Letter

Nonlinear absorption properties of indium selenide and its application for demonstrating pulsed Er-doped fiber laser

Wenqing Yang¹, Nannan Xu¹,4 and Huanian Zhang¹,2,3

¹ Shandong Provincial Key Laboratory of Optics and Photonics Devices, School of Physics and Electronics, Shandong Normal University, Jinan 250014, People’s Republic of China
² Institute of Data Science and Technology, Shandong Normal University, Jinan 250014, People’s Republic of China
³ Shandong Province Key Laboratory of Medical Physics and Image Processing Technology, School of Physics and Electronics, Shandong Normal University, Jinan 250014, People’s Republic of China
⁴ Jingjiang Photoelectric Technology Co., Ltd, Jinan 250014, People’s Republic of China

E-mail: huanian_zhang@163.com

Received 21 May 2018, revised 26 June 2018
Accepted for publication 19 July 2018
Published 21 August 2018

Abstract
Two-dimensional materials are now receiving continuously increasing attention due to their exceptional optical properties. Recently, it has been proved that indium selenide (InSe) is also an excellent photoelectric material, with wide applications in the field of photodetectors and so on. In our work, layered InSe was prepared through the liquid-phase exfoliation method, and the nonlinear saturable absorption properties of InSe were also experimentally investigated. In addition, InSe-polyvinyl alcohol film was successfully prepared and employed as an saturable absorber for demonstrating an Er-doped passive \( Q \)-switched mode-locked fiber laser for the first time. The largest \( Q \)-switched pulse energy was as high as 112.97 nJ and the narrowest mode-locked pulse duration was 2.96 ns at a repetition rate of 1.74 MHz. Our results fully indicate that InSe also has excellent nonlinear absorption properties in comparison with other commonly used 2D materials. Thus, the applications of InSe can be deeply investigated to promote the development of ultra-fast optics in the near future.

Keywords: indium selenide (InSe), passively \( Q \)-switched mode-locked fiber laser, Er-doped fiber laser

(Some figures may appear in colour only in the online journal)

1. Introduction

Pulsed fiber lasers have been attracting intensive attention due to their wide application potential in the fields of medicine, micro-machining, metrology, fiber optical sensing and telecommunications, etc [1–15]. Compared to actively \( Q \)-switched mode-locked fiber lasers, those of the passively \( Q \)-switched mode-locked variety possess attractive advantages such as low cost, compactness, simplicity and the flexibility of their design [1–10]. In the past decade, various kinds of saturable absorbers (SAs), such as semiconductor saturable absorber mirrors (SESAMs) [11–13], graphene [14–19], topological insulators (TIs) [20–23], transition metal dichalcogenides (TMDs) [24–29], black phosphorus (BPs) [30–31] and so on have already been employed for demonstrating pulsed fiber lasers. As is known, commercial
SESAMs have tunable modulation depth and central absorption wavelengths [11, 12]; however, the disadvantages of high cost, the complex fabrication process and narrow absorption bandwidth put limits on the extent of its application potential for achieving broadband tunable pulse operations. Graphene exhibits outstanding saturable absorption properties including ultra-fast recovery time, a wide absorption range and high damage threshold [14–16]. Nevertheless, graphene also has disadvantages such as its zero band gap and weak absorption co-efficiency, which significantly limit its potential application in optics-related fields, which require strong light–matter interaction. In addition, graphene-like 2D materials including TIs, TMDs and BPs have also been widely reported for demonstrating pulsed fiber lasers operating within the 1, 1.5 and 2 µm region [20–31]. These layer-structured 2D materials have suitable band-gap values, an ultra-fast recovery time, a wide absorption range and high damage threshold, and so on [14–16, 20–21, 26–28]. In brief, 2D materials have significantly promoted the rapid development of pulsed fiber lasers. Meanwhile, the acquisition of new types of saturable absorption material is still urgent.

