Nonlinear saturable absorption in antimonene quantum dots for passively Q-switching Pr:YLF laser

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Abstract
Antimonene, a new member of monoelemental two-dimensional crystals composed of antimony atoms, was predicted to possess high stability, as well as outstanding electrical and optical properties. Here, we report on the experimental results of productive, high-quality synthesis of antimonene quantum dots (AQDs) via liquid-phase exfoliation. The obtained AQDs exhibited excellent nonlinear saturable absorption. The modulation depth and the saturable intensity were found to be around 3.9% and 10.02 GW/cm², respectively. By using the AQDs saturable absorber in the 639 nm Pr:YLF laser, stable Q-switching pulsed laser was realized. The maximum output power was 83 mW and the corresponding pulse width was 255 ns with a pulse repetition rate of 227 kHz. These results suggest that AQDs are a promising candidate for nonlinear optical applications.

KEYWORDS
2D materials, Q-switch, quantum dots, saturable absorption

1 INTRODUCTION

Over the past decade, inspired by the experimental discovery of graphene, research interests among graphene-like two-dimensional (2D) materials have soared. Hundreds of 2D materials have been explored theoretically or experimentally, including graphene, hexagonal boron nitride, transition metal dichalcogenides, black phosphorus, layered oxides, etc. Based on the unique and outstanding properties of different 2D materials, numerous
applications have been demonstrated, including electronics, photonics, optoelectronics, spintronics, catalysts, sensors, solar cells, and batteries.\(^{[2–4]}\)

Among these huge 2D material families, mono-elemental 2D materials are rather rare. They are of great importance in fundamental research because of their simple chemical composition, which could avoid many extrinsic effects and reveal the intrinsic properties of 2D materials. Graphene, silicene and germanene are the mono-elemental 2D materials composed of group-IV atoms.\(^{[5]}\) All of them have zero band gaps, which limit their applications in logic switching or light emitting devices. Borophene, a Group-III mono-elemental 2D material, has been synthesized recently and it is also a metallic material. Mono-elemental 2D materials with semiconductor band structure could only be found in the group-V element family. Black phosphorus is a direct band-gap semiconductor with layer-dependent band-gap varying from 0.3 eV to 2 V.\(^{[6]}\) Although it has been proved to be valuable in nanoelectronic and optoelectronic applications,\(^{[7–9]}\) atomically thin BP is severely limited in practical use due to bad environmental stability.\(^{[10]}\)

Other 2D group-V mono-elemental materials such as antimonene have also been explored very recently. Antimonene possesses high stability according to recent theoretical and experimental studies.\(^{[11,12]}\) Though bulk antimony (Sb) is a typical semimetal, it will transform into semiconductor with the band gap of 2.28 eV when thinned to single atomic layer. Since then, more intensive efforts have been devoted to exploring antimonene and its derivatives.\(^{[13,14]}\) Recent studies predicted that antimonene might have various allotropes, which possess intriguing properties, such as high mobility,\(^{[15,16]}\) superior thermal conductivity\(^{[17]}\) and promising spintronic properties.

In recent years, various 2D materials have been widely employed in ultrafast photonics owing to their unique nonlinear optical properties.\(^{[18,19]}\) For examples, 2D titanium disulfide (TiS\(_2\)) exhibits a nonlinear optical saturable absorption from visible to mid-IR wavelengths and 2D-TiS\(_2\) based saturable absorber (SA) for ultrafast pulse generation in fiber lasers and all-optical thresholding devices have been realized.\(^{[20]}\) The passively mode-locked fiber laser using 2D CH\(_3\)NH\(_2\)PbI\(_3\) perovskite based saturable absorber has also been successfully demonstrated.\(^{[21]}\) The nonlinear optical properties of Mxenes (2D transition-metal carbides, nitrides, and carbonitrides) have also been intensively investigated recently and various nonlinear optical applications have been reported.\(^{[22]}\) As a new member of group-V 2D materials, antimonene also exhibits outstanding nonlinear optical properties\(^{[23]}\) and antimonene-decorated microfiber has been employed as SA for the passively mode-locking and Q-switching operation at the telecommunication band.\(^{[24]}\) Compared with other 2D materials based SAs, the major advantages of antimonene-based SAs include the large nonlinear absorption coefficients over broad wavelength range, as well as good stability in ambient condition.

