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GRAPHENE BASED DEVICES FOR PASSIVELY MODE-LOCKED FIBER LASER APPLICATIONS

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in fulfillment of the requirement for the degree of
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Statement of Originality

I hereby certify that the work embodied in this thesis is the result of original research and has not been submitted for a higher degree to any other University or Institution.

Date

Yang Ye

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Abstract

This thesis focuses on the fabrication methodologies of broadband graphene-based devices and the investigation of their applications in an ultrafast pulsed fiber laser at selected wavelengths. Passively mode-locked fiber lasers generating ultrashort pulses have been intensively investigated and have emerged as one of the best light sources. Depending on the operation wavelength and pulse width, their applications can include high-speed optical communication, frequency metrology, microscopy, and micromachining. In mode-locking operation, random phases of the longitudinal electromagnetic waves (modes) in the laser cavity are locked into phase with each other. To lock the phase of the modes in the laser cavity, a saturable absorber (SA), which absorbs the light at low intensity and allows the light to pass through at high intensity, is an essential component of the fiber laser cavity. The development of passively mode-locked fiber lasers has attracted extensive interest in discovering new SA materials, for which nanocarbon materials such as single-walled carbon nanotubes or graphene have shown attractive optical properties. Nanocarbon material-based SAs can be used to reshape and stabilise intra-cavity pulses through nonlinear absorption. Graphene has been shown to be a strong candidate for SAs that offers many significant advantages including intrinsic broadband operation from the ultraviolet to far-infrared region, ultrafast recovery time and high third-order nonlinearity. However, optimisation of the SA fabrication methodologies is still essential for mode-locked fiber laser performance in both the time domain and frequency domain.

In this thesis, different methodologies of SA fabrication are investigated, including spray-coating, optical-driven deposition, mechanical exfoliation of graphite, direct transfer of chemical vapour deposition (CVD) graphene and three-dimensional (3D) graphene. To investigate the broadband saturable absorption properties of the prepared SAs, fiber laser cavities operating at 1 μm , 1.55 μm and 2 μm were built for performance analysis. By spray coating or optically driven deposition of the nanocarbon materials onto fiber end facets, an SA can be simply constructed with a thin film of graphene, and such SAs have demonstrated desirable saturable absorption properties. Passively mode-locked fiber lasers using graphene-based SAs have been successfully demonstrated. Graphene-based SAs prepared by mechanical exfoliation also show similar saturable absorption properties for ultrafast laser applications. Furthermore, to control the number of graphene layers attached to the fiber end facets, CVD graphene has been used as the SA material. We have successfully demonstrated ultrafast pulse generation using the same CVD graphene-based SA at selected wavelengths. The obtained mode-locked pulses are based on ytterbium-, erbium- and thulium-doped fiber lasers at the central wavelengths of 1039, 1561 and 1971 nm, respectively. In this work, dissipative soliton pulses were generated in an all-normal-dispersion ytterbium-doped fiber laser, whereas conventional solitons were generated in the anomalous-dispersion erbium- and thulium-doped fiber lasers. The intrinsic broadband operational properties of graphene devices for all major fiber laser wavelengths ranging from 1 μm to 2 μm were validated and investigated.

As the fabrication methods and applications of graphene are actively explored, integration of individual two-dimensional graphene sheets into a

macroscopic structure is becoming essential for the applications of graphene. The next part of the thesis focuses on the fabrication and applications of novel 3D graphene-based SAs. 3D graphene is a foam-like structure that consists of multiple graphene layers integrated as a whole. In contrast to graphite, 3D graphene is obtained via a bottom-up approach using a CVD method. By carefully regulating the growth mechanism, the number of layers and crystallinity can be easily controlled, and a highly flexible and transparent structure can be realised. A 3D graphene-based SA is easy to fabricate with no additional deposition procedure because of its freestanding, flexible and elastic network structure. In addition, simple sandwiching of freestanding 3D graphene between two fiber connectors to form a 3D graphene-based SA successfully prevents possible damage to the saturable absorption material during the SA fabrication process. Stable ultrafast soliton pulse generation in a mode-locked erbium-doped fiber laser incorporating a 3D graphene-based SA was successfully demonstrated. Furthermore, to enhance the thermal damage threshold of 3D graphene-based SAs, we demonstrated an erbium-doped fiber laser passively mode-locked by a 3D graphene SA with an evanescent field interaction configuration. With the 3D graphene penetrated by a side-polished fiber to form an SA with evanescent field interaction, self-started passive mode-locking for ultrafast pulse generation in an erbium-doped fiber laser was demonstrated.

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List of Acronyms

Abbreviation	Full Expression
SA	Saturable Absorber
NPR	Nonlinear Polarization Rotation
SESAMs	Semiconductor Saturable Absorber Mirrors
1D	One-dimensional
2D	Two-dimensional
CNT	Carbon Nanotube
Er	Erbium
CVD	Chemical Vapour Deposition
DI	De-ionised
3D	Three-dimensional
Yb	Ytterbium
Tm	Thulium
FWHM	Full-width Half-maximum
DCF	Dispersion-compensation Fiber
NLSE	Nonlinear Schrödinger Equation
SMF	Single Mode Fiber
EDF	Erbium-doped Fiber
DMF	N,N-Dimethylformamide
EDFA	Erbium-doped Fiber Amplifier
PMMA	Polymethylmethacrylate
UV	Ultraviolet
NIR	Near Infrared
LD	Laser Diode
WDM	Wavelength Division Multiplexer
ISO	Isolator
PC	Polarization Controller
OC	Output Coupler

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Chapter 1

Introduction

This introductory chapter provides a basic overview of the background of mode-locked fiber lasers, the motivation and objective of the research work and a brief introduction to the contents of the dissertation. Section 1.1 introduces the background and motivation of the research. Section 1.2 states the objectives. Section 1.3 presents the major contribution in this dissertation, and section 1.4 provides an overview of the thesis.

1.1 Background and Motivation

Ultrafast optics have attracted considerable attention in fundamental research and have become one of the most active fields of optics today. Passively mode-locked fiber lasers capable of generating ultrashort pulses have been intensively studied by virtue of their widespread use in various industrial applications and scientific research fields [1, 2]. Their applications include industrial material processing [3], optical communication [4], medical diagnostics [5] and micromachining [6]. In 1963, Koester and Snitzer demonstrated the first fiber laser [7], which lased at 1061 nm and used Nd-doped glass fiber as the gain medium. Later, in 1984, the first mode-locked soliton fiber laser was reported by Mollenauer and Stolen [8]. The soliton laser was created by incorporating a length of single-mode,

polarisation-preserving fiber into the feedback loop of a mode-locked colour-centred laser. Since then, intensive investigations of passively mode-locked fiber lasers have been undertaken to generate ultrashort picosecond to femtosecond pulse trains [9-12]. In particular, all-fiber passively mode-locked lasers possess advantages of simple and compact design, high peak power, excellent beam quality, high power conversion efficiency, low cost and reliability [6, 13-17].

A saturable absorber (SA) is the key component to achieving passive mode-locking in a fiber laser. The SA is an intensity-dependent component that reshapes and stabilises the intra-cavity pulses through nonlinear absorption [4, 18, 19]. Conventionally, several different technologies are available, including nonlinear polarization rotation (NPR) [14] and discrete SAs [20]. The NPR technique is based on light interference in a laser cavity, which functions as an artificial SA [14]. However, because light interference is sensitive to phase change, mode-locking of fiber lasers by the NPR technique is relatively unstable due to environmental perturbations. An alternative method to strengthen the robustness of the laser is to apply semiconductor saturable absorber mirrors (SESAMs). SESAMs are complex quantum well devices fabricated by molecular beam epitaxy on distributed Bragg reflectors [20, 21]. However, the fabrication process of SESAMs is complex, and they have limited operational bandwidth. Such limitations have motivated research into new materials for SAs. In recent years, one-dimensional (1D) and two dimensional (2D) nanocarbon materials such as semiconducting carbon nanotubes (CNTs) and graphene have demonstrated nonlinear absorption properties [22] and can function as SAs in mode-locked fiber lasers. The CNT has a number of

interesting and useful electrical, optical, chemical and mechanical properties that allow it to be a viable candidate as a material for SAs [23]. CNT-based SAs, which can enable the mode-locking of fiber lasers at a particular wavelength, have been intensively developed in recent years [12, 24-26]. The CNT possesses unique optical properties such as sub-picosecond recovery time and mechanical [25] and environmental robustness [27]. The wavelength of the CNT-based SA is defined by choosing the CNT diameter [28], and tunability is possible by using a wide-diameter distribution [12]. However, the main drawback of the CNT-based SA is its operational wavelength, which is limited by the diameter of the CNT [12]. Contrastingly, graphene, the 2D crystal of carbon with atoms arranged in a regular hexagonal pattern [22], possesses a zero bandgap property that allows it to become a broadband absorption material [29, 30]. Graphene-based SAs have been successfully implemented in a variety of lasers including fiber lasers [29-31], solid-state lasers [32], waveguide lasers [33] and semiconductor lasers [34]. Since Bao *et al.* [30] and Hasan *et al.* [25] demonstrated the use of graphene as an SA for ultrafast pulse generation, mode-locked fiber lasers incorporating graphene as SAs have attracted considerable attention and have been actively explored [35]. Recently, Popa *et al.* demonstrated the generation of 174-fs pulses from a graphene-based erbium (Er)-doped fiber laser [36]. Graphene SAs for the mode-locking of fiber lasers at 1 μm [37] and 2 μm [38, 39] have also been reported. The broadband absorption property of graphene is attributed to the linear dispersion of the Dirac electron [40]. At the same time, the broadband saturable absorption of graphene still requires further investigation. A broadband SA that can support a fiber laser to

generate ultrafast pulses with broad spectral coverage is essential for the ultrafast optics field [21]. This need motivates research into the broadband absorption properties of graphene and the study of possible new broadband absorption materials. To fabricate a nanocarbon material-based SA to be inserted into a laser cavity, several approaches have been demonstrated for the fabrication of the graphene-based SA such as the spray coating method [41], optically-driven deposition method [24], the direct mechanical exfoliation and imprinted graphite nanoparticle method [42] and the chemical vapour deposition (CVD) graphene film transferred by de-ionised (DI) water floating and dipping method [43]. Experimental investigations of these SA fabrication methods are explored.

As the fabrication methods and applications of graphene are actively studied, integration of individual 2D graphene sheets into a macroscopic structure of graphene with three-dimensional (3D) form is becoming essential for the applications of graphene [44]. Compared with graphite, 3D graphene is obtained from a bottom-up approach via the CVD method. It is a foam-like structure consisting of multiple graphene layers integrated as a whole. The number of layers and crystallinity can be easily regulated by carefully controlling the growth mechanism, which results in a highly flexible and transparent structure [44] that is ideal for photonics applications. Hence, investigations of the applications of 3D graphene are also actively explored.

1.2 Objectives

The objectives of this research are, by conducting extensive experimental investigations on nanocarbon material, to optimise the SA fabrication method, characterise SA devices and

explore the applications of the fabricated SAs including the investigation of the broadband absorption properties of graphene for all major fiber laser wavelengths ranging from 1 μm to 2 μm in ytterbium (Yb)-, Er- and thulium (Tm)-doped fiber lasers. The generation of solitons in mode-locked fiber lasers, conventional soliton generation in anomalous-dispersion Er-and Tm-doped fiber lasers and dissipative soliton generation in normal-dispersion Yb-doped fiber lasers are studied. The application of 3D graphene as a new saturable absorption material for ultrashort pulses generation is also explored and experimentally studied.

1.3 Major Contributions

The original contributions of this thesis are as follows:

1. Exploration of different methods of fabricating SAs, such as spray coating, optical driven-deposition, direct mechanical exfoliation and the imprinted graphite nanoparticle method, the CVD graphene film transferred by DI water floating and dipping method and the transfer of 3D graphene. SA fabrication methods are compared and optimised, linear absorption properties are investigated and nonlinear saturable absorption properties are characterised.
2. Investigation of Q-switched mode-locking generation using CVD graphene based SA in an Yb-doped fiber laser. It is observed that with the appropriate PC settings, stable operation regime of Q-switched mode-locking is obtained. While the pump power increases from 161 mW to 195 mW, the repetition rate of the Q-switched mode-locked pulse decreases from 4.1 KHz to 1.0 KHz. At the same time, the laser output power is increased from $\sim 22.8 \mu\text{W}$ to $\sim 37.3 \mu\text{W}$.

3. Investigation of experimental ultrashort pulse generation using multilayer graphene as the SA in an Er-doped fiber laser. The laser operation regimes switch between mode-locking and Q-switching as the pump power varies. The graphene-based SA is prepared by mechanical exfoliation from graphite flakes and attached to the fiber end facet. The fiber laser can operate in both the passive mode-locking and Q-switching regimes with the same setup. When the laser works in the mode-locking regime, the repetition rate is 17.7 MHz for single pulse generation, and the 3-dB spectrum width is measured to be 2.1 nm at the centre wavelength of 1561.5 nm. The full-width half-maximum (FWHM) pulse width measured from the autocorrelation trace is around 1.2 ps fitted by a sech^2 pulse profile. While the laser works in the Q-switching regime, its repetition rate varies from 34.2 KHz to 52.8 KHz depending on the pump power, with an output power ranging from 0.12 mW to 6.5 mW, respectively.
 4. Investigation of the formation of dissipative solitons in a Yb-doped fiber laser without additional filtering. A CVD graphene-based SA is used to assist with pulse formation. Even without the use of a physical bandpass filter in the cavity, the dissipative soliton is still automatically formed in the laser cavity. The developed dissipative soliton laser demonstrates a 3-dB spectral bandwidth of ~ 2.72 nm, which is much narrower than the Yb-doped fiber gain bandwidth. Numerical simulations are consistent with the experimental observations. Self-started mode-locking at the total cavity dispersion of ~ 3.67 ps² is observed. A fundamental repetition rate of the generated dissipative soliton of ~ 1.23 MHz with a signal-to-noise ratio greater than 60 dB is achieved. An artificial bandpass filter introduced by the long cavity design narrows down the effective gain bandwidth of the laser and leads to dissipative soliton formation.
-

5. Investigation of the broadband saturable absorption property of CVD graphene. The ultrafast pulses are based on Yb-, Er- and Tm-doped fiber lasers at the respective central wavelengths of 1039, 1561 and 1971 nm. By using the same CVD graphene-based SA, dissipative solitons are generated at 1 μm in an all-normal-dispersion Yb-doped fiber laser, and conventional solitons are generated in anomalous-dispersion Er- and Tm-doped fiber laser cavities.
 6. Demonstration of the first experimental observation of an all-fiber passively mode-locked fiber laser incorporating 3D graphene as the SA. The 3D graphene is synthesised by a template-directed CVD method. The SA is then simply formed by sandwiching the freestanding 3D graphene between two conventional fiber connectors with no deposition process required. Such a 3D graphene-based SA is shown to be capable of producing high-quality mode-locked pulses. A passively mode-locked fiber laser is constructed, and stable output pulses with a fundamental repetition rate of ~ 9.9 MHz and a pulse width of ~ 1 ps are generated from the fiber laser. The average output power of the laser is ~ 10.5 mW while the output pulse is operating in the single pulse region. The results imply that the freestanding 3D graphene can be applied as an effective saturable absorption material for passively mode-locked lasers. To further enhance the thermal damage threshold of the 3D graphene-based SA, we demonstrate an Er-doped fiber laser passively mode-locked by 3D graphene SA functioning with evanescent field interaction realised by a side-polished fiber. The 3D graphene is synthesised by template-directed CVD. The ring laser resonator based on the Er-doped fiber is capable of generating ultrashort optical FWHM pulses of 2.45
-

nm centred at 1564.97 nm. Pulses with a duration of 1 ps are repeated with a frequency of 9.53 MHz.

1.4 Thesis Organization

The rest of the thesis is organised as follows:

Chapter 1 provides a brief introduction to the research project of the dissertation.

Chapter 2 introduces the nanocarbon materials and the fundamental principles of the mode-locked fiber laser and briefly discusses nanocarbon material-based SA devices in passively mode-locked fiber lasers.

Chapter 3 presents the fundamental theory of modelling pulse propagation in different optical fibers and components and briefly presents background knowledge on the numerical simulation of soliton operation in fiber lasers.

Chapter 4 addresses methods for nanocarbon material-based SA fabrication, including spray coating, optical-driven deposition, mechanical exfoliation of graphite, CVD graphene transfer and the direct transfer of 3D graphene.

Chapter 5 investigates the mode-locked fiber laser incorporating a multilayer graphene-based SA. First, we demonstrate Q-switched mode-locking pulses generation in an Yb-doped fiber laser. Secondly, we demonstrate an Er-doped fiber laser mode-locked by a mechanically exfoliated multilayer graphene-based SA. Two operational regimes are observed for this experiment, the mode-locking regime and the Q-switching regime. Furthermore, ultrafast pulse generation based on Yb-, Er- and Tm-doped fiber using the same CVD graphene is presented. The obtained mode-locked pulse operates at the central

wavelengths of 1039, 1561 and 1971 nm. The broadband operational properties of CVD graphene-based SA are demonstrated.

Chapter 6 presents 3D graphene, a freestanding network structure of graphene to be applied as an SA material for ultrashort pulse generation. The 3D graphene-based SA can be formed by simply sandwiching the 3D graphene between two fiber connectors. Stable mode-locking operation in an Er-doped fiber laser is observed. In addition, to enhance the thermal damage threshold of the 3D graphene-based SA, we further investigate ultrafast pulse generation by a 3D graphene SA with evanescent field interaction.

Chapter 7 concludes the dissertation and recommends possible directions for future work.

Chapter 2

Literature Review

2.1 Introduction of Graphene

Graphene is a two dimensional material, in which the carbon atoms are arranged in a regular hexagonal pattern. A single layer of graphene is a flat monolayer of carbon atoms packed to a two dimensional honeycomb lattice [45-47]. It is the basic structure element for graphitic material of all other dimensionalities as it is shown in the Figure 2-1.

