Research article

Han Pan, Hongwei Chu*, Zhongben Pan, Shengzhi Zhao, Ming Yang*, Jianwei Chai, Shijie Wang, Dongzhi Chi and Dechun Li*

Large-scale monolayer molybdenum disulfide (MoS₂) for mid-infrared photonics

<https://doi.org/10.1515/nanoph-2020-0331>

Received June 16, 2020; accepted October 19, 2020;

published online October 29, 2020

Keywords: DFT calculation; MIR pulsed laser; monolayer MoS₂; nonlinear optical properties; saturable absorber; Z-scan measurement.

Abstract: Mid-infrared (MIR) photonics has attracted tremendous interest because of its broad applications at atmospheric windows. In this work, we report high-performance MIR photonics based on large-scale and good-quality monolayer molybdenum disulfide (MoS₂). The open-aperture Z-scan measurement on the nonlinear saturable absorption features shows that the as-grown monolayer MoS₂ possesses a modulation depth of 26% and a low saturable intensity of 271 kW/cm², enabling its application as an excellent saturable absorber for the MIR pulse generation. This is further evident by the measured high effective nonlinear absorption coefficient β_{eff} (−16 cm/MW). In addition, the two-photon absorption coefficient and the nonlinear refractive index of monolayer MoS₂ are also determined by the closed-aperture Z-scan technology. As an application, we demonstrate a passively Q-switched Tm,Ho:CaLu_{0.1}Gd_{0.9}AlO₄ (Tm,Ho:CLGA) disordered crystal laser at 2.1 μm by using the monolayer MoS₂ as the saturable absorber for the first time, producing a minimum pulse width of 765 ns and a pulse repetition rate of 36 kHz. Our results demonstrate that large-scale monolayer MoS₂ is a promising candidate for the MIR photonic applications.

1 Introduction

The mid-infrared (MIR) spectrum ranging from 2 to 20 μm has diverse applications such as bio-photonics and medical photonics, absorption spectroscopy, environmental monitoring and security, and free-space communications [1]. Among these applications, owing to the important atmospheric windows and strong fundamental absorptions of molecular species, the MIR absorption spectroscopy is of great interest in civilian and military domains [2]. In such a spectroscopy, because the fundamental absorption and vibration bands of most molecules appear in the MIR region, detecting with the MIR sources is of more sensitivity and precision [2]. For real applications, MIR lasers with high power and high energy are in urgent need to achieve a sensitive and precise detection. Thus, the advent of the MIR optoelectronic and electronic devices is highly desirable for next-generation photonic applications.

It is well known that the nonlinear optical materials play an essential role in the advanced photonics as the fundamental components [3–10]. In recent years, the two-dimensional (2D) materials have emerged as promising candidates for the applications in the MIR region [11–15] because of their excellent electronic and optoelectronic properties and broadband nonlinear optical absorption. Among these 2D materials, transition metal dichalcogenides have drawn plenty of attention [16–19], as they are stable and have a direct band gap in the visible light range in a monolayer form. In particular, monolayer molybdenum disulfide (MoS₂) has demonstrated promising application in transistors, photocatalysis, solar cells, and other photonic applications [20–23]. For example, MoS₂ 2D layers have been successfully used as the nonlinear photonic devices to Q-switch and mode-lock the MIR lasers at 2–3 μm [24–32]. In these photonic applications, the device performance is strongly dependent on the quality and size of the MoS₂ layers

***Corresponding authors: Hongwei Chu** and **Dechun Li**, School of Information Science and Engineering, Shandong University, Qingdao 266237, China; and **Ming Yang**, Institute of Materials Research and Engineering, A*STAR, Singapore 138634, Singapore, E-mail: hongwei.chu@sdu.edu.cn (H. Chu), dechun@sdu.edu.cn (D. Li), yangm@imre.a-star.edu.sg (M. Yeng). <https://orcid.org/0000-0002-1231-2381> (H. Chu)

Han Pan and Shengzhi Zhao, School of Information Science and Engineering, Shandong University, Qingdao 266237, China

Zhongben Pan, Institute of Chemical Materials, China Academy of Engineering Physics, Mianyang 621900, China

Jianwei Chai, Shijie Wang and Dongzhi Chi, Institute of Materials Research and Engineering, A*STAR, Singapore 138634, Singapore

[33, 34]. The large-scale MoS₂ monolayer with good quality is highly desired as the nonlinear saturable absorber to integrate with the optical devices, which, however, is still a challenging issue.

