ZnO–Ti$_3$C$_2$ MXene Electron Transport Layer for High External Quantum Efficiency Perovskite Nanocrystal Light-Emitting Diodes

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2D transition metal carbides, nitrides, and carbonitrides called MXenes show outstanding performance in many applications due to their superior physical and chemical properties. Herein, a ZnO–MXene mixture with different contents of Ti$_3$C$_2$ is applied as electron transport layers (ETLs) and the influence of the Ti$_3$C$_2$ MXene in all-inorganic metal halide perovskite nanocrystal light-emitting diodes (perovskite NC LEDs) is explored. The addition of Ti$_3$C$_2$ makes more balanced charge carrier transport in LEDs by changing the energy level structure and electron mobility of ETL. Moreover, lower surface roughness is obtained for the ETL, thus guaranteeing uniform distribution of the perovskite NCs layer and further reducing leakage current. As a result, a 17.4% external quantum efficiency (EQE) with low efficiency roll-off is achieved with 10% Ti$_3$C$_2$, which is a 22.5% improvement compared to LEDs without Ti$_3$C$_2$.
Zn–Si–O ETL by magnetron sputtering at room temperature, which had sufficiently shallow electron affinity (≈3.2 eV) to confine excitons and sufficiently high electron mobility (≈0.8 cm^2 V^{-1} s^{-1}) to transport electrons. Consequently, the very low operating voltage and high power efficiency were achieved for CsPbX (X=Br or BrI) perovskite LEDs. \cite{8}

Recently, 2D transition metal carbides, nitrides, and carbonitrdes called MXenes with a formula of M_{n+1}X_nT_x (n=1, 2, 3; M is an early transition metal, X is carbon and/or nitrogen atom and T_x represents the surface-terminating functional groups) show outstanding performance in many applications such as supercapacitors, catalysts, conducting films, sensors, and antennas due to their superior physical and chemical properties. \cite{9} In addition, MXenes offer relatively high conductivity, high light transmittance in the visible range, and the possibility to tailor their electronic structure such as work function (WF) or bandgap. \cite{10}

All of these make MXenes possible to be the transport material in optoelectronic applications. Ti_3C_2 MXene has been incorporated into the perovskite absorber or into the ETL, modifying the chemical and/or physical properties of the interface and providing superior charge transfer paths, therefore, resulting in an enhanced power conversion efficiency in perovskite solar cells. \cite{11b}

For perovskite EML, the synthesis of CsPb_0.64Zn_0.36I_3 perovskite NCs was based on our previous work using a 0.5 feed ratio of ZnI_2 to PbI_2 (details in the Supporting Information). Figure S2a (Supporting Information) shows the UV−vis absorption and photoluminescence (PL) spectra of the as-synthesized CsPb_0.64Zn_0.36I_3 perovskite NCs whose first excitonic absorption peak and PL peak are 675 nm and 688 nm, respectively. The corresponding XRD patterns and TEM images are shown in Figure S2b,c (Supporting Information), proving the perovskite NCs with a cubic phase.

The ZnO–Ti_3C_2 (designated as ZTC) mixture are made up of the different amounts of Ti_3C_2 MXene dispersed into the ZnO NC solution. As shown in Figure S2 (Supporting Information), they formed a uniform dispersion and the color of the solution gradually darkened as the amount of Ti_3C_2 increased. The XRD patterns of different samples are shown in Figure S3 (Supporting Information). Figure S4a (Supporting Information) shows the TEM image of the ZTC. It can be seen that ZnO is attached to or scattered around the surface of Ti_3C_2. Figure S4b (Supporting Information) is the diffraction pattern without the Ti_3C_2 area and the diffraction rings of ZnO can be seen. Figure S4c (Supporting Information) is the diffraction pattern of the mixed region.
Compared with Figure S4b (Supporting Information), there are two more obvious diffraction rings, corresponding to the (101) and (110) planes of Ti$_3$C$_2$[12], indicating that there is indeed Ti$_3$C$_2$ in the mixed region. SEM images of unheated and heated ZTC are shown in Figure S4d,e (Supporting Information), respectively. Figure S4d (Supporting Information) shows that a small amount of sheet-like Ti$_3$C$_2$ is mixed on the ZnO film, and Figure S4e (Supporting Information) shows that the annealed film has uniform grains. In the process of device preparation, ZTC mixture were spin-coated onto an ITO substrate as ETL and a light output medium. In order to guarantee a high optical coupling output of the device, the light transmittance of the spin-coated ZTC mixture film cannot be greatly reduced. Subsequently, the transmittances of pristine ZnO film and ZTC film with different amounts of Ti$_3$C$_2$ were characterized and presented in Figure S5 (Supporting Information). There is no significant change in transmittance between ZTC and pristine ZnO film over the entire visible range, demonstrating that the introduction of Ti$_3$C$_2$ has a negligible effect on the optical coupling output of the device.

