Nonlinear optics of MXene in laser technologies

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Abstract

Recently, two-dimension transition metal carbides, carbonitrides, and nitrides called MXenes have attracted huge attention due to their outstanding physical properties in various fields. Here we highlight the enormous potential of MXenes as nonlinear optical materials in the laser technologies, which includes ultrafast laser pulse generation, laser frequency modulation based on four-wave mixing, and photonic diodes with time reversal symmetry breaking. Emphasis is placed on the wide spectral applicability of metallic MXene saturable absorbers in passively mode-locked femtosecond laser technologies, allowing for operation in the visible to mid-infrared range. The Z-scan analysis reveals that Ti$_3$C$_2$T$_x$ MXene has a large nonlinear optical absorption coefficient and a negative nonlinear refractive index, and notably it also possesses the higher threshold for light-induced damage with 50% increase in nonlinear transmittance. The MXene/C$_{60}$ stacked modules enable the achievement of nonreciprocal transmission of laser pulses for photonic rectification. We strongly believe that MXenes will play a significant role in advanced laser technologies such as optical information processing, molecular spectroscopy, and micromachining, while providing many insightful electro-optical applications.

1. Introduction

Nonlinear optics is one of the most intensively studied research areas in modern technologies [1]. It deals with the interaction of light with matter in which the response of materials to the light is nonlinear with respect to the magnitude of the electromagnetic field. The nonlinear optical materials can induce many intriguing physics in the laser technologies, which include four-wave mixing, saturable absorption, and optical rectification [2–5]. Saturable absorption indicates that the absorption of light decreases as the intensity of incident light increases. Soon after the infant use of dye saturable absorbers (SAs), semiconductor SA mirrors (SESAMs) have been prevailing used as the passive modulators to produce ultrashort laser pulses with high peak power through Q-switching and/or mode-locking [6]. Recently, a large amount of research efforts has been made to the study of 2D materials as novel SAs due to their excellent nonlinear optical absorption properties and wider spectral applications [7–22]. So far, graphene, 2D topological insulators (TIs), 2D transition metal dichalcogenides (TMDCs), and phosphorene, have witnessed their promising potential in this application. The latest researches indicated that tellurene and Palladium selenide can be used as SAs for large-energy mode-locked performance and air-stable operation, respectively [23, 24].

MXenes is an emerging class of 2D materials which are transition metal carbides, carbonitrides, and nitrides [25, 26]. The chemical formula of MXenes is denoted as M$_{n+1}$X$_n$T$_x$ (n = 1–3) where M is an early transition metal, X is carbon and/or nitrogen, and T$_x$ represents surface chemical termination (–OH, –O, or –F) [27]. Each of X layers in MXene is covered by M layers in the form of [MX]$_n$. MXenes have outstanding physical properties in various fields across electromagnetic interference shielding [28], electrochemical capacitors [29, 30], chemical catalysts [31], composite reinforcement [32], biosensors [33], photothermal energy conversion [34], quantum dots [35], and photothermal therapy [36] and/or detection.
[37], 2D materials like graphene have been extensively studied for water purification applications [38, 39] and MXene has been shown to exhibit superior performance in water membrane technology [40].

Recently, in addition to such intriguing properties, the excellent nonlinear optical absorption performance of MXenes was discovered and passively mode-locked femtosecond pulse lasers were readily generated using MXene SAs, opening the avenue to nonlinear photonics based on MXene 2D materials [10]. Through continued researches, the notable nonlinear optical performance of MXenes in various applications was indeed evidenced, suggesting insightful MXene-based photonic devices [41–50]. Particularly, ultrafast pulse laser can be produced using MXene SAs [10], whose signal-to-noise ratio and frequency scope were increasingly enhanced to 70 dB and 3000 nm by continued studies [47, 49].