In the present work, we report nonlinear properties and modulation characteristics in the MIR spectral band by using the large-scale monolayer MoS₂. The nonlinear linear optical characteristics were studied by exploiting the open aperture Z-scan techniques, which show a modulation depth of 26%, a low saturable intensity of 271 kW/cm², and a high effective nonlinear absorption coefficient β_{eff} of -16 cm/MW. All these results reveal that the monolayer MoS₂ is an excellent saturable absorber for the MIR pulse generation and an appealing nonlinear linear optical photonic device to modulate the optical pulses. Using the monolayer MoS₂ as the nonlinear optical saturable absorber, a passively Q-switched Tm,Ho:CLGA disordered crystal laser was realized operating at 2.1 μm in the MIR region with a recorded shortest pulse duration (765 ns) for the first time.

2 Monolayer MoS₂ preparations, characterizations, and simulation

The good-quality monolayer MoS₂ thin film was grown by physical vapor deposition (PVD) on the c-plane sapphire substrate (2-inch in diameter) using the sputtering process as documented in our previous works [34–37]. Figure 1 displays the characterization of the sputtering deposited 2-inch monolayer MoS₂. From the atomic force microscopy image (Figure 1(A)), we can see that the MoS₂ film is continuous and smooth in a large scale with a small root mean square roughness of ~ 0.21 nm. In addition, the thickness of the as-grown MoS₂ films was also measured. To expose the substrate, we used a knife to scrape off the MoS₂ film. The raised parts marked by two dashed lines in Figure 1(B) are caused by the MoS₂ stacked on the edge during the scratching process. Figure 1(C) shows the height profile of the MoS₂ film on the sapphire substrate. The unusual height bump is caused by the stacking of materials on the scratched edge. It can be seen that the thickness of the as-grown MoS₂ film was about 0.8 nm, suggesting the single layer of MoS₂. The X-ray photoelectron spectroscopy in Figure 1(D) shows the core-level peaks of Mo 3d_{3/2} (~ 233 eV), Mo 3d_{5/2} (~ 229.6 eV), and S 2s (~ 227 eV), which are the typical peaks of tetravalent Mo⁴⁺ and divalent S²⁻ in 2H-MoS₂ semiconductor [38]. We also do not observe the Mo suboxidation states from the Mo 3d core-level spectra. The thickness of the sputtered MoS₂ film can be accessed by

using Raman spectra. As shown in Figure 1(E), the relative shift between A_{1g} and E_{2g} is about 20.1 cm⁻¹, indicating the dominant monolayer MoS₂ on the sapphire substrate [22]. The sputtered monolayer MoS₂ shows strong photoluminescence (PL) emission, as shown in Figure 1(F). As the PL emission is usually determined by exciton for the low-dimensional materials, the direct band gap of monolayer MoS₂ should be smaller than the dominant emission peak of 1.87 eV. The SEM images of the monolayer MoS₂ thin film is also shown in Figure 1. The white straight line in Figure 1(G) is the substrate scratched with a knife, which is beneficial to observing the morphology of the MoS₂ film more clearly through comparison. Figure 1(H) is the SEM image after further increasing the magnification. It can be seen from both SEM images that the as-grown monolayer

Figure 1: Characterizations of the deposited monolayer molybdenum disulfide (MoS₂) on the sapphire substrate. (A) Atomic force microscopy (AFM) image, (B) AFM height image of the as-grown MoS₂ thin film, (C) line scan across the MoS₂ thin film and exposed substrate interface, (D) X-ray photoelectron spectroscopy (XPS) core-level spectra of Mo 3d, (E) Raman spectra, (F) Photoluminescence spectra, and (G and H) SEM images.

