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Q-Switched Ytterbium-Doped Fiber Laser Using Black Phosphorus as Saturable Absorber

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We demonstrate a Q-switched ytterbium-doped fiber laser (YDFL) using a newly developed multi-layer black phosphorous (BP) saturable absorber (SA). The BP SA is prepared by mechanically exfoliating a BP crystal and sticking the acquired BP flakes onto a scotch tape. A small piece of the tape is then placed between two ferrules and incorporated in a YDFL cavity to achieve a stable Q-switched operation in a 1.0 μm region. The laser has a pump threshold of 55.1 mW, a pulse repetition rate that is tunable from 8.2 to 32.9 kHz, and the narrowest pulse width of 10.8 μs. The highest pulse energy of 328 nJ is achieved at the pump power of 97.6 mW. Our results show that multi-layer BP is a promising SA for Q-switching laser operation.

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Passively Q-switched fiber lasers have gained widespread attention due to their advantages in compactness and flexibility, and potential applications in medicine, telecommunications, remote sensing and laser material processing. To date, many forms of saturable absorber (SA) have been used in generating passive Q-switching including semiconductor saturable absorber mirror (SESAM),[1] single-walled carbon nanotubes (SWCNTs)[2] and graphene.[3,4] Despite the successful implementation of these SAs, researchers are still looking for new SAs with better characteristics in terms of wavelength-independent saturable absorption, low saturable optical intensity, fiber compatibility and high damage threshold. Recently, topological insulators (TIs) and molybdenum disulfide (MoS_2) have also been identified as potential SAs for mode-locking and Q-switching.[5–7]

More recently, black phosphorus (BP), dubbed as the newly emerging allotrope, has gained much attention for potential applications in the next-generation optoelectronic devices such as sensors, field-effect transistors and solar cells.[8–10] Multi-layer BP has a similar structure as bulk graphite. In a single layer, each phosphorus atom is covalently bonded with three adjacent phosphorus atoms to form a stable honeycomb structure, and different layers are stacked together by van der Waals interaction.[11] It has the common properties of 2D materials such as wideband absorption, ultrafast carrier dynamics and planar characteristic.[12] The BP comprises only the elemental 'phosphorus' and thus it could be easily peeled off by mechanical exfoliation. In this Letter, we report a passively Q-switched ytterbium-doped fiber (YDF) laser generated using multi-layer BP-based SA, obtained by the mechanical exfoliation method. The SA is prepared by peeling bulk-structured BP 2D thin layers using a scotch tape. By inserting a small piece of the BP-SA tape into the ytterbium-doped fiber laser cavity, Q-switched pulses with a maximum pulse energy of 328 nJ are achieved, with the shortest pulse width of 10.8 μs. Mechanical exfoliation is advantageous mainly due to its simplicity and reliability, where the entire fabrication process is free from complicated chemical procedures and costly instruments.

The multi-layer BP used in our experiment was prepared by the mechanical exfoliation technique in the following. Initially, we peeled off thin flakes from a large block of commercially available BP crystal (purity of 99.995%) using the clear scotch tape. Then, we repeatedly pressed the flakes on the scotch tape to form a thin layer of BP, thin enough to transmit light with a high efficiency. Afterwards, a small piece of the BP tape was cut out and attached onto a standard FC/PC fiber ferrule end surface with the aid of index matching gel. After connecting it with another FC/PC fiber ferrule with a standard flange adapter, the all-fiber BP-based SA was finally ready. The entire preparation process is illustrated in Fig. 1.

