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Enhanced nonlinear optical response of layered WSe$_{1.4}$Te$_{0.6}$ alloy in 1 $\mu$m passively $Q$-switched laser

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Due to the composition-dependent properties of two-dimensional (2D) transition metal dichalcogenides (TMDs), alloying of existing dissimilar TMDs architecture is a novel and controllable route to tailor crystal structures with superior optical and optoelectronic properties. Here, we reported the hexagonal-phase WSe$_{1.4}$Te$_{0.6}$ alloy can enable great promise for enhanced saturable absorption response exceeding the parent component WSe$_2$ and WTe$_2$ with larger modulation depth and lower saturable intensity. These advantages allowed the 1064 nm passively $Q$-switched lasers based on WSe$_{1.4}$Te$_{0.6}$ to be more efficient, with pulse duration narrowed to 45%, and slope efficiency increased by 232%. Our findings highlighted the appropriate alloying of TMDs as an effective strategy for development of saturable absorbers.

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Transition metal dichalcogenides (TMDs) have been widely used as all-optical and optoelectronic materials for pulsed laser generation. Exploring novel saturable absorbers (SAs) with more outstanding properties is a conventional strategy, but groping towards a growth process has twists that require cumbersome experimentation and repeated trials to optimize the growth parameters. To circumvent these limitations, the improvement of existing TMDs through material engineering is therefore a very important alternative. Therefore, a number of feasible approaches, such as phase transition under high temperature/pressure, ion intercalation, alloy, defect engineering, have been actively demonstrated to effectively modify and develop the properties of TMDs. To achieve ultra-wide band and optimal application performances, an alloy is desired for tailoring their physical and chemical properties in functional applications. For example, the alloys (Bi$_2$TeSe$_2$, Bi$_2$Te$_2$Se) show lower saturable intensities compared to their undoped counterparts of Bi$_2$Te$_3$ and Bi$_2$Se$_3$. The Mo$_x$Se$_2$Se$_{2(1-x)}$(x = 0.54) nanotubes with expanded interlayer spacing of 0.98 nm exhibit high electrocatalytic hydrogen evolution reaction (HER) activity with a low onset potential of 101 mV and a Tafel slope of 55 mV$^{-1}$.$\overline{[2]}$. Also, 2H WSe$_{1.4}$Te$_{0.6}$ field effect transistor (FET) shows excellent electronic characteristics with effective hole carrier mobility up to 46 cm$^2$.V$^{-1}$.s$^{-1}$ and on/off ratios up to 10$^{6}$.$\overline{[11]}$

Different properties of WSe$_2$ (2H) from the WTe$_2$ (Td) offer a reasonable route to control the band gap and electronic properties of two-dimensional (2D) materials by the alloying technique. The alloy phases have been identified as the 2H phase, the coexistence of the 2H and Td phase, and the Td phase in the complete composition range.$\overline{[11]}$

In this Letter, we systematically investigated the composition-dependent saturable absorption properties among WSe$_2$, 2H WSe$_{1.4}$Te$_{0.6}$, and WTe$_2$. The fundamental parameters, such as modulation depth, saturation intensity, nonlinear optics (NLO) absorption coefficient $\alpha_{NL}$, and third-order NLO susceptibility $\chi^{(3)}$ were obtained by the open aperture Z-scan technique. For pulsed laser generation, we focused on the objective to find the answer to whether SA performance can be boosted by simply integrating two good 2D SAs, such as alloying WSe$_2$ and WTe$_2$ into a new crystal structure WSe$_{1.4}$Te$_{0.6}$. The stable $Q$-switched laser operated at 1064 nm was obtained. A passively $Q$-switched laser based on WSe$_{1.4}$Te$_{0.6}$ was more efficient, with pulse duration narrowed to 45% and slope efficiency increased by 232%. These results demonstrate that alloying between TMDs with dissimilar crystal structures is an interesting strategy for the design of the SAs, which have great research potential in more pulsed laser systems in the future.