This article summarized the recent progress in the nonlinear optical studies of MXenes and introduced novel photonic devices formed thereby. The following second section describes the saturable absorption performance of metallic MXenes and mode-locked femtosecond fiber and/or free space laser systems based on MXene SAs. The third section provides the detailed nonlinear optical behaviors of MXenes using an open and/or closed aperture Z-scan method. The fourth section presents the research for photonic diodes by combining SAs and reverse SAs and the performance of manufactured devices based on them.

2. Metallic MXene saturable absorbers for ultrafast laser pulse generation

The properties of metallic Ti$_2$CT$_x$, Ti$_3$C$_2$T$_x$, Ti$_3$CNT$_x$, and Ti$_4$C$_3$T$_x$ are similar to each other and these metallic MXenes are the most popular members of MXene 2D material family. Recently, the promising nonlinear optical absorption was observed in Ti$_3$CNT$_x$ and ultrafast mode-locked and/or Q-switched pulse lasers were successfully produced using Ti$_3$CNT$_x$ SAs in the fiber cavity [10].

Generally, MXene can be manufactured by a chemical etching of Al layers of MAX materials using hydrofluoric acid (HF) [27]. Then, as-prepared stacked MXenes are delaminated by sonification with adding tetrabutylammonium hydroxide (TBAOH) to obtain a solution of mono- to few-layer MXenes.

The MXene surfaces are terminated with chemical functionalities of hydroxyl groups (–OH), oxygen (–O), and fluorine (–F). The typical structure of MXene monolayers is illustrated in figure 1(a). The structure of the Ti$_3$CNT$_x$ deposited side-polished fiber module is shown in figure 1(b). The minimum insertion loss and polarization-dependent loss of the Ti$_3$CNT$_x$ SA module were 4.5 and 1.8 dB, respectively, at the wavelength of 1560 nm. The insertion loss means the loss produced by the SA module added to the laser system. It varies depending on the laser polarization and the polarization-dependent loss indicates the difference between the minimum and maximum insertion losses. The insertion loss of the Ti$_3$CNT$_x$ SA module was comparable to the reported values of some other 2D material SA modules based on microfibers or side-polished fibers [22, 51, 52].

A transmission curve of Ti$_3$CNT$_x$ was measured using a picosecond pulsed laser with a wavelength of 1560 nm and a repetition rate of 22 MHz (figure 1(c)). The following formula was used for the analysis [53]:

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

where $T(I)$ is the transmission, $\Delta T$ is the modulation depth, $I$ is the input pulse energy, $I_{sat}$ is the saturation energy, and $T_{ns}$ is the nonsaturable loss. In order to test the mode-locking performance of Ti$_3$CNT$_x$ for femtosecond laser pulse generation, an erbium-doped fiber laser incorporated with a Ti$_3$CNT$_x$ SA module was constructed and then characterized (figure 2(a)). The mode-locked laser pulses were readily produced at a pump power of 22 mW exhibiting an average laser output power of 0.05 mW. The temporal width of the output pulses was measured as 660 fs (figure 2(d)) using a two-photon absorption based autocorrelator. These results clearly showed that Ti$_3$CNT$_x$ SAs can serve as excellent mode-lockers in the laser technologies. This pulse width is in the same order of magnitude as the values obtained from the best TMDC SAs (MoS$_2$, WS$_2$, and WTe$_2$) and one order of magnitude shorter than those of the other TMDC SAs (table 1).

The time-bandwidth product of 0.4 shows that the laser pulses were slightly chirped. One important implication of the above study is that metallic MXene SAs could be operative at extremely wide spectral range due to the metallic characteristics, especially for the long wavelength laser pulses such mid-infrared and/or terahertz ones. Q-switched laser pulses were also shown to be produced using Ti$_3$CNT$_x$ SAs at the wavelength of 2000 nm by (figure 3). Generally, metallic materials are not good saturable absorbers. However, due to their 2D confined nature and fast electronic mobility, MXenes show excellent saturable absorption performance suitable to mode-locking for ultrafast pulse lasers in a broadband frequency range.