Figure 2-1 Single layer graphene

The carbon to carbon bonds in the graphene include 3 in-plane orbital perpendicular to the sheet. Since the bonding-bands and anti-bonding-bands are well separated in energy, they can be neglected in low energy regions [48]. Thus, the electric conduction in graphene is

mostly due to π electrons. Using the tight-binding approximation for the electrons, the dispersion relation between energy and 2-D wave numbers can be obtained as [49]:

$$E^{\pm}(k_x, k_y) = \pm \gamma_0 \sqrt{1 + 4 \cos \frac{\sqrt{3} k_x a}{2} \cos \frac{k_y a}{2} + 4 \cos^2 \frac{k_y a}{2}} \quad (2-1)$$

in which $a = \sqrt{3}a_{C-C}$ (with $a_{C-C} = 1.42\text{\AA}$: carbon-carbon distance) and γ_0 is the transfer integral between first-neighbor π -orbitals (typical value for γ_0 are 2.9-3.1eV). The $k = (k_x, k_y)$ vectors is the first Brillouin zone constitution the ensemble of available electronic momenta. The negative branch of this equation corresponds to the valence band (bonding π band) of graphene, while the positive branch corresponds to the conduction band (antibonding π^* band).

2.1.1 Band Structure and Optical Absorption

As shown in Figure 2-2, the valence and conduction band form two conic shapes, and touch each other at the point K [50][50]. The valence band is fully occupied while the conduction band is empty. The zero-gap linear band structure shown in Figure 2-2 is referred to as “Dirac cones”, and the K point is the “Dirac point” [51, 52]. The linear dispersion relation for both π and π^* band is given by the equation below obtained by expanding equation (2-1) in the vicinity of K or K’ points:

$$E^{\pm}(\kappa) = \pm \hbar v_F |\kappa| \quad (2-2)$$

where $\kappa = k - K$ is the wave vector around K (K') points, and $v_F = \sqrt{3}\gamma_0 a / 2\hbar \sim 10^6 m/s$ is the electronic group velocity [52]. Within this region, the bands form conical shapes with a zero-gap linear band structure are known as Dirac ones.

Figure 2-2 Band structure of graphene

Graphene exhibits a variety of unique electric properties, such as extremely high mobility due to ballistic transport of electrons, minimum conductivity, anomalous quantum hall effects, etc. [53] It also exhibits a variety of unique and unusual optical properties. Although graphene has a one atom thick layer, it can absorb a significant amount of incident light. The transmittance of single-layer graphene is calculated to be $T = (1 - 0.5\pi\alpha)^{-2} \sim 1 - \pi\alpha \sim 97.7\%$, where α is the fine structure constant ($\alpha = e^2/\hbar c \sim 1/137$). It can be seen that the absorption of $\sim 2.3\%$ is only determined by the universal physical constants, e , $\hbar$ and c , and the absorption is wavelength independent due to the zero bandgap structure of graphene. The absorption increases linearly with the number of layers N .

2.1.2 Nonlinear Optical Properties of Graphene

Nonlinear optical properties of graphene are more important in our research area. In nonlinear fiber optics, the polarization in the material can be expressed as:

$$P = \varepsilon_0 (\chi^{(1)} \cdot E + \chi^{(2)} : EE + \chi^{(3)} : EEE + \dots) \quad (2-3)$$

where ε_0 is the permittivity in vacuum and $\chi^{(j)}$ is the j th order susceptibility, $\chi^{(1)}$ is the linear susceptibility and is the dominant contribution to P . Its effects are included through the refractive index n and the attenuation coefficient α . $\chi^{(2)}$ is the second order susceptibility which is responsible for nonlinear effects such as second-harmonic generation and sum-frequency generation. The lowest order nonlinear effects in the optical fiber comes from the third order susceptibility $\chi^{(3)}$, which is responsible for phenomena such as third order harmonic generation, four-wave mixing, saturable absorption and nonlinear refraction change (Kerr effect) [54]. The third order susceptibility is very large for graphene. It is a complex quantity where the real part corresponds to the nonlinearity in the refractive index and the imaginary part is responsible for the nonlinearity in absorption.

The absorption coefficient α and the refractive index n with the 3rd order nonlinearity $\chi^{(3)}$ can be expressed as [49]:

$$\alpha = \frac{\alpha_0}{1 + I/I_s} + \alpha_{\text{int}} \sim \alpha_0 + \alpha_{\text{int}} - \alpha_0 \frac{I}{I_s} = \alpha_0 + \alpha_{\text{int}} + \frac{3\omega \text{Im}(\chi^{(3)})}{2\varepsilon_0 c^2 n_0^2} I \quad (2-4)$$

$$n = n_0 + n_2 I = n_0 + \frac{3\text{Re}(\chi^{(3)})}{4\varepsilon_0 c^2 n_0^2} I \quad (2-5)$$

where α_0 is the linear absorption coefficient, α_{int} is the intrinsic (unsaturable) absorption coefficient, and n_0 is the refractive index. I is the light intensity and I_s is the saturation intensity, ω is the optical angular frequency and n_2 is the nonlinear refractive index. The saturation intensity I_s is defined by photon energy divide by the absorption cross section σ and the recovery time τ as shown below, E_s is the saturation fluence [55]:

$$I_s = \frac{hf}{\sigma\tau} = \frac{E_s}{\tau} \quad (2-6)$$

Figure 2-3 (a) Schematic diagram of saturate absorption; (b) the typical transmission of SA.

The nonlinear absorption property of graphene is originated from its intensity-dependent absorption effect and the formula is shown in Eq. (2-4). The absorption of light decreases with increasing light intensity. When the input intensity of the incident light is low, the optical absorption by graphene is high. When the intensity of incident light is above certain lever, greater than the saturation intensity, the optical absorption by graphene is smaller. Such decrease in optical absorption is regarded as saturable absorption. The physical mechanism of such saturable absorption in graphene is schematically shown in Fig. 2-3 (a). The typical transmission of SA is shown in Fig. 2-3(b), the three main factors of saturable absorber includes modulation depth, which is the maximum change in the absorption;

saturation intensity, the intensity level that the SA saturates; and the non-saturation absorption which corresponding to the phonon scattering.

Based on the nonlinear absorption, graphene can be applied as SAs in passively mode-locked laser cavity. Current commercially available SA for passively mode-lock is SESAM. However, the SESAM has the disadvantage of having a complex and costly fabrication process, low optical damage threshold, and is not compatible with optical fibers. Compared to SESAM, graphene SAs have relatively faster recovery times, and hence can be used to generate ultrafast pulses as described in the following chapters.

2.1.3 3D Graphene

The 3D graphene (few layers) macrostructure was obtained through a direct synthesis method using template-directed CVD [56]. The growth of 3D graphene was carried out in a split tube (quartz) furnace using Cu foam as a catalytic substrate. The substrate was first annealed at 1000°C. Afterwards ethanol was bubbled into the quartz tube under constant Ar and H₂ flow, which would allow the decomposition of the ethanol to synthesize graphene film on the surface of the Cu foam [57, 58]. Next, the as-grown 3D-C/Cu sample was dip-coated with poly (methyl methacrylate) (PMMA) as a protective layer for the 3D graphene structure and subsequently immersed into hot diluted nitric acid (HNO₃) for 5 hours to completely etch off the Cu supporting structure. The result is a freestanding, ultra-light weight and flexible graphene foam, consisting of a few layers of foam-like graphene in a network, allowing charge carriers to move rapidly and with small resistance through the high-quality and continuous graphene building blocks as it is shown in Figure 2-4.

The scanning electron microscopy (SEM) images of each stage of the growth process are shown in Figure 2-4 (a) and the optical image of the 3D graphene is shown in Figure 2-4 (b). The structure of the freestanding 3D graphene can be seen to closely resemble that of the Cu foam with a porosity of 99.6% with pore diameter ranging approximately from 100 μm to 200 μm . Figure 2-4 (c) shows the transmission electron microscopy (TEM) result of the obtained 3D graphene, the average number of layers of the 3D graphene is between 3-7 layers. Figure 2-4 (d) shows the obtained Raman spectrum for the few-layer 3D graphene. There are three peaks, namely the 2D peak at 2700 cm^{-1} , the G peak at 1580 cm^{-1} and D peak at 1350 cm^{-1} [29]. The G band originates from in-plane vibration of sp^2 carbon atoms; the 2D band originates from a two phonon double resonance Raman process and is also denominated as the second-order Raman band; the D peak is reported to stand for the defect rate of graphene. In the present case, the D peak originates in the very thin character of the samples and small domain sizes, which yield a random orientation of the graphene crystals.

Figure 2-4 Characterization of 3D graphene showing (a) the scanning electron microscopy (SEM) images of each state of the growth, (b) the optical image of 3D graphene with 1.8x magnification, (c) the transmission electron microscopy (TEM) image with an average number of layers of 3-7, and (d) the Raman spectrum of the few-layer graphene foam [59].

2.2 Introduction of Passively Mode-Locked Fiber Lasers

Mode-locking is a widely used technique in optics field in which a laser can be made to generate ultrashort pulses, on the order of picoseconds or femtoseconds [60-62]. In a fiber laser cavity, there are several longitudinal modes satisfy the resonance condition and each of them oscillates in the cavity independently [63]. The relative phases of the modes are random and hence there is random fluctuation at the output intensity which will repeat itself in time period equal to the round trip time of the cavity. In mode-locking operation, the

random phases of the longitudinal modes inside the laser cavity are locked and constructive interference occurs periodically. Hence a resulting pulse train with a fundamental repetition rate corresponding to the laser cavity round-trip time is radiated as shown in Figure 2-5 [10]. There are several mode-locking techniques including active [64], and passive mode-locking [65].

Figure 2-5 Pulse train at mode-locking state

2.2.1 Fundamentals of Mode-Locking

The physics of mode locking generation is complicated, here the author only introduces the basic theory. There are numbers of longitudinal modes simultaneously operate in the laser cavity. The frequency separation among the modes is given by

$$\Delta \nu = \frac{c}{L_{opt}} \quad (2-7)$$

Where L_{opt} represents the optical length of the cavity. The separation of the modes is generally much smaller than the gain bandwidth, therefore multiples modes exist in the fiber laser. The total optical field can be written as

$$E(t) = \sum_{m=-M}^M E_m \exp[i(\phi_m - \omega_m t)] \quad (2-8)$$

where E_m , ϕ_m , and ω_m are the amplitude, phase and frequency of the m_{th} mode. If all the modes operate independently, there is no interference among different modes. So that the interference terms in the total intensity $|E(t)|^2$ averages out to zero. In this saturation, the laser operates as a multimode continuous wave laser.

Mode locking happens when the phases of various longitudinal modes are synchronized so that the neighboring modes phase differences fixed to a constant ϕ such that $\phi_m - \phi_{m-1} = \phi$. And the mode frequency is $\omega_m = \omega_0 + 2m\pi\Delta\nu$. In simplicity, if we assume all the modes have equal amplitude E_0 , the total intensity of the multi-modes can be express as:

$$|E(t)|^2 = \frac{\sin^2[(2M+1)\pi\Delta\nu t + \phi/2]}{\sin^2(\pi\Delta\nu t + \phi/2)} E_0^2 \quad (2-9)$$

The mode-locking total intensity shows a periodic function with a period of $\tau_r = 1/\Delta\nu$ which is the cavity round trip time. While mode-locking happens, the laser emits a pulse train with a fundamental repetition rate of $\Delta\nu$.

The pulse width is calculated from equation 2-3 to be $\tau_p = [(2M + 1)\Delta\nu]^{-1}$. As the modes number increase, the generated pulse width gradually narrows down. The total spectral bandwidth of all the modes locked is represented by $[(2M + 1)\Delta\nu]$, it is inverse proportional to the pulse width of the ultrashort pulses. In practice, the nature of gain broadening determines the actual relationship between the spectral bandwidth and the pulse width of the mode-locking pulses.

In general, the mode-locking can be by achieved actively or passively, named as active mode-locking and passive mode-locking. Active mode-locking requires a modulator to be inserted to modulate either the amplitude or the phase in the laser cavity [66]. Whereas passive mode locking doesn't require any active component (modulator) to be used. It generates ultrafast pulses by an all-optical nonlinear technique. Passive mode-locking utilize a nonlinear intensity dependent device to narrow down the input pulse. There are several methods to achieve passive mode-locking.

2.2.2 Saturable Absorbers (SAs) for Mode-Locking

In particular, all-fiber passively mode-locked laser owns advantages of simple and compact design, high peak power, excellent beam quality, high power conversion efficiency, low cost and reliability [6, 67-69]. To achieve passively mode-locking in a fiber laser by incorporating saturable absorbers (SAs), conventionally there are several different technologies including nonlinear polarization rotation (NPR) [14] and discrete SAs [20]. The NPR technique is based on the light interference in a

laser cavity which functions as an artificial SA [43, 70]. However, since light interference is sensitive to phase change, mode-locking of fiber lasers by NPR technique is relatively unstable due to environmental perturbation. An alternative method to strengthen the laser robustness is to apply semiconductor saturable absorber mirrors (SESAMs). SESAMs are complex quantum well devices fabricated by molecular beam epitaxy on distributed Bragg reflectors [6, 21]. However, SESAMs have complex fabrication process and limited operation bandwidth. Such limitations motivate research on new materials for SAs, such as carbon nanomaterials.

For the carbon nanomaterial such as carbon nanotube (CNT) and graphene, it shows nonlinear saturable absorption property and can be used as saturable absorber materials. However for CNT, its energy band gap depends on nanotube's diameter, it requires precisely control of CNT diameter to turn the absorption band of CNT based saturable absorbers (SAs) for mode-locking laser at a specific wavelength. Graphene, a two-dimensional material that contains a single layer of carbon atoms, has emerged as a promising material that solves the problems induced by SESAMs and CNTs.

Graphene has been demonstrated as a strong candidate for SAs which offers many significant advantages including intrinsic broadband operation from ultraviolet to the far-infrared region [71], ultrafast recovery time, and high thermal damage threshold [21, 22, 43, 71, 72].

2.3 Development of Graphene based Mode-locked Fiber Lasers

Graphene was conventionally fabricated by the mechanical exfoliation method in 2004 and from then on various kinds of fabrication methodologies are actively explored [40, 53, and 73]. Graphene based SAs have been intensively studied in a variety types of lasers including fiber lasers [29-31], solid-state lasers [32], waveguide laser [33], and semiconductor lasers [34]. Since Bao et al. and Sun et al. have successfully demonstrated mode-locked fiber lasers using graphene as SAs in [29, 30], graphene have attracted considerable attention and have been actively explored. Recently, Popa et al. have demonstrated 174 fs pulses generation from a graphene based Er-doped fiber laser [36], and Fu et al. reported broadband ultrafast pulse generation from several different fiber lasers mode-locked by graphene based SA [74]. Zhang et al. demonstrated graphene mode locked, wavelength-tunable dissipative soliton in an Er-doped fiber laser [75]. And Zhang et al. reported mode-locking of a Tm-doped fiber laser utilizing a graphene-polymer based SA operate at the center wavelength of 1.94 μm [38]. Huang et al. reported Er-doped mode-locked fiber laser based on CVD graphene saturable absorber. Optical nonlinearities of different numbers of graphene layers based SAs are compared in this work [76]. Lately, Sotor et al. demonstrated 88 fs pulses generation in a Er-doped fiber laser using graphene based SA [77] in Oct 2015.

Table 2-1 summarizes the state-of-art graphene-based mode-locked fiber laser in terms of laser key performance.

Table 2-1 State of the art of graphene based mode-locked fiber lasers

<i>Geometry</i>	<i>Linear [78] and Ring configuration [43]</i>
<i>Center Wavelength</i>	<i>1 μm [37], 1.55 μm [29, 30], 1.6 [79] and 1.94 μm [38], 2.8 μm [80]</i>
<i>Spectral Bandwidth</i>	<i>48 nm [77]</i>
<i>Pulse Width</i>	<i>Ultrashort pulse of ~88 fs [77]</i>
<i>Pulse Energy</i>	<i>6 nJ [81]</i>
<i>Repetition Rate</i>	<i>2.22GHz[82]</i>

Related to the dissipative generation at 1 μm without additional filter in the laser cavity, the wavelength of the dissipative soliton generation centered at 1069.8 nm and the 3 dB spectral bandwidth is ~1.29 nm. Our work shows a 3-dB spectral bandwidth of ~2.72 nm. We also demonstrated broadband absorption property of graphene based SA for mode-locked pulses generation from 1 μm to 2 μm using the same SA. In addition, we explored the 3D graphene based SA applications for ultrafast pulses generation.

Chapter 3

Theory of Passively Mode-Locked Fiber Lasers

Short-duration optical pulses have been extensively studied because of their applications ranging from digital communications, material processing, and diagnostic of ultrafast process. A train of equally spaced optical pulses in temporal domain can be generated from mode-locked fiber lasers. This Chapter includes the theories describing the pulse evolution in a passively mode-locked fiber laser, and the main equations that govern the pulse propagation in the laser cavity.

3.1 Introduction

In 1963, Koester and Snitzer demonstrated the first fiber laser [7]. The first fiber laser output at 1061 nm used Nd-doped glass fiber as the gain medium. In 1984, the first mode-locked soliton fiber laser was reported by Mollenauer and Stolen [8]. The soliton laser was created by incorporating a length of single-mode, polarization preserving fiber into the feedback loop of a mode-locked color centered laser. Since then, intensive investigations have been made for passively mode-locked fiber lasers to generate ultra-short pico- to femto-second pulse train.

