# **GRAPHENE OXIDE POLYMER FILM AS Q-SWITCHER AND**

## **MODE-LOCKER IN 1.5 MICRON REGION**

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# FACULTY OF ENGINEERING UNIVERSITY OF MALAYA KUALA LUMPUR

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# GRAPHENE OXIDE POLYMER FILM AS Q-SWITCHER AND MODE-LOCKER IN 1.5 MICRON REGION

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#### ABSTRACT

This project aimed to demonstrate passively Q-switched and Mode-locked Erbium-Doped Fiber Laser (EDFL) that operated in 1.5 micron region using Graphene Oxide (GO) as saturable absorber (SA) for possible applications in telecommunication, laser processing, fiber sensing and medical. A passively Q-switched EDFL was successfully demonstrated using the GO based SA. The Q-switched laser operates at wavelength between 1550nm to 1580nm with a repetition rate that can be tuned from 44.33 kHz to 61.77 kHz as the pump power is varied from 130mW to 260mW. The highest repetition rate of 61.77 kHz is achieved at a pump power of 260mW and it is observed that the Q-switched pulse produced maximum pulse energy of 0.054nJ. Nanosecond mode-locked fiber laser in three different EDFL cavities was also successfully demonstrated using GO based SA. The mode-locking threshold pump powers are 65.5, 65.5 and 60.2mW for the EDFL configured with SMF lengths of 50, 100 and 200m, respectively. The stable mode-locking operation could be sustained up to the pump power of 80.4mW for all three lasers. With 208m cavity length and total dispersion of -4.77ps<sup>2</sup>, the EDFL operates at 1566nm with repetition rate of 0.9625MHz, pulse width of 380 ns, peak power of 16.29mW, pulse energy of 7.06nJ when the pump power was fixed at 80.4mW. The repetition rates increased from 0.9625 to 3.266MHz with the reduction of cavity length from 208 to 50m. These results indicate that GO film have a great potential for pulse generation at 1.5µm.

#### ABSTRAK

Projek ini bertujuan untuk menunjukkan Q-Suis dan Mod-selakan pasif gentian laser terdop Erbium (EDFL) yang beroperasi dalam kawasan 1.5 mikron menggunakan Graphene Oxide (GO) sebagai penyerap boleh tepu (SA) bagi kegunaan seperti dalam bidang telekomunikasi, pemprosesan laser, penderiaan fiber dan juga bidang perubatan. Q-Suis EDFL pasif telah berjaya ditunjukkan dengan menggunakan GO sebagai SA. Denyut Q-Suis beroperasi di antara 1550nm ke 1580nm dengan kadar ulangan yang boleh ditala dari 44.33kHz ke 61.77kHz apabila kuasa pam berubah daripada 130mW kepada 260mW. Kadar ulangan tertinggi 61.77 kHz dicapai pada kuasa pam 260mW dan dapat diperhatikan bahawa denyut Q-Suis menghasilkan tenaga denyut yang maksimum 0.054nJ. Laser fiber modselakan nanosaat dalam tiga kaviti EDFL berbeza berjaya ditunjukkan menggunakan GO sebagai SA. Kuasa-kuasa pam ambang mod-selakan ialah 65.5, 65.5 dan 60.2mW untuk EDFL dengan panjang SMF 50, 100 dan 200m, masing- masing. Operasi mod-selakan stabil boleh dikekalkan pada kuasa pam 80.4mW untuk ketiga-tiga laser. Dengan panjang rongga 208m dan jumlah penyerakan -4.77ps<sup>2</sup>, EDFL beroperasi pada 1566nm dengan kadar ulangan 0.9625MHz, lebar denyut 380ns, kuasa puncak 16.29mW, tenaga denyut 7.06nJ apabila kuasa pam ditetapkan di 80.4mW. Kadar ulangan bertambah dari 0.9625 hingga 3.266MHz dengan pengurangan panjang rongga dari 208 hingga 50m. Keputusan ini menunjukkan bahawa filem GO mempunyai potensi yang besar untuk penghasilan denyut pada 1.5 µm.

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### LIST OF SYMBOLS AND ABBREVIATIONS

ASE	: Amplified Spontaneous Emission
Be <sub>2</sub> Se <sub>3</sub>	: Bismuth Selenide
BP	: Black Phosphorus
CNTs	: Carbon Nanotubes
CW	: Continuous Wave
DI	: Deionized Water
DWDM	: Dense Wavelength Division Multiplexing
EDF	: Erbium-Doped Fiber
EDFA	: Erbium-Doped Fiber Amplifier
EDFL	: Erbium-Doped Fiber Laser
Er <sup>3+</sup>	: Erbium
FESEM	: Feld Emission Scanning Electron Microscopy
GO	: Graphene Oxide
GVD	: Group Velocity Dispersion
$H_2O_2$	: Hydrogen Peroxide
$H_2SO_4$	: Sulfuric Acid
HCI	: Hydrochloride
KMnO <sub>4</sub>	: Potassium Permanganate
$MoS_2$	: Molybdenum Disulphide
NaNO <sub>3</sub>	: Sodium Nitrate
OC	: Output Coupler
OSA	: Optical Spectrum Analyzer

- PVA : Polyvinyl Alcohol
- RF : Radio Frequency
- SA : Saturable Absorber
- SESAMs : Semiconductor Saturable Absorber Mirrors
- SMF : Single Mode Fiber
- SNR : Signal-to-Noise Ratio
- SPM : Self-Phase Modulation
- TIs : Topological Insulators
- TMDs : Transition-Metal Dichalcogenides
- WDM : Wavelength Division Multiplexer
- WS<sub>2</sub> : Tungsten Disulphide
- Yb<sup>3+</sup> : Ytterbium
- YDFL : Ytterbium-Doped Fiber Laser