Besides the 2D layered materials, zero-dimensional quantum dot (QD), as another form of nanomaterials, exhibits unique electronic and optical properties owing to the quantum confinement in three dimensions and edge effects. Antimonene quantum dots (AQDs) as a valuable derivative of antimonene have been investigated recently.\(^{[25–27]}\) The spatial self-phase modulation effect (SSPM) based on nonlinear refractive effect of AQDs was investigated, which revealed the great nonlinear optical properties of AQDs. Potential applications in optical switcher, Kerr shutter and beam shaper were also proposed.\(^{[25]}\) Furthermore, nonlinear optical-absorption materials like AQDs could be used as the SA to achieve Q-switching or mode-locked pulsed laser generation. However, to the best of our knowledge, pulsed laser applications based on AQDs have not been reported yet.

Here, we report on the experimental results of high-yield, high-quality synthesis of AQDs from bulk antimony crystal via liquid-phase exfoliation. The synthesized AQDs with average size of 2.6 ± 0.6 nm showed good stability in N-methylpyrrolidinone (NMP). Saturable absorption of AQDs was explored by Z-scan technique, which revealed that AQDs possess a giant nonlinear refractive index of \(\sim 10^{-5}\) cm\(^2\)W\(^{-1}\) at the visible wavelength. As a proof-of-concept demonstration, AQDs were spin-coated on the output mirror of a 639 nm Pr:YLF laser cavity as the saturable absorber and Q-switching pulsed laser generation was obtained. The pulse width and repetition rate were 255 ns, 227 kHz, respectively, with a maximum output power of 83 mW. This work is the first experimental demonstration of pulsed laser generation with AQDs as the saturable absorber, which might pave the way for practical nonlinear optical applications based on AQDs.

## 2 RESULTS AND DISCUSSION

Bulk antimony is a typical layered material with weak interlayer van der Waals interactions (see Figure 1A), but strong intralayer covalent bonds. Liquid-phase sonication has been proved successful in exfoliating 2D materials with great yields.\(^{[28–31]}\) Figure 1A-C show the schematic diagrams of sonication liquid-phase exfoliation. With the help of sonication, huge amounts of few-layer nanosheets were exfoliated from the bulk sample (see Figure 1B). Further sonication could break these nanosheets into pieces and AQDs will be produced under certain conditions (see Figure 1C). The morphology of the samples during the whole exfoliation process was analyzed in
details (see Figure 1D-G). Figure 1D and E show the photograph and scanning electron microscope (SEM) images of the high-quality antimony powder and the layered structure can be observed clearly. X-ray diffraction (XRD) spectrum of antimony powder can be seen in supporting information Figure S1. Few-layer antimonene nanosheets, approximately 40 nm in length, could be found during the exfoliation process (see Figure 1F). At last, the mixture containing flakes, QDs and powder was separated by centrifugation at high speed, producing high-quality AQDs in large quantities (see Figure 1G). Figure 1H shows the AQDs suspension with 50 hours sonication time after 6000 rpm centrifugation. The Faraday-Tyndall effect could still be seen in the colorless AQDs suspension after 30 days (see Figure 1H), which means AQDs are effectively dispersed in NMP with good stability. Figure 1I shows the concentration of AQDs dispersed in NMP at different sonication time (see the Figure S2 for a detailed analysis of the concentration of AQDs). Generally, AQDs concentration increased with sonication time, but the increase slowed down after 50 hours, gradually tending to a constant. By fitting the concentration curve, maximum concentration up to 10.5 µg mL⁻¹ was obtained. Figure 1J shows the absorbance spectra of AQDs dispersed in NMP at different concentrations. The optical absorption spectra acquired from the AQDs show a broad absorption band spanning the UV and NIR regions, which is consistent with the zero band-gap of few-layer antimonene and the broadband nonlinear optical response reported in previous study.⁵²

Transmission electron microscopy (TEM) and atomic force microscopy (AFM) were employed to examine the morphology of the obtained AQDs with 100 hours sonication time. Counted from TEM image in Figure 2A, the average size of AQDs was 2.6 ± 0.5 nm (Figure 2D).
No obvious difference could be found in the average size between the AQDs obtained by 50 and 100 hours sonication times (see Figure S3). The high-resolution TEM (HRTEM) image in Figure 2B reveals the atomic structure of AQDs. The obtained AQDs have layered atomic structure consisting of buckled hexagonal rings as in the β-phase bulk antimony. The topography of the AQDs was characterized by AFM and the results are shown in Figure 2C and E. The measured heights of AQDs in Figure 2C were in the range of 2.3–3.6 nm. According to the statistical AFM analysis of 100 AQDs (Figure 2F), the average thickness was 2.8 ± 0.8 nm. Correspondingly, the number of layers could be estimated to be around 3 ± 1.

