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Abstract. We report a tungsten diselenide (WSe_2) polyvinyl alcohol (PVA)-based, saturable absorber and related experiment results of a Q-switched fiber laser. WSe_2 -PVA film is synthesized by liquid phase exfoliation method, and its saturable absorption is measured via a nonlinear transmission experiment. The result shows that WSe_2 -PVA saturable absorber has a modulation depth of 3.5%, which means it has potential for generating an ultrafast pulse laser. We apply this absorber into a ring-cavity erbium-doped fiber laser and obtain Q-switched pulses under appropriate pump power. Our work demonstrates the reliable nonlinear optical characteristics of WSe_2 and the feasibility for this two-dimensional material to be applied in the field of nonlinear optics. © 2016 Society of Photo-Optical Instrumentation Engineers (SPIE) [DOI: 10.1117/1.OE.55.8.081306]

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1 Introduction

Research advancement in some subjects might become a new driving force for another subject, which is the case with respect to material science and nonlinear fiber optics. Since the discovery of graphene in 2004,¹ the research of two-dimensional (2-D) materials has become a burst of prevailing wind, and many possible applications with these materials have been investigated. Graphene has been found to exhibit excellent nonlinear saturable absorption ability and applied in fiber laser to generate femtosecond pulses.² In recent years, many different 2-D materials have been discovered and deployed in the field of nonlinear optics, especially fiber lasers. Among these materials, transitional metal dichalcogenides (TMDs) have attracted researchers' interest. TMDs have a generalized formula as MX_2 , where M is a transitional metal atom, such as Mo or W, and X is chalcogen, such as S, Se, and Te. Due to the remarkable nonlinear optical characteristics of single-layer TMD nanosheets, many fiber laser research works combined with TMDs have been reported since 2014.³⁻⁵

Q-switching is a common method in the laser field to generate high-energy, low-frequency, and long-duration pulses. It is an important method that had been utilized to obtain pulses soon after the birth of ruby laser.⁶ Q-switched pulses earn their places in scientific research and medical treatment.^{7,8} TMD nanosheets can be used as saturable absorber (SA) to generate Q-switched pulses. In 2014, Woodward et al. reported a tunable ytterbium-doped Q-switched fiber laser based on few-layer MoS_2 , tunable from 1030 to 1070 nm.⁹ In 2015, Kassani et al. reported a WS_2 based Q-switched laser with tunable repetition rates from 82 to 134 kHz.¹⁰ In the same year, Woodward et al. achieved Q-switched pulses with an $MoSe_2$ saturable absorber.¹¹ On the road of exploring TMD materials, we will report our research results on WSe_2 and related Q-switched fiber laser in this paper. We fabricated WSe_2 -polyvinyl

alcohol (PVA) film and characterized with Raman spectroscopy and transmission electron microscopy. Saturable absorber based on WSe_2 -PVA film is fabricated and its nonlinear absorption ability is also characterized. Besides, we use this home-made SA to Q-switch an erbium-doped fiber (EDF) laser and obtain stable pulses. Our work demonstrates the nonlinear characteristics of WSe_2 nanosheets and feasibility of its application in nonlinear optics.

2 Material Preparation and Characterization

For the benefit of mass production and flexibility of usage, saturable absorber is usually fabricated with suitable substrate. One common substrate is PVA film. Liquid phase exfoliation method is adopted and the detail process is similar to Ref. 4. Briefly, 5 mg/ml WSe_2 water dispersions are prepared with sodium cholate as surfactant. Meanwhile, 50 mg/mL PVA aqueous solution is also prepared. 2 mL WSe_2 dispersions along with 10 ml PVA aqueous solution are mixed for 24 h by a magnetic stirrer. Then the mixture is processed for another 4 h by ultrasonic water bath device. After ultrasonic processing, the mixture is transferred to the surface of a clean plastic dish and dried under 50°C air condition for three to four days. Figure 1(a) shows a piece of well-fabricated WSe_2 -PVA film. The film is cut into very small pieces ($\sim 1 \times 1$ mm) for experimental usage. Scanning electron microscope (SEM) shows the flat edge of film and the thickness is ~ 22.5 μm [Fig. 1(b)].