Jhon et al indicated that all metallic MXenes such as Ti$_2$CT$_x$, Ti$_3$C$_2$T$_x$, and Ti$_4$C$_3$T$_x$ could serve as predominant SAs with ultra-broadband operation [10]. It was actually demonstrated that Ti$_3$C$_2$T$_x$, which is the most famous MXene, is an excellent SA at the broadband spectral range from visible to midinfrared.
wavelengths. Efficient mode-locking was achieved by Ti$_3$C$_2$T$_x$ SAs in the laser cavity and consequently ultrashort laser pulses were successfully produced as described in detail below [41].

In this study, few-layer Ti$_3$C$_2$T$_x$ SAs were incorporated into a ring-cavity Yb-doped fiber for laser mode-locking at the wavelength of 1000 nm (figure 4(a)). Since the cavity was at the normal dispersion, a spectral filter centered at 1064 nm with a bandwidth of 10 nm is inserted in the cavity. The typical oscilloscope trace of the mode-locked laser pulses is shown in figure 4(b). The repetition rate was 18.96 MHz at the pump power of 482 mW. The output power, peak power, and pulse energy were 9 mW, 482 W, and 0.47 nJ, respectively. The central wavelength and bandwidth of optical spectrum were measured as 1065.89 nm and 4.4 nm, respectively, and the pulse width was measured as 480 ps.

The Ti$_3$C$_2$T$_x$ SAs were also used in the same fiber ring cavity except for the use of Er-doped fiber gain medium to obtain 1500 nm laser that is the widely used light source in telecommunication applications. The bandpass filter used in a Yb-doped fiber laser system was not needed in this laser system due to the anomalous dispersion of the fiber cavity. The output power, peak power, and pulse energy obtained at a pump power of 238 mW were measured as 3 mW, 2578.6 W, and 410 pJ, respectively. The typical oscilloscopic train of pulse lasers is shown in figure 5(b) exhibiting the inter pulse time of 137.4 ns, showing a good agreement with the cavity length of 27.2 m. The laser wavelengths of 1550 to 1620 nm correspond to the conventional telecommunication bandwidth.

The autocorrelation trace of the mode-locked pulse is shown in figure 5(d) and using a squared hyperbolic secant fit, the pulse duration was measured as 159 fs. The time bandwidth product was 0.45 indicating that the mode-locked pulses were slightly chirped. This pulse duration is considerably shorter
Figure 2. (a) The schematic of the ring-cavity erbium-doped fiber laser incorporating the stacked Ti3CNTx SAs. Here, PC, EDF, WDM, and LD denote a polarization controller, an Er-doped fiber, a wavelength division multiplexing, and a laser diode, respectively. The measured (b) oscilloscope trace, (c) optical spectrum, (d) autocorrelation trace, and (e) electrical spectrum of the output pulses (SNR: signal to noise ratio, RBW: resolution bandwidth). [10] John Wiley & Sons. [© 2017 WILEY-VCH Verlag GmbH & Co. KGaA, Weinheim].

Table 1. Output performance comparison of mode-locked fiber lasers using various 2D material SAs which include MXene, graphene, phosphorene, Bi2Se3 and Sb2Te3 TIs, and TMDCs at the wavelength of 1.55 µm [10].