#### CHAPTER 1

#### **INTRODUCTION**

#### **1.1 Research Motivation**

In recent years, fiber laser attracted a lot of attentions and interests among researchers due to high power, high stability, and high reliability. By using a fiber laser, the usage and design of the laser is much more flexible. With the higher the surface of the volume ratio of fiber allows high efficiency heat dissipation. Laser based on rare-earth-doped fiber has received a great interest that simulates a new technique of fiber fabrication, as the first rareearth-doped fiber lasers have produced a few miliwatts at a wavelength of around 1 µm (Maiman, 1960). There are a few examples of rare-earth elements that have been used in fiber doped gain medium such as Erbium, Neodymium, Holmium, Ytterbium, Thulium, and Praseodymium. However, Erbium  $(Er^{3+})$  and Ytterbium  $(Yb^{3+})$  doped fiber lasers have conquered the commercial industry of fiber lasers. The operation wavelength for Erbiumdoped fiber laser (EDFL) and Ytterbium-doped fiber laser (YDFL) are ranged from 1520nm to 1560 and 1030nm to 1100nm respectively. EDFL is advantageous for optical fiber communication while YDFL is frequently used for high power laser. In this work, EDFL is used as gain medium since its can efficiently amply light in the 1550nm wavelength region, where telecom fibers have their loss minimum and dispersion (R. J. Mears, Reekie, L., Poole, S. B., & Payne, D. N., 1986) and capable to amplify data channels with the highest data rates simultaneously in dense wavelength division multiplexing (DWDM) with a low noise (Desurvire, 1987). Besides, EDFL recently has the highest demand in various areas and gained interest of many researchers since it has many advantages such as small size, low cost, high beam quality and wide tunable wavelength (Zhu, Qian, & Helmy, 2007).

In past few decades, Q-switched and mode-locked erbium-doped fiber lasers (EDFLs) have attracted much attention due to their inherent features of high stability, good mode confinement and alignment-free structure (Mao et al., 2018). Passive methods based on saturable absorbers (SAs) are more attractive compared with active Q-switching or modelocking techniques for their simple configuration without external electronic components (Wu, Zhang, Wang, Li, & Chen, 2015) (Woodward et al., 2015). As a result, there is a high motivation to develop new types of SAs. Passively Q-switched fiber lasers can find a mass application on the areas of medicine, communications, material processing and manufacturing as well as basic research and so forth due to their simple design which allows for the development of compact and cost- effective pulsed laser sources (Fermann & Hartl, 2013). Passive mode-locking is a practical technique to generate ultrafast femtosecond pulses in highly compact fiber lasers. Nano-material graphene-based SA recently, has attracted significant interest as an excellent wideband mode-locker due to its unique linear and nonlinear optical properties, such as saturable absorption characteristics for a broad wavelength range, ultrafast recover time, low saturable intensity, and pulses from sub 200 fs to a few picoseconds in the graphene mode-locked EDFLs.

Various of SAs has been demonstrated and reported for pulse generation, such as semiconductor saturable absorber mirrors (SESAMs) (H.-Y. Wang et al., 2012) carbon nanotubes (CNTs) (M.H.M.Ahmed, 2015) (Harun et al., 2012) and graphene (Ahmad, Muhammad, Zulkifli, & Harun, 2013a). SESAM has a narrow wavelength tuning range (tens of nanometers), and its modulation depth is typically less than 10% (Keller et al., 1996). Graphene is a potential absorber to take the place of the SESAMs for Q-switched or mode locked lasers. Graphene has become one of the most demanded and promising materials as a saturable absorber because of its ultrafast carrier dynamics and ultra broadband absorption. The success of graphene in photonic applications leads to the invention of new 2D materials

such as topological insulators (TIs), transition-metal dichalcogenides (TMDs) and black phosphorus (BP). Recently, TIs materials such bismuth selenide (Be<sub>2</sub>Se<sub>3</sub>) (Ahmad, Salim, Azzuhri, Soltanian, & Harun, 2015) and TMDs materials such as tungsten disulphide (WS<sub>2</sub>) (H Ahmad, NE Ruslan, MA Ismail, SA Reduan, et al., 2016), and molybdenum disulphide (MoS<sub>2</sub>) (Harith Ahmad et al., 2016) as well as black phosphorus based SA (Ismail, Kadir, Latiff, Ahmad, & Harun, 2016) have been investigated and broadly reported as they are having the same performance as graphene.

In this report, passively Q-switched fiber laser and mode-locked are demonstrated using newly developed graphene oxide polyvinyl alcohol (PVA) composite film as a SA due to its remarkable optical properties.

#### **1.2 Objectives**

This report aims to demonstrate passively Q-switched and Mode-locked EDFL that operated in 1.5 micron region using graphene oxide as saturable absorber. The following objectives are set to achieve our aim.

- To generate stable Q-switching pulse train in EDFL setup using a graphene oxide film as the saturable absorber.
- To investigate the performance of the proposed GO based mode-locked EDFL at three different cavity lengths by adding a spool of 200, 100 and 50 m long SMF inside the laser cavity.
- To analyze the changes in pulse, output power (mW), repetition rate (kHz), and pulse width (us) at the different pump power threshold for each method.

#### **1.3 Research Overview**

This research report consists of 5 chapters, which carefully demonstrate pulsed fiber lasers operation using two different method; passively Q-switched and mode-locked EDFL fiber laser that operated in 1.5 micron region using GO as SA. This first chapter introduces the topic, where motivation of this study as well as the research objectives, are also briefly described.

In chapter 2, a literature study on EDFL and saturable absorption principle are described. This chapter also describes the saturable absorption process that leads to the generation of Q-switched pulse laser and mode-locked laser as well as the important parameter to evaluate the performances of the both method.

Chapter 3 demonstrates a Q-switched EDFL using a new graphene oxide material as SA and the performance of GO PVA based SA evaluated. The results obtained indicates that GO has a new potential SA material for pulsed laser applications

Chapter 4 investigates performance of the proposed GO based mode-locked EDFL at three different cavity lengths, which was obtained by adding a spool of 200, 100 and 50 m long SMF inside the laser cavity. The result obtained indicates the superiority of easy fabrication and strong solubility of GO will facilitate potential applications for ultrafast photonics. Chapter 5 concludes the research finding of this work.

#### CHAPTER 2

#### LITERATURE REVIEW

#### 2.1 Fiber Laser

Fiber lasers are special category of solid state lasers with optical fibers as gain media, although some lasers with a semiconductor gain medium and a fiber resonator have also been called fiber lasers. Fiber lasers provide many benefits such as high reliability and high integration capability compared with other laser technologies. Right after the demonstration of the first laser by Maiman, fiber lasers were proposed and studied as promising laser configuration (Koester & Snitzer, 1964; Maiman, 1960; Snitzer, 1961). Lasers utilize the quantum effect of stimulated emission to generate light and share several common features, such as an active medium to provide gain, an optical cavity to enhance and control the optical field and pumping source to provide the energy (Siegman, 1986). However, the details of these features play an important role in differentiating the laser performance, the power scaling capabilities, stability, footprint and cost.

