## Vanadium pentoxide film for microsecond pulse generation in 1.5-µm region

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Q-switched erbium doped fiber laser (EDFL) was passively realized using vanadium pentoxide ( $V_2O_5$ ) embedded into polyethylene glycol (PEG) film as saturable absorber (SA). The laser could successfully generate stable self-starting pulses when the  $V_2O_5$  film was placed in an EDFL cavity. It operated at 1 562.4 nm wavelength. The repetition rate can be varied from 91.7 kHz to 128.2 kHz while the pulse width shrank from 10.90  $\mu$ s to 7.81  $\mu$ s with rising pump power from 110.9 mW to 166.5 mW. The pulse energy recorded was 3.2 nJ at pump power of 166.5 mW. The results indicate that the saturable absorption of  $V_2O_5$  has promising nonlinear photonic applications especially in fiber laser development for 1.5- $\mu$ m region.

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Today, a considerable attention is given to optical pulse creation in a fiber laser as a response to their various advantages and applications<sup>[1]</sup>. For instance, Q-switched erbium-doped fiber lasers (EDFLs) have gained more interests because of their advantages of alignment-free structure, good mode confinement and high stability for implementation in various areas such as material processing, microfabrication, range finding, remote sensing, communications, skin treatment and medical surgery<sup>[2-4]</sup>. Q-switching is one of the methods to generate short optical pulses in a fiber laser. Several approaches have been reported to realize the Q-switching including a passive technique based on saturable absorber (SA). Compared to the conventional active techniques, this technique is simpler, compact and efficient.

Semiconductor SA mirrors (SESAMs) were previously employed as SA due to their benefits of ultrafast recovery time, compatibility with other components, their stability, and absorption rate<sup>[5]</sup>. On other hand, they also have several drawbacks like low damage threshold, high cost, and narrow bandwidth, which delayed their development. Until now, various types of nanomaterials including graphene<sup>[6]</sup> and black phosphoros<sup>[7]</sup> have also been reported for the creation of Q-switched pulse train. Graphene was most utilized in various pulsed lasers due to its ability to work in wideband region with low saturation intensity as well as it is relatively inexpensive.

However, its application has been limited due to its small bandgap, which also makes the fabrication complex<sup>[6]</sup>.

Unlike graphene, the black phosphorus possesses the desired bandgap that gives the ability of employment in various wavelength regions. Despite that, the performance of black phosphorus (BP) rapidly degraded during its application and it gets damaged easily in natural environment<sup>[8]</sup>. Likewise, carbon nanotubes<sup>[9]</sup>, topological insulator<sup>[10]</sup>, transition metal dichalcogenide<sup>[11]</sup> and transition metal oxide (TMO)<sup>[12]</sup> materials have also drawn much interests for SA applications due to their excellent nonlinear characteristic especially in the 1.5-µm region.

However, TMO have gained greater interest in recent years due to their outstanding electronic and optical properties<sup>[12]</sup>. They are characterized by ultrafast response time, large third-order nonlinear, wide absorption band and thus can enable high performance optoelectronic devices<sup>[13]</sup>. In addition, the bandgap can be controlled by the particle size and thickness of TMO<sup>[14]</sup>. A number of TMO materials have been used for generating Q-switched or mode-locked pulses including zinc oxide<sup>[15]</sup>, titanium dioxide<sup>[16]</sup>, copper oxide<sup>[17]</sup>, and molybdenum oxide<sup>[18]</sup>.

Vanadium pentoxide (V<sub>2</sub>O<sub>5</sub>) is one of the most important transition metal oxides<sup>[19]</sup>. It was reported to have an excellent nonlinear optical absorption characteristic and thus suitable for SA applications<sup>[20]</sup>. In this letter, a Q-switched

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EDFL is obtained by means of the newly developed  $V_2O_5$  SA. The SA was prepared by the embedding of  $V_2O_5$  material into polyethylene glycol (PEG) to compose a film absorber, which was then inserted between two fiber ferrules. The stable Q-switched laser was successfully achieved with maximum repetition rate, minimum pulse width and highest energy are recorded at 128.2 kHz, 7.81  $\mu$ s and 3.2 nJ, respectively.

