SPECIAL ISSUE: Optical Gain Materials towards Enhanced Light-Matter Interactions

Magnetic-brightening and control of dark exciton in CsPbBr₃ perovskite

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ABSTRACT Exploring the fine-structure of cesium lead bromide (CsPbBr₃) perovskite nanocrystals (NCs) is not only vital to fundamental understanding of recombination mechanism of exciton but also crucial for improving the performance of quantum light emitters and spintronic devices. Herein, utilizing low-temperature magneto-photoluminescence (PL) measurement, we provide the direct PL spectral feature of the dark exciton in CsPbBr₃ single crystal, and demonstrate that the singlet dark exciton is located ~20 meV below the triplet bright exciton. Furthermore, no significant polarization effect was measured from magnetic-polarization method, indicating that there is no spin selectivity for dark exciton.

Keywords: perovskite materials, CsPbBr₃, dark exciton, magnetic field, brightening

INTRODUCTION

Over the past few years, lead halide perovskites have been regarded as a sort of revolutionary and groundbreaking materials for the field of optoelectronics, including low-cost solar cells, light-emitting diodes (LED), optical detectors and small lasers [1–14]. In comparison with hybrid organic-inorganic perovskites, all-inorganic perovskites show better stability [15–19]. Among a variety of all-inorganic perovskites, CsPbX₃ nanocrystals (NCs)

with unique properties have attracted wide attention due to the coverage of full-visible spectral range [20]. Specifically, the green-emitting perovskite, CsPbBr₃ NCs demonstrate excellent stability against oxidation and photo-excitation, leading to superior optical properties [21]. Till now, the external quantum efficiency of solution-processed CsPbBr₃-based LED devices has exceeded 20% [22]. At the meantime, CsPbBr₃ perovskites have been regarded as a promising candidate to realize electrically driven laser owing to their excellent optical gain and electronic properties [23,24]. Exploring the fine-structure of the band-edge exciton for semiconductor is vital to understand the radiative recombination mechanism, which is the basis of developing quantum technology [25–27]. Realizing controllable tuning of the fine-structure of exciton will further pave the way for quantum information process. However, in perovskite, the cognition of fine-structure of band-edge exciton is still controversial so far and more experimental efforts are needed to elucidate this issue [28–30].

It is well known that different crystalline structures have different symmetries, which directly influence their physical properties and the energy of the excitonic sub-levels [31,32]. Magnetic-photoluminescence (PL) spectroscopy is a powerful tool to address exciton properties and disclose the fine-structure of exciton [33]. Recently,

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magnetic-dependent PL dynamics spectrum has been studied to validate the existence of dark exciton in CsPbX_3 NCs; a fast-decay and a slow-decay component can be observed, which are assigned to the bright and dark exciton, respectively [34,35]. The abnormal inversion of the dark-bright splitting was also observed in CsPbX_3 perovskite arising from the Rashba effect together with strong spin-orbital coupling (SOC) [36]. In order to clearly distinguish the fine-structure of exciton, it is of great importance to provide a direct PL spectroscopic signature of the dark exciton. The dark exciton has been accurately detected and is several meV lower than the bright exciton in single FAPbBr_3 NC through magneto-PL spectroscopy [30]. However, for CsPbBr_3 perovskite, the direct PL spectral signature of the dark exciton is still lacking.

EXPERIMENTAL METHODS

Sample preparation

High-quality all-inorganic CsPbBr_3 single-crystal perovskite was synthesized by a chemical vapor deposition (CVD) method. In brief, a mixture of $\text{CsBr}/\text{PbBr}_2$ powders and a piece of sapphire (M plane) were placed at the center and the downstream of the heating zone, as reaction sources and deposition substrate, respectively. Before heating, high-purity N_2 gas (600 sccm, 1 sccm = $6 \times 10^{-5} \text{ m}^3 \text{ h}^{-1}$) was introduced to remove the O_2 . Then, the furnace was heated to 600°C and hold for 10 min for the growth of CsPbBr_3 single-crystal perovskite. The gas flow inside the quartz tube was 60 sccm and the pressure was 760 Torr (1 Torr = 1.33322×10^2 Pa).