Mode-locking is a widely used technique in optics in which a laser can be made to generate ultra-short pulses, on the order of picoseconds or femtoseconds. In a fiber laser cavity, several longitudinal modes satisfy the resonance condition and each of them oscillates in the cavity independently. The relative phases of the modes are random and hence there is random fluctuation at the output intensity which will repeat itself in time period equal to the round trip time of the cavity. In mode-locking operation, the random phases of the longitudinal modes inside the laser cavity are locked and constructive interference occurs periodically. Hence a resulting pulse train with a repetition rate corresponding to the cavity round-trip time is formed. There are several mode-locking techniques including active, passive and hybrid mode-locking. The main focus of this thesis is the passively mode-locking technique. In a passively mode-locked fiber laser, it is essential to understand how the pulse propagates in different optical fibers. Passive fiber and active fiber are the two major fiber types of a mode-locked fiber laser. For example, the most common passive fibers in a laser includes standard SMF, HI1060 fiber, dispersion-compensation fiber (DCF). In contrast to passive fibers, active fibers having an additional laser-active dopant, for instance, ytterbium, erbium, and thulium. In general, a mode-locked pulse processes high intensity and comprises many frequency components, its propagation in optical fibers simultaneously affected by the fiber nonlinearity and dispersion. These two effects can be described by nonlinear Schrödinger equation (NLSE) to model the pulse propagation along passive fibers.

3.2 Theoretical Model of Pulse Propagation in Optical Fibers

3.2.1 Pulse Propagation in Single-mode Fibers

An optical fiber is a flexible, transparent fiber made by silica or plastic to transmit light within its core region. A single-mode fiber is optical fiber which is designed to support only a single propagation mode per polarization direction for a specified wavelength. Single-mode fibers usually have a small refractive index difference between core and cladding. To achieve a total internal reflection, the refractive index of the fiber core is a bit higher than the cladding ($n_1 > n_2$). The condition for a step-index fiber single-mode guidance can be formulated using the V parameter:

$$V = k_0 a (n_1^2 - n_2^2)^{1/2} \quad (3-1)$$

Where $k_0 = 2\pi/\lambda$ with λ stands for the wavelength of light, and a is the core radius. If the V number is below 2.405, only the fundamental mode will propagate within the fiber core.

An ultrashort optical pulse train with pulse duration of picosecond or femtoseconds will form in the laser cavity when mode-locking of fiber laser happens. Such pulses generally undergo substantial spectral and temporal changes, because of chromatic dispersion and nonlinearities [83].

3.2.1.1 Fiber Dispersion

The dispersion of an optical fiber refers to the dependency of the phase velocity in an optical medium on the frequency or the propagation mode. There are mainly two types of dispersions in the single-mode fiber need to be considered, the chromatic dispersion and the polarization mode dispersion. Dispersion is generally an

intrinsic property of optical fibers, light of different wavelengths travels in optical fibers with different speed.

The chromatic dispersion of an optical medium is the frequency dependence of the phase velocity in a transparent medium. On a fundamental level, when an electromagnetic wave interacts with the bound electrons of a dielectric, the medium response, depends on the optical frequency ω [84]. In fact, there are generally two sources of chromatic dispersion in single-mode optical fibers: material dispersion and waveguide dispersion. When a ultrashort pulse propagates in an optical fiber, pulse broadening occurs due to the chromatic dispersion. Mathematically, by expanding the mode propagation constant β in a Taylor series about the pulse's carrier frequency ω_0 , the effect of the fiber dispersion on the propagation of an optical pulse is described by:

$$\beta(\omega) = n(\omega) \frac{\omega}{c} = \beta_0 + \beta_1(\omega - \omega_0) + \frac{1}{2} \beta_2(\omega - \omega_0)^2 + \dots \quad (3-2)$$

where

$$\beta_m = \left(\frac{d^m \beta}{d\omega^m} \right)_{\omega=\omega_0} \quad (m = 0, 1, 2, \dots) \quad (3-3)$$

The parameter β_0 is the propagation constant at the pulse's carrier frequency, β_1 is the inverse of the pulse group velocity and can be expressed by:

$$\beta_1 = \frac{1}{v_g} = \frac{n_g}{c} = \frac{1}{c} \left(n + \omega \frac{dn}{d\omega} \right) \quad (3-4)$$

β_2 expresses dispersion of the group velocity and is described by:

$$\beta_2 = -\frac{\lambda^2}{2\pi c} \frac{d\beta_1}{d\lambda} = \frac{1}{c} \left(2 \frac{dn}{d\omega} + \omega \frac{d^2 n}{d\omega^2} \right) \quad (3-5)$$

where n_g is the group refractive index, v_g represents the group velocity, c is the speed of light in vacuum. β_2 is the group velocity dispersion parameter, and it responsible to the pulse broadening effect when the pulse travels in a single mode fiber.

3.2.1.2 Fiber Nonlinearity

When the optical pulses with high intensity travel in the optical fiber, the fiber nonlinear effects appears. Hence, the fiber nonlinear effect should be considered when analyzing an intense light traveling in a single mode fiber. Fundamentally, the fiber nonlinearity originates from the nonlinear response of a dielectric medium to a strong electromagnetic field. It relates to the harmonic motion of bound electrons experiences in a light field [84].

Because the single mode fiber is made of symmetric molecule SiO_2 , normally do not need to consider the second-order nonlinear effects. Thus, the lowest-order nonlinear effects of optical fibers is the third order nonlinear optical susceptibility, which is responsible to the third-harmonic generation, four-wave mixing, stimulated Raman and Brillouin scattering, and optical Kerr effect. The appearance of third-harmonic generation and four-wave mixing needs to satisfy the phase-matching condition. Unless the optical fiber is specially designed, otherwise, the third-harmonic generation and four-wave mixing effects are negligible. In general, the most dominant nonlinear effect is the refractive index variation when optical fiber experiences high intensity electromagnetic field. It is called optical Kerr effect, which can be simplified by:

$$n(I) = n_0 + n_2 I \quad (3-6)$$

in which I represents the optical intensity inside the fiber. n_0 is the linear part of the refractive index, while n_2 is the nonlinear-index coefficient of the refractive index of the fiber. It relates to the third-order nonlinear susceptibility $\chi^{(3)}$:

$$n_2 = \frac{3}{8n} \text{Re}(\chi^{(3)}) \quad (3-7)$$

where $\text{Re}(\chi^{(3)})$ is the real part of $\chi^{(3)}$. The typical value of the nonlinear refractive index coefficient n_2 is $2.2 \sim 3.4 \times 10^{-20} \text{ m}^2/\text{W}$ [79], which is relatively small. Nevertheless, because the single mode fiber has a small core size, and the light travels in the core region for a long distance. The efficiency of the nonlinear effect is still dominant because of the light-fiber long distance interaction.

3.2.1.3 Nonlinear Schrödinger Equation

When optical pulses travel in an optical fiber, both of the chromatic dispersion and nonlinear optical Kerr effect affects the pulse propagation. Fundamentally, the most basic mathematical equation used to express the propagation of an optical pulses starts from the Maxwell's equations.

$$\nabla^2 \tilde{E} + \varepsilon(\omega) k_0^2 \tilde{E} = 0 \quad (3-8)$$

in which $\tilde{E}$ is the electric field expressed by:

$$\tilde{E}(r, \omega - \omega_0) \approx F(x, y) \tilde{A}(z, \omega - \omega_0) \exp(i\beta_0 z) \quad (3-9)$$

where $F(x, y)$ is the transverse distribution of propagation modes, $\tilde{A}(z, \omega - \omega_0)$ represents the pulse spectrum. And β_0 is the wave number, ω_0 is the central frequency. By considering concrete equations expressing the optical fiber's dispersion and Kerr nonlinearity, the well-known nonlinear Schrödinger Equation (NLSE) would be solved from the Maxwell's equations explained in numbers of textbooks such as Ref [84, 86]. The final equation describing the optical pulse propagation in an optical fiber is given as follows:

$$\frac{\partial A}{\partial z} + \frac{\alpha}{2} A + i \frac{\beta_2}{2} \frac{\partial^2 A}{\partial t^2} - \frac{\beta_3}{6} \frac{\partial^3 A}{\partial t^3} = i \gamma \left[|A|^2 A + \frac{i}{\omega_0} \frac{\partial}{\partial t} (|A|^2 A) - T_R A \frac{\partial |A|^2}{\partial t} \right] \quad (3-10)$$

in which $A = A(t, z)$ is the slow-varying envelop function with the local time t and the propagation distance z . β_2 and β_3 are the group velocity dispersion and third-order dispersion discussed in equation (3-2). Higher-order dispersion effects are neglectable for the pulse propagation in single mode fibers. γ is the nonlinear parameter which is expressed as $\gamma = \omega_0 n_2 / (c A_{eff})$ in which A_{eff} is the effective mode

area. $\frac{i}{\omega_0} \frac{\partial}{\partial t} (|A|^2 A)$ is the higher-order self-steepening term, and $T_R A \frac{\partial |A|^2}{\partial t}$ relates to

the raman self-frequency shift element. Since the pulse duration range studying is normally longer than 100 fs with relatively lower peak power, the third-order dispersion and higher-order nonlinear elements are very small and can be ignored. In addition, α represents the loss of the pulse during it travels within the single mode fiber, it is negligible. As a result, the final NLSE equation is given in the following:

$$\frac{\partial A}{\partial z} + i \frac{\beta_2}{2} \frac{\partial^2 A}{\partial t^2} = i \gamma |A|^2 A \quad (3-11)$$

NLSE shows a clear explanation for the optical pulse propagation in single mode optical fibers. There are a few important approximations used to obtain the equation, among them the slowly varying envelop approximation, the non-resonance approximation and the weak wave-guiding approximation are the most essential approximations [86]. It contains two important phase modulation elements resulting from the group velocity dispersion and the nonlinear effect. The nonlinearity always lead to a pulse broadening effect known as a positive frequency chirp. However, the group velocity dispersion effect can generate either positive or negative frequency chirp depending whether it is normal dispersion or anomalous dispersion. While the group velocity dispersion is negative, which means anomalous dispersion, the negative frequency chirp happens. It can balances the positive frequency chirp induced by nonlinear effect, and eventually reach a stable status in which the pulse travels in the optical fiber maintains the same pulse shape. Furthermore, the chirp introduced by the nonlinearity depends on the pulse intensity, so that the balance status can be achieved by adjusting the intensity. In short, in the anomalous dispersion laser cavity, optical soliton can be generated. And there are two requirements for the soliton formation. First, the optical intensity of the propagation light must be strong to ensure the nonlinear phase modulation to take place. And secondly, the fiber with anomalous dispersion should be used.

In the saturation of anomalous dispersion optical fiber used, the equation (3-7) has a soliton solution. It refers to the fundamental soliton, and the expression of the pulse has a hyper-secant functional form as follows:

$$A(t, z) = \sqrt{\frac{|\beta_2|}{\mathcal{N}_0}} \operatorname{sech}(t/t_0) \exp(iz/2) \quad (3-12)$$

in which t_0 is the pulse width of the soliton, and the pulse duration is expressed by

$t_{fwhm} \approx 1.756t_0$. The peak power required for the pulse formation is:

$$P_k = \frac{\beta_2}{\mathcal{N}_0} = \frac{3.11|\beta_2|}{\mathcal{N}_{fwhm}^2} \quad (3-13)$$

It is can be observed that, the pulse's peak power is a constant value, as long as the laser cavity parameters are fixed.

3.2.2 Pulse Propagation in Gain Fibers

Gain fiber is an essential component when construct a fiber laser, the gain fiber is obtained from doping rare-earth elements into the fiber. Various rare-earth ions can be doped in optical fiber, as a result the constructed fiber laser can operate in a wide wavelength range. For example, by doping Yb, Er, and Tm in an optical fiber, the resulting operation wavelength are 1000, 1550 and 2000 nm, respectively. When optical pulses propagate in rare-earth doped gain fibers, the gain effect needs to be considered beside the dispersion and nonlinear effects.

Figure 3-1 Pump schematic diagram of an Er-doped fiber

Figure 3-1 shows the pumping scheme of an Er-doped fiber, it is a three-level system. The two main efficient pumping wavelengths for the EDF are 980 nm and 1480 nm. In general, a gain fiber is characterized by the gain saturation, gain bandwidth and the small signal gain. In the amplification process, the broadened gain of the EDF is homogeneous and the gain coefficient of EDF can be written as:

$$g(\omega) = \frac{g_0}{1 - (\omega - \omega_g)^2 T_2^2 + P/P_{sat}} \quad (3-14)$$

Where g_0 represents the maximum small signal gain of the gain fiber, ω is the frequency of the incident light, ω_g is the resonant frequency of the dopant energy levels. T_2 refers to the dipole relaxation time, and the typical value of T_2 is ~ 100 fs for gain fiber. P is the power of the incident optical light, and P_s is known as the saturation power of the rare-earth-doped fiber, it depends on the dopant. If it is a three-level pumping scheme, the saturation power also relates to the pump strength. In general, if it is single pulse amplification, the term P/P_s can be negligible as it is much smaller than 1. Then the gain coefficient can be written as:

$$g(\omega) = \frac{g_0}{1 - (\omega - \omega_g)^2 T_2^2} \approx g_0 \left[1 - \frac{(\omega - \omega_0)^2}{\Omega_g^2} \right] \quad (3-15)$$

in which Ω_g represents the gain bandwidth, it is the FWHM of the gain spectrum $g(\omega)$. The gain shows by a Lorentzian profile in the above equation (3-13), in which the maximum gain happening when the incident signal frequency is same with the atomic transition frequency ω_0 . In addition, the gain bandwidth is inversely proportional to the dipole relaxation time T_2 of the fiber dopants. In practice, the typical gain bandwidth for an Er-dope fiber is 40 nm.

3.2.2.1 Gain Saturation

The laser gain medium is not possible to maintain a fix gain as the incident pump power increases. Because the upper energy level has finite density state, when the gain medium experience high input power, the gain must be decreased, the phenomenon of gain saturation takes place.

The typical gain medium upper state lifetime T_1 is around 1 ms while a short optical pulse propagates in the gain medium. The gain saturation can be ignored while the incident pulse duration well below the upper state life time. However, when the pulse duration is longer than the upper state life time, the saturated gain can be expressed as:

$$g_s(z) = \frac{g_0(z)}{1 + P_{ave}(z)/P_{sat}} \quad (3-16)$$

Where

$$P_{ave}(z) = \frac{1}{T_m} \int_{-T_m/2}^{T_m/2} |A(a, T)|^2 dT \quad (3-17)$$

in which P_{ave} is the average power of the pulses, T_m is the separation between two adjacent pulses.

3.2.2.2 Gain Dispersion

As the gain spectrum is not uniformly distributed, the incident light with different frequency will experience different gains, this phenomenon is named as gain dispersion. In order to study the effect of gain dispersion, the gain spectrum have to taking into account, as discussed in equation (3-13), the gain coefficient exhibits Lorentzian profile and can be written as:

$$\tilde{g}_f(\omega) = \frac{1}{1 - (\omega - \omega_g)^2 T_2^2} \quad (3-18)$$

where the input light frequency ω is close to the transition frequency ω_g . No matter what profile is used, the spectrum of the gain can be analyzed by Taylor expansion:

$$\tilde{g}_f(\omega) \approx 1 - (\omega - \omega_g)^2 T_2^2 \quad (3-19)$$

This equation (3-16) shows that the gain spectrum of the rare-earth doped gain fibers distributes in a nearly-parabolic profile.

3.2.2.3 Ginzburg-Landao Equation

As it is shown in above section, when an optical pulse propagates along a gain fiber, the light amplification effect must be taken into account. Fundamentally, the doped ions interacts with the light is governed by the Maxwell-Bloch equations [87]. On the other hand when the incident pulse width is larger than the dipole relaxation

time T_2 of the doped ion, by making rate equation approximation the gain term can be included into the NLSE directly.

As the gain depends on the incident light frequency ω , it can be expressed as:

$$g(\omega) = g(\omega_0) + \left. \frac{\partial g}{\partial \omega} \right|_{\omega=\omega_0} (\omega - \omega_0) + \frac{1}{2} \left. \frac{\partial^2 g}{\partial \omega^2} \right|_{\omega=\omega_0} (\omega - \omega_0)^2 + \dots \quad (3-20)$$

In which ω_0 refers to the gain's central frequency. Assume the carrier frequency of the incident pulse coincides with the ω_0 , the equation expressing optical pulse travels in the gain fibers can be written as:

$$\frac{\partial A}{\partial z} + \frac{i}{2} (\beta_2 + igT_2^2) \frac{\partial^2 A}{\partial T^2} = i \left(\gamma + \frac{i}{2} \alpha_2 \right) |A|^2 A + \frac{g}{2} A \quad (3-21)$$

in which g refers to the maximum gain coefficient, T_2 is the life time of the excited states, α_2 is the two-photon absorption coefficient. By considering the gain saturation, the peak gain coefficient g is time dependent and its variation with time is governed by [84]:

$$\frac{\partial g}{\partial t} = \frac{G - g}{T_1} - \frac{g|A|^2}{E_s} \quad (3-22)$$

where G is the small signal gain, and E_s refers to the saturation energy. T_1 is the population decay time, it is typically ~ 10 ms for an erbium doped fiber. Taking into account of that the pulse width is normally much more narrower than the population decay time, the term T_1 is negligible during the pulse amplification process, and $g(t)$ then simplifies to:

$$g(t) = G \exp \left[-\frac{1}{E_s} \int_{-\infty}^t |A|^2 dt \right] \quad (3-23)$$

The saturation energy E_s of EDF is generally about 10 μ J, and it is much greater than the pulse energy. Hence the gain saturation can be ignored. However, the excited state of the Er ions life time is typically ~ 10 ms. The pulse interval for the saturated pulsed fiber laser is around 10~100 ns, it is much smaller than the life time of the excited state of the Er. Thus, within the life time of the excited states, thousands of pulses still can passing through the gain fiber, as a result strong saturation effect could still be generated. In other words, the saturation energy required for a single pulse becomes much smaller.

