Ultrafast Terahertz Complex Conductivity Dynamics of Layered MoS2 Crystal Probed by Time-Resolved Terahertz Spectroscopy

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Ultrafast carrier dynamics, including the carrier photoexcitation and relaxation processes, plays an essential role in improving the performance of molybdenum disulfide (MoS2)-based optoelectronic devices. Herein, we investigate the photo-generated carrier dynamics in layered MoS2 crystal using a time-resolved terahertz (THz) spectroscopy. We have analyzed the ultrafast changes of the THz complex photoconductivity deduced from the peak and zero-crossing of THz waveforms. The decay time of the real part of the THz photoconductivity in layered MoS2 crystal is independent with the pump power, while the imaginary part increases with the pump power. We attribute the decay time of the real part to the carrier recombination process via phonon-assistance and the decay time of the imaginary part to the defect-assisted exciton recombination. The peak values of the complex photoconductivity show a trend of saturation with the increase of the pump power because of the many-body effect at high carrier concentration. This work deepens the understanding of the basic ultrafast physical process in MoS2 crystal, which is enlightening for the design of novel optoelectronic devices.

Keywords: layered MoS2 crystal, time-resolved terahertz spectroscopy, ultrafast carrier dynamics, exciton dynamics, terahertz photoconductivity

INTRODUCTION

Transition metal dichalcogenides (TMDs) are burgeoning layered semiconductors with a chemical formula of MX2 (M represents transition metal elements, including Ti, V, Ta, Mo, W, and Re; X represents chalcogenide atoms, such as S, Se, and Te), in which the van der Waals force connects atomic sheets. Because of the superior properties such as high carrier mobility [1], strong optical nonlinearity [2], and high mechanical strength [3], TMDs materials are advancing the development of many optoelectronic devices, including photodetectors [4], light-emitting diodes [5], field-effect transistors [6], solar cells [7], etc. As one of the most typical and important TMDs, molybdenum disulfide (MoS2) has been reported to have some unique properties. For example, MoS2 has an indirect-to-direct bandgap transition when vary the layer number from bulk to monolayer [8]; MoS2 transistor has a high on/off ratio up to 10^4; MoS2 has a strong spin-orbit coupling [9].
Therefore, many novel optical and electrical applications [2, 10, 11] are expected to be realized by MoS2.

Clarifying the carrier dynamics mechanisms of MoS2 is of key significance for developing MoS2-based optoelectronic devices. Compared with the detection techniques that investigate the photoexcited carrier properties in a static state, such as photocurrent spectroscopy [12], photoluminescence spectroscopy [13], and electroluminescence spectroscopy [14], transient absorption spectroscopy based on the optical pump-probe technology is indispensable for studying the ultrafast carrier dynamics mechanisms [15]. Specifically, many valuable conclusions have been achieved on the ultrafast dynamic properties of MoS2. For example, Wang et al. have reported the intervalley transfer, energy relaxation, and recombination of carriers in bulk MoS2 crystal by resolving the dynamic process [16]. Huang et al. demonstrated that the exciton dynamics of monolayer and few-layer MoS2 are remarkably different due to the quantum confinement effect and the surface defect effect [17]. Wang et al. proposed that the defect-assisted Auger relaxation of electron–hole recombination in MoS2 is related to the strong Coulomb interaction and the electron-hole correlation in two-dimensional MoS2 [18].

Compared with the optical pump-probe technology, optical pump-terahertz probe (OPTP) spectroscopy as another important method to probe the ultrafast process, is sensitive to terahertz (THz) conductivity instead of the static conductivity of materials. The sub-picosecond time resolution of OPTP is suitable to study the ultrafast dynamics of carrier, exciton, and phonon. Sood et al. have studied the dynamics of photoexcited carriers in a few-layered MoS2 using OPTP spectroscopy [19]. They find that the fast relaxation time occurs due to the capture of electrons and holes by defects, and the slow relaxation time is related to bounded excitons which prevent the defect-assisted Auger recombination. For comparison, MoS2 bulk crystal is an indirect bandgap semiconductor, which indicates that the position of electrons in the momentum space will change before and after the transition. In order to satisfy the conservation of momentum, there would be a large number of phonons and bounded excitons involved in the ultrafast process. Additionally, the exciton effect has been reported to be significant in TMDs [20, 21]. Therefore, understanding the exciton dynamics in MoS2 crystals is of key importance for the application development.