Fiber laser become the latest entry in solid-state technology domain, fast increasing their penetration in all sectors of industrial, medical and directed energy application space (Injeyan, Pflug, & Vespucci, 2011). The features that differentiate fiber lasers from the other existing laser technologies give superior overall performance. Due to large surface-to-volume ratio, fiber lasers present better thermal management and total elimination of thermal, lending, which plagues solid-state crystal counterparts. The well controlled spatial distribution of the signal, provided by the continuous guidance, results in superior beam quality and stability, while small quantum defect, as well as, low cavity and transmission losses result in record wall-plug efficiencies. Also, fiber lasers show turn-key operation and small foot print. Their unique properties, the output power stability and unparalleled beam

quality at high output powers, have increased their market penetration and have enabled several new applications (Hausken, 2012)

Doped fiber lasers are the main component in the fiber laser oscillator, it is used as a gain medium typically to produce wide gain spectra with reasonably high gain. Therefore, accomplished lasers with magnificent potential for broad wavelength tuning and ultra-short pulse generation is achievable. Erbium  $(Er^{3+})$  is one of the considerably employed rare earth metal element, where Er ions provide gain in a wide wavelength range around 1.5µm which is most remarkable for optical communications. Thus, erbium doped fiber (EDF) was studied through the 20th century where the first EDF was fabricated and reported in 1985 (Poole, Payne, & Fermann, 1985). In 1987 EDF amplifiers and laser have been demonstrated (R. J. Mears, Reekie, Jauncey, & Payne, 1987). This study presents an original work on developing pulsed EDFL using grapheme oxide as SA.

#### 2.2 Erbium Doped Fiber Laser

An erbium-doped fiber (EDF) is an optical fiber of which the core is doped with rareearth element erbium ion  $Er^{3+}$ . The reason why rare earth elements is used in the optical fiber is because of the glass consists of rare earth ions are optically active. Means that, they can absorb the light at one wavelength and release the coherent light at different wavelength, operating at 1550nm region. All EDFLs can be pumped with compact, efficient, and inexpensive laser diodes operating at 980nm or 1480nm. They are compatible with different fibers and fiber optic components used in communications so they have negligible coupling losses. This behavior is very useful for creating a laser, coherent broadband sources or to amplify the signal at the emission wavelength of 1550nm (Méndez & Morse, 2011). A simplified energy level diagram of  $Er^{3+}$  ion is shown in Figure 2.1. The energy levels are broadening due to the dc-Stark effect (R. Mears & Baker, 1992), which leads to relatively broad emission bandwidth. When a 974nm pump laser diode beam is fed into an EDF,  $Er^{3+}$  will be excited from the ground state  $E_1$  to higher level  $E_3$ . The excited  $Er^{3+}$  ions on  $E_3$  will rapidly decay to energy level  $E_2$  through nonradiative emission. The excited ions on  $E_2$  eventually return to ground state  $E_1$  through spontaneous emission, which produces photons in the wavelength band 1520 – 1570nm. The spontaneous emission will be amplified as it propagates through the fiber, especially when the pump laser power is increasing. As amplified spontaneous emission (ASE) covers a wide wavelength range 1520-1570nm, we can use it as a broadband light source.



Figure 2.1: Simplified energy levels of EDFL (Zhu et al., 2007).

If a laser signal with a wavelength between 1520 and 1570nm and a 974nm pump laser are fed into an EDF simultaneously as shown in Figure 2.1, there are three possible outcomes for the signal photon: stimulated absorption, stimulated emission and unabsorbed. In the stimulated emission process, signal photon excites an erbium ion from the state  $E_1$  to a higher level  $E_2$  and become annihilated. Then, the signal is amplified where signal photon stimulates an erbium ion at state  $E_2$  to decay to  $E_1$  to produce another identical photon. Signal photon can propagate unaffected through the fiber while spontaneous emission always occurs between level  $E_2$  and level  $E_1$ . When pump laser power is high enough that the population inversion is achieved between the energy level  $E_2$  and  $E_1$  of EDF, the input laser signal passing through the fiber is then be amplified. Thus, we can use EDF and pump laser to construct an optical amplifier, which called erbium-doped fiber amplifier (EDFA). The spontaneous emission also could be amplified by pump laser. So, ASE is always present is EDFA, and it is the main source of noise in these amplifiers. The laser is simply the optical amplifier with positive feedback. If the output of EDFA is fed back to its input to build a fiber loop, when pump laser power is added into the fiber loop, the EDFA is transformed to a fiber laser, which is called erbium-doped fiber laser (EDFL). The laser wavelength varies with the cavity loss. Therefore, the laser wavelength can be tuned by adjusting the cavity loss.

#### 2.3 Saturable Absorber

Saturable absorber (SA) is a nonlinear optical material where the absorption of light decreases with increasing light intensity. Optical loss can be occurred for example in a gain medium with absorbing dopant ions, when a strong optical intensity leads to depletion of the ground state of these ions. Different applications require SA with very different parameters and different devices are used. For passive mode locking as well as Q-switching, semiconductor saturable absorber mirrors (SESAM) frequently used (Keller et al., 1996). These are also suitable for passive Q-switching, particularly at lower pulse energies. For mode locking of lasers, thin layers of carbon nanotubes (CNT), single-wall nanotubes, have been used (Schmidt et al., 2008; Set, Yaguchi, Tanaka, & Jablonski, 2004; Shohda, Shirato, Nakazawa, Mata, & Tsukamoto, 2008). Such absorbers can exhibit very broadband absorption features as are desirable for broadband lases. For example, apply thin layers of nanotube to fiber ends and use them mode locking of very compact fiber lasers give high pulse repetition rates (Martinez, Fuse, & Yamashita, 2011). The recovery time of nanotube

absorbers is quite short, but substantial non-saturable losses can be a problem for some applications. In rare cases, SAs materials are used in the form of optical fiber. For example, chromium, samarium or bismuth dopants can serve this function in Q-switched fiber lasers (Dvoyrin, Mashinsky, & Dianov, 2007). Selecting a suitable SAs depending very much on the concrete circumstances on the properties of a SAs are desirable. Generally, decisions on absorber parameters should be made in the context of a comprehensive laser design processes, which considers both the dynamics of pulse generation and the limited tolerance of the absorber to high intensities or pulse energies.