The composition of the multi-layer BP tape is examined by the energy dispersive spectroscopy (EDS) on the field emission scanning electron microscopy (FESEM). Presence of the BP material on the scotch tape adhesive surface was confirmed by the presence of the high peak of phosphorus in the spectroscopy as shown in Fig. 2. The inset in Fig. 2 shows the FESEM image, which confirms the existence of uniform multi-layer phosphorus and the absence of >1 μm aggregates or voids in the tape, which otherwise result in non-saturable scattering losses. We also performed Raman spectroscopy on the prepared BP tape sample. Figure 3 shows the Raman spectrum, which is recorded by a spectrometer when a 514 nm beam of an argon ion laser is radiated on the tape for 10 ms with an exposure power of 10 mW. As shown in the figure, the sample exhibits three distinct Raman peaks at 359 cm^{-1}, 434 cm^{-1} and 460 cm^{-1},[13] correspond-

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ing to B_{2g} and vibration modes of a layered BP. While B_{2g} and modes correspond to the in-plane oscillation of phosphorus atoms in the BP layer, the mode corresponds to the out-of-plane vibration. The ratio between peak and silicon (Si) level at 520 cm^{-1} indicates that the thickness of the BP flake is ~200 nm. The thickness for single-layer BP is about 0.8 nm,[14] thus the prepared BP-based SA is expected to consist of 250 layers.

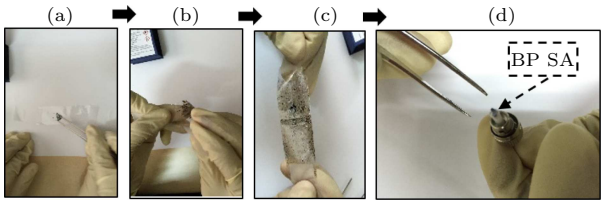

Fig. 1. The preparation flow of BP-SA by using the mechanical exfoliation method. (a) The peeled BP crystal is placed onto a sticky tape. (b) The crystal is thinned to the small flakes by repeatedly pressing the flakes onto a sticky tape. (c) The image of the BP tape after the thinning process. (d) Attaching the BP tape onto the ferrule.

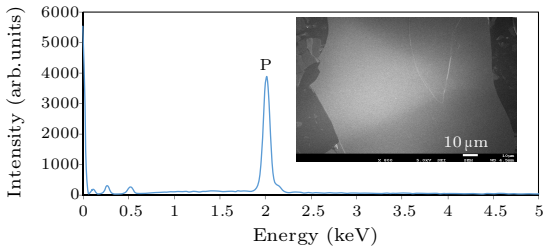

Fig. 2. EDS from the FESEM image of the prepared BP tape, which confirms the presence of phosphorus. Inset: the FESEM image.

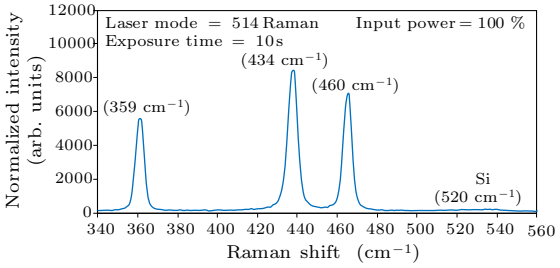

Fig. 3. Raman spectrum of the multi-layered BP tape.

The nonlinear optical response property for the multi-layer BP on the scotch tape was also investigated to confirm its saturable absorption by applying an absorption technique. A self-constructed mode-locked fiber laser (1557 nm wavelength, 1.5 ps pulsewidth, 17.4 MHz repetition rate) was used as the input pulse source. The transmitted power and a reference power for normalization are recorded as a function of incident intensity on the tape by varying the input laser power. With increasing the peak intensity, the material absorption decreases as shown in Fig. 4, confirming the saturable absorption characteristic of the multi-layer BP. The experimental results for absorption are fitted according to a simple two-level SA model,[15]

$$\alpha(I) = \frac{\alpha_s}{1 + I/I_{sat}} + \alpha_{ns},$$

where $\alpha(I)$ is the absorption, α_s is the modulation depth, I is the input intensity, I_{sat} is the saturation intensity, and α_{ns} is the non-saturable absorption. As shown in Fig. 4, the modulation depth, non-saturable intensity, and saturation intensity are 7 %, 58 % and 0.0025 MW/cm^2, respectively. Taking into account its nonlinear optical response leading to absorption saturation at relatively low fluence, the mechanically exfoliated BP meets basic criteria of a passive SA for fiber lasers.