For this work, the ammonium metavanadate (NH<sub>4</sub>VO<sub>3</sub>) compounds were used as a raw material. They were purified by using a standard chemical reaction to obtain V<sub>2</sub>O<sub>5</sub> compounds. In the process, 20 g of NH<sub>4</sub>VO<sub>3</sub> was dispersed in deionized water (500 mL). Then, a tiny amount of Triton X-100 surfactant (about 0.1 g) was added in the solution and the mixture was stirred at 90 °C for 1 h. HNO<sub>3</sub> with 35% concentration was slowly dropped into the mixture to acidify the solution. After leaving the mixture overnight, the dark brown precipitates were obtained. They were gathered and then washed in deionized water to get rid of any traces of surfactant and acid. The precipitates were then dried in hot air oven for 12 h at a constant temperature of 90 °C. Finally, dried powder was crushed and calcined at 500 °C for 5 h in a box furnace to form yellowish-brown V<sub>2</sub>O<sub>5</sub> compound (Fig.1(a)). X-ray diffraction (XRD) was then used to investigate the phase purity and structural crystallinity of V<sub>2</sub>O<sub>5</sub> as shown in Fig.1(b). The XRD profile reveals shape peaks at various  $2\theta$  positions. The peaks at 51.3°, 47.4°, 34.4°, 32.4°, 31°, 26.2°, 21.7°, 20.3° and 15.4° positions relate to the lattice planes of (002), (600), (301), (011), (310), (101), (110), (010) and (200), respectively. These peaks indicate orthorhombic crystal phase of V<sub>2</sub>O<sub>5</sub> with no impurities.

The V<sub>2</sub>O<sub>5</sub> compound was suspended in a thin PEG film so that it can be incorporated into an EDFL cavity to function as an SA device. At first, we dissolved 1.0 g of PEG powder in 120 mL deionized water and put under stirring for 2 h. Subsequently, an appropriate amount of the V<sub>2</sub>O<sub>5</sub> compound was added into the prepared uniform transparent PEG solution and the mixture was kept under constant stirring for at least 2 h to get homogenous V<sub>2</sub>O<sub>5</sub> PEG composite solution. The composite solution was cast on to the petri-disk and dried at temperature of 60 °C in an oven for a day. A light yellow solid thin film was obtained as displayed in Fig.2(a). The field emission scanning electron microscopy (FESEM) image of the V<sub>2</sub>O<sub>5</sub> film is shown in Fig.2(b), depicting the tubular morphology of V<sub>2</sub>O<sub>5</sub>. The tubular structure provides contact to the inner and outer surfaces as well as tube ends for better interaction with photons.

The SA device was constructed by simply slotting in a tiny piece of  $V_2O_5$  film between two ferrules. The film was firstly stuck onto the face of one of the FC/PC fiber ferrules with a help of index matching gel (Fig.2(c)), and then connected to another ferrule using a fiber adaptor to form a fiber compatible SA device (Fig.2(d)). We applied

an index matching gel in the connection to avoid spurious reflection in the laser cavity. The SA assembly has an insertion loss of about 1 dB.



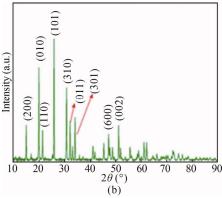


Fig.1 (a) Yellowish-brown  $V_2O_5$  compound and (b) its XRD profile

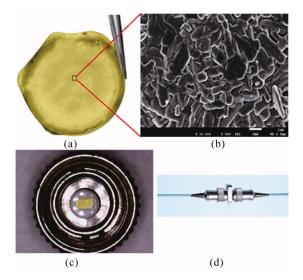
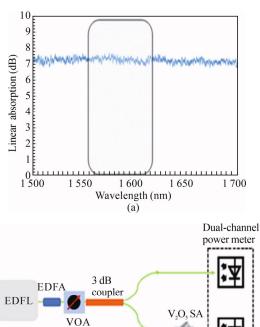


Fig.2 (a) A light yellow solid thin film of  $V_2O_5$ ; (b) FESEM image; (c) The film placed onto a fiber ferrule tip; (d) All-fiber  $V_2O_5$  SA device

The linear absorption of the  $V_2O_5$  PEG film was then examined by launching a broadband light into the prepared film. The output spectrum recorded by an optical spectrum analyzer is presented in Fig.3(a). It indicates about 7 dB absorption at 1 550 nm. The nonlinear absorption measurement was also investigated using a homemade experimental setup (Fig.3(b)). It used a light

source based on a mode locked EDFL with 1.8 MHz repetition rate and 4.62 ps pulse duration for this measurement. The 1 559 nm laser's intensity was magnified and controlled by an erbium-doped fiber amplifier (EDFA) and variable optical attenuator (VOA), respectively. The attenuated signal was then divided into two beams. One beam was propagated into the SA film while the other beam was directly connected to optical power meter as a reference. As shown in Fig.3(c), the saturation intensity of the V<sub>2</sub>O<sub>5</sub> PEG film SA is 90 MW/cm<sup>2</sup> while its modulation depth and non-saturable absorption are 7% and 49%, respectively. The optical bandgap of V<sub>2</sub>O<sub>5</sub> nanoparticles was reported at around 2.0 eV, which indicates to the peak absorption in visible region. But we also observed the absorption in 1.5-µm region because of some defects in material morphology.