In order to explore the effect of Ti$_3$C$_2$ on the electronic properties of ETL, the energy levels of ZnO/PEI and ZTC/PEI were explored by UPS measurement and the Tauc plots, the corresponding data are given in Figure 2a,b, respectively. It can be seen that the conduction band minimum (CBM) decreased from $-3.56$ to $-3.99$ eV while the bandgap remained unchanged with the increase of Ti$_3$C$_2$ loading, illustrating that Ti$_3$C$_2$ played a role of energy level regulation. Here, we designed ITO/ZTC/PEI/CsPb$_{0.64}$Zn$_{0.36}$I$_3$/TCTA/MoO$_3$/Au device structure (Figure 2c), and the corresponding energy band diagram for all functional layers is shown in Figure 2d. Changes in ZnO energy level can be attributed to the changes in surface termination groups of Ti$_3$C$_2$ after annealing$^{[11c,13]}$ (Figure S6, Supporting Information). Typically, electrons are injected into the emitting layer through the CBM of the ETL. The energy level difference between the ETL and EML is an important factor affecting the electron injection efficiency. Previous reports have used PEI as an interface modification layer to improve electron injection efficiency while blocking electron transport. The thickness of PEI as an insulating material must be moderate, otherwise, an excessive thickness will increase device resistance, causing unnecessary thermal effects, thereby reducing the device efficiency. Therefore, balanced charge transfer in the device may not be completely carried out only through adding a PEI layer.$^{[14]}$

Through the above discussion of the regulation of ETL energy level by Ti$_3$C$_2$, we can further envision the effect of which on the carrier transport in the device. The charge transfer processes in perovskite NC LEDs based on different contents of Ti$_3$C$_2$ in ETL are illustrated in Figure 3. First, for the pristine ZnO-based device (Figure 3a), there is a large potential well between ETL and EML compared with the one between HTL and EML, which will cause excessive electron accumulation, resulting in carrier recombination imbalance and further affecting device efficiency. At the beginning of the addition of 5% Ti$_3$C$_2$ (Figure 3b), the potential well between ETL and EML becomes small, but there may still be an accumulation of electrons due to the larger electron difference of ETL. When the Ti$_3$C$_2$ increases to 10% (Figure 3c), the little potential well between ETL and EML becomes a little potential barrier, which will reduce the electron injection efficiency of ZTC and promote the injection balance of electrons and holes. The accumulation of electrons at the interface between EML and HTL will be suppressed; the space charge will be reduced; the built-in electric field will be weakened. It may be manifested by an increase in device efficiency.$^{[15]}$ When 20% Ti$_3$C$_2$ is added in ZnO NCs (Figure 3d), the electron injection barrier becomes larger, which will reduce electron injection efficiency and cause charge transport to be once again unbalanced, thus degrading device performance.

**Figure 2.** a) UPS spectra, b) Tauc plots of ZnO/PEI film and ZTC/PEI film with different contents of Ti$_3$C$_2$ (from 5% to 20%) deposited on the ITO glass substrate. c) Schematic of the CsPb$_{0.64}$Zn$_{0.36}$I$_3$ perovskite NC LED configuration. d) Device energy-level diagram for each functional layer in the LEDs.
In order to determine the influence of Ti$_3$C$_2$ on the device performance of perovskite NC LEDS, devices with an architecture of ITO (200 nm) /ETL (70 nm)/CsPb$_{1.64}$Zn$_{0.36}$I$_3$ (50 nm)/TCTA (70 nm) /MoO$_3$/Au (60 nm) were fabricated (Figure S7, Supporting Information). The normalized electroluminescence (EL) and PL spectra for all sample devices are shown in Figure S8 (Supporting Information). It is noted that the EL spectra are almost consistent with the PL spectra, illustrating that the EL is indeed from the perovskite NCs without any noticeable contribution from other charge transport materials.\cite{16}

**Figure 3.** Charge carrier injection and recombination mechanisms for a) 0%, b) 5%, c) 10% and d) 20% Ti$_3$C$_2$ based devices.