| Material   | Threshold power (MW cm−2) | Modulation depth (%) | Output power (mW) | Pulse width (fs) | Refs  |
|------------|---------------------------|----------------------|-------------------|-----------------|-------|
| Graphene   | 0.08                      | 6.1                  | 0.91              | 590             | [12]  |
| Graphene oxide | 4.3                    | 8.6                  | 0.36              | 780             | [13]  |
| Phosphorene | 6.55                     | 8.1                  | NA                | 946             | [15]  |
| Bi2Se3     | 12                       | 3.9                  | 1.8               | 660             | [7]   |
| Sb2Te3     | 106                      | 3.9                  | 0.9               | 449             | [14]  |
| MoS2       | 34                       | 4.3                  | 1.78              | 710             | [16]  |
| MoS2       | 2.02                     | 10.69                | NA                | 960             | [17]  |
| WS2        | 370                      | 2.9                  | 3.9               | 595             | [18]  |
| WS2        | 600                      | 0.95                 | NA                | 1320            | [19]  |
| WS2        | NA                       | 0.6                  | 1.93              | 369             | [20]  |
| MoSe2      | 19.8                     | 0.63                 | 0.44              | 1450            | [21]  |
| MoTe2      | NA                       | 1.8                  | NA                | 1200            | [22]  |
| WTe2       | NA                       | 2.8                  | NA                | 1200            | [22]  |
| Ti3CN      | 45                       | 1.7                  | 0.05              | 660             | [10]  |

than previous values reported in the literatures and rather close to the values of commercial femtosecond fiber lasers. The stability of mode-locked lasers generated in this laser system was confirmed by a high signal-to-noise ratio (SNR > 62 dB) and the absence of jitter in the RF spectrum.

3. Nonlinear optical analysis of Ti3C2Tx MXene

Jiang et al's performed the detailed nonlinear optical analysis of Ti3C2Tx MXene using the Z-scan method [41]. A Ti3C2Tx-NMP solution was dropped onto a clean glass substrate to prepare a Ti3C2Tx film for nonlinear optical characterization.

In the Z-scan measurement, the MXene thin film with the thickness of 220 µm, being significantly smaller than the Rayleigh length, was gradually moved through the focus of the laser beam in the direction of the optical axis. The laser transmittance measured with increasing the laser intensity can provides much information on nonlinear optical properties such as nonlinear absorption, scattering, and refractive index. The nonlinear absorption can include saturable absorption, multiphoton absorption, reverse saturable absorption, and free-carrier absorption, which typically take place in a sub-picosecond scale.
To investigate its potential for broadband nonlinear optical applications, laser sources with the wavelengths of 800 nm, 1064 nm, 1550 nm, and 1800 nm were used as the input for the optical characterization. The open aperture Z-scan measurement showed that the significant nonlinear optical absorption has occurred at all examined wavelengths (figure 6).

The nonlinear optical absorption can take place through the aforementioned four types of mechanisms. By adopting the effective nonlinear absorption coefficient ($\beta_{\text{eff}}$), the absorption coefficient ($\alpha$) can be...
expressed as

\[ \alpha = \alpha_0 + \beta_{\text{eff}} I \]

where \( \alpha_0 \) is the linear absorption coefficient, and \( I \) is the laser intensity. The largest values of \( \beta_{\text{eff}} \) were measured as \(-0.297\), \(-0.206\), \(-0.358\), and \(-0.112\) cm GW\(^{-1}\) at 800, 1064, 1550 nm and 1800 nm, respectively. The imaginary part of the third-order nonlinear optical susceptibility (\( \text{Im}\chi^{(3)} \)) is related with
\( \beta_{\text{eff}} \) using the following formula [54]:

\[
\Im(\chi^{(3)}) = \frac{2\varepsilon_0 c^2 n_0^2}{3\omega} \beta_{\text{eff}}
\]

where \( \varepsilon_0 \) is the vacuum permeability, \( \omega \) is the angular frequency, \( c \) is the vacuum light speed, and \( n_0 \) is the linear refractive index \((n_0 \approx 2)\) [55]. The calculated \( \Im(\chi^{(3)}) \) showed a similar variation trend to that of \( \beta_{\text{eff}} \), being on the order of \( 10^{-21} \text{ m}^2 \text{ V}^{-2} \). The value of \( \beta_{\text{eff}} \) in Ti\(_3\)C\(_2\)T\(_x\) is at the similar level to that of graphene oxide [56] and is two orders of magnitude higher than those of black phosphorus [57] and TMDC MoS\(_2\) [58], indicating the achievement of strong optical switching. We can characterize the nonlinear refractive index of Ti\(_3\)C\(_2\)T\(_x\) MXene using the closed aperture Z-scan method [59].