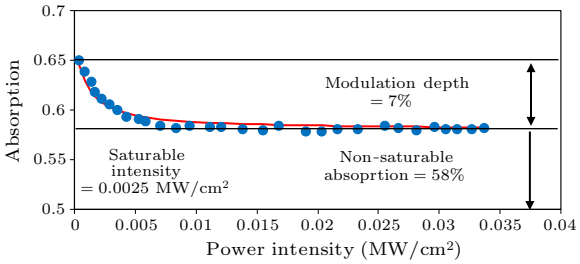

Fig. 4. Nonlinear saturable absorption profile of the multi-layer BP.

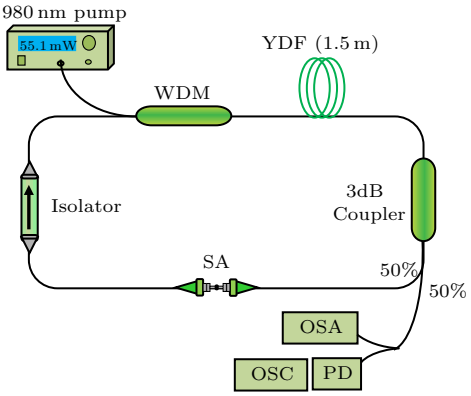

Fig. 5. Experimental setup for the Q-switched YDF fiber ring laser using SA.

The experimental setup of the proposed Q-switched YDF laser is schematically shown in Fig. 5. The ring cavity consists of a 1.5-m-long YDF, a wavelength division multiplexer (WDM), an isolator, a 3 dB output coupler and the multi-layer BP Q-switcher. The total cavity length is about 6 m. The YDF used has a core diameter of 4.0 μm, NA of 0.20 and cutoff wavelength of around 980 nm. The doping level of ytterbium ions in the fiber is 1500 ppm. The YDF is pumped by a 980 nm pump laser diode through a 980/1030 nm WDM. The multi-layer BP on the scotch tape is integrated into the cavity as a passive Q-switcher while a polarization insensitive optical isolator is incorporated to ensure unidirectional operation of the laser. The Q-switched pulse train is taken out via a 3 dB output coupler which keeps half the light oscillating in the ring cavity for both spectral and temporal diagnostics. The output is simultaneously monitored by using a 350-MHz oscillo-

scope together with a 2-GHz photodetector, a 7.8 GHz rf spectrum analyzer and an optical spectrum analyzer (OSA) with a spectral resolution of 0.02 nm. It is worth noting that the Q-switching performance such as output pulse energy of the laser is observed to be optimum with the use of the 3 dB coupler.

The YDF laser started to operate in the cw mode at the pump power of 40.5 mW, before transiting to the Q-switching operation at 55.1 mW. When the pump power was gradually increased up to 97.6 mW, a stable pulse train with an increasing repetition rate was observed. This is a typical feature of passive Q-switching. Figure 6 shows the spectral and temporal characteristics of the output Q-switching pulse train when the 980 nm pump power was fixed at 76.6 mW. The output spectrum is depicted in Fig. 6(a), which indicates that the Q-switched laser operates at 1069.4 nm with 3 dB spectral bandwidth of about 1.0 nm and the signal-to-noise ratio of about 40 dB. Figure 6(b) shows the typical oscilloscope trace of the Q-switched pulse trains. The pulse train has the period of 38.3 μs without noticeable timing jitter. Figure 6(c) reveals a typical pulse envelope, displaying an almost symmetrical Gaussian-like shape with a pulse duration of 12.2 μs. The rf output spectrum in Fig. 6(d) shows that the Q-switched pulses have a repetition rate of 26.1 kHz, which agrees with the pulse period of 38.3 μs measured in Fig. 6(b). It is also observed that the rf signal-to-noise ratio is about 23 dB with no spectral modulation, indicating that the passively Q-switching operation was stable.