To characterize the atomic structures of the fabricated film, Raman spectroscopy is used. Figure 1(c) shows measured result. A_{1g} mode can be observed at 250.7 cm^{-1} , which comes from out-of-plane motion of WSe_2 molecular.¹² E_{2g}^1 mode is found at 245.1 cm^{-1} , and it is the result of in-plane motion of WSe_2 molecular.¹² B_{2g} mode is observed at 315.6 cm^{-1} , which originates from the breakdown of translation symmetry in few-layer WSe_2 .¹² Transmission electron microscopy (TEM) gives dimension information about

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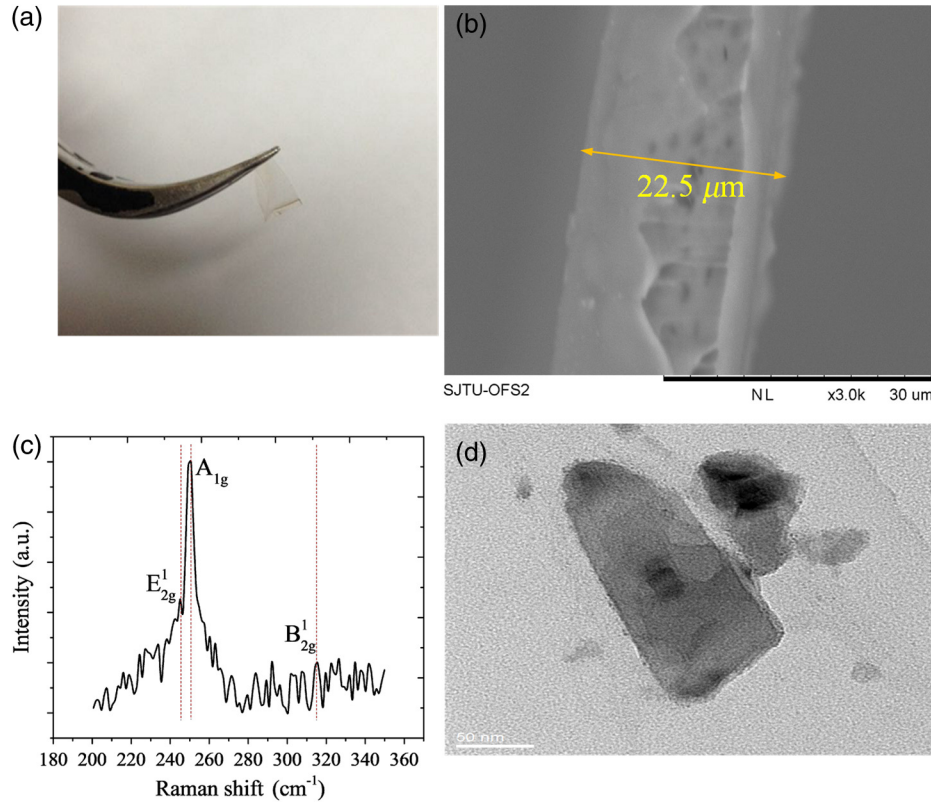


Fig. 1 (a) WSe₂-PVA film photo, (b) SEM photo of edge of WSe₂-PVA film (with ×3000 magnification), (c) Raman spectrum, and (d) TEM photo of WSe₂ nanosheet (with a scale of 50 nm).

WSe₂ nanosheets. In Fig. 1(d), the scale (white bar) stands for 50 nm. The size of nanosheet is estimated as 200 nm × 75 nm.

Nonlinear saturable absorption ability is vital to saturable absorber. To measure this characteristic, a standard two-arm experiment is carried out with WSe₂-PVA films. The setup is shown in Fig. 2(a). A mode-locked laser with tunable output power is used as optical source. The output pulses propagate through a 90:10 coupler; 10% optical power is measured by power meter 1 as a reference, while 90% optical power passes through WSe₂-PVA films and is measured by power meter 2. To adapt to the measurement range, a 10-dB attenuator is applied before power meter 2. By changing the output power of mode-locked laser, the transmission at different optical intensity can be calculated with measured data. The result is shown in Fig. 2(b). The dots are fitting with the following formula:¹³

$$T(I) = 1 - \Delta T \times \exp(-I/I_{\text{sat}}) - A_{\text{ns}} - \beta I, \quad (1)$$

where T is the transmittance, ΔT is the modulation depth, I is the intensity of laser, I_{sat} is the saturation power intensity, A_{ns} is the nonsaturable absorbance, and β is the two-photon absorption (TPA) coefficient. The modulation depth is 3.5%, saturation intensity is 103.9 MW/cm², nonsaturable absorbance is 75.1%, and the TPA coefficient is 1.77×10^{-5} cm²/MW.