#### 2.4 Graphene

Recently, graphene based SAs have shown outstanding potential for both modelocked and Q-switched fiber lasers due to its outstanding linear and nonlinear optical properties (Lv et al., 2013). Graphene has reported having a high damage threshold, an ultrabroad absorption spectral bandwidth, an ultrafast recovery time and a controllable modulation depth (Liu, Wu, Yang, & Wang, 2011; L. Zhang, Zhuo, Wang, & Wang, 2012). By using graphene SA as a Q-switcher, a Q-switched erbium-ytterbium fiber laser demonstrated with a linear cavity configuration that produced stable pulses with duration of 5.9 µs and repetition rate of 20.0 kHz (Yap, Richard, Pua, Harun, & Ahmad, 2012). Very recently, a Q-switched EDFL using a graphene SA, which was fabricated by depositing graphene on a tapered fiber emitted pulses with typical duration of  $3.89 \ \mu s$  and the pulse repetition rate was widely tuned from 10.4 to 41.8 kHz by varying the pump power has been reported (J. Wang et al., 2012). Apart from high quality pure graphene, low-cost precursor for the chemical synthesis of pure graphene; graphene oxide (GO) has also attracted much technical attention as an alternative saturable absorption material since it exhibits ultra-fast relaxation and strong nonlinear saturable absorption (Zhao et al., 2011).

#### 2.5 Q- Switching

Q-switching is a technique to generate intense short pulse laser. This technique will generate a pulse of light with a very high peak power (~ gigawatt), which is much higher than the power produced in continuous wave (CW) mode. Q-switching will produce low repetition rate pulse, high pulse energy, and broader pulse durations, compared to the mode locking technique. In Q-switching technique, we are able to achieve a single solid and short pulse trains of laser radiation (Singh, Zeng, Guo, & Cai, 2012). There are two primary methods of Q-switching: active Q-switching and passive Q-switching.

Active Q-switching requires electrically powered equipment such as electro-optics and acoustic-optics modulators where this modulator will add more cost and complexity as well as produce high loss in the cavity (H Ahmad, NE Ruslan, MA Ismail, ZA Ali, et al., 2016). Compared to active Q-switching, passive Q-switching is usually simple and costeffective achieved through the employment of SA as the Q-switcher. Besides, it is suitable to generate high pulse repetition rates. However, the pulse energies are typically lower and cannot be triggered externally. Passively Q-switched fiber lasers generate Q-switching pulses train that are applicable in the area of telecommunications, material processing, range findings, and medicine. The benefit of the Q-switched laser is the high energy per pulse as compared to mode-locked fiber lasers, and also the likelihood of a wide tuning range that can arise in the presence of various gain media. Generally, passive lasers are relatively simple in design, low cost, and provide stable and consistent output power. Figure 2.2 Simple concept of a solid-state laser with passive Q-switched.



Figure 2.2: Simple concept of a solid-state laser with passive Q-switched (Kozinc &

Marko., 2015).

#### 2.6 Mode-Locking

Mode locking is a method to obtain ultrashort pulses from lasers, which are then called mode-locked lasers (Jr., 1964). The laser resonator contains either an active element; an optical modulator or a nonlinear passive element; a saturable absorber which causes the formation of an ultrashort pulse circulating in the laser resonator. Various effects influencing the circulating pulse in the steady state are in a balance so that the pulse parameters are unchanged after each completed round trip, or often even nearly constant throughout each round trip. Each time the pulse hits the output coupler mirror, a usable pulse is emitted, so that a regular pulse train leaves the laser as shown in figure 2.3. Assuming a single circulating pulse, the pulse repetition period corresponds to the resonator round-trip time; typically several nanoseconds, whereas the pulse duration is much lower: typically between 30fs and 30ps. Because of that, the peak power of a mode-locked laser can be orders of magnitude higher than the average power. The gain medium compensates for losses, and the saturable absorber mirror (SA) enforces pulse generation. Each time the circulating pulse hits the output coupler mirror (OC), a pulse is emitted in the output.



Figure 2.3: Generation of a pulse train in a passively mode-locked laser. (Paschotta, 2008).

Methods for producing mode-locking in a laser may be classified as either active or passive. Active methods typically involve using an external signal to induce a modulation of the intra-cavity light while passive methods rely on placing some element into the laser cavity which causes self-modulation of the light. Active mode locking involves the periodic modulation of the resonator losses or of the round-trip phase change, achieved such as with an acousto-optic or electro-optic modulator. If the modulation is synchronized with the resonator round trips, this can lead to the generation of ultrashort pulses, usually with picosecond pulse durations. In some cases, the pulse duration achieved is governed by a balance of pulse shortening through the modulator and pulse broadening via other effects, such as the limited gain bandwidth.

Passive mode locking allows the generation of much shorter pulses, basically because a saturable absorber, driven by already short pulses, can modulate the resonator losses much faster than an electronic modulator: the shorter the pulse becomes, the faster the loss modulation, provided that the absorber has a sufficiently short recovery time. The pulse duration can be even well below the recovery time of the absorber. Passive mode-locking techniques do not require a signal external to the laser to produce pulses but the use of light in the cavity to cause a change in some intracavity element to produce a change in the intracavity light itselft. A commonly used device to achieve this is a saturable absorber. When placed in a laser cavity, a saturable absorber will attenuate low-intensity constant wave light. As the light in the cavity oscillates, this process repeats, leading to the selective amplification of the high-intensity spikes, and the absorption of the low-intensity light. After many round trips, this leads to a train of pulses and mode-locking of the laser (Paschotta, 2008).

Due to various applications in the fields of optical communications, optical frequency metrology, biomedicine and material processing, mode-locked fiber lasers have attracted considerable attention and become an excellent platforms for investigating fundamental physics. Ultrafast fiber lasers can be host to numerous dissipative structures and self-organization effects due to the complex dissipative interplay of linear and nonlinear effects (Chang, Ankiewicz, Soto-Crespo, & Akhmediev, 2008).