Optical spectroscopy measurement

The PL signals were recorded by a Witec Alpha 300R Plus Confocal Raman microscope, coupled with a 7 T superconducting magnet and a closed cycle optical cryostat (10 K) on an XY scanning stage. A long working distance $50\times$ objective (Olympus NA=0.45) was used for the low-temperature PL measurements. Polarization-resolved PL spectra were measured upon the excitation by the linearly and circularly polarized 514 nm laser with 10 μW .

RESULTS AND DISCUSSION

Dark exciton in 2D perovskite CsPbBr_3

In this paper, we report magneto-PL investigation of single-crystal all-inorganic CsPbBr_3 perovskite at cryogenic temperature and reveal the magnetically brightened states of the dark excitons. Moreover, polarization-dependent spectroscopy was studied to identify the spin properties of the magnetically brightened dark exciton and no significant polarization effect was observed. Two possible reasons, Pauli exclusion principle and parallel transition dipole moment, have been proposed to explain this phenomenon. These findings will not only help to optimize the model construction of exciton's fine-structure but also improve the performance of quantum light emitters and spintronic devices.

By means of high-pressure CVD method, high-quality all-inorganic CsPbBr_3 single-crystal perovskite has been synthesized with sizes ranging from nanometers to microns (Fig. 1a). And the scanning electron microscopy (SEM) images of CsPbBr_3 with different shapes can be seen in Fig. 1b and c. It is worth noting that various crystalline structures show different symmetries, which is related to the fine-structure of exciton [32]. Hence, the

Figure 1 Optical image and XRD of CsPbBr_3 . (a) Optic microscope picture of CsPbBr_3 NCs (the scale bar is 100 μm); (b, c) SEM images of CsPbBr_3 cubic column and triangular prism NCs (the scale bar is 3 μm); (d) XRD analysis of CsPbBr_3 NCs.

phase of CsPbBr₃ single-crystal was confirmed by X-ray diffraction (XRD) and it is in accordance with cubic phase (shown in Fig. 1d).

In order to reveal the existence of dark exciton, we studied the magneto-optical properties of CsPbBr₃ single-crystal *via* utilizing a home-built scanning confocal microscope system at cryogenic temperature. Fig. 2a demonstrates the magnetic-field-dependent two dimensional (2D) pseudo-color plot of PL spectra at 10 K. A 514 nm continuous-wave laser was selected as the excitation source with the applied magnetic field parallel to the PL collection axis (out-of-plane magnetic field). A new PL peak located below the bright exciton appears with increasing magnetic field from 0 to 7 T. And the same phenomenon can also be observed when the direction of magnetic field is switched. In the system of lead halide perovskite, the origin of the red-shifted emission line can be attributed to either the recombination of dark exciton or charged exciton (trion) [35,37]. In general, at zero magnetic field, the emission line of charged exciton should appear, and Zeeman effect induced two fine-structure lines could be distinguished [37]. However, it is obviously not consistent with our experimental results. Therefore, we assign the red-shifted emission line to the recombination of dark exciton.

Magnetic-brightening dark exciton in CsPbBr₃

Grasping the electronic band structure of perovskite will help us better understand the recombination mechanism of exciton. For CsPbBr₃ perovskite, the octahedral