To obtain WSe$_2$, WSe$_{1.4}$Te$_{0.6}$, and WTe$_2$ with better quality, the chemical vapor transport (CVT) method with TeCl$_4$ as the transport gas was adopted, as shown in Fig. 1(a). First, WSe$_{2-x}$Te$_{2-x}(x = 2, 1.4, 0)$ polycrystalline samples were synthesized. The 1.5 g highly pure precursor powder of stoichiometric mixture (WSe$_2$: 782 mg W, 718 mg Se; WSe$_{1.4}$Te$_{0.6}$: 726 mg W, 467 mg Se, 307 mg Te; WTe$_2$: 624 mg W, 876 mg Te) was heated to 750°C of solid state reaction in vacuum quartz tubes (8 mm inner diameter, 10 mm outer diameter, 300 mm length) for 48 h. Second, the WSe$_{x}$Te$_{2-x}(x = 2, 1.4, 0)$ monocrystals were grown by the CVT method at a double zone furnace with

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grinded polycrystalline samples. The growth procedure lasted 72 h in an evacuated and sealed quartz ampoule (8 mm inner diameter, 10 mm outer diameter, 300 mm length). The raw material and crystal growth zones were kept at 1040°C and 980°C, respectively. By optimizing the growth parameters, we synthesized high-quality crystals, as shown in Fig. 1.

The WSe1.4Te0.6 alloy was obtained by the doping Te in WSe2, where the parent components were 2H–WSe2 and Td–WTe2, as shown in Figs. 1(b) and 1(c). The crystal structure of WSe2 was hexagonal with space group symmetry P63/mmc, while WTe2 crystallized in a distorted ITd crystal structure with orthogonal symmetry Pmm21. After doping, the WSe1.4Te0.6 alloy with a 2H phase was a hexagonal crystal structure; the top view and side view are shown in Fig. 1(a). We further stripped the as-grown bulk crystals into few-layered WSe1.4Te0.6 nanosheets via liquid phase exfoliation (LPE), where 0.2 mg of grinded monocrystals was added into 4 mL acetone followed by ultra-sonication of 180 W for 20 min and centrifugation at room temperature. The final dispersion liquid was transferred to a CaF2 substrate via 2500 r/min spin coating. The terse ultra-sonication treatment in this work was a general exfoliation approach for large-scale layered nanomaterials, which can avoid the difficulty of removing the interlayered alkali metal ions brought in intercalation LPE, which results in degeneration of optical and electric properties through optical scattering or carrier trapping. Hence, terse exfoliation with ultra-sonication is more suitable than intercalation and other chemical mixing methods to prepare large quantities of pure materials for full investigation on their pure intrinsic properties. WSe2 and WTe2 samples were also treated by the same LPE and transfer processes for further comparison.

The morphology characterizations of the three as-exfoliated WSe2, WSe1.4Te0.6, and WTe2 samples by transmission electron microscopy (TEM), energy dispersive spectroscopy (EDS), and atomic force microscopy (AFM) are shown in Figs. 1(d)–1(l). TEM investigation exhibited an efficient exfoliation via LPE, where most of the samples were transparent few-layer morphology with sharp edges and predominately lateral size up to several micrometers (μm). Three general nanosheets, referring to WSe2, WSe1.4Te0.6, and WTe2, respectively. The elementary compositions of these three nanosheets were determined by EDS analysis. As shown in Figs. 1(g)–1(i), all peaks well matched the X-ray emission energy of W, Se, Te, demonstrating the absence of impurities resulting from crystal growth and exfoliation processes. The ratio of Se:Te for the ternary alloy was calculated to be 1.4:0.6, describing WSe1.4Te0.6. This ratio confirms a semiconductor phase, where WSe2xTe2−x (x~1.2–2) is for the 2H phase, according to the previous phase-transition measurement by Yu et al. TEM and SEAD characterizations of few-layer nanosheets of (d) WSe2, (e) WSe1.4Te0.6, and (f) WTe2. Corresponding full EDS scanning of (g) WSe2, (h) WSe1.4Te0.6, and (i) WTe2. AFM images and thickness measurements of typical nanosheets of (j) WSe2, (k) WSe1.4Te0.6, and (l) WTe2.