Raman spectroscopy was carried out to further study the crystal structure and quality of synthesized AQDs and the results are shown in Figure 3. Two prominent Raman peaks of AQDs can be ascribed to an out-of-plane vibrational mode (A\(_{1g}\)) at 118.5 cm\(^{-1}\), and in-plane vibrational modes (E\(_g\)) at 151.6 cm\(^{-1}\) (see Figure 3A). Compared with the Raman spectrum of bulk antimony, both E\(_g\) and A\(_{1g}\) peak frequencies of AQDs move to the higher wavenumber region (i.e., blue shift), shifted by 7.9 and 4.5 cm\(^{-1}\), respectively. This blue-shift phenomenon is quite similar to the Raman spectra variations of black phosphorus QDs and MoS\(_2\) QDs with thin thickness and small lateral dimensions.[33,34] Furthermore, the Raman spectrum of the same AQDs samples stored in ambient conditions after 3 months has been shown in Figure 3B. The E\(_g\) and A\(_{1g}\) peak positions remain the same even at the maximum resolution of the Raman spectrometer. The surface chemical composition of the AQDs was determined by X-ray photoelectron spectroscopy (XPS) after the sample had been cleaned to remove any surface contaminants)As shown in Figure S4 in supporting information(. The AQDs show the 3d\(_{3/2}\) and 3d\(_{5/2}\) doublets at 528.6 and 538.0 eV, respectively, which are the characteristics of antimony. There are also O element signals in the XPS spectrum, which results from
the surface oxidation of AQDs during sample preparation. However, this inevitable surface oxidation will not ruin the excellent nonlinear optical properties of AQDs according to the following experimental studies.

In order to study nonlinear optical absorption properties of AQDs, the open aperture Z-scan measurement was carried out. The experimental setup is shown in Figure 4A. The incident laser pulses were produced by a femtosecond laser and the average power was controlled with help of optical attenuators. Then two different laser beams were formed by beamsplitter mirror. One laser beam as the reference beam was monitored by power meter 1, while the other laser beam was focused by an objective lens into the sample and further monitored by power meter 2.

Figure 4B shows the Z-scan results of the AQDs that were obtained by 690-nm femtosecond laser (175-fs) at 4.9 and 7.2 GW cm$^{-2}$ excitation intensities, respectively. According to the NLO theory, the optical absorption satisfies

$$\frac{dI}{dz} = -\alpha(I)I$$  \hspace{1cm} (1)

where $z$ is the propagation distance in the samples, and the total absorption coefficient $\alpha(I)$ consists of a linear absorption coefficient $\alpha_0$ and a nonlinear absorption coefficient $\alpha_{NL}$:

$$\alpha(I) = \alpha_0 + \alpha_{NL}I$$  \hspace{1cm} (2)

Based on Equation (2), the relationship between the normalized transmittance $T$ and the coordinate $z$ is derived by M. Sheik-Bahae et al.\cite{35}

$$T(z) = \sum_{m=0}^{\infty} \left[ -q_0(z) \right]^m (m + 1)^{3/2}$$  \hspace{1cm} (3)

$$q_0(z) = \alpha_{NL} I_0 L_{eff} / \left( 1 + z^2/z_0^2 \right)$$  \hspace{1cm} (4)

$$L_{eff} = \left( 1 - e^{-\alpha_0 L} \right) / \alpha_0$$  \hspace{1cm} (5)

where $T(z)$ is the normalized transmittance, $I_0$ is the peak on axis intensity at focus, $z$ is the position of sample with respect to the focal position, $z_0$ is the Rayleigh length of the beam, $L_{eff}$ is the effective length and $L$ is the length of the sample. From Equation (3) to (5), the normalized transmittance can be written as\cite{36}

$$T \approx 1 - \frac{\alpha_{NL} I_0 L_{eff}}{2^{3/2} \left( 1 + z^2/z_0^2 \right)}$$  \hspace{1cm} (6)