Fig.3(d) illustrates the EDFL configuration, which was constructed for this study. It consists of the prepared V<sub>2</sub>O<sub>5</sub>-PEG SA, 2-m-long EDF, an isolator, a 90/10 output coupler, and a wavelength division multiplexer (WDM). The polarization-insensitive isolator forced the laser light to propagate in one direction inside the ring resonator. The V<sub>2</sub>O<sub>5</sub> film was slotted in between two fiber ferrules and integrated into the ring cavity to act as Q-switcher. 10% of the output power was extracted through a 90:10 coupler for analysis. The output spectrum has been detected by an optical spectrum analyzer (OSA, Yokogawa AQ6370B) with 0.02 nm spectral resolution. The frequency and time-domain of the signal can be detected by a 1.3 GHz photodetector (Thorlabs, DET10D/M) in conjunction with a 7.8 GHz RF spectrum analyzer (Anritsu) and a 350 MHz digital oscilloscope (GWINSTEK: GDS-3352), respectively. The total length of the laser's cavity is approximately 5 m.



(b)

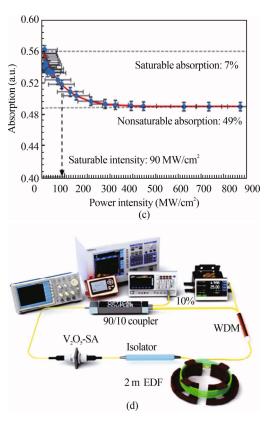


Fig.3 (a) Linear absorption curve; (b) Nonlinear measurement setup; (c) Nonlinear saturable absorption curve for the  $V_2O_5$ -SA film; (d) Laser configuration

After inserting the V<sub>2</sub>O<sub>5</sub>-PEG SA, a continuous wave laser was firstly generated when the power of pump laser reached 40 mW. As the laser diode's power was further boosted to 110.9 mW, the EDFL starts to deliver a train of Q-switched pulses. Fig.4(a) shows the output spectrum of the laser at a pump power of 138.7 mW. The laser wavelength is centered at 1 562.4 nm with a 3-dB bandwidth of 0.4 nm. Fig.4(b) illustrates the corresponding RF spectrum, which indicates that the repetition rate of the pulses is 97.2 kHz at 138.7 mW pump power. The signal-to-noise ratio of the fundamental frequency is observed to be higher than 45 dB, which confirms the stability of such Q-switched operation. Fig.5 plots the typical oscilloscope traces at pump powers of 110.9 mW, 138.7 mW and 166.5 mW, respectively. As seen, all pulses trains have a uniform profile and intensity. The pulse periods are given as 10.90 μs, 10.29 μs and 7.82 μs, which correspond to repetition rates of 91.7 kHz, 97.2 kHz and 128.2 kHz, respectively. The zoom-in images of triple pulses are depicted in Fig.5, giving a pulse width of 10.6 μs, 9.5 μs and 4.7 μs, respectively.

The evolution of the Q-switched pulses with the change of pump power is also investigated as shown in Fig.6. As demonstrated in Fig.6(a), by gradually varying the pump power from 110.9 mW to 166.5 mW, the repetition rate enlarges from 91.7 kHz to 128.2 kHz while the pulse width shortens from 10.90 µs to 7.81 µs, which is

the typical characteristic of the passively Q-switched operation. It is also found that the Q-switched pulse became unstable and disappeared as the pump power was augmented beyond 166.5 mW. This is attributed to the higher gain, which increases the laser intensity and over-saturates the  $V_2O_5$ . The Q-switched pulses train was regained when the pump power was decreased back to 166.5 mW.