Raman spectroscopy was employed to investigate the composition-dependent lattice vibration modes, as shown in Fig. 2(a). The primary mode of WTe2 was the out-of-plane (A1g(Te–W)) vibration located at 216.2 cm−1, while for WSe2, A1g(Se–W) was located at 256 cm−1. It can be seen that A1g(Te–W) and A1g(Se–W) coexist in the WSe1.4Te0.6 spectrum, but shift to high and low frequencies, respectively. It revealed that the interactions between Se and Te atoms soften the Te–W related modes and decrease the corresponding vibration frequency, similar

Fig. 1. (a) Synthesis scheme of bulk crystals by CVT with dual-temperature zones. Photographs and atomic models of the formation of 2H WSe1.4Te0.6. Photographs and atomic models of layer-structure bulks of (b) 2H WSe2 and (c) Td WTe2. TEM and SEAD characterizations of few-layer nanosheets of (d) WSe2, (e) WSe1.4Te0.6, and (f) WTe2. Corresponding full EDS scanning of (g) WSe2, (h) WSe1.4Te0.6, and (i) WTe2. AFM images and thickness measurements of typical nanosheets of (j) WSe2, (k) WSe1.4Te0.6, and (l) WTe2.
The refractive index is an important parameter of optical materials. An ellipsometer is the most direct way to identify the refractive index \( n(\omega) \) of optical film; nevertheless, it has very low spatial resolution because the measurement of film with sub-millimeter (mm) size has proven challenging. Here, we extracted refractive index \( n \) from the transmittance spectra based on the relationship of Kramers–Kronig, reflecting the relation of the real and imaginary parts of complex optical functions \( n(\omega) - 1 = \frac{1}{\pi} \int_0^\infty \frac{k(\omega') - k(\omega)}{\omega'^2 - \omega^2} d\omega' \), where \( k(\omega) \) is the extinction coefficient. It should be noted that limited transmittance spectra cannot satisfy an infinite set. Thus, a physical model is needed to extrapolate the finite transmittance spectra. We chose the Lorentz–Drude model to obtain an infinite transmittance spectrum by fitting the transmittance spectrum. The measured transmittance spectra and corresponding fitted lines based on the Drude model are shown in Fig. 2(b). There are two absorption peaks of WSe\(_{1.4}\)Te\(_{0.6}\) labelled as A, B located at the wavelength of 765 nm and 528 nm in the range of 380–900 nm, which coincides with interband excitonic transitions. A, B result from spin-orbit splitting of transitions. Also, WSe\(_2\) has two absorption peaks located at C, D. The refractive index shown in Fig. 2(c) is calculated by the relationship of Kramers–Kronig based on the fitting lines in Fig. 2(b). We perform our Tauc fit of the dependence of \( (\alpha \hbar \omega)^{0.5} \) on \( \hbar \omega \), as shown in Fig. 3(a). The optical gaps of WSe\(_{1.4}\)Te\(_{0.6}\) and WSe\(_2\), determined by the intercepts of extrapolations with the abscissa axis, are 1.28 and 1.88 eV shown with the light lines. The shortened bandgap of WSe\(_{1.4}\)Te\(_{0.6}\) results from the Te doping in WSe\(_2\), where the doping Te influences the coordination environment of W and its \( d \)–electron counts. Thus, we successfully obtained the tailored physics properties of WSe\(_{1.4}\)Te\(_{0.6}\).

![Fig. 2.](image)

Fig. 2. (a) Raman spectra of few-layer WSe\(_2\), WSe\(_{1.4}\)Te\(_{0.6}\), and WTe\(_2\) nanosheets. (b) Comparison of recorded transmittance spectra and corresponding fitted lines. (c) The refractive index of WSe\(_2\), WSe\(_{1.4}\)Te\(_{0.6}\), and WTe\(_2\) based on the relationship of Kramers–Kronig.

