Nonexponential Photoluminescence Dynamics in an Inhomogeneous Ensemble of Excitons in WSe₂ Monolayers

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The spectral and spatiotemporal dynamics of photoluminescence in monolayers of transition metal dichalcogenide WSe₂ obtained by mechanical exfoliation on a Si/SiO₂ substrate is studied over a wide range of temperatures and excitation powers. It is shown that the dynamics is nonexponential and, for times \( t > 50 \) ps after the excitation pulse, is described by a dependence of the form \( 1/(t + t₀) \). Photoluminescence decay is accelerated with a decrease in the temperature and in the energy of emitting states. It is shown that the observed dynamics cannot be described by a bimolecular recombination process, such as exciton–exciton annihilation. A model that describes the nonexponential photoluminescence dynamics by taking into account the spread of radiative recombination times of localized exciton states in a random potential gives good agreement with experimental data.

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INTRODUCTION

Atomically thin layers of transition metal dichalcogenides (TMDCs) represent a new class of semiconductor materials that have been actively investigated in recent years [1–9]. These materials include substances with an \( \text{MX}_₂ \) composition, where \( M \) is a transition metal (\( M = W, \text{Mo} \)) and \( X \) is a chalcogen (X = S, Se, Te). Transition metal dichalcogenides acquire unique properties upon a transition from a bulk crystal to a monolayer. While multilayer TMDCs are indirect-gap semiconductors, monolayer TMDCs feature a direct optical transition. The exciton binding energy in TMDC monolayers is about 200–500 meV, so that excitons form the ground state at room temperature (see [6] for a review). The unique properties of these compounds, as well as the possibility of creating heterostructures by combining monolayers of different materials [1, 3], make them promising candidates for application in optoelectronics [2–4].

Transition metal dichalcogenide monolayers are extremely two-dimensional, which, together with a high contrast in the dielectric constants of the monolayer and its environment, leads to the modification of the carrier–carrier interaction potential [10–12]. In addition, TMDCs have an unusual band structure, which is characterized by spin–valley coupling and strong spin–orbit coupling [13]. For this reason, the direct extrapolation of experience gained in the studies of traditional semiconductor systems with quantum wells is sometimes impossible, and there are still many open questions regarding the properties of TMDC monolayers. For example, the mechanisms responsible for the dynamics of photoluminescence (PL) and, in particular, for exciton recombination are still incompletely clear. Nonexponential dynamics in the PL decay and photoinduced reflection or transmission was observed in mechanically exfoliated monolayers directly deposited on a substrate [14–20]. In the cited works, this behavior was attributed to bimolecular recombination processes, in particular, exciton–exciton annihilation, which manifests itself at high pump levels in the initial stage of the decay dynamics. At the same time, exponential dynamics was observed in monolayers encapsulated between hexagonal boron nitride (h-BN) layers [21–23], which was attributed to the suppression of exciton–exciton annihilation [23].

Here, we study the PL dynamics in WSe₂ monolayers placed directly on a Si/SiO₂ substrate. On a long time scale, we observe the nonexponential PL decay that is well described by an inversely proportional relation \( 1/(t + t₀) \) and accelerates with a decrease in the temperature. We show that, contrary to common belief, the observed kinetics is unrelated to exciton–exciton annihilation and can be explained in terms of...
emission from an ensemble of exciton states with an inhomogeneous distribution of PL decay times. The decay times of exciton states feature positive correlation (i.e., increase) with their energy, which is characteristic of an ensemble of localized excitons with a spread in the localization length.

SAMPLES AND EXPERIMENTAL TECHNIQUES

WSe₂ crystals were exfoliated using an adhesive tape and transferred to a Si substrate coated with a 285-nm-thick SiO₂ layer. To more easily locate individual flakes, the substrate was furnished with binary labels made using optical lithography followed by chromium deposition. The initial search for monolayer samples was performed judging by the color of flakes viewed under an optical microscope. The surface topography of the candidates for monolayer flakes was examined using an NT-MDT Solver 47 atomic force microscope (AFM) in the semi-contact mode. Next, the sample was transferred to the setup for studying steady-state PL spectra. Figures 1a and 1b show the optical image and AFM topography of the selected flake, respectively. The lateral size of this flake is about 3 μm. According to AFM data, the step height is about 1 nm. The PL spectra confirm that this flake is a monolayer one.