In order to determine the influence of Ti$_3$C$_2$ on the device performance of perovskite NC LEDs, devices with an architecture of ITO (200 nm) /ETL (70 nm)/CsPb$_{1.64}$Zn$_{0.36}$I$_3$ (50 nm)/TCTA (70 nm) /MoO$_3$/Au (60 nm) were fabricated (Figure S7, Supporting Information). The normalized electroluminescence (EL) and PL spectra for all sample devices are shown in Figure S8 (Supporting Information). It is noted that the EL spectra are almost consistent with the PL spectra, illustrating that the EL is indeed from the perovskite NCs without any noticeable contribution from other charge transport materials.\cite{16}

**Figure 4a** presents the voltage-dependent variations of luminescence and current density for CsPb$_{1.64}$Zn$_{0.36}$I$_3$ NC LEDs with different ETLs. It can be seen that the turn-on voltages and maximum luminescence of these devices show a slight increase as the additional content of Ti$_3$C$_2$ from 0% to 10%. When the content of Ti$_3$C$_2$ continuously increased to 20%, the turn-on voltage became larger and the maximum luminescence significantly decreased. The increase in turn-on voltage is attributed to the fact that electron injection gradually changes from a potential well to a barrier, which means that the electron injection capability is lowered. The increase in luminescence is attributed to a more balanced charge carrier recombination at low Ti$_3$C$_2$ concentrations.\cite{6c} When the content of Ti$_3$C$_2$ increased, the barrier height between ETL and EML further increased, the recombination of charge carriers was again unbalanced, resulting in a sharp drop in luminescence. Similar changes were observed in EQE versus current density characters of these four devices (Figure 4b), and the corresponding electrical parameters are summarized in **Table 1**. These results, in turn, confirm our previous assumptions. The maximum EQE of 17.4% was achieved for a 10% ZTC based device, which is 1.2-fold higher than 14.2% EQE of pristine ZnO-based device. Moreover, 10% ZTC-based device has an efficiency roll-off of 22.9% at current density of 500 mA cm$^{-2}$, a 15.8% improvement over 38.7% roll-off of pristine ZnO-based device. This may be attributed to the suppression of auger recombination caused by unbalanced charge carrier injection (which favors trion formation at higher current densities).\cite{5g,15}