structure formed with lead halogen atoms plays an essential role in constructing the conduction band minimum (CBM) and the valence band maximum (VBM) [38]. The CBM mainly consists of the p-orbitals of the heavy metal Pb, and the VBM originates mainly from the Br p-orbitals and Pb s-orbitals. The strong spin-orbit interaction splits the conduction band into a 2-fold degenerated state ($J^e=1/2$) and a 4-fold degenerated state ($J^e=3/2$), while the valence band (VB) maintains 2-fold degenerated state ($J^h=1/2$). Here, J represents the total angular momentum. The band-edge exciton is formed from the coulombic interaction between an electron in the split-off state ($J^e=1/2$) and a hole in s -like VB ($J^h=1/2$). And the electron-hole exchange interaction leads to the formation of an optically active bright exciton state ($J=1$, spin-allowed) and an optically inactive dark exciton state ($J=0$, spin-forbidden), respectively [31,39]. Based on the above discussion, a three-level model including a spin-allowed bright state level, a spin-forbidden dark state level and a ground state level, has been proposed to understand the recombination mechanism of exciton [34,40]. As shown in Fig. 2b, in the absence of external magnetic field, the PL only originates from the recombination of bright exciton. However, when an external magnetic field is applied, the dark state is brightened by coupling with one of the bright triplet states combined with an extremely slow bright-dark state relaxation [30,34,40]. Thus, the external magnetic field opens a new radiative recombination channel for the dark excitons *via* mixing the dark state with the bright state (in Fig. 2c). In zero field,

Figure 2 Magnetic brightening of dark exciton. (a) Color plot of the measured emission spectrum for single crystal CsPbBr₃ at 10 K as a function of magnetic field. Emission features from the dark exciton increase with increasing magnetic field. The scale bar represents the normalized PL intensity. (b) In the absence of magnetic field, only electrons in the bright exciton states combine with holes in the VB, leading to a PL emission. (c) In the presence of an out-of-plane magnetic field, the bright exciton states ($J=1$) and dark exciton states ($J=0$) are mixed together with an extremely slow bright-dark state relaxation. Optical transitions are allowed from a dark exciton state to ground state, giving a new red-shifted emission feature.

the bright exciton PL decay is fast; when a magnetic field is applied, a slow dark exciton radiative recombination component appears, and the long-time component drastically shortens with increasing magnetic field [30,34,40], thus the PL intensity of the dark excitons increases with increasing magnetic field.

The signature of the magnetic brightened dark state was further explored by raising the magnetic field from 0 to 7 T. The emission spectra *versus* magnetic field at 10 K are plotted in Fig. 3a. Without the external magnetic field, only one emission peak attributed to the recombination of bright exciton can be observed. With the increasing magnetic field, a red-shifted emission line at ~ 2.31 eV is strikingly detected, which derives from the radiative recombination of dark exciton. Thus, the bright-dark splitting energy of CsPbBr₃ is ~ 20 meV, which is determined by the long-range electron-hole exchange interaction under the combined effect of the screening of the electron-hole interaction and quantum confinement [30,40,41]. In addition, when the magnetic field is switched to the opposite direction, the similar results can be obtained (Fig. 3b). The insensitivity of the dark exciton to the direction of the magnetic field indicates that the Pauli exclusion principle is strictly followed by dark state. Moreover, the PL intensity of dark exciton increases with the increasing magnetic field and shows a nearly linear variation tendency (Fig. 3c), which is probably due to the

enhancement of field-induced oscillator strength of the dark exciton [42]. This result is consistent well with the previous results because of a slow phonon-assisted bright-to-dark relaxation process [30,33,43]. To be sure, under the premise of ensuring the resolution of the instrument, the fine-structure of bright exciton can not be distinguished. It is because that the thermal broadening induced by electron-phonon interaction is larger than the fine-structure splitting energy.

Polarization-dependent emission spectroscopy is regarded as a vital approach to identify the spin properties of the magnetically brightened dark exciton. Magnetic-dependent circularly polarized emission spectra have been measured at 10 K and a 514 nm circularly polarized light is used as the excitation source with the power of 10 μ W. The collected right-handed and left-handed circularly polarized emission spectra in the same direction of magnetic field are shown in Fig. 4. Intriguingly, there is no significant polarization effect and both of the PL intensities increase with the increasing magnetic field. In addition, the emission spectra were recollected by reversing the direction of magnetic field and the results are basically the same as before. In order to eliminate the interference of excitation source, we have further repeated the above experiments while choosing the left-handed circular as the pumping source with the same power, and obtained the similar results.