To date, various mode-locking elements and mechanisms have been developed, including active and passive techniques and nonlinear polarization rotation (NPR) which utilizes the Kerr effect of birefringent fiber as an artificial mode locker (Tiu, Tan, Zarei, Ahmad, & Harun, 2014). In passive mode-locking various types of saturable absorbers (SAs) are used including ion doped crystals (Qiao et al., 2015), semiconductor saturable absorption mirror (SESAM) (Li et al., 2015), carbon nanotubes (CNTs) (Ahmed et al., 2014; Xu et al., 2014) and graphene (Bonaccorso, Sun, Hasan, & Ferrari, 2010). SESAMs perform better, however they are expensive, complicated in fabrication process and have a limited range of optical response, which limits their applications to a great extent. Therefore, nanomaterials based mode lockers became very popular in the past ten years. Current research in nanomaterial-based SAs primarily focuses on graphene (Ahmad et al., 2013a; Bonaccorso et al., 2010), topological insulators (Yan et al., 2015), transition-metal dichalcogenides (Ahmed, Latiff, Arof, & Harun, 2016a), and black phosphorus (Ahmed, Latiff, Arof, & Harun, 2016b).

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#### **CHAPTER 3**

#### PASSIVELY Q-SWITCHED ERBIUM-DOPED FIBER LASER

#### 3.1 Introduction

A laser could emit short pulses if the loss of an optical resonator is rapidly switched from a high to a low value. By controlling the Q-factor (quality factor) of a laser resonator, Q-switching allows the generation of laser pulses of short duration (from nanosecond to picosecond range) and high peak power. The Q-factor (dimensionless) is given by:

$$Q = \frac{2\pi f_0 \varepsilon}{P} \tag{3.1}$$

where  $f_0$  is the resonant frequency,  $\varepsilon$  is the stored energy in the cavity, and P is the power dissipated. If the Q-factor of a laser's cavity is abruptly changed from a low value to a high value, the laser will emit a pulse of light that is much more intense than the lasers' continues output. This technique is called Q-switching. There are two types of Q-switching; active and passive.

Active Q-switching uses modulation devices that change the cavity losses in accordance with an external control signal. They can be divided into three categories: mechanical, electro-optical, and acousto-optics. They inhibit laser action during pump cycle. In passive Q-switching, the laser consists of gain medium and saturable absorber. The saturable absorber absorbs light at low intensity and transmits them at high intensity. As the gain medium is pumped, it builds up stored energy and emits photons. After many round-trips, the photon flux begins to see gain, fixed loss, and saturable loss in the absorber. If the gain medium saturates before the saturable absorber, the photon flux may build, but the laser will not emit a short and intense pulse. On the contrary, if the photon flux builds up to a level that saturates the absorber before the gain medium saturates, the laser resonator will see a

rapid reduction in the intra-cavity loss and the laser Q-switches and therefore, will emit a short and intense pulse of light (J. J. Zayhowski and C. Dill, 1995).

Q-switched fiber lasers are of great interest in various applications for remote sensing, medicine, marking and machining. Compared with the active operation, the passive Q-switching owns the unique advantage of simple structure in all-fiber designing. The passive Q-switched laser can be realized by adopting a SA in the cavity. So far, many kinds of SAs have been reported, such as semiconductor saturable absorber mirrors (SESAMs) (C. Gao, 2007), carbon nanotubes (CNTs) (M.H.M. Ahmed, 2015) and graphene (Sun, 2013). SESAM has a narrow wavelength tuning range, and its modulation depth is typically less than 10% (C. Gao, 2007). The CNTs and graphene are ideal SAs for Q-switching because of their low saturation intensity, low cost and broadband wavelength operation (Sun, 2013).

Graphene is a potential absorber to take the place of the SESAMs for Q-switched or mode locked lasers. However, it is difficult to grow graphene film with high quality, which makes graphene absorbers expensive. Furthermore, graphene cannot be dissolved in water so that the efficiency for film fabrication by graphene aqueous solution is decreased. Graphene oxide has traditionally served as a precursor for graphene because of its very low cost and simple fabrication method (J. Boguslawski, 2015). In this chapter, A Q-switched EDFL is demonstrated using a new graphene oxide material as SA. The SA device is fabricated by embedding a GO material into polyvinyl alcohol (PVA) film. The GO material was obtained through a modified Hummers method from expanded acid washed graphite flakes. The graphene absorber can be applied in a broad wavelength range because of its unselective absorption.

#### **3.2** Preparation and characterization of Saturable Absorber

GO was prepared through a modified Hummers method from expanded acid washed graphite flakes. The synthesis involves the following steps. First, 5 g of graphite was added into 125 ml of H<sub>2</sub>SO<sub>4</sub>. Next, 2.75 g of NaNO<sub>3</sub> was added before start of the reaction. Subsequently, the beaker with reagents was put into water/ice bath in order to keep it below 5°C. 15 g of KMnO<sub>4</sub> was added in portions into the mixture, which was vigorously stirred. After addition of the oxidant, the beaker was heated and kept at 30-35°C with continuous stirring. Afterward it was left at room temperature for overnight. In the next step, the deionized water was added so that the temperature did not exceed 35°C. The beaker was put into a water bath at a temperature of 35°C and stirred. The mixture was then heated to 95°C and kept under these conditions for 15 min. To stop the reaction 280 ml of deionized water and 5 ml of H<sub>2</sub>O<sub>2</sub> were added. The mixture was then rinsed with HCl solution to remove the sulfate ions and with deionized water in order to remove chloride ions.

GO solution were prepared by adding GO into DI water at volume ratio of 1:10 and 1ml of polysorbate 80 solution as a surfactant. Then, the mixed solution were stirred for 2 hours until the solution homogenous followed by ultra-sonication for an hour. The host polymer were prepared by dissolving 1 g of polyvinyl alcohol powder [PVA, 40000 MW, Sigma Aldrich] into 120 ml of DI water and stirred until completely dissolved. To fabricate the saturable absorber, GO solution were mixed with PVA suspension and followed by ultra-sonication for 2 hours. Then, the mixture was carefully poured into petri dish to avoid any bubble and left dry in room temperature. After dry, the thin film was slowly peeled out. It is then cut into a small piece to attach into an FC/PC fiber ferrule. The ferrule was then matched with another fresh ferrule via a connector after depositing index matching gel onto the fiber end to construct an all-fiber SA device.