The electron mobility of ZnO films was 2.21 $\times$ 10$^{-3}$ cm$^2$ V$^{-1}$ s$^{-1}$ which is much higher than that of TCTA (2 $\times$ 10$^{-5}$ cm$^2$ V$^{-1}$ s$^{-1}$).\cite{7a,17} The large electron mobility can cause excess electron accumulation at the interface between the EML and the ETL, thereby forming a space charge and causing luminescence quenching, which ultimately leads to a reduction of device efficiency.\cite{7b} To further confirm the effect of the addition of Ti$_3$C$_2$ (electron mobility of 2.23 $\times$ 10$^{-3}$ cm$^2$ V$^{-1}$ s$^{-1}$)\cite{11b} on the carrier transport balance, the electron-only devices with device structure of ITO/ZTC/PEI (0% and 10%) /perovskite NCs /LiF/Al and the hole-only devices with device structure of ITO/PEDOT:PSS/perovskite NCs/TCTA/MoO$_3$/Au were fabricated. As shown in **Figure S9a** (Supporting Information), the current density of the electron-only device with 10% ZTC became lower than that of the electron-only device without Ti$_3$C$_2$ and was much closer to that of the hole-only device, proving that the carrier transport between electron and hole are more balanced. In addition, the electron mobility of different ETL films are estimated by fitting the space-charge-limited-current region (SCLC). As shown in **Figure S9b** (Supporting Information), the mobility of the added ZnO film with 0% to 20% Ti$_3$C$_2$ content are 1.98 $\times$ 10$^{-3}$ cm$^2$ V$^{-1}$ s$^{-1}$, 1.61 $\times$ 10$^{-3}$ cm$^2$ V$^{-1}$ s$^{-1}$, 1.16 $\times$ 10$^{-3}$ cm$^2$ V$^{-1}$ s$^{-1}$, 9.03 $\times$ 10$^{-4}$ cm$^2$ V$^{-1}$ s$^{-1}$, illustrating the addition of Ti$_3$C$_2$ reduces the electron mobility of ZnO and the current density decreases with the increase of Ti$_3$C$_2$ addition. As discussed above, we conclude that the effect of Ti$_3$C$_2$ on carrier transport balance can be divided into two aspects. On the one hand, the addition of Ti$_3$C$_2$ increases the work function of Ti$_3$C$_2$, on the other hand, the electron mobility of ETL decreases due to the lower electron mobility of Ti$_3$C$_2$, thus leading to more matched energy levels and carrier mobility between ETL and HTL. Furthermore, the ZTC layer also offers lower surface roughness than that of the ZnO layer. The AFM image of the ZnO film is shown in **Figure S10a** (Supporting Information), exhibiting root-mean-square (RMS) roughness of 3.42 nm. The RMS roughness is further reduced to 1.49 nm when 10% of Ti$_3$C$_2$ was added in the ZnO (Figure S10b, Supporting Information). The uniform ZTC films ensure low leakage current in LEDs and the semilogarithmic curve illustrated in **Figure 4a** shows that the leakage current of the 10% ZTC-based device is significantly reduced compared to the original device, which is crucial for LED’s performance. Because the reduction of leakage current means the reduction of the current flowing into the device without emission.\cite{18} The main works on the red emitting CsPb$_1$ perovskite NC LEDs are summarized in Table S1 (Supporting Information). For now, our devices have achieved the highest EQE. We also tested the repeatability and stability of these devices, as shown in **Figure S11** (Supporting Information). The average EQE$_{max}$ of the four types of devices added with 0, 5%, 10%, and 20% Ti$_3$C$_2$ are 14.2%, 15.3%, 17.3%, and 11.5%, respectively. It can be seen that the LED devices with different Ti$_3$C$_2$ contents have good repeatability. In addition, the times for LED luminances without Ti$_3$C$_2$ and with 10% Ti$_3$C$_2$ drop to half of their initial values are 9 and 23 min, respectively, illustrating a better charge transport balance can reduce the damage to the device.
Figure 4. a) Current density and luminance versus bias voltage. b) EQE versus current efficiency of CsPb$_{0.64}$Zn$_{0.36}$I$_3$ perovskite NC LEDs with different contents of Ti$_3$C$_2$.

Table 1. Electronic performance of CsPb$_{0.64}$Zn$_{0.36}$I$_3$ perovskite NC LEDs with 0–20% Ti$_3$C$_2$.

| ETL       | EL [nm] | FWHM [nm] | $V_{ON}$ [V] | $L_{max}$ [cd m$^{-2}$] | EQE $\%$ |
|-----------|---------|-----------|--------------|--------------------------|----------|
| ZnO       | 682     | 33        | 2.24         | 2678                     | 14.2     |
| ZTC (5%)  | 682     | 33        | 2.25         | 3225                     | 15.4     |
| ZTC (10%) | 682     | 33        | 2.28         | 3260                     | 17.4     |
| ZTC (20%) | 682     | 33        | 2.43         | 2483                     | 11.5     |

In conclusion, conductive sheet-like Ti$_3$C$_2$ Mxene was prepared and mixture of Ti$_3$C$_2$ and ZnO NCs were first utilized as the ETL for perovskite NC LEDs. On the one hand, the addition of Ti$_3$C$_2$ achieves charge carrier balance between ETL and HTL by changing the work function and electron mobility of ZnO. On the other hand, the ZTC film also offers lower surface roughness, thus guaranteeing uniform distribution of the perovskite NCs layer for leakage current reduction. As a result, a relatively high EQE of 17.4% and lower efficiency roll-off were achieved by using ZTC (10%) as the ETL, which is 22.5% improvement compared with the pristine ZnO-based device (14.2% EQE), showing a great promise for the applications in future display and lighting.

Supporting Information
Supporting Information is available from the Wiley Online Library or from the author.

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Conflict of Interest
The authors declare no conflict of interest.

Keywords
light-emitting diodes, MXenes, perovskite nanocrystals, Ti$_3$C$_2$

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