We fitted the Z-scan data in Figure 4B using Equation (6). The average value of $\alpha_{NL}$ is found to be about $-3.0 \times 10^{-3}$ cm/GW at different peak intensities. In our experiments, the plane mirror made of CaF$_2$ did not exhibit nonlinear optical effect under strong laser pulse, thus the contribution of $\alpha_{NL}$ only comes from the nonlinear absorption of AQDs. The imaginary part of the third-order NLO
TABLE 1 SA Results for different two-dimensional materials

| Materials | λ [nm] | $\alpha_{NL}$ [cm GW$^{-1}$] | $\text{Im}\chi^{(3)}$ [esu] | $I_{s}$ [GW cm$^{-2}$] | Reference |
|-----------|--------|-----------------------------|-------------------------------|------------------------|-----------|
| Graphene  | 800    | $-1.52\times10^{-2}$        | $-8.7\times10^{-15}$          | 583                    | [38]      |
| MoS$_2$   | 800    | $-4.6\times10^{-3}$         | $-2.52\times10^{-15}$         | 413                    | [37]      |
| MoSe$_2$  | 800    | $-2.54\times10^{-3}$        | $-1.45\times10^{-15}$         | 590                    | [38]      |
| MoTe$_2$  | 800    | $-3.7\times10^{-3}$         | $-2.13\times10^{-15}$         | 217                    | [38]      |
| BP        | 800    | $-3.4\times10^{-3}$         | $-4.90\times10^{-15}$         | 148                    | [39]      |
| AQDs      | 690    | $-3.0\times10^{-3}$         | $-4.43\times10^{-15}$         | 10.02                  | This work |

susceptibility, $\text{Im}\chi^{(3)}$ is directly related to $\alpha_{NL}$:[37]

$$\text{Im}\chi^{(3)} = \left[ \frac{10^{-7} c \alpha_{NL} \lambda^2}{96\pi^2} \right]$$

(7)

where, $c$ is the speed of light, $\lambda$ is the wavelength of the incident light, and $n_0$ is the linear refractive index. Here, we calculated the linear refractive index of antimonene at 690 nm according to Zhanget al.[25] Then, we obtained an average value of $\text{Im}\chi^{(3)} \sim -4.43\times10^{-15}$ esu, which is higher than those of some other 2D materials, such as MoS$_2 \sim -2.5\times10^{-15}$ esu,[37] MoSe$_2 \sim -1.45\times10^{-15}$ esu,[38] and MoTe$_2 \sim -2.13\times10^{-15}$ esu,[38] comparable to that of black phosphorus $\sim -4.9\times10^{-15}$ esu.[39] The detailed comparison of the SA properties of different 2D materials is presented in Table 1.

In Figure 4C, we fitted the data of normalized transmittance versus input intensity based on the saturable absorption model for one photon absorption. The transmittance $T$ has a relation with input optical intensity $I$ as:[40,41]

$$T = 1 - A_s \cdot \exp \left( -\frac{I}{I_{sat}} \right) - A_{ns}$$

(8)

Where $A_s$ is the modulation depth, $A_{ns}$ is the non-saturable components, $I_{sat}$ is the saturable intensity, and $I$ is the incident light intensity. The experimental data match well with Equation (8). By fitting the experimental data, the modulation depth and the saturable intensity were extracted to be about 3.9% and 10.02 GW cm$^{-2}$, respectively.

AQDs with large nonlinear optical absorption can be applied as the saturable absorber in passively Q-switching laser. Here, we set up a passively Q-switching Pr: YLF laser to investigate the performance of AQDs SA. The schematic of experiment setup was shown in Figure 5A. A laser diode of 444 nm with maximum power of 1 W was employed as the pump source. A 0.48% at Pr: YLF crystal ($3\times3\times6$ mm) was used as laser gain medium. The 16 mm resonator cavity was composed of a plano-concave lens M1 (R = -100 mm) and 1% transmittance plano lens M2 which was spin-coated with AQDs. Figure 5B shows the average output power versus absorbed pump power for Q-switching operation. At low pump power around the threshold absorbed pump power of 240 mW, the average output power increased rapidly and the generated pulses were unstable since the intracavity spontaneous relaxation. When the pump power exceeded 560 mW, the average output power increased linearly. The slope efficiency of output power versus pump power was 10.4% in our experiment. Figure 5C shows the pulse width and repetition rate versus absorbed pump power for Q-switching operation. When the absorbed pump power increased from the threshold to 800 mW, the pulse width was shortened from 2.1 μs to 255 ns with the corresponding repetition rate of 227 kHz. As shown in Figure 5D, the optical spectrum of the Q-switching laser was measured by a spectrometer and the wavelength was centered at 639.6 nm with 0.2 nm full width at half maximum (FWHM). Figure 5E shows the Q-switching pulse trains and single pulse profile under the absorbed pump power of 800 mW, which confirms the stability of Q-switching operation. To further investigate the stability of Q-switching, we evaluated the quality of pulse-amplitude equalization. This can be characterized by clock amplitude jitter (CAJ) which is defined as the ratio of the standard deviation $\sigma$ to the mean value (M) of the intensity histogram at the pulse peak intensity, as described below:[42,43]