#### **3.3** Configuration of the Q-Switched EDFL

The fabricated SA device was integrated into an EDFL cavity for Q-switching generation as shown in Figure 3.1. Inset figure shows the GO thin film. The laser cavity consists of a 2.4m long EDF as the gain medium, a wavelength division multiplexer (WDM), an isolator, the fabricated GO PVA SA and an 80/20 output coupler. The 980nm pump was launched into the EDF via WDM. The EDF used has an Erbium ion absorption of 23 dB/m at 980 nm. To ensure unidirectional propagation of the oscillating laser in the ring laser cavity, a polarization independent isolator was used. The laser signal was coupled out using 80:20 output coupler which keeps 80% of the light oscillating in the ring cavity for both spectral and temporal diagnostics. The output laser was tap from a 20 % port of the coupler. The spectral characteristic was measured using an optical spectrum analyzer (OSA) while the temporal characteristics were measured using a 500 MHz oscilloscope and a 7.8 GHz radio-frequency (RF) spectrum analyzer via a 1.2 GHz photodetector.



Figure 3.1: Configuration of the GO PVA film based Q-switched EDFL. Inset shows the image of GO film, which was sandwiched between two ferrules to form a SA device.

#### **3.4 Q-Switching performances**

Stable and self-starting Q-switching operation was obtained just by increasing the pump power over 39mW. There was no lasing below the threshold pump power. Such a low threshold power for Q-switching operation was most probably due to the small intra-cavity loss of the GO PVA SA. A stable pulse train with an increasing repetition rate was observed within the pump power from 39 to 96mW, which is a typical characteristic for the Q-switched laser. Figure 3.2(a) shows the output spectrum of the EDFL at the threshold pump power of 39mW. As shown in the figure, the laser operated at center wavelength of around 1563.3nm. Spectral broadening was observed in the spectrum due to the Self-Phase Modulation (SPM) effect in the laser cavity. Figure 3.2(b) shows typical oscilloscope trace of the Q-switched pulse train at pump power of 96mW. It shows the peak to peak duration or pulse period of 16.9  $\mu$ s, which is equal to the repetition rate of 61.77 kHz. The pulse width is measured to be around 10.62µs. It is also observed that the Q-switched pulse output was stable and no amplitude modulations in the pulse train was observed, which indicates that there was no self-mode locking effect during the Q-switching operation. To verify that the passive Qswitching was attributed to the GO PVA SA, the film was removed from the ring cavity. In this case, no Q-switched pulses were observed on the oscilloscope even when the pump power was adjusted over a wide range. This finding confirmed that the SA device was responsible for the passively Q-switched operation of the laser.



Figure 3.2: Spectral and temporal characteristics (a) Output spectrum at 39mW pump power (b) Typical pulses train at 96mW pump power.

Figure 3.3(a) shows the relationship between the pulse repetition rate and pulse width with pump power. As pump power increases from 39mW to 96mW, the repetition rate increases almost linearly from 44.33 kHz to 61.77 kHz. As pump power increases, more gain is provided to saturate the SA and thus increases repetition rate. In contrast, pulse duration decreases from 9.51µs to 5.57µs as the pump power increases. We observe a smaller change

of pulse width with the pump power at higher pump power. This is attributed that the SA is becoming saturated when more photons circulates inside the laser cavity as the pump power increased. The minimum attainable pulse duration is  $5.57\mu$ s, which is believed to be related to modulation depth of the SA. The pulse duration can be further decreased by shortening the cavity length and improving the modulation depth of the SA.

Figure 3.3(b) shows the relationship between the average output power and pulse energy with pump power in the proposed Q-switched EDFL. It is observed that both the average power and pulse energy increases with the increment of pump power. The average output power can be linearly increased from  $1.3\mu W - 3.34\mu W$  by tuning the pump power from 39 to 96mW. The maximum pulse energy of 0.054nJ was obtained at pump power of 96mW. The increment of pump power leads to a raise of average output power and shorten the pulse width and hence higher pulse energy is extracted in the Q-switching process.



Figure 3.3: Q-switching performances (a) Repetition rate and pulse width against pump power (b) Output power and pulse energy against the pump power.

To investigate the stability of our Q-switched pulse, the radio-frequency (RF) spectrum is obtained at the pump power of 96mW as shown in Figure 3.4. The RF spectrum shows the fundamental frequency of 61.77 kHz with a high signal to noise ratio (SNR) of 59.89dB. The SNR indicates good pulse train stability, comparable to Q-switched fiber lasers based on CNT and graphene (Ahmad, Muhammad, Zulkifli, & Harun, 2013b; Ahmed et al., 2014; Harun et al., 2012). It is expected that a better Q-switched pulse can be obtained by

optimizing the design of the cavity, including reducing the cavity length and cavity losses as well as optimizing its cavity structure and using higher quality GO based SAs.



Figure 3.4: RF spectrum at 96 mW pump power

#### **CHAPTER 4**

#### PASSIVELY MODE-LOCKED ERBIUM-DOPED FIBER LASER

#### 4.1 Introduction

Mode-locked fiber lasers are powerful sources of ultra-short pulses. They have the advantages over solid-state pulse lasers in the system robustness, beam quality, pumping efficiency, power scalability, excellent heat dissipation and easy operation, and have applications in the fields as spectroscopy, ultrafast science, medicine, machining and telecommunications (Holzwarth, 2000; Jiang, Huang, Leaird, & Weiner, 2007; Nishizawa, Chen, Hsiung, Ippen, & Fujimoto, 2004; Udem, Holzwarth, & Hänsch, 2002). Among them, Erbium-doped fiber lasers (EDFLs) have attracted much attention because of their low loss in silica based fibers, which are the medium of choice for conveying data in the current communication systems (H. Zhang et al., 2010). Passive mode-locking is an efficient and compact way of generating ultra-short pulses. The generation of mode-locked pulses is governed by various physical effects and their complex interplay, such as group velocity dispersion (GVD) compensated by self-phase modulation (SPM), gain saturation and higher-order dispersion-management. Nevertheless, it is compulsory to have a mode locking mechanism in the cavity to facilitate pulse formation.