The main methods for studying the monolayer WSe₂ flakes were steady-state and time-resolved PL in the temperature range of 10–300 K. The sample was placed in vacuum on the cold finger of a helium gas-flow cryostat. To achieve a micron spatial resolution, pump laser radiation was focused on the sample by a micro-objective lens, which was also used for collect-
ing PL. In the steady-state PL measurements, the sample was excited by a CW semiconductor laser with a wavelength of 457 nm. The PL spectra were recorded with a resolution of 0.5 meV using a spectrometer with a silicon CCD matrix cooled with liquid nitrogen. In the time-resolved PL measurements, the sample was excited at a wavelength of 400 nm by the second harmonic of radiation from a pulsed Ti:sapphire laser with a pulse duration of 2 ps. Laser radiation was focused on the sample in a spot with a diameter of 2 μm. Photoluminescence was recorded by a Hamamatsu streak camera combined with a spectrometer. For spectrally resolved and spatially resolved measurements, the spectrometer grating was set to the first or zero diffraction order, respectively. The time and spectral resolutions in these experiments were up to 5 ps and 1.5 meV, respectively.

RESULTS AND DISCUSSION

Figure 1c shows the PL spectra of the WSe₂ monolayer obtained at temperatures of 300 and 10 K. The position and shape of the spectral lines coincide with published data for exciton PL [16, 24–30]. We note that the low-temperature PL spectra reported in [27–30] exhibit additional low-energy lines that are lacking in our case and are, apparently, associated with defects in the prepared layers. We investigate WSe₂ monolayers placed directly on a Si/SiO₂ substrate without h-BN encapsulation. As a result, the spectrum is inhomogeneously broadened even at low temperatures, which makes impossible the separation of neutral and charged exciton contributions to PL [31]. The presence of trion PL is indicated by the asymmetry of the spectral line. Figure 1d shows the temperature dependence of the position and width of the PL line. As the temperature increases, we observe redshift and line broadening associated with a decrease in the band gap and with thermal broadening, respectively.

Figure 2a shows the dynamics of emission from a WSe₂ monolayer at different temperatures upon pulsed laser excitation at a wavelength of 400 nm. The dynamics is nonexponential. It features a pronounced fast stage, after which the decay rate decreases. The initial part of the dynamics is shown in more detail in the inset of Fig. 2a. The fastest component of the dynamics is characterized by a decay time of less than 5 ps. The contribution of the fast component increases (the decay kinetics accelerates) as the temperature decreases. The contribution of the fast component also increases with a decrease in the optical transition energy where PL is detected; this is true for both low and high temperatures (Figs. 2c and 2d, respectively).

The emission dynamics for different excitation powers is shown in Fig. 2b. Qualitative changes in the dynamics are difficult to distinguish: it remains non-exponential for both high and low excitation powers.

TEST OF THE BIMOLECULAR RECOMBINATION HYPOTHESIS

The nonexponential PL dynamics at times $t \gtrsim 50$ ps is well described by an inversely proportional relation $I(t) \sim 1/(t + t_0)$, where $t_0$ is a constant. Fits of kinetic curves by functions of this form are shown in Fig. 2b by red solid lines. For comparison, the dashed line shows the best fit with a biexponential function over the same time interval for a pump power of $P = 3$ mW; however, this curve deviates significantly from the experimental data. The time dependence of the PL intensity of the form $I(t) - 1/(t + t_0)$ was observed in many studies for monolayers of WSe₂ [16, 19, 32], WS₂ [18–20], MoSe₂ [15, 17], and MoS₂ [14, 18, 19]. This dependence was explained by the impact of a bimolecular process requiring the participation of two excitons in recombination. One such process is exciton–exciton annihilation (Auger recombination), whereby one of the excitons recombines nonradiatively by transferring energy to the second exciton, which can dissociate. The dynamics of the exciton concentration $n$ upon bimolecular recombination is described by the equation

$$\frac{dn}{dt} = -Cn^2 - \frac{n}{\tau}. \quad (1)$$

Here, $C$ is the constant that determines the bimolecular recombination rate, and the second (linear) term describes the radiative recombination of excitons with a time constant $\tau$. The solution of this equation is

$$n(t) = (C\tau + 1/n_0) \exp(t/\tau - C\tau) - 1,$$  \quad (2)