Recently, indium selenide (InSe) with a few layers of atom thickness has been widely reported due to its excellent optoelectronics properties [32–36]. InSe belongs to the family of layered metal chalcogenide semiconductors. Each of its layers has a honeycomb lattice that effectively consists of four covalently bonded Se–In–In–Se atomic planes [33–35]. The layers are held together by van der Waals interactions at an interlayer distance of ≈ 0.8 nm [34–36]. Studies have proved that the band gap of InSe greatly varies with a decreasing number of layers, which is in agreement with the density functional theory [34–36]. As reported, few-layered InSe has an indirect band gap of 1.4 eV. Meanwhile, the bandgap value is 1.2 eV for bulk InSe, which is due to the suppression of interlayer coupling as its thickness approaches a few atomic layers [36–40]. Additionally, Bandurin et al. [32] employed exfoliation and the subsequent encapsulation of few-layer InSe in an inert (argon) atmosphere, which allowed them to fabricate InSe structures and field effect devices down to a monolayer, proving that InSe can exhibit previously unattainable quality and stability under ambient conditions. In 2017, based on the pulsed-laser deposition method, Yang et al. designed wafer-scale InSe nanosheets and demonstrated InSe-based broadband phototransistors within the region from ultraviolet to near-infrared, and their findings suggest that pulsed-laser-deposition-grown InSe would be a promising choice for future device applications [36]. Lei et al. studied the evolution of the electronic band structure and efficient photo-detection in atomic layers of InSe, and a strong photoresponse of 34.7 mA W−1 as well as a fast response time of 488 µs for few-layered InSe were recorded, with their results suggesting that InSe is a good material for thin film optoelectronic applications [37]. However, the nonlinear absorption properties of InSe have not been investigated.

In this paper, we demonstrated InSe-based passively Q-switched mode-locked fiber lasers for the first time. The nonlinear absorption properties of the InSe film were investigated experimentally. Stable Q-switched pulses were obtained with the repetition rate varying from 5.56 to 13 kHz, the minimum pulse duration was 8.3 µs and the maximum pulse energy was 112.97 nJ. Mode-locked generation with a minimum pulse width of 2.96 ns was also obtained. Our experiment results fully prove that InSe can be a potentially excellent SA for obtaining pulsed fiber lasers.

2. Preparation and characteristics of InSe-based SA

Figure 1 shows the preparation progress of the film-type InSe-polyvinyl alcohol (PVA) SA. As shown, liquid-phase exfoliation and spin coating methods were employed to prepare the InSe solution and the film-type InSe-PVA SA, respectively. Firstly, 0.1 g InSe powder was added to 40 ml 30% alcohol for the preparation of the InSe solution. The InSe solution was placed in a high-power ultrasonic cleaner for 6 h and centrifuged for 20 min; thus, a solution with a few-layered InSe nanosheet is obtained. Then, the InSe solution and 4 wt% PVA solution were mixed at a volume ratio of 3:4 and placed in an ultrasonic cleaner for 3 h to prepare the uniform InSe-PVA solution. A 100 µl InSe-PVA solution was spin-coated onto a sapphire substrate for the formation of the InSe-PVA film. Finally, a 1 × 1 mm² film was cut off from the substrate and placed at the end of the fiber as a proposed mode-locker.

The layered structure and morphology of the used InSe powder were characterized by transmission electron microscopy (SEM) (Sigma 500). The SEM image of the tested InSe is shown in figure 2(a), and the InSe powder reveals a typical layered structure. This structure suggests that single or few-layer InSe nanosheets can be expected after the progress of liquid-phase exfoliation. A TEM image (recorded by a JEM-2100) of the InSe solution produced after centrifugation is provided in figure 2(b), and it is obvious here that layered InSe
nanosheets were successfully obtained in the experiment. The thickness characteristics of the prepared InSe nanosheets were tested with an atomic force microscope (Bruker Multimode 8), as shown in figures 2(c) and (d). The thicknesses of the samples marked 1–4 are all about 4 nm, meanwhile, those of the samples 5 and 6 are about 3.1 nm, which indicates that the layers of the InSe nanosheets number about 3–4.