In this work, we use the OPTP technique to explore the dynamics of photo-induced carriers in layered MoS2 crystal. The complex photoconductivity is calculated from the pump-induced THz amplitude and phase changes. The real and imaginary parts of the photoconductivity is fitted by the exponential model. The real part-related time constant \( \tau_1 \) of \(~80\) ps is independent of the pump power, while the imaginary part-related time constant \( \tau_2 \) increases from \(110\) to \(260\) ps as the pump power increases. The former is explained by the phonon-assisted carrier recombination process and the latter is induced by the defect-assisted exciton recombination. Additionally, with the increase of pump power, the peak values of the real and imaginary parts of the complex conductivity exhibit a trend of saturation, which is attributed to the many-body effect. These results deepen the understanding of carrier dynamics in MoS2 crystals.

### EXPERIMENTAL SECTION

The freestanding layered MoS2 crystal sample (SPI Supplies) is 90 \(\mu\)m in thickness, and its size is approximately \(8 \times 8\) mm. This crystal sample is hexagonal 2H polytype with good crystalline quality as proved from the X-ray diffraction measurement in our previous work [20]. The Raman spectrum (SmartRaman confocal-micro-Raman module) is used to investigate the phonon characteristics of samples. The light source for the OPTP experiment is a Ti:sapphire femtosecond laser, which has a repetition rate of 1 kHz, a central wavelength of 800 nm, and a pulse width of 35 fs. The beam generated by the femtosecond laser is divided into three parts for the THz wave generation, THz wave detection, and optical pump functions [21]. 1) The THz radiation is generated from the air plasma by a two-color method under 800 and 400 nm laser excitation. The generated THz wave was focused onto the sample by a pair of off-axis parabolic mirrors in a transmission configuration. 2) The THz wave is probed by an electro-optic sampling method using a zinc telluride (ZnTe) (110) crystal as the THz detector. A delay line is used to measure the time domain signal of THz electric field \(E(t)\). 3) The pump beam is focused onto the MoS2 sample in a transmission geometry. A pump delay line is used to change the delay time \(t_{\text{pump}}\) between the pump and probe pulses. The sample is measured in a normal incident angle for both the THz wave and the pump laser. All experiments were measured in a nitrogen environment to avoid THz absorption by atmospheric water vapor.

### RESULTS AND DISCUSSION

The Raman spectrum of layered MoS2 crystal under 532 nm excitation is shown in Figure 1A. The peaks \(E_{2g}\) and \(A_{1g}\) are two Raman modes, indicating in-plane and out-of-plane vibrations. The frequencies of these two modes are at around 379.2 and 403.8 m\(^{-1}\), which are consistent with the characteristic peak positions of MoS2 crystal according to previous report [22]. Since there is a close relationship between the photoexcitation process and the band structure of materials, first-principles calculation is performed to study the band structure of MoS2 crystal (The calculation software is Quantum Espresso). The Perdew-Burke-Ernzerh of generalized gradient approximation is used for the exchange-correlation potential. We use the ultrasoft pseudopotential to describe the electron–ion interactions and the ultrasoft pseudopotential incorporate the electron orbital of Mo 4s5p4s5p4d and O 2s2p. The in-plane lattice constants are set as \(a = 3.166\) Å and \(c = 18.41\) Å. Monkhorst–Pack k-mesh of 15 × 15 × 15 is set for sampling the Brillouin zone. The kinetic energy cutoffs of the plane waves for charge density and basis function are set to 25 and 300 Ry. The van der Waals corrections are described by the vdW-DF method. As shown in Figure 1B, the valence band maximum is at the \(\Gamma\)
point, and the conduction band minimum lies at the symmetry point between K and Γ. The transition from valence band maximum to conduction band minimum is indirect [8]. Thus, MoS2 crystal is an indirect semiconductor with a bandgap energy of 1.29 eV. Additionally, there is a direct transition with bandgap energy of 1.9 eV from valence band K point to conduction band K point, which cannot be realized under the 800 nm (1.55 eV) laser excitation.