MoS₂ film features the excellent uniformity. All these characterizations confirm the good quality of the deposited large-scale monolayer MoS₂ on the sapphire substrate.

To further investigate the band gap of monolayer MoS₂, we also simulated the band structure of monolayer MoS₂ with density functional theory calculation based on Vienna ab initio Simulation Package (VASP) code. The projected augmented wave pseudopotentials are used, as implemented in the VASP. In the calculations, Perdew–Burke–Ernzerhof of generalized gradient approximation function is used for exchange–correlation interaction. The cut-off energy of plane-wave basis for calculation was set as 400 eV. Optimization of geometric structure was relaxed until the total energy and maximum residual force are 10^{-6} eV and 0.02 eV/Å. K-point meshes of $14 \times 14 \times 1$ have been used. Figure 2(A) displays the model of monolayer MoS₂. The result is shown in Figure 2(B), which indicates that the direct band gap of monolayer MoS₂ is about 1.8 eV. The simulated result is in good agreement with the PL spectrum.

An open-aperture Z-scan technology was exploited to measure the nonlinear saturable absorption (SA) features of the monolayer MoS₂ at 2 μm . The laser source used in nonlinear absorption measurements is a home-made Q-switched laser with a pulse width of 50 ns at a repetition rate of 3 kHz. A lens with a focal length of 200 mm is used as a focusing device. Then the sample is put behind the lens, moving along the z-axis. With the different location of the sample on the z-axis, the pump laser intensity on the sample will change accordingly. To avoid the thermal effect on the sample, a pulsed laser with a low frequency and a low power is used in our case, which can efficiently reduce the heat accumulation on the MoS₂ film [39], and the low power density incident on the MoS₂ also decreases the influence of thermal-induced nonlinearity. In addition, the focal length of the used lens is large, which can cause the slow beam caustic to reduce the heat accumulation. Moreover, the large size of the uniform monolayer MoS₂ film benefits the thermal diffusion.

As Figure 3(A) shows, the normalized transmission curve exhibits a symmetrical peak. The maximum peak

presents a strong nonlinear SA in the MoS₂ at the focus ($Z = 0$). An effective nonlinear absorption coefficient β_{eff} was introduced to characterize the nonlinear absorption, which can be expressed as follows [10, 40]:

$$T = \sum_{m=0}^{\infty} \frac{[-q_0(z, 0)]^m}{(m+1)^{1.5}}, \quad m \in \mathbb{N} \quad q_0(z, 0) = \frac{\beta_{\text{eff}} L_{\text{eff}} I_0}{(1 + z^2/z_0^2)} \quad (1)$$

where L_{eff} is the effective length, I_0 represents the on-axis peak intensity, and z_0 is the Rayleigh range. Therefore, the nonlinear absorption coefficient β_{eff} was estimated as $-16 \pm 0.5 \text{ cm/MW}$, which is comparable with the previous works [41]. The relationship between the transmission of MoS₂ saturable absorber and incident laser intensity was recorded as displayed in Figure 3(B). The measurement data were fitted by the equation as following [9–11]:

$$T = 1 - \Delta T \exp(-I/I_s) - T_{\text{ns}}, \quad (2)$$

in which, ΔT represents the modulation depth, and I_s and T_{ns} represent the SA intensity and the nonsaturable losses, respectively. The modulation depth, SA intensity, and nonsaturable losses of 26%, 271 kW/cm², and 7% were obtained, respectively. Note that the monolayer MoS₂ materials feature a direct band gap of 1.87 eV, which is much larger than the photon energy at 2 μm . Therefore, the SA response of monolayer MoS₂ at 2 μm comes from the defect energy levels and subbandgap generated by the edge effect [28, 41, 42]. In comparison with the previous results shown in Table 1, the monolayer MoS₂ thin film used in our work exhibits stronger nonlinear optical response, such as larger modulation depth and lower saturation intensity. This indicates that the as-grown monolayer MoS₂ by the PVD method could be a potential saturable absorber for Q-switched lasers around 2 μm . In comparison with the few-layered MoS₂, the monolayer MoS₂ possesses more point defects because of the stronger quantum confinement effect caused by size reduction and more non-stoichiometric ratio. These point defects including atomic vacancy defects and antisite defects will form a large number of defect energy levels, which may result in a strong nonlinear SA to mid-infrared lasers. In addition,

Figure 3: (A) Normalized transmission versus the position at the light peak intensity of 1.25 MW/cm² and (B) the intensity-dependent nonlinear saturable absorption of single-crystalline monolayer molybdenum disulfide (MoS₂) at 2 μm.

