TITANIUM CARBIDE AND TITANIUM ALUMINIUM CARBIDE SATURABLE ABSORBERS FOR PULSE GENERATION IN ERBIUM-DOPED FIBER LASER

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ABSTRACT

The nonlinear optical properties of various materials have been widely investigated to develop an all-fiberized laser cavity. The existing saturable absorber (SA) materials own a few limitations such as band gap-dependent wavelength, narrow lasing bandwidth, low optical damage tolerance, and weak nonlinear absorption. Hence, this study has developed MXene $Ti_3C_2T_X$ and MAX phase Ti_3AlC_2 as SA in a fiber laser cavity. The SA materials were prepared by a solution casting method and the D-shaped fiber was fabricated by using a mechanical wheel technique. The SA materials were characterized using field-emission scanning electron microscopy, energy-dispersive x-ray spectrometer, and Raman spectroscopy to confirm their elemental constituent. A twin-balanced detector technique examined the nonlinear absorption of SA devices, while linear absorption measurement confirmed the operating wavelength of the SAs. Linear and nonlinear absorption of the prepared SA devices exposed strong saturable absorption properties in the 1.55-µm region. An erbium-doped fiber laser cavity was developed and optimized to generate a continuous-wave laser. The Q-switched and mode-locked lasers were successfully generated using the SAs developed based on D-shaped fiber and thin film structure in the erbium-doped fiber laser cavity, indicating the compatibility of such SA devices in the all fiber-based cavity. The SA device with the highest nonlinear absorption of 3% was realized with MXene $Ti_3C_2T_X$ coated on D-shaped fiber. All SA devices own strong optical properties, thus generating powerful Q-switched and mode-locked lasers. An improvement in the pulsed laser's parameters and nonlinear absorption properties of the material was achieved with D-shaped fiber as SA in the laser cavity. The MAX phase Ti_3AlC_2 deposited onto D-shaped fiber generated a mode-locked laser with a pulse width of 2.21 ps compared to its thin film counterparts, which initiated a mode-locked laser with a 3.68 ps pulse width. The use of ternary metal carbides, which are MXene $Ti_3C_2T_X$ and MAX phase Ti_3AlC_2, proved the development of a SA with strong nonlinear absorption, high optical damage threshold, band gap-independent wavelength, and broad operational bandwidth. The short-pulsed lasers in the 1.55-µm regime are essential for various applications such as optical fiber communications, remote sensing, material processing, and laser cutting technology.

ABSTRAK

Ciri-ciri optik tak linear untuk berbagai-bagai bahan telah dikaji secara menyeluruh bertujuan untuk pembangunan kaviti laser gentian. Bahan penyerap boleh tepu yang sedia ada mempunyai beberapa kelemahan seperti jurang jalur yang bergantung kepada panjang gelombang, lebar jalur laser yang sempit, toleransi yang rendah terhadap kerosakan optik, dan penyerapan tak linear yang lemah. Justeru, kajian ini membangunkan MXene $Ti_3C_2T_X$ dan MAX phase Ti_3AlC_2 sebagai bahan penyerap boleh tepu dalam kaviti laser gentian. Bahan penyerap boleh tepu disediakan dengan kaedah penuangan larutan dan gentian berbentuk D dihasilkan menggunakan kaedah roda mekanikal. Bahan penyerap boleh tepu ini diuji dengan mikroskop imbasan elektron pancaran medan, spektrometer sinar-x penyebaran tenaga, dan spektroskopi Raman untuk mengesahkan unsur-unsur bahan tersebut. Teknik pengesan kembar terimbang telah digunakan untuk mengkaji ciri tak linear penyerap boleh tepu, manakala ukuran serapan linear mengesahkan panjang gelombang operasi penyerap boleh tepu. Serapan linear dan tak linear bahan penyerap boleh tepu yang telah disediakan berupaya bertindak sebagai penyerap boleh tepu di kawasan laser 1.55-µm. Kaviti laser gentian terdop Erbium telah dibangunkan dan dioptimumkan untuk menjana laser gelombang selanjar. Laser suis Q dan laser selakan mod berjaya dihasilkan menggunakan penyerap boleh tepu berdasarkan gentian berbentuk D dan struktur filem nipis dalam kaviti laser gentian terdop Erbium, menunjukkan kesesuaian peranti penyerap boleh tepu dalam kaviti berasaskan gentian. Peranti penyerap boleh tepu dengan penyerapan tak linear tertinggi sebanyak 3% telah dihasilkan dengan gentian berbentuk D disaluti MXene $Ti_3C_2T_X$. Semua peranti penyerap boleh tepu yang disediakan mempunyai ciri-ciri optik yang unggul, seterusnya menjanakan laser suis Q dan laser selakan mod berkuasa tinggi. Penambahbaikan dalam parameter laser denyut dan ciri-ciri penyerapan tak linear bahan telah dicapai dengan gentian berbentuk D sebagai penyerap boleh tepu dalam kaviti laser. MAX phase Ti_3AlC_2 dimendapkan diatas gentian berbentuk D telah menjanakan laser selakan mod dengan lebar denyut 2.21 ps berbanding filem nipis yang mencetuskan laser selakan mod dengan lebar denyut 3.68 ps. Penggunaan karbid logam terner seperti MXene $Ti_3C_2T_X$ dan MAX phase Ti_3AlC_2 telah menghasilkan penyerap boleh tepu dengan penyerapan tak linear kuat, mempunyai ambang kerosakan optik yang tinggi, jurang jalur yang tidak bergantung kepada panjang gelombang, dan kendalian lebar jalur yang luas. Laser denyut pendek dalam rejim 1.55-µm adalah penting untuk pelbagai aplikasi seperti komunikasi gentian optik, penderiaan jauh, pemprosesan bahan, dan teknologi pemotongan laser.