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![Fig. 3.](image)

Fig. 3. (a) Determination of the Tauc optical gap. The spectral dependence of \( \sqrt{\alpha \hbar \omega} \) on \( \hbar \omega \). (b) The open-aperture Z-scan results of WSe\(_2\), WSe\(_{1.4}\)Te\(_{0.6}\), and WTe\(_2\) nanoflakes.

The features of the nonlinear response were compared by open aperture Z-scan measurement with a Ti:sapphire femtosecond optical parametric oscillator (OPO) laser as the exciting source. Figure 3(b) shows the Z-scan results of WSe\(_2\), WSe\(_{1.4}\)Te\(_{0.6}\), and WTe\(_2\) SAs at 1070 nm. The transmittance of the WSe\(_{1.4}\)Te\(_{2-x}\) nanosheets equation is expressed in the form of

\[
T = 1 - A_s \times \exp \left( -\frac{I}{I_s} \right) - A_{ns},
\]

where \( I_s \) is the saturation intensity, \( A_s \) is the modulation depth, and \( A_{ns} \) is the nonsaturable loss. Based on Eq. (1), we fitted the Z-scan data in Fig. 3(b) and obtained \( I_s \) and \( A_s \). According to the NLO fitting parameters in Table 1, the saturation intensity of WSe\(_{1.4}\)Te\(_{0.6}\) is less than those of WSe\(_2\) and WTe\(_2\). Thus, the intensity of ternary WSe\(_{1.4}\)Te\(_{0.6}\) is two orders lower than that of WTe\(_2\). Also, the modulation depth of WSe\(_{1.4}\)Te\(_{0.6}\) is 3.5 times deeper than that of the binary WTe\(_2\) \( I_s \) of the WSe\(_{1.4}\)Te\(_{2-x}\), \(~1\) GW/cm\(^2\) is two orders of magnitude less than in the previous work of MoS\(_2\), \(~100\) GW/cm\(^2\). We obtained a larger saturable response in WSe\(_{1.4}\)Te\(_{0.6}\) nanosheets, resulting from optical bandgap of the alloy covering 1.0 \( \mu \)m. The stronger photon absorption of WSe\(_{1.4}\)Te\(_{0.6}\) SA can increase the number of absorbed photons to produce more electrons at the same laser intensity. Thus, the saturable intensity of WSe\(_{1.4}\)Te\(_{0.6}\) SA is lowered.
Based on the theory of NLO, the propagation equation based on the Beer–Lambert format is shown as

\[
dI/dz' = -\alpha(I) I,
\]

where \(dz'\) is the propagation distance in the sample, and the absorption coefficient \(\alpha(I)\) is expressed as \(\alpha(I) = \alpha_0 + \alpha_{NL} I\). In view of Eq. (2), \(\alpha_{NL}\) of the WSe\(_2\)Te\(_{2-x}\) nanosheets can be obtained. The value of \(\alpha_{NL}\) is \(\sim 10^4\) cm/GW.

The relationship between the imaginary part of the third-order nonlinear optics susceptibility \(\text{Im}\chi^{(3)}\) and \(\alpha_{NL}\) is represented as in Refs. [9, 14]

\[
\text{Im}\chi^{(3)}(\text{esu}) = \left(10^{-7}cn^2/96\pi^2\right) \cdot \alpha_{NL}(\text{cm/W}),
\]

where \(c\) is the speed of light, \(\lambda\) is the laser light wavelength, and \(n\) is the refractive index calculated by the relationship of Kramers–Kronig. The figure of merit (FOM) for the third-order NLO is defined as

\[
\text{FOM} = \frac{\text{Im}\chi^{(3)}}{\alpha_0}.
\]

Fitting the data with Eq. (2), the parameters are listed in Table 1. \(\text{Im}\chi^{(3)} \sim 10^{-6}\) esu is three orders larger than topological insulators Bi\(_2\)Te\(_{3-x}\) SAs of \(10^{-9}\) esu[9]. Compared to previous works, the FOM of WSe\(_{1.4}\)Te\(_{0.6}\) is two orders of magnitude larger than that of graphene, graphene oxide, MoS\(_2\)/N-methyl-2-pyrrolidone (NMP) dispersions \(\sim 10^{-15}\) esu·cm[16]. The FOM value of the WSe\(_{1.4}\)Te\(_{0.6}\) SA was larger than those of WSe\(_2\) and WTe\(_2\) SAs. Thus, the enhanced NLO is achieved at 1070 nm.