The structure characterizations of the InSe nanosheets were also investigated by using a Raman spectrometer (Horiba HR Evolution) and x-ray diffraction (XRD) (D8 Advance Bruker). As shown in figure 3(a), obvious Raman shifts at 115.1, 175.8 and 224.4 cm⁻¹ were recorded, in agreement with previous works [36–37]. The powder diffraction XRD spectrum from the InSe is shown in figure 3(b), and as shown, the peaks, which correspond to the (002), (004), (101), (006) and (008) planes in InSe, are recorded [36], respectively. In particular, the high diffraction peak located at the (004) plane indicates that InSe nanosheets with a well-layered structure and high crystallinity were successfully prepared.

Based on a commonly used balanced twin-detector method, which has been reported in our previous works [28–29], the nonlinear optical saturable absorption response
of the InSe-PVA film was investigated. The pump source was a home-made nonlinear polarization rotation mode-locked Er-doped fiber laser with 560 fs pulses at 1580 nm with a repetition rate of 33.6 MHz, and the maximum average output power was 5.57 mW after one-stage amplification. The recorded experimental results are shown in figure 4. Additionally, based on the following conventional formula [27–29]:

\[ T(I) = 1 - T_{ns} - \Delta T \times \exp(-I/I_{sat}) \]

where \( T \) is transmission, \( T_{ns} \) is nonsaturable absorbance, \( \Delta T \) is the modulation depth, \( I \) is the input intensity of the laser and \( I_{sat} \) is the saturation intensity. The saturation intensity and modulation depth of the InSe-PVA film, which were obtained by fitting the experimental results, are 60.1 MW cm\(^{-2} \) and 3.4%, respectively.

3. Experimental details

The experimental setup of the pulsed fiber laser is schematically shown in figure 5. A ring cavity was used in this experiment, which was pumped by a 980 nm laser diode (LD) through a 980/1550 nm wavelength division multiplexer (WDM). A piece of 1 m long erbium-doped fiber (Er80-8/125) with an absorption of 110 dB m\(^{-1} \) at 1530 nm was used as a gain medium. Two polarization controllers (PCs) were used to adjust the polarization state in the cavity. A polarization-insensitive isolator (PI-ISO) was used to ensure unidirectional light propagation. A 95:5 fiber coupler was used to export the output laser from its 5% port. Additionally, it is a well-known fact that the formation of soliton mode-locked pulses is the result of the dynamic balance between the total cavity dispersion, the laser gain, the total cavity loss, the parameters of the SA and a variety of nonlinear effects. In our experiment, when the total cavity length was 118.1 m, mode-locked operation was detected by adjusting the state of the PCs and the pump power. Thus, the total cavity length was chosen to be about 118.1 m. The laser characteristics were simultaneously monitored by a 3 GHz photo-detector combined with a 500-MHz oscilloscope (DPO3054), a radio-frequency (RF) spectrum (R&S FPC1000), an optical spectrum analyzer (AQ-6317) and a power meter (PM100D-S122C).

4. Results and discussions

In the experiment, firstly, without inserting the InSe SA into the laser cavity, by changing the cavity parameters such as the pump power and the rotation of the PCs, no pulsed generation was observed. Afterwards, when the InSe SA was inserted into the laser cavity, pulsed generations were observed as we increased the pump power, which proved that the InSe SA was responsible for the generation of the pulsed lasers.

In the experiment, when the pump power ranged from 190–412 mW, by adjusting the state of the PCs, stable Q-switched pulse trains were generated. The typical characteristics of the InSe-based Q-switched Er-doped fiber laser under a pump power of 412 mW are shown in figure 6. The optical spectrum, which was recorded with a resolution of 0.05 nm, is shown in figure 6(a), and the central wavelength is 1532.2 nm. In addition, to test the stability of the passively Q-switched fiber laser, the radio frequency (RF) spectrum was measured by a spectrum analyzer. The RF spectrum is shown in figure 6(b) located at a fundamental repetition rate of 13 kHz, and the signal-to-noise ratio is about 36 dB. The RF result proves that the passively Q-switched operation has high stability. The corresponding typical oscilloscope trace under a pulse repetition rate of 13 kHz and a temporal pulse shape with a width of 10.62 µs at a pump power of 412 mW are shown in figures 6(c) and (d), respectively.

