Enhanced nonlinear optical response of layered $WSe_{1.4}Te_{0.6}$ alloy in 1 µm 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 WSe₂ and WTe₂, 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^[1,2], ion intercalation^[3,4], $alloy^{[5,6]}$, defect engineering [7,8], 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 (Bi₂TeSe₂, Bi₂Te₂Se) show lower saturable intensities compared to their undoped counterparts of Bi_2Te_3 and $Bi_2Se_3^{[9]}$. The $MoS_{2x}Se_{2(1-x)}(x=0.54)$ 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[10]}. Also, 2H WSe_{1.4}Te_{0.6} field effect transistor (FET) shows excellent electronic characteristics with effective hole carrier mobility up to 46 cm² · V⁻¹ · s⁻¹ and on/off ratios up to $10^{6[11]}$.

Different properties of WSe₂ (2H) from the WTe₂ (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 1Td phase,

and the 1Td phase in the complete composition range^[11]. In this Letter, we systematically investigated the composition-dependent saturable absorption properties among WSe₂, 2H WSe_{1.4}Te_{0.6}, and WTe₂. The fundamental parameters, such as modulation depth, saturation intensity, nonlinear optics (NLO) absorption coefficient $\alpha_{\rm NL}$, and third-order NLO susceptibility ${\rm Im}\chi^{(3)}$ 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 WSe₂ and WTe₂ 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 $WSe_{14}Te_{0.6}$ 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 WSe₂, WSe_{1.4}Te_{0.6}, and WTe₂ with better quality, the chemical vapor transport (CVT) method with TeCl₄ as the transport gas was adopted, as shown in Fig. <u>1(a)</u>. First, WSe_xTe_{2-x}(x = 2, 1.4, 0) polycrystalline samples were synthesized. The 1.5 g highly pure precursor powder of stoichiometric mixture (WSe₂: 782 mgW, 718 mg Se; WSe_{1.4}Te_{0.6}: 726 mg W, 467 mg Se, 307 mg Te; WTe₂: 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_xTe_{2-x} (x = 2, 1.4, 0) monocrystals were grown by the CVT method at a double zone furnace with



Fig. 1. (a) Synthesis scheme of bulk crystals by CVT with dualtemperature zones. Photographs and atomic models of the formation of 2H WSe_{1.4}Te_{0.6}. 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) WSe_{1.4}Te_{0.6}, and (f) WTe₂. Corresponding full EDS scanning of (g) WSe₂, (h) WSe_{1.4}Te_{0.6}, and (i) WTe₂. AFM images and thickness measurements of typical nanosheets of (j) WSe₂, (k) WSe_{1.4}Te_{0.6}, and (l) WTe₂.

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. <u>1</u>.

The WSe_{1.4}Te_{0.6} alloy was obtained by the doping Te in WSe₂, where the parent components were 2H–WSe₂ and Td–WTe₂, as shown in Figs. <u>1(b)</u> and <u>1(c)</u>. The crystal structure of WSe₂ was hexagonal with space group symmetry P63/mmc, while WTe₂ crystallized in a distorted 1Td crystal structure with orthogonal symmetry Pmn2₁. After doping, the WSe_{1.4}Te_{0.6} alloy with a 2H phase was a hexagonal crystal structure; the top view and side view are shown in Fig. <u>1(a)</u>. We further stripped the as-grown bulk crystals into few-layered WSe_{1.4}Te_{0.6}

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 CaF_2 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. WSe_2 and WTe_2 samples were also treated by the same LPE and transfer processes for further comparison.

The morphology characterizations of the three asexfoliated WSe₂, WSe_{1.4}Te_{0.6}, 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-layered morphology with sharp edges and predominately lateral size up to several micrometers (μm) . Three general nanosheets, referring to WSe₂, $WSe_{1.4}Te_{0.6}$, and WTe_2 are represented in Figs. 1(d)-1(f), respectively. Insets show the corresponding selected area electron diffraction (SAED), clearly exhibiting the crystal structure to be monocrystalline 2H, 2H, and Td phases for the obtained WSe₂, WSe_{1.4}Te_{0.6}, 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 $WSe_{1.4}Te_{0.6}$. This ratio confirms a semiconductor phase, where $WSe_{x}Te_{2-x}$ $(x \sim 1.2 - 2)$ is for the 2H phase, according to the previous phase-transition measurement by Yu *et al.*^[11]. The AFM images and corresponding height profiles of WSe₂, $WSe_{1,4}Te_{0,6}$, and WTe_2 nanosheets are shown in Figs. 1(j)-1(l). The thicknesses of WSe₂, WSe_{1.4}Te_{0.6}, and WTe₂ nanoflakes were about 5.16, 5.26, and 5.65 nm, corresponding to 8, 8, and 9 layers.

Raman spectroscopy was employed to investigate the composition-dependent lattice vibration modes, as shown in Fig. <u>2(a)</u>. The primary mode of WTe₂ was the outof-plane ($A_{1g(Te-W)}$) vibration located at 216.2 cm⁻¹, while for WSe₂, $A_{1g(Se-W)}$ was located at 256 cm⁻¹. It can be seen that $A_{1g(Te-W)}$ and $A_{1g(Se-W)}$ coexist in the WSe_{1.4}Te_{0.6} 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. 2. (a) Raman spectra of few-layer WSe₂, WSe_{1.4}Te_{0.6}, and WTe₂ nanosheets. (b) Comparison of recorded transmittance spectra and corresponding fitted lines. (c) The refractive index of WSe₂, WSe_{1.4}Te_{0.6}, and WTe₂ based on the relationship of Kramers–Kronig.

to other alloying ternary TMDs, e.g., $MoS_{2x}Se_{2(x-1)}$ and $WS_{2x}Se_{2(x-1)}^{(\underline{12},\underline{13})}$.