where $n_0$ is the initial concentration of excitons. When the contribution of the bimolecular process is dominant, which is the case for $t \ll \tau$ and $Cn_0\tau \gg 1$, the exciton concentration follows an inversely proportional time dependence $n(t) = n_0/[1 + Cn_0\tau]$, whereas in the limiting case of $t \gg \tau$, the exciton concentration decays exponentially: $n(t) = (C\tau + 1/n_0)^{-1}\exp(-t/\tau)$. Note that we observe no transition to exponential decay even for times as long as $t = 600$ ps. Therefore, if the bimolecular process is indeed dominant, the condition $t \ll \tau$ should be valid. In this case, the ratio of PL intensities for two different excitation powers and, correspondingly, different initial exciton concentrations $n_0$ and $\bar{n}_0$ should decrease with time from the value $n_0/\bar{n}_0$ to 1: $\bar{I}(t)/I(t) = 1 + (\bar{n}_0/n_0 - 1)/(C\bar{n}_0\tau + 1)$. The inset of Fig. 2b shows the ratio of two kinetic curves recorded for pump powers differing by a factor of 3. Evidently, this ratio changes only at the initial stage of the kinetics ($t \lesssim 50$ ps) and is constant at longer times in the region where the PL intensity is inversely proportional to the time. By fitting the experimental curves to the dependence $I(t) \sim 1/(t + t_0)$, we can determine the expected bimolecular recombination coefficient $C = -(dn/dt)/n^2$. Its dependence on the excitation power is shown in Fig. 3a. The coeffi-
cient $C$ is plotted in arbitrary units; only its relative change in various experiments has physical meaning. An increase in the excitation power is equivalent to an increase in the initial concentration and should not be accompanied by a significant change in the coefficient $C$. Meanwhile, our measurements show that $C \sim 1/P$. Figure 3b shows the dependence of $C$ on the optical transition energy at two different temperatures. Contradictions appear in this case as well: contrary to our observations, an increase in the energy or temperature should lead to the delocalization of excitons and an increase in the efficiency of the bimolecular process, i.e., an increase in $C$ [33]. Finally, the spatial distribution of excitons should change considerably in the case of bimolecular recombination. Areas with a higher initial exciton concentration should be emptied more rapidly, which should lead to the effective broadening of the spatial distribution of excitons and of the PL spot. The diffusion of excitons should only enhance this broadening. The measured and calculated spatial distributions of PL normalized to the peak value are shown in Fig. 3c for different times. Experi-
mentally, we observe no significant increase in the width of the PL spot, while the calculation predicts considerable broadening, which, however, should be limited by the size of the monolayer flake. Therefore, in our case, the dynamics of the PL decay at times $t \geq 50$ ps and, in particular, the time dependence $I(t) \sim 1/(t + t_0)$ cannot be explained in terms of a bimolecular process and, apparently, has some other nature.

**LINEAR MODEL OF THE NONEXPONENTIAL DYNAMICS**

Since the character of the PL dynamics at $t \geq 50$ ps is independent of the excitation power, the dynamics of the exciton concentration is described by linear equations. In this case, the intensity decreases with time nonexponentially because we observe the intensity of emission from an inhomogeneous ensemble of states where each state exhibits exponential decay but the decay time $\tau$ is different for different states. Then,

$$I(t) = \int_0^\infty n_0(\tau) \exp(-t/\tau) d\tau,$$

where $n_0(\tau) d\tau$ is the concentration of excitons at time $t = 0$ in states characterized by decay times between $\tau$ and $\tau + d\tau$. In particular, to obtain the dependence $I(t) \propto 1/(t + t_0)$, which is close to the experimental one, the function $n_0(\tau)$ should have the form $n_0(\tau) \propto n_0 \exp(-t_0/\tau)/\tau$, where the time $t_0$ corresponds to the maximum of the distribution $n_0(\tau)$ and may be considered as the characteristic time of the PL dynamics. The dependence of this time on the energy of the emitting state at different temperatures is shown in Fig. 3d. This dependence confirms the conclusion that the dynamics slows down with an increase in the energy of the emitting states or temperature.

Nonexponential dynamics associated with the inhomogeneity of recombination times is found in many systems [34–42]. In the vast majority of them, nonexponential dynamics is associated with the need for electron tunneling toward recombination centers or with donor–acceptor recombination. In this case, recombination should speed up with an increase in the temperature, which contradicts our observations. We note that the slowing down of the PL dynamics with increasing temperature was also reported in other studies for WSe$_2$ [43, 44], MoS$_2$ [45], and MoSe$_2$ [44] and, apparently, is general for TMDC monolayers.

Before determining the character of the distribution of decay times $\tau$ and find $I(t)$, let us give a few remarks about the nature of emitting states. The exciton ground state in WSe$_2$ monolayers is dark, while the bright state is $\sim 40$ meV higher in energy [43, 46]. It was shown in [46] that the ground state can nevertheless emit at a nonzero angle to the normal. Presumably, this state determines the PL dynamics, at least at low temperatures. A significant role in the PL dynamics in WSe$_2$ monolayers at low temperatures is played by trion states [44], which are characterized by short PL decay times. Biexciton states can also contribute to PL in monolayer WSe$_2$. In our case, their contribution to the dynamics at $t \geq 50$ ps is insignificant, since the corresponding intensity is a quadratic function of the pump power (see Fig. 2b).