Table 1: Comparisons of saturable absorption properties of molybdenum disulfide (MoS₂) saturable absorber in the mid-infrared (MIR) band.

λ (nm)	Saturable absorber	Modulation depth (%)	Saturable absorption intensity (MW/cm ²)	Ref.
2840	Few-layered MoS ₂	4.2	—	[27]
2021	Few-layered MoS ₂	21	0.8	[28]
1977	Few-layered MoS ₂	7.2	—	[30]
2090	Large-sized monolayer MoS ₂	26	0.27	This work

compared with few layers, the in-band relaxation rate in monolayer is increased because of defect-assisted scattering [43, 44], and the defects of the monolayer MoS₂ will cause a direct state with a local band gap, reducing the influence of the phonon effect [45]. All abovementioned factors will benefit the modulation performance of monolayer MoS₂ as a saturable absorber for MIR lasers.

To further investigate the nonlinear optical property of monolayer MoS₂, we increased the laser intensity to observe the optical limiting phenomenon. The decreased transmittance at high intensities is due to the two-photon absorption (TPA) effect, which leads to the reversed SA

(RSA) prevailing over the SA behavior. Considering the effects of both SA and RSA, the experimental data are fitted by the equation as follows [46]:

$$T = \left[1 - \frac{\alpha_0 L I_s}{I_s + I_0 / (1 + z^2 / z_0^2)} - \beta_{\text{TPA}} L I_0 / (1 + z^2 / z_0^2) \right] / (1 - \alpha_0 L) \quad (3)$$

here, I_s is the SA intensity, z_0 is the diffraction length of the beam, and β_{TPA} is the TPA coefficient. The normalized transmittance curve of monolayer MoS₂ is shown in Figure 4(A). The TPA coefficient of monolayer MoS₂ at 2 μm was also obtained as $-31 \pm 0.8 \text{ cm/MW}$.

Furthermore, a closed-aperture (CA) Z-scan measurement was also implemented to determine the nonlinear refractive index. For the CA Z-scan, an aperture is placed in front of the sample to confine the transmitted beam incident on the detector. The nonlinear refractive index (n_2) is estimated by fitting the normalized transmittance from the measurement data with the following equation as follows [46]:

$$T_{\text{close/open}} = 1 + \frac{4(z/z_0)\Delta\phi}{((z/z_0)^2 + 9)((z/z_0)^2 + 1)} \quad (4)$$

where $\Delta\phi \cong n_2 I_0 k d$ represents the nonlinear phase shift induced in the samples with n_2 , I_0 , k , and d , denoting the nonlinear refractive index, peak intensity, wave-vector, and the thickness of the sample, respectively. Figure 4(B)

Figure 4: (A) Reversed saturable absorption (RSA) phenomenon at the light peak intensity of 2.35 MW/cm² (B) The results of closed-aperture (CA) Z-scan measurements. Lines: fitting curves; dots: normalized transmission data.

displays the normalized transmittance obtained by CA Z-scan measurement. The nonlinear refractive index n_2 of monolayer MoS₂ at 2 μm is $-25 \pm 0.6 \times 10^{-4} \text{ cm}^2 \text{ MW}^{-1}$.