If we transfer T2 into frequency domain, the previous equation is written as:

$$\frac{\partial A}{\partial z} + \frac{i}{2} \left(\beta_2 + i \frac{g}{\Omega_g^2} \right) \frac{\partial^2 A}{\partial T^2} = i \left(\gamma + \frac{i}{2} \alpha_2 \right) |A|^2 A + \frac{g}{2} A \quad (3-24)$$

in which Ω_g is the bandwidth of the gain. In practice, the optical fiber loss (~ 0.2 dB/km) should also be considered for a long distance light transmission. Then equation (3-21) further transfers:

$$\frac{\partial A}{\partial z} + \frac{i}{2} \left(\beta_2 + i \frac{g}{\Omega_g^2} \right) \frac{\partial^2 A}{\partial T^2} = i \left(\gamma + \frac{i}{2} \alpha_2 \right) |A|^2 A + \frac{1}{2} (g - \alpha) A \quad (3-25)$$

in which α is the loss of the optical fiber. If ignore the fiber loss α and gain g , the equation back to NLSE. Equation (3-22) is named as the cubic Ginzburg-Landau equation (GLE). By taking into account of the three dimensionless variables

$$\xi = \frac{z|\beta_2|}{T_0^2}, \quad \tau = \frac{T}{T_0}, \quad u = \left(\frac{\gamma T_0^2}{|\beta_2|} \right)^{1/2} A \quad (3-26)$$

Equation (3-22) can represent into a dimensionless form

$$i \frac{\partial u}{\partial \xi} = \frac{i}{2} \mu u + \frac{1}{2} \left(s + id \frac{\partial^2 u}{\partial \tau^2} - (1 + iu_2) |u|^2 u \right) \quad (3-27)$$

in which $s = \text{sgn}(\beta_2) = \pm 1$, d , μ and μ_2 are defined in the following form:

$$d = \frac{gT_2^2}{|\beta_2|}, \quad \mu = (g - \alpha) \frac{T_0^2}{|\beta_2|}, \quad \mu_2 = \frac{\alpha_2}{2\gamma} \quad (3-28)$$

In the case of the fiber operates with anomalous dispersion, $s = -1$, the solution of equation (3-24) is a chirped hyper-secant profile that further becomes the following form [88]:

$$\mu(\xi, \tau) = N_s \text{sech}(p\tau) \exp[iK_s \xi - iq \ln(\cosh p\tau)] \quad (3-29)$$

in which N_s , p , q , K_s are constants relate to the term d . This above equation of GLE is known as dissipative soliton solution, it is realized as a complex energy balance of dispersion, nonlinearity, gain and loss. It can be observed that, unlike the solitons in normal optical fibers, these dissipative solitons show the property of frequency chirp due to the effect of the doped fiber gain.

3.3 Theoretical Model of Pulse Shaping by Saturable Absorbers

SA is the essential cavity component for ultrashort pulses generation of the fiber laser, which contributes in the pulse shaping effect because of its intensity dependent absorption. When an incident light interact with a material, the absorption of the incident light decreases while the incident light intensity increases, such phenomena is known as saturable absorption property. When a SA interact with the incident optical pulse, the absorption coefficient can be expressed as:

$$\alpha = \alpha_{ns} + \alpha_s \quad (3-30)$$

where α_{ns} stands for the coefficient of the non-saturable absorption, and it corresponds to the intrinsic loss of the SA. α_s is the saturable absorption coefficient,

and it is related to the incident pulse intensity. In the case of a SA with slow response time, the below equation can be used to describe its property [89]:

$$\frac{d\alpha_s(z, T)}{dT} = -\frac{\alpha_s - \alpha_0}{\tau_{sa}} - \alpha_0 \frac{|A(z, T)|^2}{E_{sa}} \quad (3-31)$$

in which α_0 is the modulation depth of the SA, τ_{sa} express the recovery time, and E_{sa} is the saturation energy of the SA. If we assume recovery time of the SA to be negligible, equation (3-27) can be solved and results to an equation to describe the saturable absorption coefficient of a SA with fast response time:

$$\alpha_s(z, T) = \frac{\alpha_0}{1 + |A(z, T)|^2 / P_{sa}} \quad (3-32)$$

3.4 Theoretical Model of Mode-Locked Fiber Lasers

The previous sections of this chapter introduces the theoretical models of pulse propagation in ordinary optical fibers and gain fibers, as well as the modeling of the SA. In this section, the conditions for mode-locking and modeling of the mode-locked fiber laser will be discussed.

3.4.1 Mode-locking Condition

Figure 3-2 shows a typical configuration of a pulsed fiber laser constructed by the main components pump, gain medium, SA, single mode fiber, polarization controller and output coupler. However a fiber lasers have with such configuration is possible to operate in either mode-locking or Q-switching regime. The operation states is determined by the intra-cavity pulse energy.

Figure 3-2 Typical configuration of a mode-locked fiber laser.
LD: pump laser diode; WDM: wavelength-division multiplexer;
ISO: isolator; SA: saturable absorber; PC: polarization
controller; OC: output coupler.

The condition for a fiber laser to operate in mode-locking regime is written as [68]:

$$E_p^2 > E_{sat,g} E_{sa} \alpha_0 \quad (3-33)$$

in which E_p is the energy of the pulse, $E_{sat,g}$ is the saturation energy of the doped gain fiber and E_{sa} is the saturable absorber's saturation energy. α_0 stands for the SA's modulation depth. And in the same fiber laser, it can operate in both Q-switching or mode-locking regime depends on the pulse energy.

3.4.2 Master Equation for Mode-locked Fiber Lasers

In the case of the pulse energy satisfy the mode-locking condition, the final pulse formation results from the combination effects of all the optical components [1, 90]. If we make an assumption that in the fiber laser the changes of an optical pulse within one round trip is negligible, the master equation of the mode-locked fiber laser can be expressed as [91]:

$$T_R \frac{\partial A}{\partial T} + \frac{i}{2} (\bar{\beta}_2 + i \bar{g} T_2^2) L_R \frac{\partial^2 A}{\partial t^2} = i \bar{\gamma} L_R |A|^2 A + \frac{1}{2} (\bar{g} - \bar{a}) L_R A - \frac{\alpha_0/2}{1 + |A(T, t)/P_{sa}|} A \quad (3-34)$$

where $\bar{\beta}_2$, $\bar{g}$, $\bar{\gamma}$ and $\bar{a}$ are the average dispersion, gain, nonlinearity and loss of the fiber laser, respectively. T_R represents the laser cavity round-trip time. L_R is the total length of the cavity. t relates to the frame of the propagating pulse and T is the time of the propagation. And normally t is much lesser than the propagation time T .

We numerically simulated the laser operation. By circulating the optical light in the cavity and considering the effects of each optical element in the cavity, we explicitly considered the cavity gain, loss, nonlinearity and dispersion. We simulated the laser operation for the anomalous dispersion condition, near zero dispersion condition, and normal dispersion condition. The central wavelength of the simulation is set at 1560 nm. Figure 3-3 (a) and (b) shows simulation result for a ring cavity with a net cavity dispersion of -0.1 ps^2 . Soliton pulses generated as it is shown in the figure. A relatively narrow pulse width ($<1 \text{ ps}$) can be generated. The simulation result matches our experimental observations. Figure 3-3 (c) and (d) shows simulation result for a ring cavity with a net cavity dispersion near zero ps^2 . The pulse have a wider pulse width as compare to the soliton pulses generated. Figure 3-3 (e) and (f) shows simulation result for a ring cavity with a net cavity dispersion in the normal dispersion region. Positive net cavity dispersion is simulated at 0.02 ps^2 . Dissipative soliton pulses can be generated, the generated pulse is highly chirped, which it is typical for dissipative pulses. Experimental result on dissipative soliton generation is discussed in chapter 5.3.

Figure 3-3 Simulated result for (a, c, e) the optical spectra with cavity dispersion of -0.1, 0, and 0.02 ps²; (b,d,f) single pulse with cavity dispersion of -0.1, 0, and 0.02 ps².

For our experiment, the Yb-doped fiber laser operates in the normal dispersion condition with gain-bandwidth ~40 nm. Our spectral width is much smaller than the gain-bandwidth, hence the gain bandwidth in our Yb-doped fiber laser is not the limiting factor. The spectral width is limited by the narrowest spectral filtering

bandwidth, and it is a combination effect from the device bandwidth and filtering effect in the cavity.

For fiber lasers operate at the anomalous dispersion condition, the pulse width is a balancing effect from the nonlinearity and the dispersion. The SA contributes to the initial startup of the mode-locking operation. When the pulse propagates in the cavity the positive dispersion from the gain medium tends to broaden the pulse width, however the negative dispersion from the SMF tends to compress the pulse width. The pulse maintains its shape by the combination effect from all the optical components in the laser cavity.

3.5 Summary

In this chapter, the theories relate to a passively mode-locked fiber laser is introduced. Pulse propagation in single-mode and gain fibers are discussed. For the pulse propagation in passive fiber, dispersion, nonlinearity and NLSE is introduced. For the pulse propagation in an active fiber, gain saturation, dispersion and GLE is discussed. The model of a SA is introduced in the third section. And in section 3.4, we introduces the condition and the master equation of a mode-locked fiber laser.

Chapter 4

CNT and graphene based saturable absorber fabrication

4.1 Introduction

As the research of passively mode-locked fiber laser develops, the optimization of the SA fabrication methodologies become more and more important. This chapter focuses on the methodologies to integrate nanocarbon materials such as CNT or graphene with optical fibers to fabricate SA devices. They include spray coating, optical-driven deposition, mechanical exfoliation of graphite, and direct transfer of CVD graphene and 3D graphene. After the fabrication of the SAs, characterization of the device is also demonstrated.

4.2 Fabrication Methods

4.2.1 Spray Coating

Mechanical spray coating is a fast, low-cost, simple fabrication method to spray deposit a graphene film onto target fiber devices. It makes use of spray gun to deposit the dispersions

of graphene onto a fiber patch cord end facet (fiber ferrule) with N₂ gas. A thin layer of graphene film can be formed at the fiber ferrule by spray coating method.

Kish Graphite flakes are used to prepare the solution-dispersed layers of graphene, the graphite flakes are commercially available and can be purchased from Graphene Laboratories. The graphite flakes are exfoliated for 12 hours by ultrasonication in N,N-Dimethylformamide (DMF). DMF is capable of overcoming the strong van der Waals force and insures the homogenous dispersion of the few layer graphene with relative aggregate free distribution [92, 93], hence DMF is chosen as dispersant in the experiment. After the conduction of ultrasonication the resulting solution is then kept aside overnight to precipitate the unexfoliated graphitic particles. To remove the large aggregates, centrifugal separation is then conducted at room temperature for the obtained graphene dispersion at 4500 rpm for 1 hour. High quality graphene dispersion is obtained by collecting only the supernatant homogenous part. The graphene dispersion is observed to be stable against sedimentation and there is no further aggregation for a period of weeks.

Figure 4-1 (a) Absorption spectrum of graphene solution. (b) Raman spectra of graphene flakes [94].

The UV-visible absorption spectrum is measured by a spectrophotometer (Perkin-Elmer Lambda 950). Figure 4-1(a) shows the absorption spectra measurement of the graphene dispersed solution. The absorption spectrum shows featureless and flat over a wide spectral range from 700 nm to 1700 nm as expected [95, 96]. Raman measurement is also conducted for the graphene dispersed solution using WITec Raman microscopy. The samples for Raman measurement are prepared by diluted and drop-cast the solution onto a Si wafer with 300 nm thermally grown SiO₂. After that, the sample wafers are dried overnight at room temperature. And then the Raman measurements are performed at 532 nm excitation using a Raman microscopy. The spot size of the laser beam of the Raman microscopy is about 1 μ m diameter measured by a 100 $\times$ objective lens. Figure 4-1(b) plots the Raman spectrum of the prepared samples of the graphene dispersed solution. It shows three characteristic peaks: D ($\sim$ 1344 cm⁻¹), G ($\sim$ 1581 cm⁻¹), and 2D ($\sim$ 2703 cm⁻¹). The broader linewidth of the 2D-band represents the characteristic of few layer graphene flakes [97][90]. An increasing of the number of defects in the graphene based samples would result in an increasing of the D-band intensity. In our case, low intensity of D peak implies that as-produced graphene flakes possess a low defect-level [98].

Subsequently, as it is shown in Figure 4-2, a small volume of the prepared graphene solution is transfused into a spray gun. The spray gun is pumped with N₂ gas, and the fiber patch cords are fixed on the target holder. The fiber ferrules are cleaned by fiber cleaner before the deposition process. During the spray coating process, a heating gun is used simultaneously to dry up the DMF solution.

Figure 4-2 Setup for mechanical spray coating

By controlling the spray time and the N₂ gas pump pressure, a thin layer of graphene can be deposited on the fiber ferrules. The graphene-based fiber devices show an insertion loss of 1.4 ± 0.2 dB.

The spray coating method is relatively simple and easy to handle and can be used for mass production, however the deposition uniformity on the fiber ferrules is hard to control. In addition, during the spray coating process, a portion of the graphene solution sprayed outside the fiber patch cord end facet is wasted.

4.2.2 Optical-driven Deposition

Optical deposition of carbon nano-sized material has an advantage of being more efficient than other deposition methods, such as spraying, direct synthesis and polymer embedding. Optical deposition is a simpler and more cost effective handling technique for mass production of carbon nano-sized material-based optical devices. It enables area selective deposition of nanocarbon materials onto a core region of an optical fiber end facet. This fabrication method is relatively controllable and cost efficient [24, 99, 101]. The fabrication

of CNT-based and graphene based optical devices using optical-driven deposition method is introduced in this section.

Light guided by an optical fiber into a nanocarbon material-dispersed solution can be used to deposit nanocarbon material onto the fiber end via optical trapping and heat convection effect [99]. The force induced by the optical radiation on the nanocarbon materials suspended in liquids results in the deposition to take place [100]. Suspensions of micron-sized dielectric particles can generate strong artificial Kerr nonlinearities, in which optical gradient force is sufficient to move particles into regions of high optical power [24]. The optical forces on the nanocarbon materials suspended in liquid includes the gradient force, which acts perpendicular to the laser beam to move the particles from a region of lower intensity to a higher intensity region, and the scattering force operating in the direction of the beam's k vector. The optical power will generate heat in the suspensions as well, and thermophoretic motion of the material is from the hot areas (heated solution) to the cold area (fiber ferrule end facet) [24] as it is shown in Figure 4-3.

Figure 4-3 Schematic illustration of optically driven deposition of nano-particles.

Figure 4-4 shows the experimental setup for optical deposition of nanocarbon material on the fiber facet. In this experiment, a tunable laser operating at the wavelength of 1550 nm is employed as a light source. A high power erbium-doped fiber amplifier (EDFA) is connected to the tunable laser to amplify the laser beam. The amplified light from the EDFA delivers a maximum optical power of approximately ~23 dBm at 1550 nm. The power of the reflected light from the fiber end is measured by an optical power meter through a circulator. By controlling the injected optical power, the pattern of the deposition can be controlled. In general, for our experiment, a lower power leads to core region deposition, whereas a higher power normally leads to a ring pattern deposited around the

core region. The reflectivity drastically increases after the first deposition of an entanglement leading to an increase in the power reading of the power meter. The deposition is achieved by the optimization of the injected power while monitoring the reflection. In addition, the thickness of the deposition can be controlled by the duration of the deposition.

Figure 4-4 Experimental setup for optical deposition

Figure 4-5 shows the change in reflectivity during the deposition process of CNTs dispersed solution. In this experiment, the power of the amplified input light is ~21 dBm. The CNT deposition on fiber end starts 12 seconds later when the fiber end is put into the solution. Once the CNTs are optically trapped and deposited onto the fiber end, an enhanced reflection is observed. The reflectivity is increased more by than ~ 10 dB once the deposition occurs.

Figure 4-5 Plot of optical reflectivity against time for CNT deposition on fiber ends.

Figure 4-6 Microscopic image of fiber end surface after optical deposition of CNTs.

The microscopic image of the deposited fiber end in Figure 4-6 shows that CNTs are successfully deposited on the end surface. Due to the high injection power, a ring pattern is formed at the fiber end. The overall insertion loss of this CNT-based device is 1.23 dB.

Since CNTs are deposited around the core region where CNTs mostly interact with the evanescent field of the propagating light, the CNTs can avoid thermal damage in high power applications. Such CNT-deposited devices have potential applications in mode-locked lasers which can sustain the high intra-cavity power and enable high output power.

By using the same optical deposition setup as CNT deposition, here we demonstrate the manipulation of graphene on optical fiber ends. Graphene dispersed in ethanol solution is used in the experiment. The launched light delivers an optical power of 12 dBm. Compared to the CNTs dispersed in DMF solution, graphene dispersed in ethanol solution is found to require less power for the deposition to take place. Figure 4-7 shows the reflectivity change during the deposition process. The graphene deposition on the fiber end starts 20 seconds after the fiber is immersed into the solution. The reflectivity is increased by around 12 dB as shown in Figure 4-7. It takes 15 seconds to stabilize after the deposition starts. The microscopic image of the deposited fiber end is shown in Figure 4-8. Similar to the case for CNT, graphene layers are also successfully deposited on the end surface. After deposition, the insertion loss measured is 1.46 dB.

Figure 4-7 Plot of optical reflectivity against time for graphene deposition on fiber ends.

Figure 4-8 Microscopic image of fiber end surface after deposition of graphene.

Similar to the optically driven deposition of CNTs, the physical mechanisms responsible for optical deposition of graphene to take place are optical trapping and the thermal effect. Optical trapping is caused by the interaction of the intensity gradient of the laser beam which consists of the gradient and scattering forces. The thermal effect includes thermodiffusion force and thermally driven convection flow caused by the local heating of the solution by the laser energy. The thermal diffusion force is generated to move nanoparticles along a temperature gradient from the hot liquid suspension heated by the laser to the cold fiber ferrule end facet.