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LIST OF ABBREVIATIONS

2D - Two-Dimensional
ASE - Amplified Spontaneous Emission
Bi_2Se_3 - Bismuth Selenide
BP - Black Phosphorus
CW - Continuous Wave
DI - Deionized
DSF - D-shaped Fiber
EDF - Erbium-doped Fiber
EDFA - Erbium-doped Fiber Amplifier
EDFL - Erbium-doped Fiber Laser
EDS - Energy Dispersive X-ray Spectroscopy
EM - Electromagnetic
Er^{3+} - Erbium
FESEM - Field Emission Scanning Electron Microscope
FT - Fourier Transform
FWHM - Full-Width at Half-Maximum
GO - Graphene Oxide
GVD - Group Velocity Dispersion
HF - Hydrofluoric
LD - Laser Diode
MCVD - Modified Chemical Vapor Deposition
MF - Microfiber
ML - Mode-Locked
MMF - Multimode Fiber
MoS_2 - Molybdenum Disulfide
Nd^{3+} - Neodymium
NPs - Nanoparticles
OSA - Optical Spectrum Analyzer
PC - Polarization Controller
PEO - Polyethylene Oxide

PMMA - Polymethyl Methacrylate
PVA - Polyvinyl Alcohol
PVDF - Polyvinyl Difluoride
QS - Q-switched
RF - Radio Frequency
RFSA - Radio Frequency Spectrum Analyzer
SA - Saturable Absorber
SEM - Scanning Electron Microscope
SESAMs - Semiconductor Saturable Absorber Mirrors
SHG - Second Harmonic Generation
SMF - Single-Mode Fiber
SNR - Signal-to-Noise Ratio
SPM - Self-Phase Modulation
SPR - Surface Plasmon Resonance
SWCNT - Single-Walled Carbon Nanotube
TBP - Time Bandwidth Product
TF - Thin Film
THG - Third Harmonic Generation
TIR - Total Internal Reflection
TIs - Topological Insulator
TMDCs - Transition Metal Dichalcogenides
TPA - Two Photon Absorption
WDM - Wavelength Division Multiplexer
WS_2 - Tungsten Disulfide
VBW - Video Bandwidth

LIST OF SYMBOLS

α - Absorption Coefficient
α_{cf} - Phase-Amplitude Coupling Factor
β_2 - Group Velocity Dispersion Coefficient
β_3 - Third Order Dispersion
ε_0 - Permittivity of Vacuum
σ_{cs} - Effective Cross-Sectional Area
δ - Ratio of Saturable to Non-Saturable Loss
τ_p - Pulse Width
τ_{rex} - Relaxation Time
η - Energy Extraction Efficiency
ω - Angular Frequency
$\Delta\omega$ - Frequency Offset
ϕ - Photon Flux
ϕ_{nl} - Nonlinear Phase Constant
χ - Linear/Nonlinear Susceptibility
λ - Wavelength
λ_c - Center Wavelength
λ_D - Near-Zero Dispersion Wavelength
$\Delta\lambda_{3dB}$ - 3-dB Spectral Bandwidth
AL_G - Volume of Pumped Gain Medium
c - Speed of Light
d_p - Penetration Depth
D_w - Waveguide Dispersion
D_m - Wavelength-dependent Fiber Dispersion
E - Energy
E_A - Saturation Energy of Saturable Absorber
E_F - Normalized Electric Field
E_L - Saturation Energy of Gain Medium
E_p - Pulse Energy
E_{stored} - Energy Stored