3 Q-Switching Operation

The nonlinear transmission experiment in Sec. 2 shows a good saturable absorption ability of our home-made saturable absorber. To further demonstrate this characteristic, it is applied to Q-switch an EDF laser. The setup of fiber laser is shown in Fig. 3. The pump source is a 980 nm diode laser with tunable output power. A 980/1550 nm

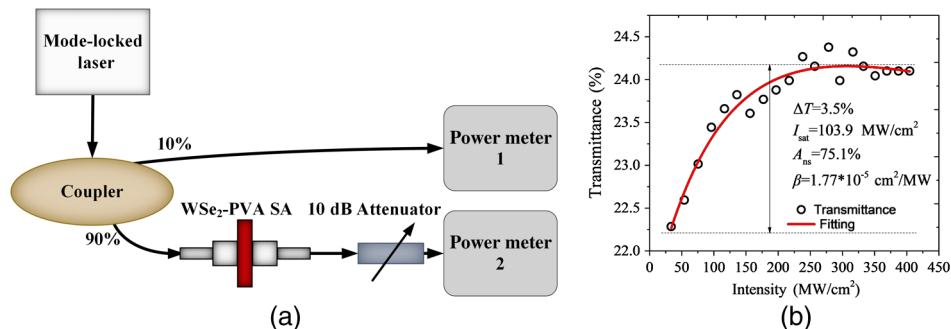


Fig. 2 (a) Setup of nonlinear transmission experiment and (b) nonlinear transmission experiment result.

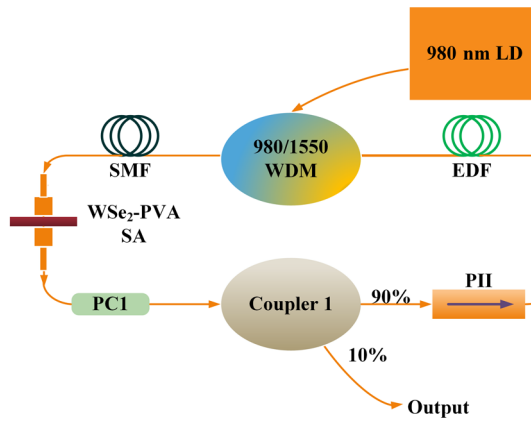


Fig. 3 Setup of WSe₂ Q-switched EDF laser. LD, laser diode; WDM, wavelength division multiplexer; SMF, single-mode fiber; EDF, erbium-doped fiber; PC, polarization controller; PII, polarization-independent isolator.

wavelength division multiplexer couples the pump light into the ring cavity. The gain medium is a section of EDF. Two polarization controllers (PC1 and PC2) are used to optimize polarization state and birefringence of intracavity light in order to stabilize Q-switching operation. A 90/10 coupler extracts 10% optical power as output. A polarization-independent isolator ensures the unidirectional running of fiber laser. To measure parameters of output pulses, an optical spectrum analyzer (YOKOGAWA AQ6370C) is used to monitor the optical spectrum. A photodetector is used to transform the optical signals into electrical signals. An oscilloscope (Agilent Technologies, DSO9254A) is used to detect waveform in time domain and an electric spectrum analyzer (ROHDE & SCHWARZ) is used to measure power spectrum. WSe₂-PVA saturable absorber is constructed by sandwiching three pieces of WSe₂-PVA films by a pair of flat connector/physical contact (PC) connectors.

The laser operates on continuous-wave (CW) mode when pump power is <40 mW. As the pump power increases over 40 mW, the laser outputs unstable Q-switched pulses without a uniform amplitude. When the pump power exceeds 140 mW, the Q-switching operation shows a good stability, which has little phase jitter and amplitude fluctuation. The amplitude of Q-switched pulses start to decrease along with the increase of pump power when pump power exceeds 240 mW. At last, if the pump power exceeds 300 mW, the

laser turns back to CW mode and no pulse would be generated. If we remove the WSe₂-PVA films, which are sandwiched between fiber connectors, the laser would work on CW mode no matter what the pump power is. This shows WSe₂-PVA films are indispensable to generate Q-switched pulses in this ring-cavity fiber laser.