Graphene as the first-discovered two-dimensional material is most widely used for passive mode-locking, due to its excellent properties of saturable absorption with ultrabroad operation waveband (from visible to mid infrared), ultrafast recovery time, low saturation intensity, and controllable modulation depth. Therefore, graphene is one of the most effective and popular SAs for passive mode-locking. However, at the moment, their polymer composites are lacking high power capacitance while the deposition procedure is not easy to be precisely controlled with a limited modulation capability (Hasan et al., 2009). In chapter, a mode-locked EDFL is demonstrated using a newly developed GO PVA composite film as a SA. A mode-locking pulses train with nanosecond pulse width was successfully achieved with three different cavity lengths. The proposed GO SA has advantages of easy fabrication, low price, and amphipathic properties as a mode-locker.

#### 4.2 Fabrication and characterization of saturable absorber

GO was prepared through a modified Hummers method from expanded acid washed graphite flakes (Yu, Zhang, Bulin, Li, & Xing, 2016). The synthesis involves the following steps. First, 5 g of graphite was added into 125 ml of H<sub>2</sub>SO<sub>4</sub>. Next, 2.75 g of NaNO<sub>3</sub> was added before start of the reaction. Subsequently, the beaker with reagents was put into water/ice bath in order to keep it below 5°C. 15 g of KMnO<sub>4</sub> was added in portions into the mixture, which was vigorously stirred. After addition of the oxidant, the beaker was heated and kept at 30-35°C with continuous stirring. Afterward it was left at room temperature for overnight. In the next step, the deionized water was added so that the temperature did not exceed 35°C. The beaker was put into a water bath at a temperature of 35°C and stirred. The mixture was then heated to 95°C and kept under these conditions for 15 min. To stop the reaction 280 ml of deionized water and 5 ml of H<sub>2</sub>O<sub>2</sub> were added. The mixture was then rinsed with HCl solution to remove the sulfate ions and with deionized water in order to remove chloride ions.

GO solution were prepared by adding GO into DI water at volume ratio of 1:10 and 1 ml of polysorbate 80 solution as a surfactant. Then, the mixed solution were stirred for 2 hours until the solution homogenous followed by ultra-sonication for an hour. The host polymer were prepared by dissolving 1 g of polyvinyl alcohol powder [PVA, 40000 MW, Sigma Aldrich] into 120 ml of DI water and stirred until completely dissolved. To fabricate the SA GO solution were mixed with PVA suspension and followed by ultra-sonication for 2 hours. Then, the mixture was carefully poured into petri dish to avoid any bubble and left dry in room temperature. After dry, the thin film was slowly peeled out. Fig. 4.1(a) shows the image of the film, which has a thickness of around 50  $\mu$ m. It is then cut into a small piece to attach into an FC/PC fiber ferrule. The ferrule was then matched with another fresh ferrule via a connector after depositing index matching gel onto the fiber end to construct an all-fiber SA device. We also investigate different characteristics of the GO SA, which can potentially affect the performance of the fiber laser. Morphological property of the film was investigated with field emission scanning electron microscope (FESEM) and the image is shown in Figure 4.1(b). We can see there are no obvious air holes or bubbles in the GO-PVA polymer film, which indicates the excellent uniformity of the SA.



Figure 4.1: Image of the film (a) real image (b) FESEM image

Figure 4.2(a) illustrates the Raman spectrum of the GO PVA film, which was excited by an Argon ion laser operating at 514nm. In the experiment, the laser power was fixed at 50mW and the exposure time was 10 seconds. The spectrum reveals two prominent peaks of GO at 1357cm<sup>-1</sup> and 1606cm<sup>-1</sup>, which are assigned to D and G bands, respectively. The D band corresponds to the structural imperfections created by the attachment of hydroxyl and epoxide groups on the carbon basal plane. The G band is obtained due to ordered sp<sup>2</sup> bonded carbon. It is worthy to mention that the 2D band was hardly observed in the spectrum. The 2D band was normally observed at 2700cm<sup>-1</sup> for graphene material. This indicates that there was no graphene on the GO film. The nonlinear absorption characteristic for the GO film was also performed by launching a self-constructed mode-locked Erbium-doped fiber laser (wavelength of 1552nm, pulse width of 1.5ps, repetition rate of 17.4 MHz). By performing a balance twin-detector measurement, the modulation depth of GO SA was 11 % at 1558nm. The absorption plot against input pump power is illustrated in Figure 4.2(b). The nonsaturable loss was 9 % and the saturation intensity was 1.3 MW/cm<sup>2</sup>.



Figure 4.2: GO PVA film characteristics (a) Raman spectrum (b) nonlinear absorption curve.

#### 4.3 Experimental setup

Figure 4.3 shows the schematic experimental configuration of the GO based modelocked erbium-doped fiber laser. The ring cavity included a piece of 3m erbium-doped fiber (EDF), which was pumped by a 980 nm laser diode via a wavelength division multiplexer (WDM). A 99/1 output coupler was used to couple out the output while maintaining 99% of the oscillating light inside the cavity. The SA device was constructed by sandwiching the prepared GO PVA film between two connector ferrules via a fiber adapter. A small amount of index matching gel was used to stick the film onto the ferrule while reducing the insertion loss of the SA device. A 200, 100 and 50 m long SMF pieces are used to construct ring cavities with different lengths of 208, 108 and 58 m respectively. Since we used 3 m piece of EDF, therefore the three cavities have 205, 105 and 55 m of SMF fiber. The SMF and polarization controller (PC) were used inside the cavity to introduce nonlinearity and to control the state of polarization of oscillating light so that the production of mode-locked laser can be facilitated and realized. An optical spectrum analyzer (OSA) with a resolution of 0.02nm, a 7 GHz radio-frequency analyzer, and a 500 MHz oscilloscope with a 2.5 GHz photo-detector were employed to monitor the laser output simultaneously.



Figure 4.3: Schematic laser configuration for the mode-locked EDFL.

#### 4.4 Laser Performance

In this study, the performance of the proposed GO based mode-locked EDFL was investigated at three different cavity lengths, which was obtained by adding a spool of 200, 100 and 50m long SMF inside the laser cavity. When the EDFL operation was initiated with pump power over 15.9mW, the laser first went to continuous wave (CW) operation. The output power shows a linear increase with pump power. When the pump power was increased to over threshold and the PC position was carefully adjusted, the laser came to the stable mode-locking regime. The mode-locking threshold pump powers are 65.5, 65.5 and 60.2mW for the EDFL configured with SMF lengths of 50, 100 and 200m, respectively. The stable mode-locking operation could be sustained up to the pump power of 80.4mW for all three lasers. The threshold is lowest at 200m SMF due to the higher nonlinearity effect in the cavity which supports the ultra-short pulses generation. Fig. 4.4 shows the typical pulses train for the mode-locked EDFL at three different SMF lengths; 200, 100 and 50 m when the pump power was fixed at 70.6mW. It is observed that the repetition rates vary with different cavity

length. The repetition rates were 0.9625, 1.786 and 3.266 MHz with 200, 100 and 50 m long SMF spool in the cavity, respectively as shown in Figure4.4. The observed repetition rates are in full agreement with the cavity lengths.