In figure 7(a), we show the pulse width and the pulse repetition rate as a function of the pump power. When the power increased from 190 mW to 412 mW, the pulse width decreased from 23.5 to 10.6 µs, the narrowest pulse duration was 8.3 µs under a pump power of 385.4 mW. According to the following equation [9]:

\[ \tau = 3.52T_R/\Delta T \]

where \( T_R \) is the cavity-round trip time and \( \Delta T \) is the SA modulation depth. So the pulse width can be further narrowed by decreasing the length of the cavity (our cavity length is more than 100 m) and increasing the SA modulation depth. Thus, our further works will explore the potential of InSe in obtaining short-pulse-width passively Q-switched fiber lasers.
Meanwhile, the pulse repetition rate increased from 5.58 kHz to 13 kHz. In general, the repetition rate of the Q-switched laser always changed linearly according to the increase of the pump power. As shown in figure 7(a), the repetition rate is not linear at 300 mW, and the repetition rate shows a suddenly increasing trend. The results might indicate that when the pump power is higher than 300 mW, the nonlinear effect in the fiber increases due to the high pulse energy, which will lead to the instability of the average output power and the pulse repetition rate. The average output power and the pulse energy as a function of the pump power are shown in figure 7(b).

As shown, the output power increased from 502.3 µW to 1.425 mW, and the pulse energy increased from 90.34 nJ to 112.97 nJ.

Brief results of the passively Q-switched ring-cavity Er-doped fiber lasers based on different 2D materials as SAs are described in table 1. As described, our results show similar performance compared with the Q-switched laser based on BP as the SA. The experiment results show that InSe SA exhibits good nonlinear saturable absorption properties.

By adjusting the state of the PCs, dual-wavelength mode-locked operations have also been achieved within...
the mentioned ring-cavity under a pump power of 270 mW with an average output power of 0.86 mW. Firstly, the typical dual-wavelength emission spectrum of the mode-locked laser is shown in figure 8(a), and an obvious dual-wavelength spectrum located at 1566.68 and 1567.48 nm was recorded. However, typical traditional soliton-like pulse shapes with characteristic Kelly-sideband peaks were not obtained. Figure 8(b) depicts the typical pulse train of the mode-locked laser, and the pulse-to-pulse time was 574.7 ns, corresponding to a pulse repetition rate of 1.74 MHz, which matches well with the cavity length of 118.1 m. A single pulse sharp is shown in figure 8(c), and the pulse width was 2.96 ns; this wide pulse width also indicates that the mode-locked operation obtained in our work is not a traditional soliton mode-locked one. It is obvious that for an Er-doped mode-locked fiber laser, the ns-level pulse width can be compressed significantly by optimizing the dispersion parameters. The wide pulse width was mainly due to the large net dispersion value caused by the long laser cavity length. It has been mentioned that in our experiment, when the total cavity length is 118.1 m, mode-locked operation can be recorded due to the dynamic balance between the total cavity dispersion, the laser gain, the total cavity loss, the parameters of the SA and a variety of nonlinear effects. Thus, to improve the quality of the mode-locked pulse, a relatively small net dispersion value is essential. In the future, we will do our best to compress the pulse width on the basis of shortening the cavity length and optimizing the parameters of the InSe SA.

Additionally, stability is one of the most important parameters for mode-locked lasers. To test the stability of the InSe-PVA-based mode-locked laser, the radio frequency (rf) spectrum was also recorded and depicted in figure 8(d). The radio frequency spectrum is located at a fundamental

