoparticles, it has issues with reducing agents such as toxicity, high cost, and impurities (Zhang et al., 2010). Due to the shortcomings of the aforementioned fabrication techniques, this study proposes a method that uses commercially available copper (Cu), aluminium (Al), and brass 3-dimensional (3D) printer filament. Copper (Cu), Aluminum (Al), and Brass SAs may be made at room temperature using minimum apparatus and materials in a straightforward and time-saving technique.

Many SA materials, ranging from topological insulators to metal nanoparticles, have been developed using synthetic polymers such as polydimethylsiloxane (PDMS), polyvinyl alcohol (PVA), polyethylene oxide (PEO), and polymethyl methacrylate (PMMA) (Aziz et al., 2017; Ng et al., 2020; Nady et al., 2018; Zhang et al., 2018). However, with today's growing environmental concern, a biodegradable and compostable material is in high demand. As an alternative to traditional host polymers, a chitin-based bio host polymer is used as a binder to create copper (Cu), aluminium (Al), and brass chitin SAs. The fabricated copper (Cu), aluminium (Al), and brass SAs's optical characteristics, physical characteristics and laser performance in the Q-switching regime were reported and examined in terms of their repetition rate, pulse width, peak power, pulse length, and signal to noise ratio (SNR).

1.3 Research Objectives

This research aims to generate pulsed fiber laser in Erbium-doped fiber laser in ring cavity by using metal based 3D filament for a 3D printer as a passive saturable absorber. Hence, the objectives of this research are:

1. To investigate and fabricate different metal based three-dimensional filaments as passive saturable absorbers using bio-compatible host polymer
2. To characterize the fabricated saturable absorber in terms of physical and optical properties
3. To evaluate the performance of the fabricated saturable absorber for pulsed laser generation

1.4 Research Scope

This research focuses on observing the performance and stability of metal based passive saturable absorbers in a Q-switched regime by investigating the generated pulsed. Throughout the experimental works, no mode-locking pulsed was obtained and this due to the laser cavity is not fully optimized. The developed passive saturable absorber is opaque, which require higher input pump power. A commercially available Copper, Aluminium and Brass 3D printer filament were used as the starting material in fabricating the SA. The optical properties of the developed saturable absorber will be characterized using Energy Dispersive Spectroscopy (EDS). In contrast, its physical properties characterization, such as surface morphology and thickness measurement, will be done using Field Emission Scanning Electron

Microscope (FESEM) and 3D measuring laser microscope, respectively. The next step is to integrate the fabricated metal based passive saturable absorber in EDFL in the ring cavity. By increasing the input pump power, the output will be characterized. The respective production includes central operating wavelength (λ), pulse energy (P_E), average output power (A_{op}), peak power (P_p), pulse width (τ_p), repetition rates (R_r), instantaneous peak power and signal to noise ratio (SNR).

1.5 Significant of Study

This research will significantly impact the research field, especially in the photonics area. The findings from this research will help widen the scope of the current trend in investigating metal based three-dimensional filament for 3D printers as starting material for fabricated metal based SA, which reduces the time and complexity during fabrication. The fabrication process using metal based three-dimensional filament could reduce several steps from the conventional process by using powders-based starting material. In this work, the introduction of chitin as the host polymer as a binder will direct the research toward utilizing naturally sourced polymers and subsequently creating a more sustainable environment for the benefit of humankind compared to the synthetic polymer such ad polyvinyl alcohol (PVA) and polyethylene oxide (PEO).

1.6 Overview

This study is organized into five chapters. Chapter 1 introduces research, including the research background problem statement, research objectives, the scope of research, and study significance. Chapter 2, the research literature review, includes the theory of Q-switching and the saturable absorber theory. This chapter also discusses each variable, function, and process used in this experiment. A previous study that used metal based SA is being compiled together to be used as a reference for this study.

Chapter 3 addresses the methods used to perform this experiment. The analysis includes the research workflow chart, the equipment used in the experiment and the cavity suggested for the experiment. The characterization of optical properties and physical properties has been reported. The fiber laser cavity used in this experiment is also introduced in Chapter 3.

Chapter 4 is a compilation of data that had been recorded with proper discussion. The performance of the Q-switching pulse laser was also being elaborated on in this chapter. The research is wrapped up in Chapter 5, including suggestions for future work based on the findings.

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LIST OF PUBLICATIONS

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