Because the deposition process for CNTs and graphene are similar, it is believed that another possible reason for the difference in required optical power should be the use of different types of the solvent. Optical deposition of CNTs onto the fiber end-face using CNT-ethanol solution has shown that optical power required by the deposition is ~10 dBm [24][24], whereas the deposition of graphene using graphene-DMF solution is ~23 dBm [102][95]. From our experiment, deposition of CNTs using CNT-DMF solution requires ~21 dBm of optical power whereas deposition of graphene using graphene-ethanol solution only requires 12 dBm. It is found that the solution plays an important role for the optical deposition of nanoparticles to take place.

The optical-driven deposition method enables the possibility to selectively deposit on the target area with high efficiency. The pattern of the deposition can be controlled by the injected optical power. In addition, the modulation depth of the nanocarbon material-based SA can be managed by controlling the deposition pattern. Nanocarbon material-based SA with ring pattern deposition can be used to enhance the thermal damage threshold in the mode-locked fiber laser applications.

4.2.3 Mechanical Exfoliation of Graphite

Mechanical exfoliation is a process to separate graphene layers using mechanical force from graphite flakes. An adhesive tape is generally used to peel off the graphene layers attached. Since Novoselov et al. managed to obtain and characterize few and single layer graphene based on mechanical exfoliation of graphite using scotch-tape method in 2004 [103], the ease of production and low cost make mechanical exfoliation of graphite the most popular method to prepare multi-layer graphene [42, 104].

The graphite flake is first placed onto an adhesive tape and peeled repeatedly to reduce the number of layers, where multi-layer graphene is obtained after repeating the steps mentioned above. Then the obtained graphene layers are transferred to a FC/APC fiber connector end facet by simply pressing the fiber ferrule to the adhesive scotch tape. Therefore, on the fiber end facet, a graphene layer is formed. Finally, the graphene-coated connector is connected to another cleaned FC/APC connector through a conventional adapter. The steps of the mechanical exfoliation of graphite are shown in Figure 4-9. The insertion loss induced by this multi-layer graphene coating is measured to be ~3.5 dB. The prepared optical device is then used as a SA in the mode-locked fiber laser cavity.

Figure 4-9 Steps of mechanical exfoliation of graphite method

The mechanical exfoliation of graphite using adhesive tape is an easy and quick method to fabricate multilayer graphene-based devices. However this method has a lack of controllability on the produced graphene layer number. In the second place, the purity of the produced multi-layer graphene is hard to be guaranteed due to exfoliating agent remnants [105]. Thirdly, the exfoliation method may introduce stain and various types of defects during the transfer of graphene layers to fiber ferrules. The performance of the multi-layer graphene-based devices might be affected by these defects as they break translational or rotational symmetry.

4.2.4 CVD Graphene Transfer

Although graphene is initially discovered by mechanical exfoliation method from graphite, it is hard to control the number of layers of graphene. An alternative method to obtain single layer or a few layers of graphene is CVD method [57, 106-109].

The CVD graphene film is fabricated on the surface of thin copper foil substrate [110]. First, the substrate is pretreated in high temperature to improve the quality and enlarge the grain

size of Cu foil. Next, the samples are heated up to 1000°C under the flow of Ar/Hydrocarbon gas/H₂ mixture for a few minutes. Then, the copper substrate covered by graphene film is cooled down to room temperature. Finally, a thin layer of polymethylmethacrylate (PMMA) is spin coated onto the graphene on Cu substrate as a support layer. Subsequently, the thin Cu foil is dissolved in the aqueous solution of ammonium persulfate. The obtained CVD graphene/PMMA film is cleaned by repeatedly using deionized water.

The graphene film float on the surface of deionized water is transferred on the fiber ferrule by immersing the fiber ferrule in the deionized water as shown in Figure 4-10. The CVD graphene-based SA is formed by sandwiching CVD graphene film between two FC/APC fiber ferrule connector end facets via a conventional adapter. The average number of layers of the CVD graphene we used is ~5 layers.

Figure 4-10 Transfer of CVD graphene to fiber ferrule

Figure 4-11 shows the linear transmittance spectrum of the CVD graphene measured by an UV-Visible-NIR spectrophotometer (Perkin-Elmer Lambda 950). The result shows a wavelength independent absorption over a wide spectral range between 500 nm and 1700 nm as expected, indicating a broadband saturable absorption property of the CVD graphene. The average optical transmittance between 500 and 1700 nm is ~ 86%.

Figure 4-11 Absorption spectrum of CVD graphene

The CVD graphene transfer method ensures accurate control of the number of graphene layers attached at the fiber ferrule, hence precisely controlling the modulation depth of the prepared device for the mode-locked fiber laser applications.

4.2.5 3D Graphene Transfer

As the fabrication methods and applications of graphene are actively explored, integration of individual two-dimensional graphene sheets into a macroscopic structure becomes more and more essential for the applications of graphene [44]. 3D graphene is a foam-like structure that consists of multiple graphene layers integrated as a whole. In contrast to

graphite, 3D graphene is obtained from bottom-up approach through CVD method. By carefully controlling the growth mechanism, the number of layers and crystallinity can be easily controlled and result in a highly flexible and transparent structure [44], which is ideal for photonics applications. A series of graphene based composites and microstructures have been fabricated using various methods such as chemically derived graphene sheets or direct synthesis of 3D graphene macrostructure by template-directed CVD. The latter method enables the fabrication of an interconnected flexible network of graphene as the fast transport channel of charge carriers for high electrical conductivity [44, 110]. The fabrication advantage of 3D graphene relies in the free-standing structures obtained. In contrast to graphene films, which often require to be handled with a substrate as a support, this foam-like structures are easier to handle and can be produced uniformly in large scale.

Figure 4-12 Fiber end facet type 3D graphene based SA

A fiber end facet type 3D graphene based SA is easy to fabricate without any additional deposition procedure because of its freestanding, flexible and elastic network structure. In addition, simple sandwiching of freestanding 3D graphene in between two fiber connectors to form a 3D graphene based SA successfully prevents the possible damage of the saturable absorption material during the SA fabrication process as it is shown in Figure 4-12.

The linear transmittance spectrum of the 3D graphene sample is further measured by an UV-Visible-NIR spectrophotometer (Perkin-Elmer Lambda 950). The result shows a wavelength independent absorption over a wide spectral range from 500 to 2200 nm, which is similar to that of graphene. The average optical transmittance between 500 and 2200 nm is ~82% as it is shown in Figure 4-13. The absorption properties of the 3D graphene are found comparable to those of multi-layer CVD graphene [112, 113].

Figure 4-13 Transmission spectrum of the 3D graphene measured by a spectrophotometer.

Furthermore, in order to enhance the thermal damage threshold of 3D graphene based SAs, we further fabricate 3D graphene SA with evanescent field interaction configuration. With the free-standing 3D graphene penetrated by a side-polished fiber to form a SA with evanescent field interaction, we produced a side-polished fiber type 3D graphene-based SA as shown in Figure 4-14.

Figure 4-14 Side-polished fiber type 3D graphene based SA.

4.3 Characterization of Fabricated SAs

A femto-second pulsed laser is then applied as a light source to characterize the nonlinear absorption of the prepared nanocarbon material based SAs as it is shown in Figure 4-15. The maximum output power of the pulsed laser source is ~ 9.5 mW at a center wavelength of 1561.9 nm. The output pulse trains exhibited a repetition rate of ~ 67.1 MHz with a pulse width of ~ 440 fs. A variable attenuator is connected to the output coupler of pulsed laser to tune the output power of the laser. A 90/10 coupler is connected to the attenuator where 10% of the power is coupled to the power meter as a reference power, and 90% of the power is coupled through the CVD graphene based SA and monitored by another power meter. The output power levels, with and without the SA, are measured simultaneously.

Figure 4-15 Experimental setup for the characterization of SAs

The modulation depth is estimated by the following procedure, adjust the variable attenuator to tune the output of the laser increasing from zero to its maximum. Measure and record the two power meter readings simultaneously. Take the data recorded from 10% port of the coupler as the reference power level, we can calculate the estimated power level before the light pass through the SA (90% coupler port power level). Transmission of the SA can be calculated by using the power reading after and before the SA. Plot out the Transmission change according to input power increment. The modulation depth is finally estimated by the maximum change in the transmission of the SA.

Figure 4-16 shows the saturable absorption measurement result for spray coated SA, the modulation depth of the device is 2.1%. And the non-saturation loss of the device is ~26.5%.

Figure 4-16 The nonlinear transmission measurement of the spray coated graphene-based SA.

Figure 4-17 shows the saturable absorption measurement result. The transmission rate is increased from 61.7% to 62.6% as the input power increases from 0 to 18 mW. Since CNTs are deposited around the core region where CNTs mostly interacts

with the evanescent field of propagating light, the modulation depth of the fabricated ring pattern CNT based SA is $\sim 1\%$.

Figure 4-17 The nonlinear transmission measurement of the CNT-based SA deposited with ring pattern by a femto-second pulsed laser.

Figure 4-18 shows the nonlinear absorption of the mechanical exfoliated graphene based SA. The modulation depth is measured to be $\sim 3\%$, with a non-saturation loss of $\sim 46.8\%$.

Figure 4-18 The nonlinear transmission measurement of the mechanical exfoliated graphene-based SA

Figure 4-19 shows the nonlinear absorption of the CVD graphene based SA. As the measurement is conducted for the whole SA rather than the material, the results

show a higher background loss. Our 3D graphene based SA shows a modulation depth of $\sim 2.3\%$, and a non-saturable loss of $\sim 17.3\%$, as shown in Figure 4-16.

Figure 4-19 The nonlinear transmission measurement of the CVD graphene based SA.

Figure 4-20 shows that of the nonlinear absorption of the 3D graphene based SA. Our 3D graphene based SA shows a modulation depth of $\sim 3.2\%$.

Figure 4-20 The nonlinear transmission measurement of the 3D graphene based SA

The nonlinear absorption property of graphene is originated from its intensity-dependent absorption effect and the formula is shown in Eq. (2-4). The absorption

of light decreases with increasing light intensity. There are 3 main factors of a SA, including modulation depth, saturation intensity and non-saturation loss. A comparison table of the characteristics of the fabricated SAs using five methods is shown in Table 4-1. For spray coating, optical-driven deposition and mechanical exfoliation methods, the characteristics of the fabricated SAs will be different for different thickness and configuration of materials attached to the fiber connector end facet. Therefore, in our research, we focus our research on the CVD graphene based and 3D graphene based SAs.

Table 4-1 Characteristics of the fabricated SAs

	Spray Coating	Optical-driven deposition	Mechanical Exfoliation	CVD graphene	3D graphene
Modulation depth	~2.1 %	~1 %	~3 %	~2.3%	~3.2 %
Saturation Intensity	~82 MW/cm ²	~67 MW/cm ²	~58 MW/cm ²	~215 MW/cm ²	~50 MW/cm ²
Non-saturation loss	~26.5 %	~38.4 %	~46.8 %	~ 17.4 %	43.7 %

This section shows the characterization of CNT based SA, CVD graphene based SA, and 3D graphene based SA. The CNT based SA is fabricated using optical-driven deposition, a ring pattern for evanescent-field interaction is achieved by this method. The formation of CNTs in a ring pattern on the end-facet of the connector enables an effective interaction between the CNTs and the propagated light in the fiber laser cavity. Although such SA has a low modulation depth of ~1%, it still owns notable advantages including higher thermal damage threshold [114], polarization insensitive, and easy fabrication. This type of SA can withstand relatively high cavity optical power. We demonstrated that thermal damage threshold of the ring-pattern CNT based SA improved by ~130% compared with

those CNT based SAs covering the entire core region [114]. For the CVD graphene based SA, the modulation depth can be controlled by controlling the number of layers of the CVD graphene. In general, the modulation depth increases with increasing thickness of the CVD graphene. However, the non-saturable loss of the device also increases. A higher non-saturable loss of the SA results in higher cavity loss if we implement the fabricated device in the pulsed fiber laser cavity. 3D graphene based SA shows a modulation depth of $\sim 3.2\%$ in this section, it also can be controlled by adjusting the configuration of the 3D graphene. It shows comparable recovery time and saturation power with CVD graphene based SA. The most remarkable advantage of 3D graphene based SA is easy to fabricate, and to ease the difficulty to handle nano-scaled materials. The characteristics of the 3D graphene based SA such as modulation depth, damage threshold, saturation power also can be controlled.

4.4 Summary

This chapter mainly focuses on the fabrication and characterization of nanocarbon material-based SA devices. The nanocarbon materials mainly include CNT and multilayer graphene fabricated from mechanical exfoliation, CVD graphene and 3D graphene. There are five methods introduced in this chapter: spray coating, optical-driven deposition, mechanical exfoliation of graphite, and direct transfer of CVD graphene and 3D graphene.

Chapter 5

Mode-Locked Fiber Lasers Incorporation Multilayer Graphene Based SA

This chapter mainly presents the investigation of multilayer graphene based SAs in mode-locked fiber lasers. In section 5.1, Er-doped fiber laser mode locked by mechanical exfoliated multilayer graphene based SA is demonstrated. The laser is able to switch its operation regime between mode-locking and Q-switching. And in section 5.2, Yb-doped fiber laser generating Q-switched mode-locking optical pulses are demonstrated. Then mode-locking of fiber lasers using CVD graphene based SA is investigated in Yb-, Er-, and Tm-doped fiber laser. A single CVD graphene based SA is able to generates ultrashort pulses at 1 μm , 1.55 μm and 2 μm . Dissipative soliton generation in Yb-doped fiber laser is achieved, and conventional soliton operation in Er- and Tm-doped fiber lasers is demonstrated. The broadband absorption property of graphene is evidenced experimentally.

5.1 Generation of Q-switched mode-locked pulses from Yb-doped Fiber Laser

5.1.1 Introduction

Here we demonstrated generation of Q-switched mode-locked pulses from a passively mode-locked Yb-doped fiber. The laser cavity is constructed entirely of fiber and fiber components with normal GVD at 1060 nm, a graphene based SA is used to initiate and sustain the generation of optical pulses. The graphene film used in the experiment was synthesized by CVD, and it is predominantly multi-layered (~5 layers). The CVD graphene based SA was prepared by sandwiching CVD graphene film between two FC/APC fiber ferrule connector end facets through a conventional adapter. The prepared device was then used as a SA in the mode-locked erbium-doped fiber laser cavity. In the experiment, it was found that with the appropriate PC settings, operation regime of Q-switched mode locking is observed. While the pump power increased from 161 mW to 195 mW, the repetition rate of the Q-switched mode-locked pulse decreased from 4.1 KHz to 1.0 KHz. We demonstrated Q-switched mode-locking, it is a train of pulses with cavity repetition rate inside a giant Q-switched envelop [115]. Laser pulses generates from Q-switched mode-lock show the advantages of high pulse energy [68]. Q-switched mode-lock pulses own the potential application in nonlinear frequency conversion, precise fabrication of microstructures [116], and surgery [21]. Q-switched mode-lock operation in Erbium doped fiber laser is demonstrated [117], here we demonstrated Q-switched mode-lock pulse generation in normal dispersion Yb-doped fiber laser cavity.

5.1.2 Experimental Setup

An all-fiber Yb-doped fiber ring laser is constructed to generate the Q-switched mode-locking pulses and the experimental setup is shown in Figure 5-1. The laser cavity was constructed using standard 1060 nm SMF based components. A 3.6 m long single-mode Yb-doped fiber with group velocity dispersion parameter β_2 of 0.0209 ps²/m and peak core absorption of 80 dB/m at 975 nm was employed as the gain medium in the laser cavity. A 980/1060 nm single mode WDM coupler was used to couple the 980 nm pump light into the laser cavity, and a polarization PC was employed to adjust the cavity polarization and optimize the mode-locking condition in the cavity. Additional 150 m Hi-1060 SMF was inserted to the laser cavity. A polarization-independent ISO was applied to ensure unidirectional light propagation in the laser cavity. A CVD graphene based SA was used in the cavity. 10% of the optical power at the emission wavelength was coupled out as the laser output from the laser cavity by a 1 × 2 optical coupler. The net cavity dispersion was ~3.5 ps². The characteristics of the output pulses including the optical spectrum, the repetition rate and the pulse width were observed using an optical spectrum analyzer (Ando AQ6317B), a 2GHz photo-detector (Thorlabs DET01CFC) followed by a signal source analyzer (Rohde & Schwarz FSUP26).

Figure 5-1 Schematic of all-fiber ring laser mode-locked by CVD graphene based SA. LD: pump laser diode; WDM: wavelength-division multiplexer; YDF: ytterbium-doped fiber; ISO: isolator; PC: polarization controller; OC: output coupler; SA: CVD graphene based saturable absorber

5.1.3 Experimental Result

By adjusting the PC within the cavity manually, an operation regime of Q-switched mode locking can be obtained. Figure 5-2(a) shows the optical spectrum change while the pump power increased from 161 mW to 195 mW, the 3 dB spectral bandwidth when pump power was set at 161 mW was 2.45 nm. Figure 5-2 (b) shows the observed Q-switched mode-locked trace from oscilloscope, and Figure 5-2 (c) shows a close-up view of the Q-switch mode-locked pulse. The temporal width and repetition rate of the Q-switched envelop were observed to be $\sim 41.4 \mu\text{s}$ and $\sim 4.1 \text{ kHz}$ respectively while the pump power was set at 161 mW. Figure 5-2 (d) shows the laser output power and repetition rate change while the pump power increased from 161 mW to 195 mW; the laser output power was increased from

$\sim 22.8 \mu\text{W}$ to $\sim 37.3 \mu\text{W}$, and the repetition rate of the Q-switched mode-locked pulse was decreased from 4.1 KHz to 1.0 KHz respectively.

