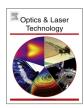
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#### Full length article

# Femtosecond mode-locked erbium-doped fiber laser based on MoS<sub>2</sub>–PVA saturable absorber



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#### ARTICLE INFO

Article history: Received 16 November 2015 Accepted 7 March 2016 Available online 16 March 2016

Keywords: Fiber laser MoS<sub>2</sub> Polymer composite Mode-locked laser

#### ABSTRACT

We fabricate a free-standing few-layer molybdenum disulfide (MoS<sub>2</sub>)-polymer composite by liquid phase exfoliation of chemically pristine MoS<sub>2</sub> crystals and use this to demonstrate a soliton mode-locked Erbium-doped fiber laser (EDFL). A stable self-started mode-locked soliton pulse is generated by fine-tuning the rotation of the polarization controller at a low threshold pump power of 25 mW. Its solitonic behavior is verified by the presence of Kelly sidebands in the output spectrum. The central wavelength, pulse width, and repetition rate of the laser are 1573.7 nm, 630 fs, and 27.1 MHz, respectively. The maximum pulse energy is 0.141 nJ with peak power of 210 W at pump power of 170 mW. This result contributes to the growing body of work studying the nonlinear optical properties of transition metal dichalcogenides that present new opportunities for ultrafast photonic applications.

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#### 1. Introduction

Ultrafast fiber lasers have potential applications in many fields such as industry, medicine, remote sensing and optical communication [1,2]. Therefore, we observe a tremendous increase in research interest on developing new saturable absorbers (SAs) for passive mode locking in recent years because of their advantages of excellent mechanical stability, easy implementation and low cost. To date, various type of SAs have been intensively investigated and implemented to realize passive mode-locked pulses, such as nonlinear-optical loop mirror (NOLM) [3], nonlinear polarization rotation (NPR) [4], semiconductor saturable absorption mirrors (SESAMs) [5] and single wall carbon nanotubes (SWCNTs) saturable absorbers (SAs) [6]. In the NOLM approach, a long fiber must be used to produce sufficient nonlinear phase shifts. The NPR technique utilizes dispersion and nonlinearity management to generate laser. However, it is often sensitive to ambient factors such as vibration and temperature, which limits its practical applications. SESAMs require complex design to improve their damage threshold and work only in a narrow wavelength range. A simpler and cost-effective alternative relies on SWCNTs. However, SWCNTs have a low damage threshold, and tend to bundled entangled morphology.

Most recently, graphene have been extensively used for

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generating passive O-switching and mode locking applications [7,8]. Compared to the previous SAs, graphene has the advantages of ultra-fast recovery time and broadband saturable absorption. But the absence of band-gap and the low absorption co-efficiency (2.3%/layer) of graphene have also restraint its applications. These limitations lead to the intensive research on other two-dimensional (2D) materials which can complement the graphene. The success of graphene as inspired the exploration of other graphenelike 2D materials such as topological insulators (Bi2Te<sub>3</sub>, Bi<sub>2</sub>Se<sub>3</sub> etc.) for laser photonics applications. Zhao et al. reported ultrashort pulse generation in mode locked fiber lasers using SAs based on Bi<sub>2</sub>Te<sub>3</sub> and Bi<sub>2</sub>Se<sub>3</sub> [9,10]. Also, black phosphorous (BP) has recently joined in the family of 2D materials. The multi-layer BP exhibit significant saturable absorption under femtosecond excitation at both 400 nm and 800 nm band and picosecond pulse at 1562 nm and 1930 nm band [11]. Currently, transition-metal dichalcogenides (TMDs), molybdenum disulfide (MoS2) has also attracted much attention of fiber laser research as a SA for ultrafast laser applications, due to their thickness dependent band-gap and optical properties [12,13]. Also, it was reported that MoS2 nanosheets in dispersions have stronger saturable absorption response than graphene dispersions [13].

To data there are only few reported works on using few layer of  $MoS_2$  as SA [12–18]. For instance, Du, Juan, et al. demonstrated dissipative solitons by few-layer  $MoS_2$ -SA functioned with evanescent field Interaction in ytterbium-doped fiber laser (YDFL) at 1042.6 nm with pulse duration of 656 ps [15]. Liu et al. reported the generation of  $\sim$ 710 fs pulse with repetition rate of 12.09 MHz centered at 1569.5 nm wavelength by  $MoS_2$ -based SA fabricated by

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solution processed using intercalation [16]. Xia et al. proposed multilayer MoS<sub>2</sub> prepared by chemical vapor deposition (CVD), which resultant output soliton pulses have central wavelength, pulse width, and repetition rate of 1568.9 nm, 1.28 ps, and 8.288 MHz, respectively [17]. Also, Zhang et al. used MoS<sub>2</sub> for tunable wavelength from 1535 to 1565 nm with pulse width of 960 fs [18].

The bulk and few-layer MoS<sub>2</sub> possess discernible properties. Thus, it is imperative to exfoliate the few-layer material from the bulk material. Several approaches including micromechanical cleavage, chemical vapor deposition (CVD) and solution based methods are available to exfoliate the MoS<sub>2</sub> layers from the bulk materials. Among these methods, liquid phase exfoliation (LPE) method is considered to be the most efficient and cost effective approach since it does not require any post chemical treatments of the prepared solvents and permits large scale production. Luo et al. demonstrated the broadband absorption properties of MoS<sub>2</sub> from LPE fabrication technique which obtained the Q-switching pulse at 1, 1.5 and 2 µm regions [19]. In this paper, we fabricate few-layer molybdenum disulfide (MoS<sub>2</sub>)-polymer composite by LPE of chemically pristine MoS<sub>2</sub> crystals and use this to demonstrate a femtosecond mode locked Erbium-doped fiber laser (EDFL). The free-standing SA film was integrated in the EDFL ring cavity by sandwiching it between two fiber ferrules via a connector to achieve soliton mode-locking pulses with pulse width of 630 fs, repetition rate of 27.1 MHz, pulse energy of 0.141 nJ and peak power of 210 W. The achievement of femtosecond pulses confirmed that the MoS<sub>2</sub> could be indeed a good candidate for an ultrafast saturable absorption device.

