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## GRAPHENE OXIDE-POLYETHYLENE OXIDE (PEO) FILM AS SATURABLE ABSORBER ON MODE-LOCKED ERBIUM DOPED FIBER LASER GENERATION

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Graphical abstract



## Abstract

Mode-locked erbium doped fiber laser by using graphene oxide (GO) that obtained by oxidation and ultra-sonification process of natural graphite was reported. GO produced was dissolved in water and mixed with Polyethylene Oxide (PEO) to form film. When the fabricated GO-PEO film saturable was employed in the proposed EDFL configuration, the laser generates 1.25 ps pulses at a repetition rate of 21.8 MHz with pump input of 70 mW. The stable mode-locking operation was observed within pump input power of 70 mW until 175 mW. Soliton-like spectrum was achieved with prominent Kelly-sideband and centre wavelength of 1558.6 nm.

Keywords: Graphene oxide, mode-locked, passive saturable absorber, erbium-doped fiber

## Abstrak

Mod terkunci daripada laser gentian didopkan erbium telah dilaporkan dengan menggunakan grafin oksida (GO) yang dihasilkan melalui proses pengoksidaan dan ultrasonifikasi serbuk grafit semulajadi. GO yang dihasilkan tersebut dilarutkan kedalam air dan dicampurkan dengan polietilen oksida (PEO) untuk membentuk lapisan filem. Seterusnya, lapisan filem penyerap boleh tepu GO-PEO yang dihasilkan tadi telah digunakan di dalam konfigurasi EDFL dan pembentukan denyutan laser yang mempunyai ciri-ciri seperti kadar denyutan sebanyak 1.25 ps dan kadar pengulangan sebanyak 21.8 MHz terhasil daripada kemasukan kuasa pam 70 mW. Didapati, mod terkunci yang terhasil adalah paling stabil pada kuasa pam diantara 70 mW hingga 175 mW. Spektrum seperti-soliton telah dicapai dengan keadaan Kelly-sideband yang menonjol dan dengan panjang gelombang tengah pada 1558.6 nm.

Kata kunci: Grafin oksida, Mod terkunci, penyerap boleh tepu pasif, laser gentian didopkan erbium

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## **1.0 INTRODUCTION**

To date, passively mode-locked fiber lasers offer compactness, low cost, stability as well as many other advantages in micromachining, medicine, applications in metrology and so on [1-2]. In order to generate passively mode-locked fiber laser, a variety type of saturable absorber (SA) have been studied and used [3-6]. Recently, graphene has been favourable saturable absorbers (SAs) to achieve mode-locking in fiber lasers. It offers characteristics such as ultrafast recovery time and capable to

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## Full Paper

achieve broadband operation with its intrinsic property due to the linear dispersion of Dirac electrons property [7-9].

There are a few reports on techniques to produce graphene-based SA (GSA) and are able to achieve mode-locking. For example, chemical vapor deposition (CVD) method, liquid phase exfoliation (LPE) method and Hummers method that produce graphene-oxide (GO) to be used as GSA [10-12]. Of late, GO-based passively mode-locked fiber lasers have been demonstrated in rare-earth doped fiber lasers {[9], [13-15], and GO based SA is interesting as it can be considered as an insulating and disordered analogue of a highly conducting crystalline graphene [11]. In addition, GO can be dissolved in water for its carboxyl and hydroxyl groups, thus allowing it to be mixed with polymer composite very well in the aqueous solution [16]. Hence, thin film form is easy to achieve.

In this report, we demonstrate a mode-locked EDFL in ring cavity, employing GO-PEO as SA. A stable mode-locking operation started after supplying pump power of 70 mW and the operation remained until pump power supplied over 135 mW. At 70 mW the proposed laser generates a soliton-like spectrum with 3 dB band width of 2.2 nm and centre wavelength of 1558.2 nm. The laser generates pulse-train with repetition rates of 21.8 MHz, pulse width of 1.25 ps and total pulse energy of 0.015 nJ.