The characteristic recombination time of neutral excitons in TMDCs $\tau_0$ is quite short [33]. An exciton can emit if its total wave vector lies within the light cone $|k| < \omega/c$ (where $\omega$ is the photon frequency) and, therefore, its kinetic energy is close to zero. In thermal equilibrium, the exciton energies are distributed in the range of $\sim k_B T$, and the decay time of the total exciton concentration can be estimated as $\tau \sim \tau_0 k_B T/(\hbar^2 \omega^2/2mc^2) \gg \tau_0$ [47], where $m \approx 0.8 m_0$ is the exciton mass [22] and $m_0$ is the free electron mass. This explains a quite long-term PL dynamics and the increase in the characteristic PL decay time with increasing temperature. In our case of nonencapsu
localized WSe$_2$ on a Si/SiO$_2$ substrate, there is inhomogeneity associated with fluctuations in the potential landscape where excitons move. This inhomogeneity manifests itself in the broadening of the PL spectrum. Potential fluctuations lead to the localization of excitons and spreading of exciton states in $k$ space on the order of $\delta k^2 \sim 1/L^2$, where $L$ is the characteristic localization length (Fig. 4). Then, the radiative lifetime of a localized state is $\tau \sim \tau_n c^2/\omega^2 L^2$. If we assume that the inhomogeneous broadening of the spectrum is caused by the spread in localization lengths $L$, the energy of a localized state (measured from the bottom of the potential well) is $E \sim h^2/2mL^2 \sim (\tau/\tau_0)(\hbar\omega)^2/2mc^2$. Therefore, in a system where inhomogeneity is due to a spread in the size of the localizing potential wells, the characteristic exciton recombination time is

$$\tau/\tau_0 \sim \alpha E,$$

where $\alpha = 2mc^2/(\hbar\omega)^2 \approx 400$ meV$^{-1}$. If not only variations in the size of the localizing potential wells but also variations in their depths are taken into account, there appears a spread in the values of $\tau$ corresponding to the same energy in the spectrum, but the overall trend, described by Eq. (4), remains unchanged. For trion states, there is no requirement that the total wave vector be close to zero, because the momentum can be transferred to the remaining carrier upon recombination. For this reason, the trion PL decay rate is fairly high [44] and trions do not contribute significantly to the dynamics at $t \gtrsim 50$ ps. Anyhow, taking into account trion states that have a short PL decay time and are lower in energy fits into the trend of an increase in the decay time with the energy set by Eq. (4). We note that the increase in the characteristic PL decay time with an increase in the energy of emitting states is confirmed by experimental data (Figs. 2c, 2d).

Let us determine the total PL intensity by summing the intensities $f \exp(-E/k_B T)\exp(-t/\tau)/\tau$ of emission from individual states whose occupancy, under the assumption of thermal equilibrium, is described by the Boltzmann distribution $f \exp(-E/k_B T)$:

$$I(t) = \int_0^\infty \frac{dE}{\tau} \exp\left(-\frac{E}{k_B T}\right) \exp\left(-\frac{t}{\tau}\right) g(E) dE$$

$$= A \int_0^\infty \exp\left(-\frac{\tau}{\alpha\tau_0 k_B T}\right) \frac{t}{\tau} d\tau$$

(5)

Here, we use $\tau/\tau_0 = \alpha E$. $f_0$ is a constant that determines the occupancy; $g(E)$ is the density of states, which, for simplicity, can be assumed constant like that of free-motion states in a two-dimensional system; and $A = f \alpha/k_0$.

The red lines in Fig. 2a show the results of calculations by Eq. (5). These should be compared with the experimental data for the dynamics of the PL intensity at times $t > 50$ ps; it is reasonable to expect that thermal equilibrium is established in the system and rapid nonlinear processes, including those related to bie excitons and to bimolecular recombination, fade out on this time scale. The calculations were performed with the value $\tau_0 = 1.3$ ps, which is in reasonable agreement with the available experimental data on the recombination time of excitons with zero wave vector [33]. Note good agreement of the calculated curves with experimental data. In particular, the calculations yield nonexponential dynamics that slows down with time and is close to $1/(t + t_0)$ and reproduce changes in the dynamics with temperature.

A more complete analysis should include all possible exciton states with different combinations of electron and hole spins, excitons consisting of an electron and a hole occupying different valleys [46, 48, 49], and also trion states [44]. Excitons with different spin or valley structures may not reach thermal equilibrium between them, which further complicates the analysis. However, to explain the experimentally observed long-term PL dynamics at $t \gtrsim 50$ ps, which is nonexponential, slows down with time, is independent of the excitation power, and accelerates with a decrease in temperature, only two conditions have to be met: (i) the observed PL should originate from a set of emitting states characterized by different PL decay times $\tau$, and (ii) there should be positive correlation between the radiative lifetime $\tau$ and the energy $E$ of a given state. Neither a constant density of emitting states nor a linear, nor even unambiguous, dependence $\tau(E)$ is required. On a shorter time scale ($t \lesssim 50$ ps), nonlinear processes such as bimolecular recombination can also make a significant contribution to the dynamics, as noted in [14, 17, 18, 32].

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