f - Repetition Rate

g - Gain

h - Planck’s Constant

I - Intensity-dependant Input

I_{sat} - Saturation Intensity

k - Frequency-Dependent Wavenumber

l - Total Cavity Loss per Round Trip

l_{int} - Optical Intensity

L - Laser Cavity Length

L_T - Thickness of Material

n - Refractive Index

N - Integral Multiple

N_{mod} - Number of Modes

N_t - Density of Absorbing Unit

P - Intracavity Power

P_{avg} - Average Power

P_{int} - Pulse Intensity

P_L - Induced Polarization

P_p - Peak Power

q - Saturable Absorber Loss Coefficient

ΔR - Modulation Depth

S_p - Pulse Shape Factor

T - Pulse Period

T_r - Cavity Round Trip Time

V - Normalized Frequency

V_g - Group Velocity

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CHAPTER 1

INTRODUCTION

1.1 Background of Study

In 2004, Geim and Novoselov discovered an atomically thin layer of carbon called a few-layer graphene [1]. Graphene owns excellent condensed matter properties with ballistic electron mobility, which prevents lattice dislocation and crystal imperfection at high temperatures. Thus, it is widely used as a SA in the broad near-infrared spectrum due to its overlapped conduction-to-valence band and ultrafast relaxation time (~200 fs) [2-4]. As promising as it is, graphene owns a few limitations in terms of bandgap alteration ability and low modulation depth (<2.3 % per layer) [5]. The latter contributed to a low second-order susceptibility of its structure [6]. In contrast, TMDCs such as molybdenum disulfide (MoS_2) possess a high second-harmonic generation (SHG) and third-harmonic generation (THG) with recovery time nearly as fast as graphene (~30 fs) [7]. Both SHG and THG are strongly related to the thickness of MoS_2, thus, optimizing a few layers thickness of such material might enhance the performance of pulses generated [8]. The modification of TMDCs layer can also convert their bandgap from indirect to direct structure, which is suitable for near-infrared laser generation. Conversely, layer-dependent wavelength operation gives rise to a complicated preparation procedure resulting in the development of other two-dimensional (2D) material-based SA.

Few years ago, many research works have been conducted on investigating the physical and optical properties of BPs [9, 10]. Thanks to its excellence charge-carrier mobility (10^5 cm^2/Vs) and thermodynamically stable phases, numerous semiconductor applications have been developed [11]. Its ability to generate pulses in the near-infrared region is proven, as a naturally occurring BPs covers wide near-infrared region due to a suitable bandgap of such allotrope (0.3 eV). Unfortunately, BPs' compatibility as a SA diminishes over time as it is very sensitive to the environment. Exposure of BPs to air for a few hours might reduce its saturable absorption properties, making it useless for pulse generation. Therefore, the surge for a new SA material which is not only easy to prepare, yet preserve the excellent nonlinear optical properties, wide operating bandwidth, and high damage threshold is still essential.

Recently, an extensive study on a newly synthesizes 2D material, MXene, has been widespread [12]. $Ti_3C_2T_X$, one of MXene, exhibits an excellent nonlinear absorption alike graphene with two orders of magnitude larger than MoS_2 and BPs [13], indicates its fast-optical switching capability. Not only that, $Ti_3C_2T_X$ holds a high damage threshold of 70 mJ/cm^2, comparable to most metal NPs [14]. Therefore, avalanche research interest had revealed the potential of MXene as a Q-switcher and mode-locker in a 1-, 1.3-, 1.55- and 2-µm laser cavity [15-18]. However, the ability of its bulk-counterparts, MAX phase (layered metal carbides and/or nitrides) for the generation of pulses in a near-infrared region is not fully explored. MAX phase Ti_3AlC_2 is as unique as its precursor with good electrical conductivity, thermodynamically stable material, high damage tolerance at room temperature, good mechanical strength, and excellent oxidation-resistance [19-21]. The latter seems favorable for pulse generation as it did not easily oxidize in the air due to a dense alumina layer within the material. Unlike 2Ds' includes MXene, MAX phase is synthesis based on a simple solution-casting method, a mixture of MAX phase Ti_3AlC_2 and polyvinyl alcohol (PVA) was magnetically stirred to produce a SA device for pulse generation. Herein, this work investigated the optical properties of ternary metal carbides, MXene $Ti_3C_2T_X$, and its bulk-counterparts MAX phase Ti_3AlC_2 as a pulse generator in an all-fiberized EDFL cavity.