Figure 4.4: Typical pulses train at pump power of 70.6 mW for the EDFL configured with three different SMF spool lengths (a) 200m (b) 100m and (c) 50 m

Figure 4.5 shows optical spectra for the three different cavity lengths examined. It is observed that the spectrum shifts towards the shorter wavelengths with decreasing cavity lengths. This is attributed to lower losses with smaller cavity. The laser operates with one peak with centre wavelength of 1566 and 1564nm for 208 and 58m cavities respectively, whereas 108m cavity shows a dual wavelength output at 1563.3 and 1564.5nm as shown in Figure 4.5. This is due to strong suppression of mode competition at this length. The SMF

used has a group velocity dispersion (GVD) of -23.4ps<sup>2</sup>/km whereas the EDF has a GDV of 40ps2/km. Therefore calculated total dispersion are -4.77ps<sup>2</sup>, -2.337ps<sup>2</sup> and -1.167ps<sup>2</sup> for 208, 108 and 58m cavities respectively. Since the total GVD is negative, the laser operates in the anomalous dispersion regime. It is observed from the optical spectrum that the laser is a stretched pulse laser. This formation of stretch pulse is attributed to the incorporation of SA.



Figure 4.5: Output spectra of the mode-locked EDFL at pump power of 70.6 mW for different SMF lengths

The repetition rate and pulse width remain constant for the entire pumping range, but a reduction in pulse width is observed with 100m SMF as the pump power is increased as shown in Figure 4.6(a). This is due to dual wavelength generation. The observed pulse durations are 380 and 124 ns for 208 and 58m cavities while for 108 m it is in the range of 178 to 160ns. Calculated pulse energies and peak powers for the three different cavities are plotted against pump power to examine the behavior of the pulse energy and power as shown in Figure 4.6(b), (c) and (d). It's been observed that both pulse energy and peak power increase with increasing pump power. For 208m cavity, the observed pulse energies and peak powers are in the range 5.07 to 7.06nJ and 10.59 to 16.29mW respectively, with the pump power in the range of 60.2 to 80.4mW. Similarly for 108 m cavity the observed pulse energies and peak pulse powers are in the range of 2.67 to 3.44nJ and 13.15 to 18.94mW with the corresponding pump power range of 65.5 to 80.4mW. Likewise for 58 m cavity the observed pulse energies and peak pulse powers are in the range of 1.60 to 2.06nJ and 10.72 to 14.64mW with the corresponding pump power range of 65.5 to 80.4mW.

![](_page_42_Figure_1.jpeg)

Figure 4.6: Mode-locking performances (a) pulse width against pump power at three different cavity lengths (b) pulse energy and peak power against pump power at 208 m cavity length (c) pulse energy and peak power against pump power at 108 m cavity length

(d) pulse energy and peak power against pump power at 58 m cavity length

It is observed that the pulse energy decreases with the decreasing cavity lengths or increasing repetition rate. This is attributed to the mode-locking process does not change the average energy in the laser beam, but instead simply redistributes this energy in time. To verify the stability of the pulsed lasers, radio frequency (RF) spectra are taken for all three cavity lengths and are shown in Figure 4.7. It is observed that with 208m cavity the SNR is 54.02dB, with 108m cavity SNR is 53.5dB and with 58m cavity SNR is 52dB. These high SNR ratios for all the lasers show the stability of the lasers. These results are comparable with those of other passive saturable absorbers and the superiority of easy fabrication and hydrophilic property of GO will facilitate its potential applications for ultrafast photonics. By further optimization of the cavity design and improvement on the GO based SA, we could generate mode-locked pulses with narrower pulse width and larger pulse energy.

![](_page_43_Figure_1.jpeg)

Figure 4.7: RF spectra for the EDFL configured with three different SMF spool lengths (a) 200m (b) 100m and (c) 50 m

#### CHAPTER 5

#### CONCLUSION

Passively Q-switched and Mode-locked EDFL are demonstrated using a new graphene oxide material as SA. GO has advantages of easy fabrication, low price, and amphipathic properties as a Q-switcher and mode-locker in 1.5µm region. The SA device is fabricated by embedding a GO material into PVA film and obtained through a modified Hummers method from expanded acid washed graphite flakes.

A passively Q-switched EDFL operating at 1563.3 nm was successfully demonstrated based on GO PVA SA. Employing this device into an EDFL cavity, stable Q-switched pulses generation achieved within a pump power range of 39 to 96mW. Through fine increasing the pump power, the repetition rate could be changed from 44.33 kHz to 61.77 kHz, and pulse duration from 9.51  $\mu$ s narrow to 5.57  $\mu$ s. The pulse energy was 0.054nJ at pump power of 96mW.

Passively mode-locked fiber laser successfully demonstrated in three different EDFL cavities using GO based SA. A 200, 100 and 50m long SMF pieces are used to construct ring cavities with different lengths of 208, 108 and 58 m respectively. The mode-locking threshold pump power was lowest at the longest cavity length. With 208 m cavity length and total dispersion of -4.77 ps<sup>2</sup>, the EDFL operates at 1566 nm with repetition rate of 0.9625 MHz, pulse width of 380 ns, peak power of 16.29mW, pulse energy of 7.06nJ when the pump power was fixed at 80.4mW. The repetition rates increased from 0.9625 to 3.266 MHz with the reduction of cavity length from 208 to 50 m. On the other hand, the pulse width was lower with a shorter cavity length. The signal to noise ratio of the electrical signal spectrum was more than 50 dB for all lasers, which indicates their excellent stability.

From the first experiment, results shows that GO is a new potential SA material for pulsed laser applications. From the second experiment, results shows that the superiority of easy fabrication and strong solubility will facilitate potential applications of GO for ultrafast photonics.

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