We will discuss the ultrafast THz complex conductivity dynamics of MoS2 by using the OPTP technique in the following parts. The pump laser power is 20 mW. As shown in Figure 2A, the THz electric-field transmission waveform through the unexcited layered MoS2 crystal \( E_{\text{ref}} \) is labeled as the blue curve, and the photo-induced THz waveform change \( \Delta E \) at pump delay time \( t_{\text{pump}} = 20 \) ps is labeled as the yellow curve. The peak and zero-crossing values of the THz waveform are sensitive to absorption and phase change, respectively [23]. The former represents the real component of the complex conductivity and the latter represents the imaginary component of the complex conductivity. Specifically, the THz waveform changes at the peak and zero-crossing positions as a function of pump-probe delay time depict the photoexcitation dynamics of MoS2 crystal, as shown in Figure 2B. Because the photon energy of the incident light is larger than the bandgap of MoS2 crystal, real carriers will generate after excitation. We can see the THz waveform at both the peak and zero-crossing positions exhibit ultrafast optical response, indicating photoinduced carrier generation and recombination processes.

According to the peak and zero-crossing changes of the THz waveform, the photoconductivity can be calculated by the formula [24]:

\[
\Delta \sigma(t_{\text{pump}}) = -n_{\text{air}} + n_{\text{THz}} \frac{\Delta E(t, t_{\text{pump}})}{E(t_{\text{max}})},
\]

where \( n_{\text{air}} = 1 \) is the THz refractive index of air, \( n_{\text{THz}} = 2.95 \) is the THz refractive index of the MoS2 crystal [22], \( Z_0 = 377 \, \Omega \) is the...
free space impedance, and $d = 90 \, \mu m$ is the thickness of the MoS$_2$ crystal. Using Eq. 1, the photoconductivity of MoS$_2$ with different pump power from 5 to 30 mW can be obtained. The real and imaginary parts of the photoconductivity are shown in Figures 3A,B, respectively. The sub-picosecond abrupt changes observed at $\sim 32$ ps in Figures 3A,B are related to the process that the photo-induced carriers are excited from the valence band to the conduction band. Subsequently, the slow changes in Figure 3 correspond to the relaxation processes of photoconductivity. The real and imaginary parts of the complex conductivity reflect the absorption and chromatic dispersion properties of materials, respectively. According to previous reports, the THz absorption properties of MoS$_2$ are mainly decided by the photo-generated carriers [20], and the THz chromatic dispersion properties of MoS$_2$ could be contributed from the polarization effects of bound charges such as excitons [25, 26]. Here, the binding energy of exciton in MoS$_2$ crystals is approximately 0.1 eV [27], which is larger than the thermal energy ($\sim 25$ meV) at room temperature. Due to the strong Coulomb interactions among carriers, extraordinary exciton effects have been observed in TMDs such as MoS$_2$ [17], WSe$_2$ [21], and WS$_2$ [20]. Therefore, the exciton effect could be important for the imaginary part of photoconductivity of MoS$_2$ crystal due to the polarization effect.

Next, the time constants deduced from the pump delay time dependent complex conductivity are analyzed to reveal the relaxation dynamics of the photoexcited carriers and excitons in MoS$_2$ crystal. The experimental data in Figure 3 are exponentially fitted by $\Delta \sigma_{RE/IM} = \exp((t - t_0)/\tau_{1,2})$, where $\tau_1$ ($\tau_2$) is the time constant of the real (imaginary) part of the photoconductivity. The obtained $\tau_1$ and $\tau_2$ with different pump power are shown in Figure 4A as depicted by blue and yellow dots, respectively. The time constant $\tau_1$ is approximately 80 ps, independent of the pump power. In comparison, the time constant $\tau_2$ increases linearly from 110 to 260 ps with the increase of the pump power. In MoS$_2$ crystal, there are many possible relaxation processes. For the fast relaxation processes with a duration of sub-picosecond or several picoseconds, there are carrier-carrier scattering, carrier-phonon scattering, and exciton-exciton scattering in TMD materials [17, 28]. However, these fast processes cannot be identified from our experiment because of the limited time resolution. The time constant $\tau_1$ and $\tau_2$ can mainly be attributed to the slow relaxation processes. For the time constant $\tau_1$, it has been reported that the phonon-mediated recombination time of free carriers is independent on the pump power in layered WSe$_2$ crystal, monolayer MoS$_2$, and suspended graphene [17, 21, 29]. Hence, the decay time $\tau_1$ could be attributed to the phonon-assisted free carrier recombination. For the time constant $\tau_2$, it has been reported that defect-assisted exciton recombination can result in an increase of decay time with pump power [20, 21]. The Auger processes for exciton capture by defects are believed to be important in most bulk semiconductors with high carrier densities [18]. Therefore, the time constant $\tau_2$ could be governed by the exciton recombination via defect-assisted Auger process.