1.2 Problem Statement

In recent years, pulsed laser generation has been a widely discovered area since it is essential for industrial applications such as metal cutting technology, laser ablation, remote sensing, and laser eye surgery. The main component used for ultrashort pulse laser generation is a saturable absorber material. Over the years, researchers have implemented various materials as saturable absorbers such as graphene, molybdenum disulfide, tungsten disulfide, metal nanoparticles, and many more into the laser cavity to generate the pulse. However, the existing saturable absorber owns a narrow operating bandwidth, weak nonlinear optical properties, and a low damage threshold.

Therefore, this work proposed MAX phase Ti_3AlC_2 and MXene $Ti_3C_2T_X$ for pulse generation in a 1.55 µm regime. Not only do they have strong optical properties such as fast-optical switching capability, high damage threshold, excellent nonlinear absorption, but they also own a broad operating wavelength. This work outlined the investigation of MAX phase and MXene optical properties to prove their good saturable absorption properties and incorporated those materials in a fiber laser cavity for pulse generation. Eventually, it enabled a pulsed laser with good spectral and temporal characteristics.

1.3 Research Objectives

The main aim of this work is to develop a saturable absorber material based on the MAX phase and MXene and to optimize a Q-switched and mode-locked fiber laser cavity. The success of this work is evaluated based on the three main objectives as follows,

(a) To synthesis Ti_3AlC_2 and $Ti_3C_2T_X$, and determine the surface morphology, elemental constituent, structural fingerprint, and optical properties of the SA devices such as linear and nonlinear absorption.

(b) To develop and generate Q-switched and mode-locked lasers by incorporating the D-shaped fiber and the thin film structure of SA materials inside the erbium-doped fiber laser.

(c) To determine the temporal pulse performance such as pulse width and repetition rate, and to calculate the output power, pulse energy, peak power, and slope efficiency of the pulsed lasers with Ti_3AlC_2 and $Ti_3C_2T_X$ as saturable absorber in erbium-doped fiber laser.

1.4 Scope of Study

A solution-casting method was utilized to synthesis the SA in the form of solution, and a thin film SA was attained directly after the process. A homemade mechanical wheel technique was utilized to fabricate the D-shaped fiber structure, which was followed by the deposition of the prepared SA solution onto its polished region. Later, the characterization of material with Raman spectroscopy, FESEM and EDS proved the existence of Ti_3AlC_2 and $Ti_3C_2T_X$ composition in the as-prepared SA devices. The SAs based on the MAX phase Ti_3AlC_2 and MXene $Ti_3C_2T_X$ revealed strong optical properties, as evidenced by the linear and nonlinear absorption measurement in the 1.55-µm region. Erbium-doped fiber laser cavity was developed and optimized to generate continuous-wave laser in 1.55 µm regime. All saturable absorbers were incorporated into the cavity to generate QS and ML, and the better-pulsed performance achieved with the incorporation of D-shaped fiber-based SA inside the laser cavity.

1.5 Significance of Study

This work introduced a homemade mechanical wheel technique to fabricate a D-shaped fiber structure, which is essential to improve laser performance in a fiber laser configuration. In addition, this thesis synthesized MAX phase Ti_3AlC_2 and MXene $Ti_3C_2T_X$ for pulse generation in a 1.55 µm regime. The materials own good saturable absorption properties, a high damage threshold, and a broad operating wavelength. The potential of MAX phase Ti_3AlC_2 and MXene $Ti_3C_2T_X$ as passive saturable absorbers in EDFL can also be beneficial for the researcher in this field since it can also be used for pulsed generation in 1 and 2-µm laser cavities. The comparison between D-shaped fiber structure and thin film as SA is also essential to realize the pulsed laser with a better output performance. The generated Q-switched and mode-locked laser can be used for industrial applications such as remote sensing, laser beam machining, and high precision material processing.