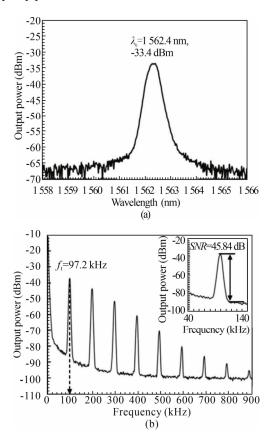


Fig.4 (a) Output spectrum and (b) RF spectrum of the Q-switched EDFL at 138.7 mW pump

Fig.6(b) shows the average power and pulse energy versus pump power. It is obtained that the output power increases monotonously from 0.24 mW to 0.40 mW with pump power. On the other hand, the single pulse energy also grows from 2.6 nJ to 3.2 nJ with the increase of pump power from 110.9 mW to 166.5 mW. The light conversion efficiency was about 0.3%. The low efficiency might be due to the large cavity loss induced by the SA film. To confirm whether the Q-switching operation is purely induced by the  $V_2O_5$  PEG film, the SA was replaced with a pure PEG film. However, Q-switched pulses were not observed in any cases, in despite of tuning laser diode over a full range.

Tab.1 compares  $V_2O_5$  SA with other materials for the formation of Q-switched laser. As seen, the  $V_2O_5$  SA exhibits the highest repetition rate. By slightly modifying the EDFL cavity, mode-locked pulses could also be realized by the  $V_2O_5$  SA. The mode-locking operation requires a better design of laser cavity to balance the

nonlinear effect with the group velocity dispersion.

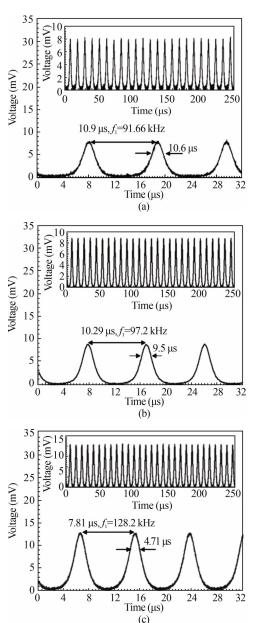
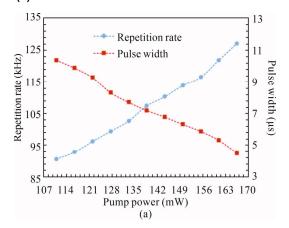


Fig.5 Typical pulse trains of the Q-switched EDFL at different pump powers: (a) 110.9 mW, (b) 138.7 mW and (c) 166.5 mW



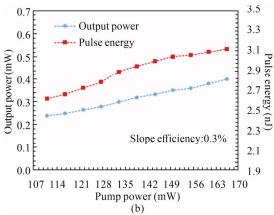


Fig.6 (a) Repetition rate and pulse width against pump power; (b) Output power and pulse energy at various pump powers

Tab.1 Q-switching performance comparison

SA	Maximum pump power (mW)	Repe- tition rate (kHz)	Pulse width (µs)	Pulse energy (nJ)	λ (nm)	Ref.
Graphene	151.47	67.8	6.02	206	1 558.3	[21]
CNT	209.6	70.4	4.5	81.3	1 563.1	[22]
BP	170	44.33	7.04	134	1 552.9	[23]
$MoS_2$	170	38.43	5.02	141.3	1 551.4	[23]
$MoS_2$	170	41.4	13.5	184.7	1 560.0	[11]
$WSe_2$	300	49.6	3.1	33.2	1 560.0	[24]
$Ti_2AlN$	106	41.55	2.52	7	1 557.0	[25]
$Ti_2AlC$	74	27.45	4.88	22.58	1 560.4	[26]
$V_2O_5$	166.5	128.2	7.81	3.2	1 562.4	This
						work

In conclusion, Q-switched pulses were succesfully demonstrated in EDFL cavity using the new SA based on V<sub>2</sub>O<sub>5</sub> film. The SA was obtained by embedding the synthesized V<sub>2</sub>O<sub>5</sub> into a PEG polymer film. It has a modulation depth, saturation intensity and non-saturable loss of about 7%, 90 MW/cm<sup>2</sup> and 49% respectively. Q-switching operation was obtained from pump power range of 110.9—166.5 mW. The pulse repetition rate indicates an increasing trend from 91.7 kHz to 128.2 kHz, whereas the pulse width shows a decreasing trend from 10.90 µs to 7.81 µs. The highest pulse energy of 3.2 nJ is obtained at pump power of 166.5 mW. The V<sub>2</sub>O<sub>5</sub> SA device has advantages in terms of ease fabrication, robustness and stability, and thus it has promising potential for various mesaurement and optical communication applications.

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## **Statements and Declarations**

The authors declare that there are no conflicts of interest related to this article.

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