3 MIR photonic device: saturable absorber

In this section, we would like to address the MIR photonic application of the monolayer MoS₂ as a saturable absorber to produce short MIR laser pulses. The laser cavity was a compact plane-concave resonator with a length of 15 mm, as shown in Figure 5. The input mirror M1 (radius of curvature: 200 mm) was coated for high reflectance at 2.1 μm and antireflectively coated for the pump wavelength at 794 nm. The gain medium was a polished *c*-cut Tm,Ho:CaLu_{0.1}Gd_{0.9}AlO₄ (Tm,Ho:CLGA) disordered crystal. In the experiment, the laser crystal was mounted into a water-cooled copper sink cooled at 15 °C. The large-scale

monolayer MoS₂ was placed near the output coupler (OC) as the saturable absorber to generate the short MIR pulses. The OC was a plane mirror with a $T = 1\%$ at 2.1 μm . The pump source was a commercial fiber-coupled diode laser emitting the radiation at 794 nm (Coherent Inc., USA). The diameter and the numerical aperture of the coupled fiber were 400 μm and 0.22, respectively. The pump beam was focused into the gain crystal with a waist of 100 μm by a 1:1 optical imaging system. A longpass filter was put behind the OC to block the residual pump beam. The pulse temporal behavior was recorded by a DPO 7104C digital phosphor oscilloscope with a high-speed photodetector. A laser power meter was used to measure the average output power (Figure 5).

First, we investigated the free running of the Tm,Ho:CLGA disordered crystal laser without the monolayer MoS₂ as the saturable absorber. The threshold absorbed pump power for the lasing was 2.38 W. The maximum output power was 86 mW, with a slope efficiency of 4.3%, as shown in Figure 6(A). When the monolayer MoS₂ was inserted into

Figure 5: An illustration of the passively Q-switched Tm,Ho:CLGA laser with large-size monolayer MoS₂ as a saturable absorber.

Figure 6: (A) The output power and (B) the pulse duration and the pulse repetition rate versus the absorbed pump power; (C) typical temporal pulse profile and (D) the stable pulse train at an absorbed pump power of 4.26 W.

the laser resonator working as the saturable absorber, the stable passive Q-switching operation can be achieved by slightly aligning the mirrors. In this case, the threshold power for the laser operation is slightly increased to 2.7 W, while the stable Q-switching operation starts at 2.98 W. Under the highest absorbed pump power of 4.26 W, the maximum output power for the passive Q-switching operation is 56 mW, corresponding to a slope efficiency of 3.3%. As Figure 6(B) shows, the pulse duration decreases monotonically from 4 μ s to 765 ns, while the pulse repetition rate increases from 15 to 36 kHz. Once the output power, the pulse duration, and the pulse repetition rate were given, the pulse energy and the peak power can be estimated. The corresponding highest pulse energy and the maximum peak power are 1.55 μ J and 2.03 W at an absorbed pump power of 4.26 W, respectively. The temporal pulse profile and stable pulse train with the pulse width of 765 ns at the repetition rate of 36 kHz are displayed in Figure 6(C) and (D), respectively. From the recorded pulse train, the pulse peak-to-peak fluctuation is estimated as 4.6% in the RMS error method, demonstrating a stable Q-switching operation.

The comparisons of the Q-switched laser performance at around 2 μ m between our work and previous reported works have also been summarized in Table 2. The good laser performance is mainly attributed to the strong SA properties of monolayer MoS₂, such as large modulation depth and low saturable intensity. The resonator and saturable absorber can be further optimized to have a better performance, such as a shorter pulse duration and higher pulse energy. In addition, during the laser experiments, the monolayer MoS₂-based SA worked stably always even at a higher pump power, which indicated its relative high damage threshold.

Table 2: Comparisons of the Q-switched laser performance at 2 μ m with previous reported works.

Crystal	λ (nm)	Saturable absorber	Pulse width (ns)	Single pulse energy (μ J)	Ref.
Tm:GdVO ₄	1902	Few-layered MoS ₂	800	2.08	[26]
Tm,Ho:YAP	2129	Few-layered MoS ₂	435	5	[28]
TDFL	2032	Few-layered MoS ₂	1760	1	[29]
Tm:CLNGG	1979	Few-layered MoS ₂	4840	0.72	[30]
Tm,Ho:CLGA	2090	Large-sized monolayer MoS ₂	765	1.55	This work

MoS₂, molybdenum disulfide.