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 $n(\omega) - 1 =$ $\frac{2}{\pi}P\int_0^\infty \frac{\omega'k(\omega')-\omega k(\omega)}{\omega'^2-\omega^2} d\omega', \text{ where } k(\omega) \text{ is the extinction coeffi$ cient^[14,15]. 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-Lorentz 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^[16]. Also, WSe₂ 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. $2(b)^{14}$. We perform our Tauc fit of the dependence of $(\alpha \hbar \omega)^{0.5}$ on $\hbar \omega$, as shown in Fig. <u>3(a)</u>^[17,18]. The optical gaps of $WSe_{14}Te_{0.6}$ 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^[11]. Thus, we successfully obtained the tailored physics properties of $WSe_{1.4}Te_{0.6}$.



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 WSe₂, WSe_{1.4}Te_{0.6}, and WTe₂ 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 <u>3(b)</u> shows the Z-scan results of WSe₂, WSe_{1.4}Te_{0.6}, and WTe₂ SAs at 1070 nm. The transmittance of the WSe_xTe_{2-x} nanosheets equation is expressed in the form of $\frac{9.16.19.20}{2}$

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

where I_s is the saturation intensity, A_s is the modulation depth, and $A_{\rm ns}$ 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 $WSe_{14}Te_{0.6}$ 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_xTe_{2-x} , $\sim 1 \text{ GW/cm}^2$ is two orders of magnitude less than in the previous work of MoS_2 , ~100 GW/cm^{2[16]}. We obtained a larger saturable response in $WSe_{1.4}Te_{0.6}$ nanosheets, resulting from optical bandgap of the alloy covering 1.0 µm. The stronger photon absorption of WSe₁₄Te_{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.

Sample	n	$lpha_{ m NL}(10^4~{ m cm/GW})$	$\text{Im}\chi^{(3)}(10^{-9} \text{ esu})$	$FOM(10^{-13} \text{ cm} \cdot \text{esu})$	$I_s({ m GW/cm^2})$	$A_s(\%)$
WSe ₂	23	-1.35	-22.87	25.41	0.53	3
$WSe_{1.4}Te_{0.6}$	24	-1.60	-29.26	25.73	0.08	7.8
$WTe_{2.}$	12	-1.50	-6.46	5.97	4.43	2.2

Table 1. Results of Z-scan Measurements at 1070 $\rm nm$

Table 2. Parameters of WSe_xTe_{2-x} Saturable Absorbers Based *Q*-switched Lasers

Parameter	WSe_2	WTe_2	$\mathrm{WSe}_{1.4}\mathrm{Te}_{0.6}$
Pulse duration (ns)	1220	776	556
Repetition frequency (kHz)	117.1	172.1	164
Average power (mW)	30.3	50.2	82.3

Based on the theory of NLO, the propagation equation based on the Beer–Lambert format is shown as $\stackrel{[9,16]}{=}$

$$dI/dz' = -\alpha(I)I,$$
(2)

where dz' is the propagation distance in the sample, and the absorption coefficient $\alpha(I)$ is expressed as $\alpha(I) =$ $\alpha_0 + \alpha_{\rm NL} I$. In view of Eq. (2), $\alpha_{\rm NL}$ of the WSe_xTe_{2-x} nanosheets can be obtained. The value of $\alpha_{\rm NL}$ is ~10⁴ cm/GW. The relationship between the imaginary part of the third-order nonlinear optics susceptibility $\text{Im}\chi^{(3)}$ and $\alpha_{\rm NL}$ is represented as in Refs. [9,14] $\text{Im}\chi^{(3)}(\text{esu}) =$ $(10^{-7} c\lambda n^2/96\pi^2) \cdot \alpha_{\rm NL} ({\rm cm/W})$, where c is the speed of light, λ 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 FOM = $|\text{Im}\chi^{(3)}/\alpha_0|$. 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 $Bi_2Te_xSe_{3-x}$ 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 $\sim 10^{-15} \, \text{esu} \cdot \text{cm}^{[16]}$. The FOM value of the $WSe_{1,4}Te_{0,6}$ SA was larger than those of WSe₂ and WTe₂ 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 µm with the gain medium of



Fig. 4. Experimental setup of the LD pumped passively Q-switched laser.

Nd-doped $Y_3Al_5O_{12}$ (Nd:YAG). As shown in Fig. <u>4</u>, the laser diode was coupled in a fiber with a core diameter of 105 µm and numerical aperture of 0.22. The pump beam was focused at 105 µm 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



Fig. 5. (a) Output power with increasing incident pump powers. (b) Variations of pulse repetition rates and pulse duration with increasing pump powers. (c) Typical temporal pulse trains of *Q*-switched lasers. (d) Single pulse traces.



Fig. 6. Optical spectrum of a $WSe_{1.4}Te_{0.6}$ Q-switched laser.

1.06 μ m). 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 – CaF₂ 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 $1.22 \ \mu s$, 556 ns, and 776 ns for WSe₂, WSe_{1.4}Te_{0.6}, and WTe₂ 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 WSe₂. Importantly, the slope efficiency of a $WSe_{1,4}Te_{0,6}$ SA is steeper than that of WSe_2 and WTe₂ SAs, as shown in Fig. 5(a). The slope efficiencies of WSe₂, WSe_{1.4}Te_{0.6}, and WTe₂ *Q*-switched lasers are corresponding to 44.6%, 97.7%, and 75.7%. The slope efficiency is increased to 232% compared to the WSe₂ 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 WSe₂ and WTe₂ 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 WSe₂ and WTe₂ SAs, including large modulation depth and low saturable intensity, which are desirable for pulsed lasers. Compared to the WSe₂ and WTe₂ 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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