To verify whether the enhanced nonlinear absorption by doping strategy can be boosted in the pulsed laser, we compared the three SAs by building passively Q-switched lasers at the wavelength of 1 \(\mu\)m with the gain medium of Nd-doped Y\(_3\)Al\(_5\)O\(_{12}\) (Nd:YAG). As shown in Fig. 4, the laser diode was coupled in a fiber with a core diameter of 105 \(\mu\)m and numerical aperture of 0.22. The pump beam was focused at 105 \(\mu\)m by a doublet lens. The cavity structure was a concave-planar structure consisting of a high-reflectivity input mirror (\(R = 200\) mm) and a part-transmission planar output coupler (\(T = 18\%\) at Nd:YAG).

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1.06 μm). The total cavity length was 12 mm. The gain medium was wrapped by a copper heat sink kept at 21°C by the water. Q-switching operation was realized by inserting the as-prepared SAs — CaF$_2$ plates after increasing the pump power exceeding thresholds.

The obtained average output power, pulse duration, and repetition rate, depending on incident pump power, are shown in Figs. 5(a) and 5(b). As shown in Table 2, the shortest pulse durations of 1.22 μs, 556 ns, and 776 ns for WSe$_2$, WSe$_{1.4}$Te$_{0.6}$, and WTe$_2$ were obtained, respectively, with the corresponding average output powers of 30.3 mW, 82.3 mW, and 50.2 mW, and repetition frequencies of 117.1 kHz, 164 kHz, and 172.1 kHz. It is exciting to note that output power/pulse duration of the alloy, the key properties for Q-switched lasers, is much higher/narrower than those of WSe$_2$ and WTe$_2$. It is worth noting that a WSe$_{1.4}$Te$_{0.6}$ SA can compress laser pulse width to 45% of WSe$_2$. Importantly, the slope efficiency of a WSe$_{1.4}$Te$_{0.6}$ SA is steeper than that of WSe$_2$ and WTe$_2$ SAs, as shown in Fig. 5(a). The slope efficiencies of WSe$_2$, WSe$_{1.4}$Te$_{0.6}$, and WTe$_2$ Q-switched lasers are corresponding to 44.6%, 97.7%, and 75.7%. The slope efficiency is increased to 232% compared to the WSe$_2$ SA. It is well consistent with the lower saturable intensity of the WSe$_{1.4}$Te$_{0.6}$ SA in the Z-scan results. As shown in Fig. 5(b), the pulse durations decrease with the pump power, but the repetition frequencies increase with the absorbed pump power. The optical spectrum of the WSe$_{1.4}$Te$_{0.6}$ SA-based Q-switched laser is shown in Fig. 6. The central wavelength is located at 1064 nm, and the FWHM is about 0.78 nm. The central wavelengths and FWHM of WSe$_2$ and WTe$_2$ SAs were not changed. Thus, Q-switched lasers based on the doping engineering SAs can reduce energy consumption and improve key parameters such as pulse width, slope efficiency, and average output power across a broadband spectral range.

In this work, we have experimentally demonstrated the transition of the nonlinear optical properties from pure WTe$_2$ and WSe$_2$ to the structure of alloying Te into WSe$_2$. By controlling the doping of TMDs, we have achieved improved nonlinear response sensitivity exceeding WSe$_2$ and WTe$_2$ SAs, including large modulation depth and low saturable intensity, which are desirable for pulsed lasers. Compared to the WSe$_2$ and WTe$_2$ SAs, WSe$_{1.4}$Te$_{0.6}$ can lower the laser’s threshold, narrow the pulse duration, and reduce the power consumption. Our promising findings can open up exciting opportunities to research novel materials by alloying between TMDs with dissimilar crystal structures possessing more outstanding properties for optics and optoelectronic applications.

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