To make sure the laser is working in the MIR region, the oscillating wavelength of the monolayer MoS₂ saturable absorber Q-switched Tm,Ho:CLGA disordered crystal laser was recorded by an IR spectroscopy meter (Avantes, the Netherlands). As Figure 7(A) shows, the peak of oscillating wavelength locates at $\sim$ 2090 nm with a full-width at half-maximum (FWHM) of 20 nm. Compared with the broad spontaneous emission spectrum of Tm,Ho:CLGA disordered crystal ($\sim$ 300 nm) [47], the linewidth of MoS₂-based Q-switched laser is much narrower, which indicates that the stable laser operates in the constructed solid-state laser device [48]. Moreover, the beam caustic versus the position is shown in Figure 7(B). From the experimental data, the beam quality M^2 from the monolayer MoS₂ saturable absorber Q-switched Tm,Ho:CLGA laser at 2.1 μ m can be estimated as <1.1 under the highest absorbed pump power of 4.26 W, indicating good optical properties of the large-size monolayer MoS₂-based saturable absorber.

It is worth mentioning that the monolayer MoS₂ possesses higher thermal conductivity, which benefits the thermal diffusion [49]. Meanwhile, owing to the horizontal extension of materials in large-sized MoS₂, the MoS₂ around the beam will help reduce the heat accumulation of the MoS₂ illuminated by the beam. Both factors make the large-sized monolayer MoS₂ good thermal stability, which ensures good modulation performance. On the other hand, the excellent uniformity of large-sized MoS₂ thin-films ensures the continuity and repeatability of laser experiment. What's more, under the premise of ensuring uniformity, large-size MoS₂ thin films can break through the limitations of the manufacturing process to achieve large-scale preparation and tunable thickness, which is also very important for promoting the application of MoS₂ nanofilms in optoelectronic fields.

4 Conclusions

In conclusion, we have demonstrated the application of large-scale monolayer MoS₂ for the MIR photonic applications. The PVD monolayer MoS₂ shows a large modulation depth of 26% and a low saturable intensity of 271 kW/cm², indicating that the monolayer MoS₂ is an excellent saturable absorber for the MIR pulse generation. The effective nonlinear absorption coefficient β_{eff} is measured to be -16 cm/MW. Furthermore, the TPA coefficient and the nonlinear refractive index of monolayer MoS₂ are also determined as -31 ± 0.8 cm/MW and $-25 \pm 0.6 \times 10^{-4}$ cm² MW⁻¹, respectively. Based on the large nonlinear absorption coefficient and modulation depth, we demonstrate a passively

Figure 7: (A) The working spectrum of the single-crystalline monolayer molybdenum disulfide (MoS₂) Q-switched Tm,Ho:CLGA laser and inset: spontaneous emission spectrum of Tm,Ho:CLGA crystal; (B) the beam quality M^2 factor at the absorbed pump power of 4.26 W.

Q-switched Tm,Ho:CaLu_{0.1}Gd_{0.9}AlO₄ (Tm,Ho:CLGA) disordered crystal laser at 2.1 μm by using the monolayer MoS₂ as the saturable absorber for the first time, which shows remarkable performance such as a short pulse width of 765 ns and pulse repetition rate of 36 kHz. Our results unravel that sputtered large-scale monolayer MoS₂ is a promising candidate for the MIR photonic devices.

Acknowledgments: The authors would like to thank Dr. Na Qi and Mr. Daozhi Li from Shandong University for their help in the morphology characterization and the DFT simulation.

Author contribution: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.

Research funding: This work is partially supported by National Natural Science Foundation of China (NSFC) (61575109, 12004213), Fundamental Research Fund of Shandong University (2018TB044), and Foundation of President of China Academy of Engineering Physics (YZJLX2018005). D.C., S.W., J.C., and M. Y. acknowledge the funding support from A*STAR Science and Engineering Research Council PHAROS 2D Program (SERC Grant No. 152-70-00012).

Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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