1.6 Organization of this thesis

This thesis is structured in 5 main chapters, starting from the theoretical and current progress on pulsed laser, the work continues to investigate the optical properties and the lasing application of the as-prepared SA, and comparison on pulsed laser performance between two SA implementation methods concludes the finding. This chapter describes the story of laser and why it is worth to expand the current work on this field. The motivation of this thesis was also elaborated, together with the proposal on how to solve the existing problems. This section also states the significance and scope of the study.

Chapter 2 elaborates various physical phenomena such as a quasi-three-level erbium-doped fiber laser system, the principle of Q-switching, the principle of mode-locking, the principle of soliton, a saturable absorption mechanism, and evanescent field interaction. This section also contains current progress on various SA materials and its application as a pulse generator in the EDFL cavity.

Chapter 3 comprises the methodology of this research. The preparation of both SA, MAX phase Ti_3AlC_2, and MXene $Ti_3C_2T_X$ are elaborated together with the fabrication of the D-shaped fiber structure. Followed by the linear absorption measurement to confirm the ability of SA devices to initiate pulse in a 1.55 μm regime. Later, the balanced-twin detector technique was introduced to investigate saturable absorbers' optical properties. Finally, this chapter described the development and optimization of an erbium-doped fiber laser cavity.

The as-stated objectives are covers in **Chapter 4**, starting with the EDS, FESEM, and Raman spectrum to confirm the existence of the MAX phase Ti_3AlC_2 and MXene $Ti_3C_2T_X$ in the SA devices. It also details the linear and nonlinear optical profiles of the SA devices. In the end, it describes the generation of QS and ML in

EDFL by incorporating ternary metal carbides-based SA. All the SA devices generate Q-switched and mode-locked with exceptional laser parameters. The laser parameters such as pulse width, repetition rate, output power, pulse energy, and slope efficiency of all the generated lasers are recorded and compared.

Chapter 5 concludes and discusses the results achieved from the previous chapter, including the optical properties, and generated laser performances of SA devices. The improvement of QS and ML by using a D-shaped fiber structure proposed a new SA implementation method in an all-fiberized laser cavity. A few limitations and future works are also suggested for the better temporal pulse characteristics of the pulsed laser.

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LIST OF PUBLICATIONS

Publications based on the main scopes of this thesis:

1. **Jafry, A. A. A.,** Muhammad, A. R., Kasim, N., Rosol, A. H. A., Rusdi, M. F. M., Ab Alim, N. N. N., Harun, S. W., & Yupapin, P. (2021). Ultrashort pulse generation with MXene Ti3C2Tx embedded in PVA and deposited onto D-shaped fiber. *Optics & Laser Technology, 136, 106780.* **(ISI indexed, Q1, IF:3.233)**

2. **Jafry, A. A. A.,** Rosol, A. H. A., Kasim, N., Muhammad, A. R., Rulaningtyas, R., Yasin, M., & Harun, S. W. (2020). Soliton mode-locked pulse generation with a bulk structured MXene Ti3AlC2 deposited onto a D-shaped fiber. *Applied Optics, 59(28), 8759-8767.* **(ISI indexed, Q3, IF: 1.961)**

3. **Jafry, A. A. A.,** Kasim, N., Nizamani, B., Muhammad, A. R., Yusoff, R. A. M., Harun, S. W., & Yupapin, P. (2020). MAX phase Ti3AlC2 embedded in PVA and deposited onto D-shaped fiber as a passive Q-switcher for erbium-doped fiber laser. *Optik, 165682.* **(ISI indexed, Q2, IF: 2.187)**

4. **Jafry, A. A. A.,** Krishnan, G., Kasim, N., Zulkipli, N. F., Samsamnun, F. S. M., Apsari, R., & Harun, S. W. (2020). MXene Ti3C2Tx as a passive Q-switcher for erbium-doped fiber laser. *Optical Fiber Technology, 58, 102289.* **(ISI indexed, Q2, IF: 2.212)**

5. **Jafry, A. A. A.,** Kasim, N., Rusdi, M. F. M., Rosol, A. H. A., Yusoff, R. A. M., Muhammad, A. R., . . . Harun, S. W. (2020). MAX phase based saturable absorber for mode-locked erbium-doped fiber laser. *Optics & Laser Technology, 127, 106186.* **(ISI indexed, Q1, IF:3.233)**