## 2.0 EXPERIMENTAL

#### 2.1 GO-PEO FIlma Saturable Absorber Fabrication

Graphene oxide (GO) was first produced by mixing sulfuric acid: Phosphoric acid (H<sub>2</sub>SO<sub>4</sub>:H<sub>3</sub>PO<sub>4</sub>) in ratio of 320 mL:80 mL, graphite flakes, and Potassium permanganate (KMnO<sub>4</sub>), 18 g, by using a magnetic stirrer. After adding all the materials slowly, the onepot mixture was left for stirring for 3 days to allow the oxidation of graphite. The color of the mixture changes from dark purplish green to dark brown. Later, H<sub>2</sub>O<sub>2</sub> solution was added to stop the oxidation process, and the color of the mixture changes to bright yellow, indicating a high oxidation level of graphite. The graphite oxide formed was washed three times with 1 M of HCl aqueous solution and repeatedly with deionized water until a pH of 4-5 was achieved. The washing process was carried out using simple decantation of supernatant via a centrifugation technique with a centrifugation force of 10,000 g. During the washing process with deionized (DI) water, the graphite oxide experienced exfoliation, which resulted in the thickening of the graphene solution, forming a GO gel. The GO gel was then mixed with DI water to obtain a graphene oxide solution.



Figure 1 Fabrication of GO-PEO composite solution; (a) Mixing of GO solution with the PEO host polymer; (b) Ultrasonic bath of the mixed suspension GO with host polymer

To prepare the polymer, 1 g PEO (average molecular weight of  $1 \times 10^6$  g/mol) is dissolve in 120 ml DI water using hot plate stirrer with the aid of magnetic stirrer. From the experiment, it will take around three hours to fully dissolve the PEO in DI water. GO-PEO composite was fabricated by adding a different quantity of dispersed GO suspension containing GO into a solution of 1 g PEO in deionized water and thoroughly mixed them using an ultrasonification process. In this experiment, the mixer is put into ultra-sonic bath (Branson 2510, 40 kHz) for about one hour to produce a stable GO-PEO composite solution (Figure 1). In this work, only small amount of the PEO solution (0.6 ml - 2 ml) are used to minimize the use of GO. This mixture of PEO and GO is then left dry at room temperature to obtain GO-PEO film as seen in Figure 2 (a). Figure 2 (b) is the Field Emission Scanning Electron Microscope (FESEM) image of the GO in 500 nm dimension. It shows a well-defined and interconnects graphene sheets forming a porous and loose sponge like structure. Figure 2a shows the FESEM image of graphite flakes with an average lateral size of 1 mm.



Figure 2 (a) GO-PEO film after let dry at room temperature; (b) FESEM image of GO

Figure 3 shows the Raman spectra of GO which clearly discerned that the D peak of GO located at 1359 cm<sup>-1</sup> and the G peak located at around 1600 cm<sup>-1</sup>. The D band is due to defect-induced breathing mode of sp<sup>2</sup> rings and the G band is due to the first order scattering of the  $E_{2g}$  phonon of sp<sup>2</sup> carbon atoms [17]. As observed the G band of the GO is located at a higher frequency compare to graphite 1580 cm<sup>-1</sup> and corresponds to the finding reported [18]. The  $(I_D/I_G)$  intensity ratio for GO is 0.85 which is the measure of disorder degree and is inversely proportional to the average size of the sp<sup>2</sup> clusters [19].



Figure 3 Raman spectrum of the GO

The saturable absorber to be inserted inside the laser cavity was fabricated by cutting a small part of the GO-PEO thin-film around 2×2 mm<sup>2</sup>. Then, the cut thin film was transfreed on to the end face of an optical fiber ferrule that was pigtailed at its other end and sandwiched by an additional fiber ferrule with deposition of index-matching gel on both fiber ferrules. The insertion loss of the GOSA was measured to be around 3 dB at 1550 nm.