Figure 5-2 Characteristics of Q-switched mode locking regime, the output of the fiber laser showing (a) the optical spectrum change according to the pump power, (b) the measured oscilloscope trace, (c) a close-up view of a Q-switched, mode-locked pulse, and (d) the output power and repetition rate change with respect to the pump power.

Figure 5-3 shows the changes of the Q-switched mode-locked pulse train and the fundamental RF spectrum while the pump power was set at 161mW and 190 mW respectively. As shown in Figure 5-3(b, and d) for the fundamental beating frequency of 1.23 MHz, A clean Q-switched mode-locked fiber laser with high extinction ratio of $\sim 63 \text{ dB}$ against the noise was demonstrated. The inset of Fig5-3(d) shows the RF spectrum at a pump power of 190 mW with a scan range of 30 MHz. Stable Q-switched mode-lock pulses were being generated.

Figure 5-3 Q-switched mode-locking pulse train and fundamental RF spectrum at pump power of (a-b) 161 mW, and (c-d) 190 mW. Inset: the RF spectrum for 30 MHz scan range.

5.1.4 Summary

We have demonstrated that CVD graphene can be used as a SA to produce Q-switched mode-locking pulses in an Yb-doped fiber laser. The generated optical pulses are found stable. We believe that such a functional photonic device can find more applications in various photonic fields.

5.2 Erbium-doped Fiber Laser Mode-locked by Mechanical

Exfoliated Multilayer Graphene based SA

5.2.1 Introduction

The passively mode-locking of an erbium doped fiber laser using mechanical exfoliated multilayer graphene SA is investigated in this section. The laser operation regimes switches between mode-locking and Q-switching are also investigated. The multilayer atomic graphene is fabricated from commercially available graphite flakes in this experiment. Mechanical exfoliation method is used to prepare the multilayer graphene based SA. The graphite flake is first placed onto a scotch tape and peeled repeatedly to reduce the number of the layers, where multi-layered graphene is obtained after repeated steps mentioned above. The prepared device is then used as a SA in the mode-locked fiber laser cavity.

5.2.2 Experimental Setup

In our experiment, the multi-layered graphene is fabricated from commercially available graphite flakes. We use mechanical exfoliation method to obtain the multi-layered graphene SA. The graphite flake is first placed onto a scotch tape and peeled repeatedly to reduce the number of the layers, where multi-layered graphene is obtained after repeated steps mentioned above. Then the obtained graphene sheets were transferred to a FC/APC fiber connector end facet by simply pressing the fiber ferrule to the scotch tape. Therefore, on the fiber end facet, a graphene layer can be formed. Finally, the graphene-coated connector is connected to another cleaned FC/APC connector through a conventional

adapter. The insertion loss induced by this multi-layered graphene coating is measured to be 3.5 dB. The prepared device is then used as a SA in the mode-locked fiber laser cavity.

Figure 5-4 shows the ring cavity passively mode-locked fiber lasers setup incorporating the prepared graphene-based SA. The total length of the laser cavity is estimated to be 17 m. A 0.6m long erbium-doped fiber is used as the gain medium in the laser cavity. A 980/1550 single mode WDM coupler is used to launch a 980 nm pump light into the laser cavity, where an isolator is applied to ensure the unidirectional operation of the laser. A polarization controller (PC) is used to adjust and optimize the mode-locking condition in the cavity. 10% of the optical power is coupled out as the laser output. The total cavity dispersion is in anomalous region with -0.37ps^2 . The characteristics of the output pulses are observed using an optical spectrum analyzer, a photo-detector followed by an oscilloscope, and an autocorrelator.

Figure 5-4 Experimental setup of the ring-cavity fiber laser. WDM: wavelength-division multiplexer; EDF: erbium-doped fiber; OC: output coupler; SA: graphene based saturable absorber.

5.2.3 Mode-Locking Regime

The laser starts to mode-lock at a pump power of 90.3 mW, producing multiple pulses in a round trip time in our experiment. After self-starting, the pump power is reduced to 34.4 mW to reach a single-pulse operation mode at a fundamental repetition rate of 17.7 MHz corresponding to the cavity length. The output spectrum and the autocorrelation trace at a pump power of 38.9 mW are shown in Figure 5-5 and Figure 5-6, respectively. At this pump power level, the average power of the laser output is 0.12 mW. The 3-dB spectrum width is measured to be 2.1 nm at a center wavelength of 1561.5 nm. The full-width half-maximum (FWHM) pulse width measured from the autocorrelation trace is around 1.2 ps fitted by a sech^2 pulse profile. The laser operation in mode-locking regime is found stable. When the SA is removed from the laser cavity, mode-locking operation is found not possible even with further increase the pump power to 100 mW. The observation confirms that the SA provides the required mechanism to initiate and sustain the passively mode-locking operation, where the graphene-layer is the key component. At a relatively higher pump power of ~70.5 mW, the laser operates in a multiple-pulse mode resulting in a higher harmonic repetition rate at the multiple of the fundamental round trip frequency.

Figure 5-5 Output spectrum of the fiber laser in mode-locking regime with 39-mW pump power.

Figure 5-6 Autocorrelation trace of the output pulses in the passively mode-locking regime.

5.2.4 Q-Switching Regime

With further increment of the pump power, the laser shifted from the mode-locked regime to a Q-switched regime. When the laser is pumped at a pump power higher than 93 mW, the laser starts to operate in Q-switch regime. The output spectrum and waveform are observed using optical spectrum analyzer and photodetector with

oscilloscope. In this operation regim, the output pulse repetition rate increases from 34.2 KHz to 52.8 KHz while the pump power increases from 93 mW to 295 mW, respectively, as shown in Figure 5-9. Same as the mode-locking case, no pulse generation is observed when the SA is removed from the cavity, confirming that the multi-layered graphene-based SA is responsible for the Q-switching of the laser. Fig. 5-7 and Figure 5-8 show the output spectrum of the laser and output pulse train at the input pump power of 250 mW, respectively.

Figure 5-7 Output spectrum of the fiber laser in Q-switching regime with 250-mW pump power [118].

Figure 5-8 Output pulse train of the fiber laser operating in Q-switching regime [118].

As the input pump power increases from 34 mW to 295 mW, the output power of the laser increases from 0.12 mW to 6.5 mW. The average output power of the laser increase linearly with the pump power as shown in Figure 5-9.

Figure 5-9 Plot of the laser output power and the output pulse repetition rate against the input pump power [118].

5.2.5 Summary

In summary, we have demonstrated pulse generation of an erbium-doped fiber laser incorporating multi-layered graphene saturable absorber. The graphene-based SA is

prepared by mechanical exfoliation from graphite flakes and attached to fiber end facet. The fiber laser is able to operate in both passively mode-locking and Q-switching regimes with the same setup. When the laser works at mode-locking regime, the repetition rate is 17.7 MHz for single pulse generation. While it works in the Q-switching regime, its repetition rate varies from 34.2 KHz to 52.8 KHz depending on the pump power, with an output power of 0.12mW to 6.5mW, respectively. The laser efficiency was ~22%. However, for the mechanical exfoliated multilayer graphene, the exact number of layers is hard to estimate. And during the transfer process of the multilayer graphene, defects at the graphene layer is possible to happen.

5.3 Dissipative Soliton Generation in Yb-doped Fiber Laser Incorporating CVD Graphene

5.3.1 Introduction

In this section, we demonstrate the generation of dissipative solitons in Yb-doped fiber laser. Our results show that by carefully optimizing the cavity gain, loss, dispersion and nonlinearity; stable dissipative soliton pulses are able to generate from an Yb-doped all-fiber laser with large dispersion without an additional filter. Zhao et al. have demonstrated an experimental work on dissipative soliton generation in Yb-doped fiber laser incorporated with CVD graphene and without additional bandpass filter [37]. The wavelength of the dissipative soliton generation centered at 1069.8 nm and the 3 dB spectral bandwidth is ~1.29 nm. In this section, we numerically and experimentally demonstrate the generation of dissipative soliton in Yb-doped fiber ring laser with graphene based SA without

additional filter. The CVD graphene based SA was fabricated by sandwiching CVD graphene film in between of two fiber ferrule connector end facets via a conventional adapter. We found that with the appropriate polarization controller (PC) settings, self-started mode-locking with dissipative soliton pulses was generated. The developed dissipative soliton laser demonstrates simpler configuration: single pumping, all-fiber cavity, and no incorporation of additional filter. Importantly, the 3-dB spectral bandwidth of ~ 2.72 nm is observed that is twice as much as the previous demonstrations [37].

The laser cavity is constructed entirely of fiber and fiber components with normal GVD at 1060 nm, a graphene based SA is used to initiate and sustain the generation of optical pulses. The graphene film used in the experiment was synthesized by CVD, and is predominantly multi-layered (~ 5 layers). In the experiment, it was found that with the appropriate PC settings, when the pump power was increased to 213 mW, self-started mode-locking with dissipative soliton pulses was observed first with multiple pulses generated. Single pulse generation was obtained by decreasing the pump power to 151 mW. The fundamental pulse repetition rate of ~ 1.23 MHz with a signal to noise ratio larger than 60 dB was observed for dissipative soliton generation. The all-normal-dispersion Yb-doped fiber laser operates in the dissipative soliton regime, as confirmed by the steep spectral edges in the output optical spectrum. Numerical simulations well reproduce the experimental observations.

5.3.2 Experimental Setup

An all-fiber Yb-doped fiber ring laser is shown in Figure 5-10. The laser cavity was constructed by standard 1060 nm SMF based components. The laser cavity consists of a piece of 3.6-m single-mode Yb-doped fiber as the gain medium with group velocity parameter β_2 of 20.9 ps²/km and peak core absorption of 80 dB/m at 975 nm. A 980/1060 nm single-mode WDM was used to couple 980 nm pump light into the laser cavity. A PC was employed to adjust the cavity polarization and optimize the mode-locking condition in the cavity. A 150-m Hi-1060 SMF with group dispersion parameter β_2 of 21.9 ps²/km was inserted to the laser cavity to promote the start of mode-locking by increasing the random pulse surge of the background noise. A polarization-independent ISO was applied to ensure unidirectional operation within the laser cavity. A CVD graphene based SA with a modulation depth of ~2.3% was used in the cavity. It was prepared by sandwiching CVD graphene film between two FC/APC fiber ferrule connector end facets via a conventional adapter. The average number of layers of the CVD graphene was ~5 layers. 10% of the optical power at the emission wavelength was coupled out as the laser output from the laser cavity by an optical coupler. The calculated total cavity dispersion β_2 was ~3.67 ps², which was in the normal dispersion region. The total length of the laser cavity is approximately 168 m. The characteristics of the output pulses including the optical spectrum, the repetition rate and the pulse width were observed using an optical spectrum analyzer (Ando AQ6317B), a 2-GHz photo-detector (Thorlabs DET01CFC) followed by a signal source analyzer (Rohde & Schwarz FSUP26).

Figure 5-10 Schematic of all-fiber ring laser mode-locked by CVD graphene based SA. LD: pump laser diode; WDM: wavelength-division multiplexer; YDF: Yb-doped fiber; ISO: isolator; PC: polarization controller; OC: output coupler; SA: CVD graphene based saturable absorber [119].

5.3.3 Experimental Result

Although there is no additional spectral filter and no dispersion compensation element in the laser cavity, a stable dissipative soliton pulse still formed. During the experiment, it was found that with appropriate PC settings and when the pump power was increased to 213 mW, self-started mode-locking with multiple pulses generated was observed. Single pulse generation was obtained by decreasing the pump power. Figure 5-11(a) shows the spectrum of the soliton pulses. Sharp steep spectral edges with center wavelength located at ~ 1038.9 nm was observed. The 3-dB spectral bandwidth gradually increased from ~ 1.59 nm to ~ 2.72 nm as the pump power is increased from ~ 155 mW to ~ 171 mW. The radio frequency (RF) spectrum of the output pulses for the fundamental repetition rate is shown in Figure 5-11(b), which corresponds to the laser cavity length of 168 m, and the fundamental peak corresponding to a cavity repetition rate of ~ 1.23 MHz. The signal to noise

ratio of the fundamental repetition rate is larger than 60 dB above the noise level, indicating that stable dissipative soliton pulses were being generated. The RF spectra shown was measured at a resolution of 100 Hz with a 2 MHz span. The inset of Figure 5-11(b) shows the laser output pulse train measured by a photo-detector followed by an oscilloscope. Single pulses were confirmed to be generated. The pulse-to-pulse separation was found to be ~811 ns, which matches the cavity roundtrip time, indicating that the pulses were formed as a result of mode-locking.

Figure 5-11 Dissipative soliton output of the fiber laser showing (a) the optical spectra with different pump power and (b) the fundamental RF spectra. Inset shows the output pulse train measured by a photo-detector [119].

It was found that negligible variation of the laser output parameters was observed over several hours of operation. When the CVD graphene based SA was removed from the laser cavity, mode-locking operation was found to be impossible even with a further increase in the pump power to ~250 mW. This observation indicates that the SA helps in the passive mode-locking operation of the fiber laser.

5.3.4 Numerical Simulation

To get an insight on the dissipative soliton evolution in the laser cavity, we numerically simulated the laser operation. The complex Ginzburg-Landau equation was used to describe the light propagation in the fibers [120]:

$$\frac{\partial A}{\partial z} + \frac{i}{2}(\beta_2 + ig_s T_2^2) \frac{\partial^2 A}{\partial T^2} + \frac{1}{2}(\alpha - g_s)A = i\gamma|A|^2 \quad (5-1)$$

where A is the envelop of the optical pulse, β_2 is the second-order dispersion coefficient of the fiber, T_2 is the dipole relaxation time, α is the fiber loss which is negligible, and γ represents the nonlinearity of the fiber. g_s is the saturable gain of the fiber, for undoped fibers $g_s = 0$. For the Yb-doped fiber, we further considered the gain saturation as:

$$g_s = \frac{g_0}{1 + \frac{E_{pulse}}{E_s}} \quad (5-2)$$

where g_0 is the maximum small signal gain in the gain medium and E_s is the normalized saturation energy. By circulating the light in the laser cavity and taking into account the action of each optical component in the cavity, we explicitly considered the cavity's dispersion, nonlinearity, loss, and gain. To get possibly close to the experimental conditions of our laser, the following parameters were used for the simulations: dipole relaxation time $T_2 = 0.1 \text{ ps}$; nonlinear fiber coefficient, $\gamma_{YDF} = 1 \text{ W}^{-1}\text{km}^{-1}$ and $\gamma_{SMF} = 0.8 \text{ W}^{-1}\text{km}^{-1}$; fiber dispersion parameter $\beta_{2_YDF} = 20.9 \text{ ps}^2/\text{km}$ and $\beta_{2_SMF} = 21.9 \text{ ps}^2/\text{km}$; Yb-doped fiber bandwidth of 40 nm;

small signal gain $g_0 = 1.5$; saturation energy $E_s = 0.3 \text{ nJ}$; and a center wavelength of $\lambda = 1039 \text{ nm}$.

Since a CVD graphene based SA was used in the experiment, the saturable absorption effect was also taking into account in the laser cavity simulation, the saturable absorption coefficient that is depends on the peak power of pulse is shown below:

$$\alpha_s = \frac{\alpha_0}{1 + \frac{P}{P_{sat}}} \quad (5-3)$$

where α_0 is the modulation depth of the SA, and P_{sat} represents the saturation power. We used the following simulation parameters in the simulation: the modulation depth is $\alpha_0 = 2.3\%$; and the saturation power of $P_{sat} = 150 \text{ W}$.

Figure 5-12 Numerical simulation of the fiber laser showing (a) the optical spectrum and (b) the corresponding output pulse [].

The numerical simulations were able to reproduce the experimental observations. Figure 5-12(a) shows the numerical simulation result of the optical spectrum of the fiber laser, the center wavelength is located at $\sim 1039 \text{ nm}$ and the 3-dB spectrum width is $\sim 1.3 \text{ nm}$. Figure 5-12(b) shows the generated corresponding single pulse

with a pulse width of ~ 100 ps. The simulated dissipative soliton pulses can be observed to be highly chirped, and it is well known that the chirped dissipative soliton pulses can be compressed outside the laser cavity. Due to the broad gain bandwidth of the Yb-doped fiber (~ 40 nm), a bandpass filter is generally required to assist in the formation of the dissipative solitons in the Yb-doped fiber laser. However in our Yb-doped fiber laser, the dissipative soliton can be automatically formed without adding in an extra filter. The spectral filtering is possible provided from the intracavity birefringent filter of the laser [25].

5.3.5 Summary

We have demonstrated the generation of dissipative soliton pulses in an Yb-doped fiber laser. CVD graphene was used as a SA to passively mode-lock the fiber laser. And the dissipative soliton pulses generated were found to be stable. The mode-locked pulses are highly chirped, which is a typical feature of dissipative solitons. The dissipative solitons are the consequence of the combined effects of the cavity's gain, loss, dispersion and fiber nonlinearity. Self-started mode-locking was observed. The center wavelength of the soliton pulse was located at ~ 1038.9 nm and the 3-dB spectral bandwidth gradually increased from ~ 1.59 nm to ~ 2.72 nm as the pump power was increased from ~ 155 mW to ~ 171 mW. Laser cavity repetition rate was ~ 1.23 MHz, while the signal to noise ratio of the fundamental repetition rate was ~ 60 dB above the noise level, indicating that dissipative soliton pulses generated were stable. It was shown that the dissipative soliton operation could still be achieved in the laser cavity even without the insertion of a bandpass filter. It is believed that, an artificial bandpass filter could naturally introduced in the cavity,

and it narrows down the effective gain bandwidth of the laser consequently. In the end it results in dissipative soliton formation.