| SAs    | Wavelength (nm) | Max pulse energy (nJ) | Repetition (kHz) | Min pulse width (μs) | Max output power (mW) | Refs        |
|--------|-----------------|-----------------------|-----------------|--------------------|-----------------------|------------|
| Graphene | 1556.17     | 16.7                  | 3.3–65.9        | 3.7                | 1.1                   | [18]       |
| SWNTs  | 1555–1560     | 14.1                  | ~5–~16          | ~7                 | ~225 μW               | [10]       |
| SESAMs | 1561.9        | 49.3                  | 1.72–7.59       | 30                 | 8.37                  | [12]       |
| BP     | 1562.87       | 94.3                  | 6.983–15.78     | 10.32              | ~1.5                  | [31]       |
| Bi2Se3 | 1565.14       | 15                    | 4.508–12.88     | 13.4               | ~150 μW               | [22]       |
| MoS2   | 1519.6–1567.7 | 160                   | 8.77–43.47      | 3.3                | 5.91                  | [25]       |
| InSe   | 1532.2        | 112.97                | 5.56–13         | 8.3                | 1.425                 | Our work   |

Figure 8. (a) The optical spectrum, (b) pulse train, (c) single pulse trace and (d) RF output spectrum.
repetition rate of 1.74 MHz with a bandwidth of 1.5 MHz, a resolution of 300 Hz and a signal-to-noise ratio of >45 dB.

Additionally, the RF spectra within a wide span are also conducive to testing the stability of a mode-locked laser. The RF spectra within bandwidths of 10 and 50 MHz were recorded and are shown in figures 9(a) and (b). As shown, within wide bandwidths, the RF spectra also exhibit high signal-to-noise ratios. Thus, in conclusion, the results shown in figures 8(d) and 9 prove that mode-locked pulses with high stability were obtained in our work.

In particular, it is obvious that the operating photon energies in our work (0.81 eV for Q-switched operation and 0.79 eV for mode-locked operation) were all lower than the bandgap of the SA material InSe (1.2–1.4 eV). In other words, sub-bandgap absorption phenomena were recorded and were responsible for the passively Q-switched and mode-locked operation, which also indicates that the nonlinear optical absorption of the prepared InSe was contributed by sub-bandgap absorption. Actually, passively Q-switched and mode-locked fiber lasers modulated by sub-bandgap absorption phenomena have been widely reported before [41, 42]. The sub-bandgap absorption phenomenon can be explained as follows: in a perfect crystal, there is no sub-bandgap absorption. However, in a finite system, the sub-bandgap absorption at low photon energies can also be realized, which are attributed to the energy levels within the bandgap arising from the edge-state [43]. Thus, in our opinion, the sub-bandgap absorption observed in our work can also be attributed to the edge-state absorption of the InSe.

Additionally, as mentioned, the central wavelengths of the Q-switched operation and the mode-locked operation were 1532.2 and 1566.5 nm, respectively. The difference indicates that the InSe SA may have a slightly high polarization-dependent loss. In the experiment, based on the experimental setup shown in figure 10, the characteristic of the polarization-dependent loss was tested. As shown, a continuous-wave 1560 nm Er-doped fiber laser was used as the laser source. A PC was employed to adjust the polarization state of the 1560 nm laser. The laser was divided into two parts by a 10:90 OC. Output one was used as a reference light and output two was the output power after the SA. In the experiment, we found that the output two port power changed slightly with the adjustment of the polarization state of the pump source, indicating that the wavelength difference between the Q-switched mode-locked lasers was not due to the polarization dependent loss of the SA. In our opinion, the formation mechanisms of Q-switched mode-locked lasers are not the same, which may have led to the wavelength difference. Thus, our future work will focus on the precise reasons for this discrepancy. In addition, based on the same setup shown in figure 10, the insert loss of the SA was tested to be 1.8 dB, and the large insert loss caused high threshold power during Q-switched mode-locked laser operation.

In conclusion, InSe was employed as an SA in the generation of passively Q-switched mode-locked fiber lasers for the first time. For the Q-switching operation, the maximum output power was 1.425 mW, and the repetition rate varied from 5.56 kHz to 13 kHz. The narrowest pulse duration was 8.3 µs, corresponding to a pulse energy of 112.97 nJ. In addition, dual-wavelength mode-locked operations were also recorded in the experiment. Our results clearly show that InSe could be a new 2D optoelectronic material with potential applications for obtaining pulsed fiber lasers.

**Acknowledgments**

The authors acknowledge support from the China Postdoctoral Science Foundation (2016M602177), the Shandong Provincial
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