#### 2.2 Experimental Setup Configuration

The configuration of the implied mode-locked EDFL as shown in Figure 4 consists of a 1 m long Erbium doped fiber (EDF) as the gain medium, a wavelength division multiplexer (WDM), a ThorLab polarization controller (PC), an isolator, a 95:5 coupler and a 10 m long single mode fiber (SMF) in a ring configuration. The fabricated GO-PEO film SA was inserted in between the gain medium and the SMF. The EDF was pumped by the 1480 nm laser diode (LD) via the 1480/1550 WDM. Incorporation of an isolator in the cavity is crucial in order to ensure unidirectional of the oscillating light. PC was integrated to adjust light polarization inside the laser configuration. We employed a 10 m long SMF to add cavity loss in this configuration. 5 % of the output of this laser was tapped out from the configuration via the 95:5 coupler while keeping 95 % of the laser propagating inside the ring cavity. The optical spectrum analyser (OSA) is used for the spectral analysis of the modelocked EDFL with a spectral resolution of 0.02 nm, while the oscilloscope is used to observe the output pulse train of the mode-locked operation in the form of electrical signal via a 6 GHz bandwidth photodetector. The pulse width is measured using Alnair intensity based autocorrelator and also characterized via a radio frequency (RF) spectrum analyser.



Figure 4 Experimental setup proposal of the mode-locked EDFL

## **3.0 RESULTS AND DISCUSSION**

The EDFL was operating in continuous wave (CW) mode when we operate the laser without GO-PEO film SA. After incorporation of the SA, unstable modelocked train was observed. In order to stabilize the mode-locked operation, the SMF was employed. In this work, the stable mode-locking regime is achieved at the pump power of 70 mW. Figure 5 (a) shows the soliton-like output spectrum of the EDFL with prominent Kelly side-bands at the pump power of 70 mW. It has centre wavelength of 1558.6 nm with approximately 2.2 nm of 3 dB bandwidth with the peak power of -27 dBm. Figure 5 (b) displays the pulse train of the mode-locked EDFL at the pump power of 70 mW. The repetition rate achieved at this pump power was 21.8 MHz with peak-to-peak separation  $(t_{ptp})$  of 244.2 ns. The pulse width (T) of the mode-locked operation was obtained from the trace of autocorrelator as shown in Figure 5 (c). We can observe that our experimental data follows the sech<sup>2</sup> fitting almost perfectly from the autocorrelation trace. T achieved was 1.25 ps and the calculated time-bandwidth product (TBP) is 0.34. This value is a bit far of the theoretical value sech<sup>2</sup> profile of modelocked pulses TBP of 0.315 signifying that the pulses produced was chirped at the same pump power of 70 mW. Measurement of the average output power at this pump power is done via the optical power meter. Average output power of 0.363 mW was measured translated to generated peak power of 11.3 W and pulse energy of 0.015 nJ.



**Figure 5** Output traces of: (a) OSA, (b) oscilloscope with frequency 21.8 MHz, and (c) autocorrelator; at the fix input pump power of 70 mW

Stable mode-locked pulses can be obtained from the pump power of 70 mW until 175 mW. The modelocking operation of the proposed EDFL was diminished after supplying pump power of more than 175 mW. Figure 6 below displays the average output power of the mode-locked EDFL. The figure shows the output power has an almost linear relationship to the input power and the laser efficiency is calculated to be 0.4 %.



Figure 6 Output power as a function of input power of the  $\mathsf{EDFL}$ 

## 4.0 CONCLUSION

Unwavering passively mode-locked EDFL operated at 1558.6 µm was reported by employing graphene oxide/PEO film as the saturable absorber. The proposed mode-locked EDFL generates a soliton-like spectrum at the pump power of 70 mW with 3 dB bandwidth of 2.2 nm. The repetition rate achieved at

this pump power was 21.8 MHz accompanied by pulse width of 1.25 ps. TBP value calculated was 0.34 signifying that the pulses produced was slightly chirped. Measurement of the average output power at this pump power is done via the optical power meter. Average output power of 0.363 mW was calculated translated to generate pulse energy of 0.015 nJ.

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