5.4 Erbium-doped Fiber Laser Mode-locked by CVD Graphene based SA

5.4.1 Introduction

The ultrafast optical pulses generation at the communication wavelength (1550 nm) is demonstrated in this section. As mentioned in the earlier section, CVD graphene can assist a fiber laser system to generate ultrashort pulses [76, 121]. The CVD graphene based SA is easy to fabricate and integrate into an all-fiber structure. In addition, compare to other graphene based SA fabrication methods such as spray coating and optical-driven deposition, the modulation depth of the SA device can be accurately controlled through carefully control the number of the graphene layers. In this section, we utilize the same CVD graphene as it is in section 5.2. The CVD graphene based SA is shown in Figure 5-13, the number of the graphene layers is around 5 layers. A CVD graphene layer is sandwiched by two fiber connectors through an adapter, the modulation depth of the fabricated SA is ~2.3%.

Figure 5-13 CVD graphene based SA

5.4.2 Experimental Setup

An all-fiber Er-doped fiber ring laser is shown in Figure 5-14. The laser cavity was constructed using standard single-mode fibers based components. A 0.6 m long single-mode Er-doped fiber (nLight LIEKKI Er110-4/125) with group velocity dispersion parameter β_2 of 0.012 ps²/m and peak core absorption of 110 dB/m at 1530 nm was employed as the gain medium in the laser cavity. A 980/1550 nm single mode wavelength-division multiplexer (WDM) coupler was used to launch a 980 nm pump light into the laser cavity, and a polarization controller (PC) was employed to adjust the cavity polarization and optimize the mode-locking condition in the cavity. A polarization-independent isolator (ISO) was applied to ensure unidirectional light propagation in the laser cavity. A CVD graphene based SA was used in the cavity. 10% of the optical power at the emission wavelength was coupled out as the laser output from the laser cavity by a 1 × 2 optical coupler. The total length of the laser cavity is approximately 11.9 m corresponding to a fundamental repetition rate of 17.37 MHz. The net cavity dispersion was ~ -0.24 ps². The characteristics of the output pulses including the optical spectrum, the repetition rate and the pulse width were observed using an optical spectrum analyzer (Ando AQ6317B), a 2GHz photo-detector (Thorlabs DET01CFC) followed by a 350 MHz oscilloscope (Agilent Technologies), and an autocorrelator (Alnair Labs HAC-200).

Figure 5-14 Schematic of all-fiber ring laser mode-locked by CVD graphene based SA. LD: pump laser diode; WDM: wavelength-division multiplexer; EDF: Er-doped fiber; ISO: isolator; PC: polarization controller; OC: output coupler; SA: CVD graphene based saturable absorber

5.4.3 Experimental Result

Self-started mode-locking is also observed when the pump power reaches 35 mW. The pump power is then gradually decreased, pulse train remains stable during the decreasing process because of the hysteresis effect [122]. Single pulse was generated while the input power increased to 26.7 mW. The mode-locked fiber laser operation is found stable. The typical spectrum of the mode-locked pulses is shown in Figure 5-15, where the center wavelength is located at 1564.97 nm and the 3-dB spectral bandwidth is measured to be ~2.28 nm. Spectral sideband can be observed from the spectrum, it shows that the fiber laser is in anomalous dispersion regime, and operates in the conventional soliton mode-locking mode. The output pulse train measured by an oscilloscope is shown in Figure 5-16, the cavity repetition rate of ~17.37 MHz is observed. The fundamental repetition rate corresponds to the laser

cavity length. The autocorrelation trace of the output pulse was measured to be ~ 1 ps with a sech^2 pulse shape fitting as shown in Figure 5-17, where the solid red line is the sech^2 pulse fitting and the black circles are the experimental data.

Figure 5-15 Output spectrum of the fiber laser at the pump power of 26.7 mW

Figure 5-16 Output pulse train of the fiber laser

Figure 5-17 Autocorrelation trace of the output pulses

5.4.4 Summary

In summary, we demonstrate that multilayer CVD graphene can exhibit saturation absorption at the communication wavelength. The Er-doped fiber laser generates stable ultra-short pulses in the conventional soliton operation regime. Self-starting mode-locked lasing was observed, and the resultant pulse train with a fundamental repetition rate of ~ 17.37 MHz and a pulse width of ~ 1 ps was achieved. The laser efficiency was $\sim 52\%$, and the time-bandwidth product of the generated pulse is ~ 0.36 . The conventional soliton operation is found stable.

5.5 Thulium-doped Fiber Laser Mode-locked by CVD Graphene based SA

5.5.1 Introduction

Mid-infrared fiber lasers that can generate ultrashort pulses have various applications, such as material processing, communication, chemical and biomedical sensing and military applications as we discussed in the earlier sections. In this section, we report ultrashort pulse generation in Tm-doped fiber laser at long wavelength. Long wavelength ultrafast fiber lasers have the potential applications in various field, such as medical diagnostics, laser surgery, gas detection, material processing, long-range radar and free-space optical communications [38, 39, 123]. In our study, graphene has shown excellent property to generate ultrashort pulses, it is easy to fabricate and integrate into all-fiber structure. It shows nonlinear absorption property in 1 and 1.55 μm , to further verify the broadband saturable absorption property, a Tm-doped fiber laser operates at long wavelength (2 μm) is constructed and tested in this section. We used the same CVD graphene based SA to generate ultrashort pulses at the center wavelength of 1971.3 nm. The constructed fiber laser operates in anomalous dispersion region, conventional soliton operation is observed.

5.5.2 Experimental Setup

The experimental setup of the CVD graphene based passively mode-locked fiber laser is as shown in Figure 5-18. A 3.5 m long TDF was used as the gain medium.

The laser was pumped by a 1550 nm tunable laser source connected to a high power erbium doped fiber laser. A 1550/2000 nm single mode WDM is used to couple the pump light into the laser cavity. A polarization controller (PC) was employed to adjust the cavity polarization and optimize the mode-locking condition in the cavity. A polarization-independent isolator (ISO) was applied to ensure unidirectional light propagation in the laser cavity. A CVD graphene based SA was used in the cavity. The average number of layers of the CVD graphene is ~5 layers. 10% of the optical power at the emission wavelength was coupled out as the laser output from the laser cavity by a 1×2 optical coupler. The net cavity dispersion was $\sim -0.58 \text{ ps}^2$.

The characteristics of the output pulses including the optical spectrum, the repetition rate and the RF spectrum were observed using an optical spectrum analyzer (Ando AQ6317B), a 2GHz photo-detector (Thorlabs DET01CFC) followed by a 350 MHz oscilloscope (Agilent Technologies), and a signal source analyzer (Rohde & Schwarz FSUP26).

Figure 5-18 Schematic of all-fiber ring laser mode-locked by CVD graphene based SA. PUMP: pump laser diode; WDM: wavelength-division multiplexer; TDF: Tm-doped fiber; ISO: isolator; PC: polarization controller; OC: output coupler; SA: CVD graphene based saturable absorber

5.5.3 Experimental Result

Conventional soliton is generated in the fiber laser cavity. Figure 5-19 shows the optical spectrum of the mode-locked pulses, then central wavelength is located at 1971.3 nm. The 3-dB bandwidth of the spectrum is ~4.3 nm. The laser output pulse train is measured by an oscilloscope, and the result shows in Figure 5-20. The RF spectrum measured by a signal source analyzer is shown in Figure 5-21, the fundamental repetition rate is ~19.23 MHz. The mode-locking is in stable operation mode.

Figure 5-19 Output spectrum of the fiber laser

Figure 5-20 Output Pulse train measured by oscilloscope

Figure 5-21 RF spectrum of the optical pulses

5.5.4 Summary

In summary, we show that multilayer CVD graphene can exhibit saturation absorption at the long-wavelength fiber lasers. The Tm-doped fiber laser generates stable ultra-short pulses in the conventional soliton operation regime. Self-starting mode-locked lasing was observed, and the resultant pulse train with a fundamental repetition rate of ~ 19.237 MHz was achieved. The laser efficiency was $\sim 13\%$.

Chapter 6

Mode-Locked Fiber Laser Incorporating 3D Graphene Based SA

In this chapter, we investigated the ultrashort pulses generation using 3D graphene based SA. As the 3D graphene is a free standing material, formation of 3D graphene based SAs are direct and simple. Two types of 3D graphene based SAs are investigated in this chapter. Section 6.1 introduces fiber end facet type SA, it is fabricated by simply sandwiching the 3D graphene in between of two fiber ferrule connectors through a conventional adapter. For the first time, we demonstrate that 3D graphene can be used as a SA to mode-lock fiber lasers. Section 6.2 discusses evanescent-field interaction of 3D graphene achieved by a side-polished fiber. The SA is formed by penetrate the free standing 3D graphene by a side-polished fiber. By this evanescent-field interaction configuration, the optical power allowed in the laser cavity is higher. By using 3D graphene, we are able to control the characteristics of the SA device, and we are able to control the modulation depth and non-saturable absorption of the device. It enables the possibility to optimize the SA characteristic. The 3D structure of the material ease the burden of handling nano-scale materials. In contrast to graphene films, which often require to be

handled with a substrate as a support, this foam-like structures are easier to handle and can be produced uniformly in large scale. It enables the possibility to precisely control the nonlinear characteristics of the SA to match the applications for different laser performance improvement goals.

6.1 Fiber End face Integrated with 3D Graphene

6.1.1 Introduction

Graphene has been demonstrated as a strong candidate for SAs which offers many significant advantages including intrinsic broadband operation from ultraviolet to the far-infrared region [71], ultrafast recovery time, and high thermal damage threshold [21, 22, 43, 71, 72]. Graphene was conventionally fabricated by the mechanical exfoliation method in 2004 and since then various kinds of preparation methods are intensively explored [40, 53, 73]. Graphene based SAs have been successfully implemented in a variety of lasers including fiber lasers [29-31], solid-state lasers [32], waveguide laser [33], and semiconductor lasers [34]. Since Bao *et al.* and Sun *et al.* have demonstrated in [29, 30], mode-locked fiber lasers incorporating graphene as SAs have attracted considerable attention and have been actively explored. Recently, Popa *et al.* have demonstrated generation of 174 fs pulses from a graphene based erbium-doped fiber laser [36], and Fu *et al.* reported broadband ultrafast pulse generation from several different fiber lasers mode-locked by the same graphene based SA [74]. In order to fabricate a graphene based SA to be inserted into a laser cavity to interact with propagating light, several approaches have been demonstrated for the fabrication of the graphene based SA such as spray

coating method [41], optically-driven deposition method [100], direct mechanical exfoliation and imprinted graphite nano-particle method [42], CVD graphene film transferred by de-ionized (DI) water floating and dipping method [43]. However, it is difficult to control the number of graphene layers that needs to be transferred to the fiber connector end precisely when using the first three methods, and hence the performance of the prepared SAs is hard to estimate. Additionally, the first two methods require an extra procedure to prepare graphene based solution. The last method of CVD graphene film transferred to fiber connector end involves a process of floating and transferring graphene thin film in DI water and drying procedure. During the transfer process of the graphene layers to the fiber connector end facets, the mechanical exfoliation method and CVD graphene film method hold a possibility of damaging the graphene structure.

As the fabrication methods and applications of graphene are actively explored, integration of individual two-dimensional graphene sheets into a macroscopic structure becomes more and more essential for the applications of graphene [44]. 3D graphene is a foam-like structure that consists of multiple graphene layers integrated as a whole. In contrast to graphite, 3D graphene is obtained from bottom-up approach through CVD method. By carefully controlling the growth mechanism, the number of layers and crystallinity can be easily controlled and result in a highly flexible and transparent structure [44], which is ideal for photonics applications. A series of graphene based composites and microstructures have been fabricated using various methods such as chemically derived graphene sheets or direct synthesis of 3D graphene macrostructure by template-directed CVD. The latter method enables

the fabrication of an interconnected flexible network of graphene as the fast transport channel of charge carriers for high electrical conductivity [44, 111]. The fabrication advantage of 3D graphene relies in the free-standing structures obtained. In contrast to graphene films, which often require to be handled with a substrate as a support, this foam-like structures are easier to handle and can be produced uniformly in large scale. In this work, we report on a new type of SA for the passive mode-locking of fiber lasers with simple fabrication method. We demonstrate that 3D graphene can be used as a SA to mode-lock fiber lasers. A 3D graphene based SA is easy to fabricate without any additional deposition procedure because of its freestanding, flexible and elastic network structure. In addition, simple sandwiching of freestanding 3D graphene in between two fiber connectors to form a 3D graphene based SA successfully prevents the possible damage of the saturable absorption material during the SA fabrication process. In this paper, we obtained 3D graphene through a direct synthesis method, characterized the optical properties of 3D graphene and sandwiched the freestanding 3D graphene thin film between two optical fiber connectors to be used as a SA in a mode-locked fiber ring laser. For the first time, to the best of our knowledge, we demonstrate a 3D graphene SA to generate stable ultra-short soliton pulses in a mode-locked erbium-doped fiber laser. We believe that by varying and optimizing the number of graphene layers of the 3D graphene, the saturation absorption strength can be modulated over a wide range to obtain optical pulses with shorter pulse width.

6.1.2 Experimental Setup

A 1.55- μm ring cavity passively mode-locked fiber laser was further constructed to investigate the performance of the fabricated 3D graphene based SA as shown in Figure 6-1. The fabricated 3D graphene was cut to $\sim 2\text{ mm} \times 2\text{ mm}$ in size in preparation process. And the 3D graphene based SA was prepared by direct placed freestanding 3D graphene between two FC/APC fiber connector end facets through a conventional adapter. The prepared device was then used as a SA in the mode-locked erbium-doped fiber laser cavity. Our 3D graphene based SA shows a modulation depth of $\sim 3.2\%$, a saturation intensity of $\sim 50\text{ MW/cm}^2$, and a non-saturable loss of $\sim 43.7\%$, as discussed earlier in Chapter 4. It has been reported that the modulation depth of single-layer CVD graphene is $\sim 1\%$ with a non-saturable loss of $\sim 1.9\%$ [33-34]. Furthermore, the modulation depth of graphene based SAs can be increased by stacking multiple layers of graphene [7]. A 21-layer graphene based SA fabricated through CVD was reported to have a modulation depth of $\sim 2.93\%$, a saturation intensity of $\sim 53.25\text{ MW/cm}^2$, and a non-saturable loss of $\sim 53.05\%$ [35]. Our fabricated 3D graphene based SA shows comparable optical properties to that of multi-layer CVD graphene based SAs.

Fig. 3 shows the ring cavity passively mode-locked fiber laser setup incorporating the prepared 3D graphene based SA. The total length of the laser cavity was estimated to be $\sim 20.9\text{ m}$. A 0.6 m long erbium-doped fiber (nLight LIEKKI Er110-4/125) with group velocity dispersion parameter β_2 of $0.012\text{ ps}^2/\text{m}$ and peak core absorption of 110 dB/m at 1530 nm was used as the gain medium in the laser cavity. The other fibers were all standard single mode fibers with a length of ~ 20.3

m. A 980/1550 nm single mode wavelength-division multiplexer (WDM) coupler was used to launch a 980 nm pump light into the laser cavity, where an polarization-independent isolator (ISO) was applied to ensure the unidirectional light propagation in the laser cavity. A polarization controller (PC) was employed to adjust the cavity polarization and optimize the mode-locking condition in the cavity. 50% of the optical power at the emission wavelength was coupled out as the laser output from the laser cavity by a 1×2 optical coupler. The total cavity dispersion β_2 was managed in the anomalous region with -0.42 ps^2 for soliton shaping. The characteristics of the output pulses including the optical spectrum, the repetition rate and the pulse width were observed using an optical spectrum analyzer (Ando AQ6317B), a 2GHz photo-detector (Thorlabs DET01CFC) followed by a signal source analyzer (Rohde & Schwarz FSUP26), and an autocorrelator (Alnair Labs HAC-200), respectively.

Figure 6-1. Schematic of all-fiber ring laser mode-locked by 3D graphene based SA. LD: pump laser diode; WDM: wavelength-division multiplexer; EDF: erbium-doped fiber; ISO: isolator; PC: polarization controller; OC: output coupler; SA: 3D graphene based saturable absorber; SMF: single mode fiber.

6.1.3 Experimental Result and Discussion

In the experiment, when the pump power was increasing up to ~ 35.6 mW, continuous wave lasing was observed with an appropriate PC setting. The pump power threshold for single-soliton generation was found to be ~ 130.03 mW. Self-started mode-locking was also observed. To achieve a single soliton with a relatively high output power, the pump power was further increased to ~ 135.1 mW. The spectrum of the soliton pulses is shown in Figure 6-2(a), where the center wavelength is located at 1561.4 nm and the 3-dB spectral bandwidth is ~ 2.3 nm. The autocorrelation trace of the output pulse was measured to be ~ 1.01 ps with a sech^2 pulse shape fitting as shown in Figure 6-2(b), where the solid red line is the sech^2 pulse fitting and the black circles are the experimental data. Figure 6-2(c) shows the radio frequency (RF) spectrum of the output pulses for the fundamental repetition rate which corresponds to the laser cavity length, and the fundamental peak corresponds to the cavity repetition rate of ~ 9.9 MHz; the signal to noise ratio of the fundamental repetition rate is larger than 80 dB above the noise level, indicates that stable soliton pulses were being generated. The wideband RF spectrum to show the pulse train of ~ 9.9 MHz repetition rate of the mode-locked pulses is given in Figure 6-2(d). The laser average output power coupled out through a 50% coupler from the laser cavity was found to be ~ 10.5 mW while the pump power was set at ~ 135.1 mW. Therefore, the output pulse energy was estimated ~ 1 nJ for single pulse operation of the mode-locked laser. The output power of the constructed fiber laser is believed to be limited by the damage threshold of the 3D graphene based SA. One further improvement could be utilizing

evanescent-field interaction of the propagating light and the 3D graphene, which could be realized by using side-polished fibers [13] or tapered fibers [36] together with the 3D graphene to form the SA. The laser operation for soliton pulse generation was found to be stable. We also measured the stability of the laser output, negligible variation of the laser output parameters was observed over several hour periods of operation.