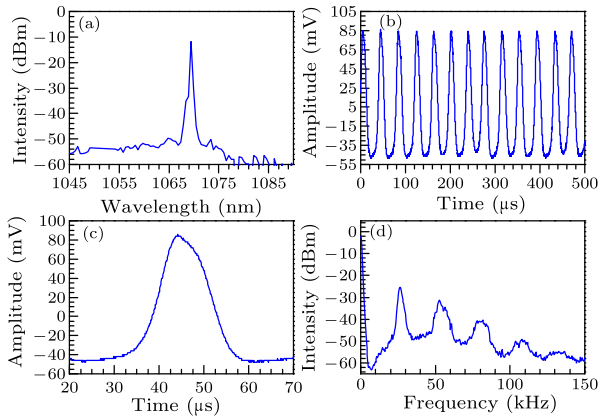

Fig. 6. Spectral and temporal characteristics of the output Q-switching pulse train at the pump power of 76.6 mW: (a) output spectrum, (b) typical pulse train, (c) typical single pulse envelope, and (d) rf spectrum.

Figure 7 shows the recorded pulse repetition rate and the pulse duration as a function of the pump power. The repetition rate of the Q-switching pulses linearly increases from 8.1 to 32.9 kHz as the pump power is increased from 55.1 to 82.0 mW. However, the repetition rate reduces as the pump power is further increased before the pulse train becomes unstable, and disappears as the pump power is increased above 97.6 mW. Meanwhile the pulse duration significantly decreases from 17.9 to 10.8 μs as the pump power is increased from from 55.1 to 97.6 mW. The pulse duration could be further narrowed by optimizing the parameters such as shortening the cavity length and improving the modulation depth of the BP Q-switcher. It is observed in Fig. 8 that both average output power and pulse energy almost linearly increase with the input pump power up to the pump power of 97.6 mW. The maximum average output power and pulse energy are 10 mW and 328 nJ, respectively. The experiment is also repeated for different locations of the BP tape sample. It is found that a small deviation of tape or BP layer thickness does not change the optical properties of the BP SA and the Q-switching performance. We believe that the pulse energy produced by the laser could be further improved by optimizing the SA parameters of BP tape and the cavity design. The output pulse energy could be further improved if the cavity loss and SA loss can be further improved. The similar performance of Q-switching is also obtained in the erbium-doped fiber laser (EDFL) and the thulium-doped fiber laser (TDFL) setups, which operates at 1550 nm and 1920 nm, respectively. This shows that the proposed BP SA can be used as a wideband SA.

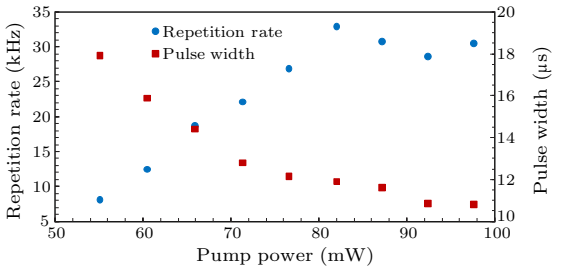

Fig. 7. Repetition rate and pulse width of the proposed Q-switched YDFL against the pump power.

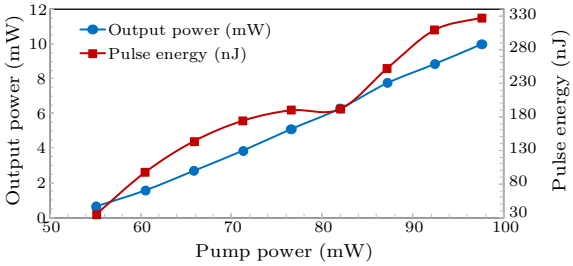

Fig. 8. Output power and pulse energy of the proposed Q-switched YDFL against the pump power.

In conclusion, we have experimentally demonstrated that a multi-layer BP tape can be used as an effective SA for passive Q-switching. The BP tape is prepared by the mechanical exfoliation method and sandwiched between two fiber ferrules via a fiber adapter to form a fiber-compatible SA. Stable Q-switched pulses at 1069.4 nm are successfully obtained. The laser registers a pump threshold of 55.1 mW and a minimum pulse duration of 10.8 μs. The pulse repetition rate could be varied over a wide range of frequencies, from 8.2 to 32.9 kHz, by adjusting the pump power. The highest pulse energy of 328 nJ is obtained at the pump power of 97.6 mW. Our ex-

perimental results suggest that a multi-layer BP is a promising material for pulsed laser applications.

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