Output power is dependent on pump power as shown in Fig. 4(a). The output power of Q-switched pulses can be adjusted from -11.27 dBm (0.07 mW) to 0.91 dBm (1.23 mW) with the change of pump power. The modes in regions I and V are CW mode. Regions II and IV are unstable Q-switching region, while in region III, the laser operates on stable Q-switching mode. Pulse duration and repetition rate are both pump power dependent. Their relations are shown in Fig. 4(b). When pump power is tuned from 60 to 300 mW, the pulse duration could be tuned from 3.1 to 7.9 μ s while repetition rate could be adjusted from 4.5 to 49.6 kHz. By calculation, the single pulse energy can be tuned from 13.5 to 33.2 nJ.

Figure 5 shows the detailed laser parameters, which are measured under 200 mW pump power. Figure 5(a) depicts the pulse train, and Fig. 5(b) shows a single pulse profile. The output power is -3.41 dBm, repetition frequency is 26.907 kHz, and pulse duration is 3.976 μ s. This corresponds to a single pulse energy of \sim 17 nJ. The optical spectrum is shown in Fig. 5(c). The central wavelength is 1560 nm. To investigate the stability of Q-switching operation, the electrical spectrum has been measured. In Fig. 5(d), the extinction ratio (with a resolution of 100 Hz) at fundamental frequency is 46.7 dBm, which means a relatively good Q-switching operation stability. However, there are many spikes existing on the optical spectrum peak, which means CW lasing with different longitudinal modes compete with each other. The existence of CW lasing could be the reason that limits the extinction ratio of Q-switching operation. Actually, applying an optical filter might be helpful in suppressing parasite CW lasing and improving extinction ratio.¹⁴

4 Discussion

According to Ref. 11, single-layer WSe₂ has a direct bandgap of 1.65 eV and bulk state has an indirect bandgap of 1.2 eV. The bandgap values correspond to wavelengths between 753 and 1035 nm. However, the wavelength of generated Q-switched pulse is 1560 nm, which is out of that range. This might come from sub-bandgap absorption of

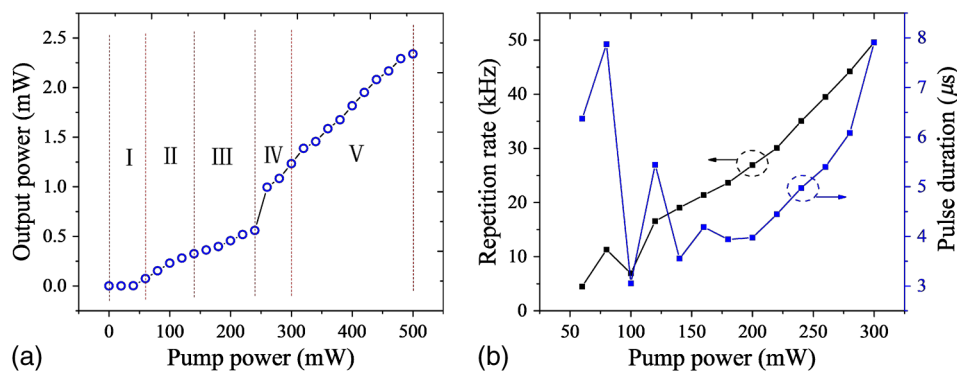


Fig. 4 (a) Output power versus pump power and (b) repetition rate and pulse duration versus pump power.