Additionally, the peak value of photoconductivity related to the carrier quantity has been discussed. Figure 4B shows the pump power dependence of the maximum (minimum) of the real (imaginary) part of photoconductivity. Both the real and imaginary parts exhibit enhanced absolute values with the increase of the pump power, and then present a saturable trend at the high pump power region. Because the photoconductivity is associated with the free carriers and
excitons, the enhancement of the peak values indicates that the quantity of photo-induced carriers and excitons increase with the pump power. Then, equation $\Delta \sigma \propto P / (P + P_s)$ ($P$ is the pump power and $P_s$ is the saturation pump power) [21] is used to fit the experimental data. The saturation pump power $P_s$ of the real and imaginary photoconductivity are 3.6 and 5.7 mW, respectively. These results suggest that both photo-induced carriers and excitons are generated before the pump power of 3.6 mW; then, the photo-induced carriers are saturated and excitons are continuously generated before 5.7 mW; at last, both the carriers and excitons become saturated due to the possible many-body effect at high carrier concentration [30, 31]. The carrier density is calculated as [16]:

$$N_0 = \left[ 1 - \frac{(n_0 - 1)^2 + \kappa_0^2}{(n_0 + 1)^2 + \kappa_0^2} \right] \alpha_0 F_0 / \hbar \omega,$$

(2)

where $n_0 = 4.83$ is the real part of the refraction index, $\kappa_0 = 0.78$ is the imaginary part of the refraction index, and $\alpha_0 = 1.23 \times 10^7$ m$^{-1}$ is the absorption coefficient [22, 32]. The $F_0$ is the peak energy fluence of the pump pulse, which can be calculated by


\[ F_0 = 4 \ln(2) P/(\pi f w^2) \]

where \( P \) is average pump power, \( f = 1 \) kHz is the repetition rate of the laser, and \( w = 2.5 \) mm is the radius of focus pump spot. According to Eq. 2, the numbers of photo-induced carriers are calculated to be 1.95×10^{25}, 3.9×10^{25}, 5.85×10^{25}, 7.8×10^{25}, 9.75×10^{25}, 11.7×10^{25} m^{-2}, with 5, 10, 15, 20, 25, and 30 mW pump power, respectively.

Finally, the frequency-dependent photoconductivity is obtained by the fast Fourier transform of the time-domain signals. The real and imaginary parts of the photoconductivity are measured at the delay time of 5 ps with a variable pump power of 5, 10, 20, and 30 mW as shown in Figure 5. The Drude model can be used to describe the free carrier motion and the Smith term for exciton species. Combined with the Lorentz model (exciton species) to describe the photo-induced complex conductivity. The fitting formula of the Drude-Smith-Lorentz model is described as follows [21]:

\[
\sigma_{D-S-L} = \frac{D_0 \tau}{1 - i\omega\tau} \left( 1 + \frac{C}{1 - i\omega\tau} \right) + \frac{S\omega}{i(\omega_0^2 - \omega^2) + \omega^2} \tag{3}
\]

where \( D_0 \) is the Drude weight, \( \tau \) is the free carrier relaxation time, \( \omega \) is the angular frequency, \( C \) ranging from -1 to 0 is related to the degree of carrier scattering, \( S \) is the oscillator strength, \( \omega_0 \) is the resonant frequency, and \( \gamma \) is the damping coefficient. From Figure 5, the curves calculated with Eq. 3 fit well with the dotted experimental data. The fitting coefficients are given in Table 1. The relaxation time \( \tau \) increases with the increase of pump power. The constant \( C \) has no pump power dependence, suggesting that the carrier backscattering is not affected by the pump power. In addition, the frequency \( \omega_0 \) of the oscillator response has no obvious change with the increase of pump power.

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