Figure 3 Magnetic field-dependence of emission from dark exciton. (a, b) PL spectra as a function of magnetic field. The spectra show emission from the bright exciton (2.33 eV) and dark exciton (2.31 eV). (c) PL intensity of emission feature from dark exciton as a function of magnetic field.

Figure 4 Polarization-resolved PL spectra of dark exciton emission vs. magnetic field. (a–d) Polarization-resolved PL spectra upon right-handed (a, b) and left-handed (c, d) circularly polarized light excitation in an out-of-plane magnetic field. The dark exciton emission increases with the increasing magnetic field, but no degree of polarization is observed. (e, f) Right- and left-handed circularly polarized PL intensity upon polarization-resolved light excitation as a function of magnetic field.

For the sake of making the experimental results more intuitive and clearer, under the condition of right-handed circular excitation, we have extracted the relationship between the PL intensity and the magnetic field for right-handed and left-hand circularly polarized emission spectra, respectively, as shown in Fig. 4a and b. In all cases, the same nearly linear tendency has been observed and the intensities of the left-handed and right-handed circularly polarized PL of dark excitons are almost the same, which indicates that there is no significant polarization effect. For left-handed circularly polarized light excitation, similar results can be obtained (see Fig. 4c and d). And the PL intensity ration of the dark excitons upon

right- and left-hand excitation is shown in Fig. 4e and f. Two possible reasons have been proposed to explain this phenomenon. (i) The magnetically brightened dark exciton originates from a coupling effect between the dark state and one of the bright states [30]. When the transition dipole moment of the coupled bright state is parallel to the magnetic field, the emission line has no clear polarization characteristic. Because the polarization properties of dark state are determined by the coupled bright state, the dark exciton shows no polarization effect. (ii) The electrons in the dark energy level strictly follow the Pauli exclusion principle and the VB of perovskite is a 2-fold degenerated state. Considering that the number of

electrons with opposite orientation fall back from the dark state to ground state is equal, therefore there is no helicity parameter. Hence, there is no significant polarization effect for dark exciton.

CONCLUSION

In conclusion, we have investigated the magneto-PL spectra of all-inorganic CsPbBr₃ single-crystal at the cryogenic temperature to probe the dark exciton directly and reveal the polarization properties of the dark exciton. We demonstrate that the dark state is located below the bright state with a bright-dark splitting energy of ~20 meV, which is beneficial to constructing the theoretical model of the fine-structure of the band-edge exciton in the system of perovskite. Moreover, the emission from bright exciton is not inhibited by dark exciton, indicating that it has great potential in the field of quantum light sources. Furthermore, the non-polarization effect of dark exciton would help to better understand the magnetic field-induced spin-dependent generation and recombination process in perovskite-based materials. These findings will help to improve the performance of quantum light emitters and spintronic devices.

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Conflict of interest The authors declare no competing financial interests.

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二维钙钛矿材料CsPbBr₃中暗激子的磁场调控特性研究

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摘要 二维钙钛矿材料作为21世纪最热门的材料之一,其优异的光电特性使其在太阳能电池、激光器等方面具有巨大的应用潜力。二维无机钙钛矿结构CsPbX₃ (X=Cl, Br, I)的激子性质对其光电特性具有决定性的作用。本工作针对CsPbBr₃中的暗激子进行了详细的研究,通过施加面外磁场,实现了对CsPbBr₃中暗态激子的激活,并发现暗态激子的荧光强度随磁场增加而呈现线性增强。本工作揭示了CsPbBr₃暗激子的磁场调控行为和内在物理机制,对深入理解其光电特性具有重要的科学意义。