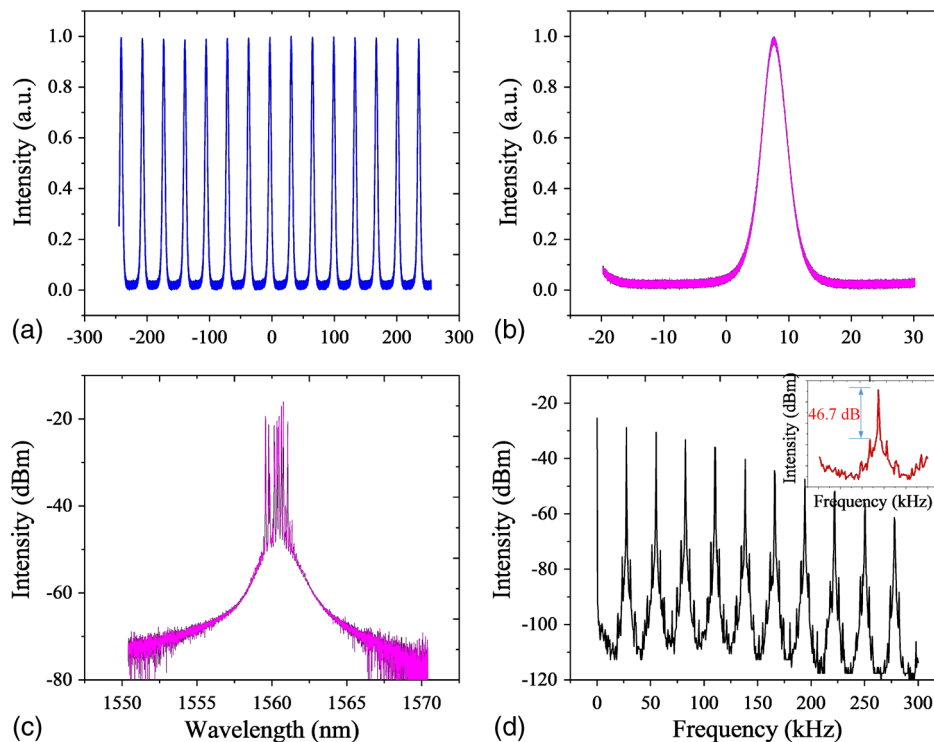


Fig. 5 Parameters of Q-switched pulses under 200 mW pump power: (a) pulse train, (b) single pulse profile, (c) optical spectrum, and (d) electrical spectrum (inset shows a 46.7 dB extinction ratio at fundamental frequency).

edge states.^{9,15} Sub-band absorption has been studied by introducing point defects with WS₂ recently.¹⁶ As a similar property and structure, WSe₂ could also display analogous sub-bandgap absorption. The size of our WSe₂ nanosheets is $\sim 200 \times 75$ nm; this limit size leads to large edge-to-surface ratio, which means the existence of large quantity of edge states. Besides, during the fabrication process, we cannot avoid the introduction of defects, which results in defect states.¹⁷ The absorption happened at edge states and defect states could be the origin of 1560-nm photons.

Pulse duration of Q-switched laser was determined by the cavity round-trip time or the cavity length. For a cavity with short length, sub-microsecond pulse width can be obtained.¹⁸ For long cavities, pulse widths of a few microseconds were usually reported. For example, carbon nanotube based Q-switched fiber laser with 20-m-long cavity generated pulses with 7 μ s duration.¹⁹ An 8-m-long graphene based Q-switched fiber laser outputs pulses with width tunable between 4 and 18 μ s.²⁰ For our laser, we had constructed a relatively long cavity with a cavity length of ~ 17 m. As a result, the pulse width of our laser was above 1 μ s. To further decrease the pulse width, the way is to shorten the cavity length. The amplitudes of the Q-switched pulse become unstable when the pump power exceeds 260 mW. The melting point of WSe₂ is 800°C²¹ and thermal conductivity is 0.9 W/(m · K).²² However, the melting point of PVA is 228°C; it has a glass transition temperature of 85°C,²³ and its thermal conductivity is 0.2 W/(m · K).¹⁴ Therefore, PVA is sensitive to high temperature. Under high pump power, the area where laser irradiates directly absorbs much heat and might change its physical state. As a result, this introduces instability to Q-switching operation under high pump power. The substrate of saturable absorber limits the performance of

WSe₂; a possible solution could be adopting some other robust substrates, such as fluorine mica.¹⁴ Besides, with the idea of evanescent field interaction, depositing WSe₂ on side-polished fiber or microfiber might be another choice to make a saturable absorber suitable for high pump power operation.^{24–27}

5 Conclusion

In conclusion, we have characterized the home-made WSe₂-PVA film and measured its saturable absorption ability. The results show that WSe₂-PVA saturable absorber has a modulation depth of 3.5%, which has the potential to generate pulse laser. We insert this SA into a ring-cavity EDF laser and obtain Q-switched pulses under appropriate pump power. Our work demonstrates the reliable nonlinear optical characteristics and the feasibility of this 2-D material for application in nonlinear optics.

Acknowledgments

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