The normalized transmittance was obtained using the following fitting formula:

\[
T_{\text{norm}}(z) = \frac{1}{1 - 4x/(1 + x^2)^2 \Delta \Phi + 4/(1 + x^2)^3 \Delta \Phi^2}
\]

where \( x = z/z_0 \), \( z \) is the Z-scan displacement, \( \Delta \Phi = 2\pi n_2 I_0 L_{\text{eff}}/\lambda \) is the phase change, \( n_2 \) is the nonlinear refractive index, \( L \) is the physical thickness of the sample, \( L_{\text{eff}} = (1 - \varepsilon_0/\varepsilon_0) / \alpha_0 \) is the effective thickness of the sample, \( I_0 \) is the on-axis intensity at focus, \( z_0 = \pi \omega_0^2/\lambda \) is the Rayleigh length of the Gaussian beam, \( \lambda \) is the wavelength, and \( \omega_0 \) is the beam waist. The peak-valley appearance in the transmittance curves indicates that Ti\(_3\)C\(_2\)T\(_x\) MXene has negative nonlinear refractive indices, which were measured as \(-4.66\), \(-3.47\), \(-4.89\) and \(-9.85 \times 10^{-20} \text{ m}^2 \text{ W}^{-1} \) at 800, 1064, 1550 and 1800 nm, respectively.

The real part of the third-order nonlinear optical susceptibility (Re\(\chi^{(3)}\)) is related with nonlinear refractive index as given below [54]:

\[
\text{Re}\chi^{(3)} = \frac{4\varepsilon_0 c n_0^2}{3} n_2
\]

The \( \text{Re}\chi^{(3)} \) were calculated as \(-6.61\), \(-4.93\), \(-6.93\) and \(-13.9 \times 10^{-22} \text{ m}^2 \text{ V}^{-2} \) at the wavelengths of 800, 1064, 1550 and 1800 nm, respectively. Although the result would vary depending on the specific experimental condition, the measured nonlinear refractive index of Ti\(_3\)C\(_2\)T\(_x\) close to that of graphene.

4. MXene/C\(_{60}\) based photonic diodes

Dong et al demonstrated that Ti\(_3\)C\(_2\)T\(_x\) MXene SAs can provide a photonic diode if it is combined with a reverse saturable absorber (RSA) such as fluorene C\(_{60}\) [42]. The time-reversal symmetry in the electromagnetic wave equation implies the intrinsic reciprocity of light transmission. However, the time-reversal symmetry could be broken when we use a bilayer structured material composed of SA and RSA layers. In other words, while either of SA or RSA exhibits spatially reciprocal light transmission, the transmission would be nonreciprocal if a laser with the input fluence \((F_{\text{in}})\) greater than \(I_0\) propagates in perpendicular to the SA/RSA bilayer.

The bilayer composed of Ti\(_3\)C\(_2\)T\(_x\) SA and C\(_{60}\) RSA was indeed shown to exhibit excellent photonic rectification suitable to photonic diode applications. In the forward direction (Ti\(_3\)C\(_2\)T\(_x\) followed by C\(_{60}\), the pulse laser intensity transmitted through a Ti\(_3\)C\(_2\)T\(_x\) SA will increase if the input fluence \((F_{\text{in}})\) is greater than saturation intensity \((I_0)\). The transmitted laser will be attenuated by a C\(_{60}\) RSA due to the multiphoton absorption. However, the overall response of laser transmission could be higher than linear transmittance if the SA effect overwhelms the RSA effect. On the other hand, in the reverse direction (C\(_{60}\) followed by Ti\(_3\)C\(_2\)T\(_x\)), even though the a pulse laser input with the same magnitude of fluence as that of the forward direction was applied to a C\(_{60}\) RSA, the intensity of the transmitted laser will be smaller than the \(I_0\) due to the multiphoton absorption in C\(_{60}\). Consequently, if it is the case, the transmission of laser through the SA/RSA bilayer can be severely suppressed.