$$\text{CAJ} = \frac{\sigma}{M} \times 100\%.$$  

(9)

The CAJ of the pulse train in Figure S5 (time scale of 800 μs) was calculated to be 6.2%, revealing good intensity stability.

3 CONCLUSION

In summary, we successfully produced a large amount of AQDs by liquid-phase exfoliation method. The saturable absorption of AQDs was assessed firstly. With a lateral size of 2.6 nm and a thickness of about 2.8 nm, the high-quality AQDs exhibited 3.9% and 10.02 GW cm$^{-2}$ in modulation depth and saturable intensity, respectively. The saturable absorber based on AQDs was used in the 639 nm Pr:YLF laser cavity. Stable Q-switching pulsed laser emission was realized and the slope efficiency was up to 10.4%. The maximum output power was 83 mW and the corresponding
pulse width was 255 ns with a pulse repetition rate of 227 kHz. AQDs as new nonlinear optical materials might be applied in pulsed lasers, optical switcher and Kerr shutter applications. Our results suggest that AQDs are a promising candidate for nonlinear optical applications in the future.

### 4 | EXPERIMENTAL SECTION

#### 4.1 | Preparation of AQDs

200 mg of antimony powder was added into 200 mL of NMP in a reagent bottle. Then the bottle enclosing mixture was sonicated in a bath for 10–100 hours at the power of 180 W. The dispersion was centrifuged for 20 minutes at speed of 6000 rpm and then the supernatant containing AQDs was isolated gently.

#### 4.2 | Characterization of AQDs

The AQDs suspension was dropped onto a carbon-coated holey carbon support film with 300 mesh copper grid for TEM measurement or onto a clean Si substrate for Raman measurement. TEM images were taken on an FEI Titan T20, operated at 200 KV, while HRTEM images were taken.
using an FEI Titan 80–300 Cs−corrected electron microscope operated at 300 KV. Raman measurements were performed with a Horiba Labram HR800 Raman spectrometer with an excitation wavelength of 532 nm. The AFM (Bruker Multimode-8) sample was prepared as follows: after the AQDs suspension in NMP was centrifuged at 10000 rpm for 10 minutes, the supernatant was removed and the precipitate was re-dispersed in ethanol (The boiling point of NMP is 203°C). This process was repeated twice and then the collected suspension was dropped onto 300 nm thick SiO2 substrate cleaned with piranha solution (H2SO4/H2O2 = 3:1) for AFM measurement.

4.3 Measurements of nonlinear optical absorption properties of AQDs

The NLO properties of our AQDs samples were measured by Z-scan technique in the femtosecond regimes with linearly polarized 175-fs pulses (80 MHz repetition rate) at 690 nm generated from a mode-locked Nd:YAG laser. The AQDs suspension was dropped onto plane mirror (CaF2). The sample was placed at the focus. The reflected and transmitted pulse energies were measured simultaneously with two power detectors.

4.4 Measurements of Q-switching laser based on AQDs

A laser diode (444 nm) was served as pump source. To avoid the damage of gain medium (Pr:YLF) and reduce the effect of thermal lens, a water cooled system was adopted. In this system, the gain medium was wrapped with indium foil and mounted in a copper holder circulated 21°C water. The compact cavity length is about 16 mm, the laser resonator was composed of a high reflectivity mirror and part transition mirror. The part transition mirror coated with AQDs film was also served as the saturable absorber.

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DATA AVAILABILITY STATEMENT

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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SUPPORTING INFORMATION

Additional supporting information may be found online in the Supporting Information section at the end of the article.

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