Figure 6-2. Characteristics of the output of the fiber laser showing (a) the optical spectrum, (b) the autocorrelation trace, (c) the fundamental RF spectrum, and (d) the wideband RF spectrum.

When the 3D graphene based SA is removed from the laser cavity, mode-locking operation was found to be impossible even with a further increase in the pump

power to ~200 mW. This observation confirms that the SA provides the required mechanism to initiate and sustain the passive mode-locking operation, where the 3D graphene is the key component.

6.1.4 Summary

In summary, we report that 3D graphene can exhibit saturation absorption. We have presented a simple, convenient method to form 3D graphene based SAs to generate high power and stable ultra-short pulses. Self-starting mode-locked lasing was observed, and the resultant pulse train with a fundamental repetition rate of ~9.9 MHz and a pulse width of ~1.01 ps was achieved. The average output power of the laser was found to be ~10.5 mW with corresponding output pulse energy of ~1 nJ for single pulse operation. The laser efficiency was ~16%. Such a freestanding 3D graphene offers a new solution to high quality SAs for fiber lasers with relatively simple and cost effective fabrication process.

6.2 Evanescent-field interaction with 3D Graphene using a side-polished fiber

As discussed in the previous section, 3D graphene shows nonlinear saturable absorption property and ultrashort pulses can be generated using a fiber end facet type SA. However, if the laser cavity operates at high power, the 3D graphene attached to the fiber end facet is vulnerable to thermal damage. Hence to prevent possible damage of the 3D graphene while the laser cavity processes high power,

we investigated evanescent-field interaction with the material using a side-polished fiber in this section.

6.2.1 Introduction

The common method to fabricate a nanocarbon material based SA is to sandwich a thin film in between of two fiber ferrule connectors. In this way, the fiber core region is fully covered by the nanocarbon material. As a result, the optical power direct interact with the deposited material. As it is shown in figure 6-3, light is not uniformly distributes in the optical fiber core region. It is typically reaches its' peak at the center of the core and gradually tails off at the interface between the core and the cladding. The distribution of the light extends into the cladding region as well. Hence, the central part of the fiber core region carries higher optical power direct interacts with the deposited material. In general, the thermal damage threshold of the nanocarbon material is relatively low in this type of configuration. The generated heat could damage the nanomaterial attached to the central of the fiber core [124]. Therefore, the mode-locked fiber laser average output power level is limited to a few mW levels and the peak power in a range of less than hundreds of Watt. In this section, we investigated evanescent-field interaction of the free-standing 3D graphene utilized the side-polished fiber to achieve mode-locking of a fiber laser.

Figure 6-3 Light distribution in an optical fiber

It has been reported that three-dimensional (3D) graphene which is a freestanding material, shows nonlinear saturable absorption property and ultrashort pulses can be generated using a fiber end facet type SA in the previous section. Such a freestanding 3D graphene offers a new solution to high quality SAs for fiber lasers with relatively simple and cost effective fabrication process. However, the fiber end facet type 3D graphene based SAs are vulnerable to thermal damage in a high-power laser cavity, which is same as the fiber end facet type CNT based SA [125]. One possible solution for the problem is employ evanescent field interaction of laser with the carbon nanostructures such as the freestanding 3D graphene. The interaction between the 3D graphene and evanescent field in a long-distance format can be achieved by simply penetrating a piece of freestanding 3D graphene by a side-polished fiber. We present for the first time, to the best of our knowledge, a soliton pulse generation in passively mode-locked fiber laser with a saturable absorber based on 3D graphene together with a side-polished fiber. Stable ultra-

short soliton pulses with full width at half maximum of 2.45 nm centered at 1564.97 nm was generated.

6.2.2 Experimental Setup

A 1.55- μm ring cavity passively mode-locked fiber laser was further constructed to investigate the performance of the fabricated 3D graphene based SA. The setup of the mode-locked fiber laser is depicted in Figure 6-4. The laser consists of a 0.6 m long Er-doped fiber (nLight LIEKKI Er110-4/125) with group velocity dispersion parameter β_2 of 0.012 ps²/m and peak core absorption of 110 dB/m at 1530 nm, it is pumped by a 980 nm laser diode via a 980/1550 nm wavelength division multiplexer (WDM). The net cavity dispersion was ~ -0.46 ps². The signal was coupled out using 50/50 output coupler (OC). A polarization-independent isolator (ISO) is applied to ensure the unidirectional light propagation in the laser cavity. Fiber-based polarization controller (PC) allows to adjust the intra-cavity polarization and optimize the mode-locked condition.

Figure 6-4 Schematic of all-fiber ring laser mode-locked by 3D graphene based SA. LD: pump laser diode; WDM: wavelength-division multiplexer; EDF: erbium-doped fiber; ISO: isolator; PC: polarization controller; OC: output coupler; SA: 3D graphene based saturable absorber.

The 3D graphene (few layers) freestanding macrostructure was obtained through a direct synthesis method using template-directed CVD as previous reported. The fabricated 3D graphene is in cuboid shape with the dimension of $1\text{cm} \times 1\text{cm} \times 3\text{cm}$. The SA is formed by simply penetrate the 3D graphene by the side-polished fiber as it is shown in Figure 6-5.

Figure 6-5 SA formed by 3D graphene and side-polished fiber

The characteristics of the output pulses including the optical spectrum, the repetition rate and the pulse width were observed using an optical spectrum analyzer (Ando AQ6317B), a 2GHz photo-detector (Thorlabs DET01CFC) followed by a signal source analyzer (Rohde & Schwarz FSUP26), an oscilloscope and an autocorrelator (Alnair Labs HAC-200), respectively.

6.2.3 Experimental Result

Self-started mode-locking was observed. Single pulse was generated while the input power increased to 36.7 mW. The spectrum of the soliton pulses measured by an optical spectrum analyzer is shown in Figure 6-6(a), where the center wavelength is located at 1564.97 nm and the 3-dB spectral bandwidth is ~2.45 nm. The laser output pulse train measured by a photo-detector followed by an oscilloscope shows in Figure 6-6(b). Figure 6-6(c) shows the RF spectrum of the output pulses, the fundamental peak corresponds to the cavity repetition rate of ~9.53 MHz; the signal to noise ratio of the fundamental repetition rate is larger than 60 dB above the noise level, indicates that stable soliton pulses were being generated. The autocorrelation trace of the output pulse was measured to be ~1 ps with a sech^2 pulse shape fitting as shown in Figure 6-6(d), where the solid red line is the sech^2 pulse fitting and the black circles are the experimental data.

Figure 6-6 Characteristics of the output of the fiber laser showing (a) the optical spectrum, (b) the pulse train, (c) the fundamental RF spectrum, and (d) the autocorrelation trace.

6.2.4 Summary

We present for the first time, to the best of our knowledge, a soliton pulse generation in passively mode-locked fiber laser with a saturable absorber based on three dimensional graphene together with a side-polished fiber. The ring laser resonator based on Er-doped fiber was capable of generating ultrashort optical pulses with full width at half maximum of 2.45 nm centered at 1564.97 nm. The pulses with duration of 1 ps were repeated with a frequency of 9.53 MHz. The laser efficiency was ~23%. Compare to other nano carbon materials, such as CNT and

graphene, the 3D graphene is a free-standing structure. The handling of 3D graphene, and the fabrication of 3D graphene based SAs are rather simple and straightforward. The results imply that the freestanding 3D graphene can be applied as a saturable absorption material. And in addition, we believe that by varying and optimizing the number of graphene layers of the 3D graphene free standing structure, the saturation absorption strength can be modulated and precisely controlled over a wide range to obtain optical pulses with shorter pulse width.

Chapter 7

Conclusions and Future Works

In this dissertation, fabrication methodologies of broadband graphene-based devices and their applications in ultrafast pulsed fiber lasers at selected wavelengths have been extensively investigated. This chapter summarises the accomplishments of the research and presents some suggestions for future work.

7.1 Conclusions

In summary, this thesis focuses on the investigation of graphene-based SAs in passively mode-locked fiber laser applications to achieve better performance of ultrafast fiber laser systems. This research comprises three major aspects: design and fabrication of graphene-based devices, investigation of the broadband absorption property of graphene-based SAs in mode-locked fiber lasers at selected wavelengths, and exploration of 3D graphene-based SA devices applied in mode-locked fiber lasers.

First, different methodologies of graphene-based SA fabrication were investigated to achieve better performance of mode-locked fiber laser applications. These methodologies include spray coating, optical-driven deposition, and mechanical exfoliation of graphite, transfer of CVD graphene and the direct transfer of 3D graphene. The main achievements

of the nanocarbon material-based SAs in ultrafast fiber laser applications are summarised as follows.

Q-switched mode-locking of an Yb-doped fiber laser using CVD graphene based SA is investigated. The laser operation is stable, the repetition rate of the Q-switched mode-locked pulse decreased from 4.1 KHz to 1.0 KHz when the pump power increased from 161 mW to 195 mW, and the laser output power was increased from $\sim 22.8 \mu\text{W}$ to $\sim 37.3 \mu\text{W}$.

A multilayer graphene-based SA fabricated by mechanical exfoliation is experimentally demonstrated that is capable of mode-locking an Er-doped fiber laser. The multilayer graphene is fabricated from commercially available graphite flakes. An adhesive tape is used to separate and reduce the number of graphene layers. The insertion loss induced by this multilayer graphene-based SA is $\sim 3.5 \text{ dB}$. By inserting the fabricated multilayer graphene SA in the laser cavity, the fiber laser can operate in two regimes. The laser operation can be switched between the mode-locking regime and the Q-switching regime with the same setup. The laser operates in the mode-locking regime while the pump power is at $\sim 34.4 \text{ mW}$ with a single pulse generated. The fundamental repetition rate is $\sim 17.7 \text{ MHz}$, and the 3-dB spectrum width is $\sim 2.1 \text{ nm}$ at the centre wavelength of $\sim 1561.5 \text{ nm}$. The pulse width is measured to be $\sim 1.2 \text{ ps}$. With further increments of the pump power, the laser shifts from the mode-locking regime to the Q-switching regime, during which the output pulse repetition rate increases from 34.2 KHz to 52.8 KHz as the pump power is increased from 93 mW to 295 mW.

Dissipative solitons are generated in a Yb-doped fiber laser using a CVD graphene-based SA without additional filtering. To achieve this, an additional bandpass filter is generally indispensable. However, by designing a laser cavity with large all-normal dispersion and using a CVD graphene-based SA, we can generate dissipative solitons without an additional physical bandpass filter. The CVD graphene-based SA is fabricated by sandwiching thin-film graphene in between two FC/APC fiber ferrule connector end facets via a conventional adapter. The average number of graphene film layers required is ~ 5 layers. The CVD graphene-based SA has a modulation depth of $\sim 2.3\%$. Self-started mode-locking is observed with a 3-dB spectral bandwidth of ~ 2.72 nm at the centre wavelength of ~ 1038.9 nm, which is twice that stated in a peer report. A sharp, steep spectral edge is observed that indicates the generation of dissipative solitons. The fundamental repetition rate is 1.23 MHz, which corresponds to a laser cavity length of 168 m. Numerical simulation of the dissipative evolution in the laser cavity can reproduce the experimental observations. The generated mode-locked pulses are highly chirped, which is a typical feature of dissipative solitons.

The broadband saturable absorption property of graphene-based SAs is experimentally demonstrated. The linear transmittance spectrum of the CVD graphene is measured by an UV-visible NIR spectrophotometer. The results are featureless over a wide spectral range from 500 nm to 2000 nm. By sandwiching a CVD graphene film between two fiber ferrule connectors, a CVD graphene-based SA is used to investigate the broadband saturable absorption property. In fact, ultrafast fiber lasers operating at broadband range are in high demand for different applications, such as spectroscopy and biomedical diagnosis. Here, we use a single SA to generate ultrafast pulses at three selected wavelengths. We constructed

three fiber laser cavities, which use Yb-, Er- and Tm-doped fiber as the gain mediums. The three lasers can operate at the central wavelengths of 1039, 1561 and 1971 nm, respectively. The constructed Yb-doped fiber laser operates in the anomalous dispersion region with dissipative soliton pulses generated, whereas the Er- and Tm-doped fiber lasers operate in the normal dispersion regime with conventional soliton pulses generated. The ultrafast pulses generated at all essential wavelengths between 1 μm to 2 μm provide evidence that the CVD graphene-based device possess an intrinsic broadband operation property.

For the first time, an all-fiber passively mode-locked fiber laser incorporating 3D graphene as a saturable absorber is presented. The 3D graphene is synthesised by a CVD method and is a foam-like structure that consists of multiple graphene layers integrated as a whole. The fabricated 3D graphene possesses a free-standing structure, it is easier to handle, and it can be produced uniformly at a large scale. The average number of layers of the 3D graphene ranges from 3 to 7 layers. The average optical transmittance between 500 to 2200 nm is $\sim 82\%$ as measured by spectrophotometry. A femtosecond pulsed laser was used to characterise the nonlinear absorption of the 3D graphene-based SA device. The device shows a modulation depth of $\sim 3.2\%$, a saturation intensity of $\sim 50 \text{ MW/cm}^2$, and non-saturable loss of $\sim 43.7\%$. The free-standing 3D graphene is cut into a proper size and directly sandwiched between two fiber ferrule connectors to form the SA device. A 1.55- μm ring cavity passively mode-locked fiber laser was further constructed to investigate the performance of the fabricated 3D graphene-based SA. The spectrum of the soliton pulses is generated at the centre wavelength of 1561.4 nm, and the 3-dB spectral bandwidth is $\sim 2.3 \text{ nm}$. The autocorrelation trace of the output pulse is measured to be $\sim 1.01 \text{ ps}$ with a sech^2 pulse shape fitting. The RF spectrum of the

output pulses for the fundamental repetition rate, which corresponds to the laser cavity length, is also measured, and the fundamental peak corresponds to the cavity repetition rate of ~ 9.9 MHz. The signal-to-noise ratio of the fundamental repetition rate is greater than 80 dB above the noise level, indicating that stable soliton pulses are being generated. The laser average output power coupled out through a 50% coupler from the laser cavity is ~ 10.5 mW when the pump power is set at ~ 135.1 mW. Therefore, the output pulse energy is estimated to be ~ 1 nJ for single-pulse operation of the mode-locked laser. The laser operation for soliton pulse generation is stable. The output power of the 3D graphene-based fiber laser is believed to be limited by the damage threshold of the 3D graphene-based SA. One further improvement could be to use evanescent-field interaction of the propagating light and the 3D graphene, which could be realised by using side-polished fibers or tapered fibers together with the 3D graphene to form the SA.

We further demonstrate an Er-doped fiber laser passively mode-locked by a 3D graphene SA functioning with evanescent field interaction realised by a side-polished fiber. Self-started mode-locking is also observed. A single pulse is generated while the input power is increased to 36.7 mW. The centre wavelength of the generated laser pulses is located at 1564.97 nm, and the 3-dB spectral bandwidth is ~ 2.45 nm. The autocorrelation trace of the output pulse is measured to be ~ 1 ps with a sech^2 pulse shape fitting. The RF spectrum of the output pulses for the fundamental repetition rate, which corresponds to the laser cavity length, and the fundamental peak corresponds to the cavity repetition rate of ~ 9.53 MHz. The

signal-to-noise ratio of the fundamental repetition rate is ~60 dB above the noise level, indicating that stable soliton pulses are being generated.

7.2 Future Works

Our study shows that by using different nanocarbon material-based SAs, the same laser cavity can work in two different states: mode-locking or Q-switching. Different modulation depths induced by different SAs might be responsible for such phenomena. Further study on how the modulation depth for nanocarbon material-based SAs will affect the lasing condition will be undertaken. Because of the broadband absorption property of graphene, our research will mainly focus on the analysis of graphene-based SA modulation depth.

From the current stage of research, we have confirmed that 3D graphene can be used as an SA at the communication wavelength. Further studies of the applications of 3D graphene-based pulsed lasers at different wavelengths for non-telecommunications applications will be continued. For example, the 3D graphene-based SA is believed to be able to obtain mode-locking in either an Yb- or Tm-doped fiber laser to generate output pulses at 1 μm and 2 μm range, respectively. Unlike the Er-doped fiber laser, which can work in an anomalous dispersion region in which classical solitons are easy to obtain, the Yb-doped fiber laser works in an anomalous cavity dispersion region, and mode-locking for dissipative soliton generation will require further study. Upon successful mode-locking of a Yb-doped fiber laser with a 3D graphene-based SA, a theory and simulation study of dissipative soliton pulse generation, which is likely to occur while the passively mode-locked fiber laser is working in all-normal-dispersion Yb-doped ring fiber laser mode,

needs to be performed. Our aim is to develop a high-energy, high-speed, and low-noise ultrafast fiber laser that is more attractive for real-life applications. By optimising the laser cavity configuration and nanocarbon material-based SAs, we aim to achieve a laser system that works at a higher pulse energy and higher output power by evanescent field interaction of nanocarbon nanomaterial with optical power within the cavity.

The applications of the mode-locked fiber laser incorporating a nanomaterial-based SA device can also be further explored. Flat supercontinuum generation using a 3D graphene-based mode-locked Yb-doped fiber laser operating at 1 μm or a Tm-doped fiber laser operating at 2 μm as a pump light source can also be studied in the future.

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5. Y. Yang, K. K. Chow, "Graphene-based all-normal-dispersion ytterbium-doped fiber laser for 1- μ m dissipative soliton generation," IEEE Photonics Journal, to be submitted.
6. Y. Yang, K. K. Chow, "Generation of Q-switched mode-locked pulses in Yb-doped Fiber Laser by graphene based saturable absorber," to be submitted.

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7. Y. Yang, H. H. Liu, K. K. Chow, "All-normal-dispersion dissipative soliton ytterbium-doped fiber laser with multilayer graphene saturable absorber," *IEEE Photonics Conference (IPC)*, pp. 234-235, 4-8 Oct. Reston, USA, 2015.
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