A ’space–time slicing’ model can theoretically reproduce the rectification performance of the MXene/C\(_{60}\) bilayer based on the following light propagation equation:

\[
\frac{dI}{dz} = -\alpha(I) I
\]

without priori assumptions, wherein \( \alpha(I) = \frac{I_0}{1 + I/I_0} \) for Ti\(_3\)C\(_2\)T\(_x\) MXene and \( \alpha(I) = \alpha_0 + \beta I \) for C\(_{60}\) in which \( \beta \) is the two-photon absorption coefficient. This model can describe the transmission behavior of Ti\(_3\)C\(_2\)T\(_x\) MXene/C\(_{60}\) bilayer using experimentally determined parameters such as \( I_0 \) and \( \beta \) but without any free parameters. In the forward bias (figure 7(a)), a 7 ns Gaussian pulse is sliced into multiple parts with a time width of 70 ps. Each of temporal slices of the Gaussian pulse first propagates through SA (sliced in space with
Figure 7. Optical diode action in a Ti<sub>3</sub>C<sub>2</sub>T<sub>x</sub>-MXene/C<sub>60</sub> bilayer. Schematics for (a) forward and (b) reverse bias configurations of the Ti<sub>3</sub>C<sub>2</sub>T<sub>x</sub>-MXene/C<sub>60</sub> bilayer. (c) Nonlinear transmission results obtained from the Z-scan method. [41] John Wiley & Sons. [© 2018 WILEY-VCH Verlag GmbH & Co. KGaA, Weinheim].

1 nm thickness for each slice) and the output intensity is calculated by solving the propagation equation using the Runge–Kutta method.

The transmitted laser pulse from the n<sup>th</sup> spatial slice acts as an input for the n+1<sup>th</sup> spatial slice. At the final MXene layer, the output intensity is calculated by solving the propagation equation. For the reverse bias, the order of solving the propagation equation is reversed. The intensity of laser pulses passed through Ti<sub>3</sub>C<sub>2</sub>T<sub>x</sub> MXene/C<sub>60</sub> bilayer had completely different magnitudes, depending on the bias direction. Nonlinear transmission curves obtained using the space-time slicing model showed a good agreement with the experimental observation of nonlinear optical absorption of a Ti<sub>3</sub>C<sub>2</sub>T<sub>x</sub> MXene/C<sub>60</sub> bilayer (figure 7(c)).

5. Conclusions

The promising potential of MXenes in nonlinear optics has been highlighted by introducing several notable studies of MXene-based laser technologies. It was shown that metallic MXenes, which are typically titanium carbonitrides and carbides, can serve as excellent SAs for mode-locked ultrafast laser generation. Noticeably, the metallic MXenes have proven to have the predominant broadband applicability due to their metallic characteristics. The systematic studies using the Z-scan method revealed that in addition to their outstanding saturable absorption, Ti<sub>3</sub>C<sub>2</sub>T<sub>x</sub> MXene have a large negative nonlinear refractive index and a higher threshold for light-induced damage. Especially, MXene is supposed to have a prominent potential at a long wavelength range in laser technologies, surpassing other 2D materials. It was demonstrated that by employing MXene SAs as nonlinear optical components, photonic rectification operation can be achieved in SA/RSA devices, indicating their promise in developing novel photonic devices based on 2D materials. This review suggests that MXenes could play a remarkable role in various laser technologies beyond the scope described here, which include terahertz photonics, plasmonic applications, and broadband all-optical modulation devices.

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