#### 2. Preparation and characteristics of MoS<sub>2</sub>

The liquid phase exfoliation (LPE) process for MoS<sub>2</sub> exfoliation consists of 2 steps. First, bulk MoS2 crystals are mixed with a solvent that has a similar surface energy with MoS<sub>2</sub> ( $\sim$ 75 mJ m<sup>-2</sup>) [20]. In this work, we thus use Dimethylformamide (DMF) solvent with an initial concentration of 5 mg/ml. The second step is to apply ultrasound, which exfoliates the material by creating local pressure variations sufficient to overcome the weak van der Waals forces between the atomic layers of the bulk crystal [21,22], producing a dispersion enriched in few-layer flakes [22]. We prepare the MoS2 dispersion by ultra-sonicating the solution for 24 h. The resultant dispersion is centrifuged for 1 h at 3000 rpm in a swinging bucket rotor. This acts to sediment the un-exfoliated material as larger flakes descend more rapidly through the centrifuge cell than the exfoliated few-layer flakes [23]. The upper 80% of the dispersion, which now primarily contains few-layer flakes, is decanted for composite fabrication.

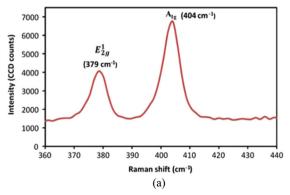
The free-standing MoS<sub>2</sub>-polymer composite film is prepared by

mixing 10 ml of the few-layer  $MoS_2$  dispersion with 20 ml of aqueous polyvinyl alcohol (PVA) solution with concentration of 10 mg/ml. The solution was obtained by dissolving 200 mg of PVA in 20 ml of deionized water. The  $MoS_2$ -PVA solution mixture, which is now 30 ml, is then stirred using a magnetic stirrer and heated continuously at a temperature of 80 °C till the solution is reduced to approximately 10 ml. This process takes approximately 7 h to complete. The reduced  $MoS_2$ -PVA mixture is then poured into a glass substrate and dried in an oven at  $\sim$ 80 °C for another 4 h to form a  $\sim$ 40 µm thick free-standing composite film.

We performed Raman spectroscopy on the  $1 \times 1 \text{ mm}^2$  piece of fabricated MoS<sub>2</sub>-PVA film and retain used for following experimental setup. So, multiple measurement for Raman spectroscopy is not necessary for different position on the small area of  $1 \times 1 \text{ mm}^2$  film. Fig. 1 shows representative Raman spectra for polymer composite film and mechanical exfoliated MoS<sub>2</sub>. Fig. 1 (a) shows the Raman spectrum, which is recorded by a spectrometer when a 514 nm beam of an Argon ion laser is radiated on the film for 10 seconds with an exposure power of 50 mW. As shown in the figure, the sample exhibits two characteristic peaks. in parallel with two phonon modes; out-of-plane vibration of Sulfide atoms at 404 cm<sup>-1</sup>, and in-plane vibration of Molybdenum and Sulfide atoms at 379 cm<sup>-1</sup>, with a frequency difference of 25 cm $^{-1}$ . It is observed that the  $A_{1g}$  mode due to the out-of-plane motion blue shifts after complete exfoliation, indicating that the few layer MoS<sub>2</sub> with thicknesses in the range of  $2\sim3$  layers had been successfully fabricated [24,25]. The absence of a noticeable shift in the peak positions from those measured for the exfoliated MoS<sub>2</sub> indicates that the material structure is unaffected by its inclusion in the composite as shown in Fig. 1(b).

#### 3. Experimental setup

We use this free-standing MoS2-PVA composite to build and test an ultrafast laser. We develop a soliton mode-locked EDFL consisting of all-fiber integrated components for an alignmentfree and compact system, as shown in Fig. 2. The MoS<sub>2</sub>-PVA SA is integrated into the fiber laser cavity by sandwiching a  $\sim 1 \times 1 \text{ mm}^2$ piece of the fabricated film between two fiber connectors, adhered with index matching gel. The mode-locked EDFL has total cavity length of 7.3 m which operates in anomalous fiber dispersion of -0.094 ps<sup>2</sup>. The cavity length consists of a 1.8 m long Erbium-doped fiber (EDF), 5 m long standard single mode fiber (SMF-28), and a segment of 0.5 m HI 1060 wavelength division multiplexer (WDM) fiber with group velocity dispersion (GVD) of 27.6, -21.7, and -48.5 ps<sup>2</sup>/km, respectively. The EDF has core and cladding diameters of 4 µm and 125 µm respectively, a numerical aperture of 0.16 and Erbium ion absorption of 23 dB/m at 980 nm. It is pumped by a 980 nm laser diode (LD) via a 980/1550 nm WDM. An



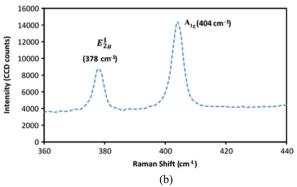


Fig. 1. Raman spectrum of (a) MoS<sub>2</sub>-polymer composite film and (b) mechanical exfoliated MoS<sub>2</sub>.

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