Photo-Diodes Based on CH$_3$NH$_3$PbCl$_3$ Perovskite Single Crystals by Epitaxial Growth for Ultraviolet Photo-Detection

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INTRODUCTION

Recently, Organic-inorganic hybrid methylammonium lead halide perovskite (MAPbX$_3$, where MA = CH$_3$NH$_3$, and X = Cl, Br, I) perovskite have attracted a lot of attention due to their excellent optoelectronic properties, such as tunable band gap, low trap density, and long diffusion length [1, 2]. MAPbX$_3$ have been proved to have potential in a number of optoelectronic devices, such as photodetectors, light-emitting diodes, and solar cells [3–9]. There are reports on the visible and near infrared detection applications of MAPbI$_3$ and MAPbBr$_3$ single crystals because of their suitable band gap and high optical absorption coefficient [10, 11]. MAPbCl$_3$ single crystals are wide-bandgap (2.9 eV) semiconductors [12] which are insensitive to visible light while having a high absorption for ultraviolet (UV) radiation (wavelength ~100–400 nm) [13]. These characters make MAPbCl$_3$ single crystals suitable in applications of UV detection [14–16]. To best our knowledge, little work carried out to study the optoelectronic characteristics of photo-diode based on MAPbCl$_3$, which is important to develop optoelectronic device based on PSCs. Therefore, it is necessary to obtain a photo-diode based on MAPbCl$_3$ with high performance.

The properties of conventional semiconductor could be adjusted by impurity doping, similarly, heterovalent metals doping into PSCs is an efficient way to change the electrical and optical properties [17–21]. MAPbCl$_3$ is proved to be a n$^-$ type semiconductor [12], while doping Bi$^{3+}$ into MAPbX$_3$ would move Femi level close to conduction band [22, 23]. Inspired by the above work, we
suppose to fabricate \( n^-n^+ \) photo-diodes based on \( n^- \)-type MAPbCl\(_3\) single crystal substrate with \( n^+ \)-type Bi-doped MAPbCl\(_3\) epitaxial layer. Different to conventional heterojunction, the PSCs homojunction has low lattice mismatch rate, continuous band alignments, and low trap densities [24–26] which allow the device to have a lower dark current when the device is under reverse bias.

In this work, a bulk MAPbCl\(_3\) single crystal with cubic shape and high crystallization quality was synthesized by inverse temperature crystallization (ITC) method [27], and we fabricated a UV photo-diode by growing Bi-doped MAPbCl\(_3\) epitaxial layer on MAPbCl\(_3\) single crystal substrate. This photo-diode shows a low dark current density of \(-2.15 \mu A cm^{-2}\) at \(-20 V\) bias voltage while its photocurrent reaches \(-82.69 \mu A cm^{-2}\) under 393 nm LED with the illumination of 20 mW cm\(^{-2}\). Due to the successful fabricating of the \( n^-n^+ \) junction, the responsibility reaches 404.7 mA/W, \(~1-2\) orders of magnitude higher than pervious reported photodetector based on MAPbCl\(_3\) [14, 28], and its detectivity reaches 4.87 \times 10^{11} \text{ Jones}, which is \(~1-3\) orders of magnitude faster than that of the previously reported MAPbCl\(_3\) photodetectors [14, 15, 28].

**MATERIALS AND METHODS**

**Materials**

Lead chloride (PbCl\(_2\), 99%) were purchased from Sigma Aldrich. N, N-Dimethylformamidel (DMF, 99.5%), and Dimethyl sulfoxide were purchased from Chinese reagents. Bismuth Chloride (BiCl\(_3\), 98%) and Methylamine hydrochloride (MACl, 98%) were purchased from Aladdin. All commercial products were used as received.

**Fabrication of Device**

1 mol/L MACl and 1 mol/L PbCl\(_2\) were dissolved in 50 ml dimethylformamide (DMF) and dimethylsulfoxide (DMSO) mixed solution (1:1 v/v) to act as precursor solutions. The poly tetra fluoroethylene (PTFE) filters with a 30 \( \mu \)m pore size were used to filter the solutions. Then, the precursor solutions, after being filtered, were transferred to a crystallizing dish by dropper. The crystallizing dish was placed on a programmable heating station (IKA-RET control-visc). The temperature was set to rise 0.5\(^\circ\)C per hour from 40 to 60\(^\circ\)C, the whole process is shown in Figure 1A. After two days, the pristine MAPbCl\(_3\) single crystal could be obtained, Figure 1B shows the MAPbCl\(_3\) substrate with the size of 6.95 \times 6.96 \times 2.52 mm.

Similarly, 1 mol/L MACl, 1 mol/L PbCl\(_2\), and 0.1 mol/l BiCl\(_3\) were dissolved in 10 ml dimethylformamide (DMF) and dimethylsulfoxide (DMSO) mixed solution (1:1 v/v) to prepare for epitaxial growth. The pristine MAPbCl\(_3\) single crystal was put into the epitaxial solution. After the heating station temperature kept at 60\(^\circ\)C for 4 h, the MAPbCl\(_3\) with epitaxial layer could be obtained, and Figure 1C is a photograph of MAPbCl\(_3\) single crystal with epitaxial layer after polishing the edges of the crystal, obviously, the color of the epitaxial layer is yellow and the thickness of the epitaxial layer is nearly 0.52 mm. To fabricated the photo-diodes, the gold electrode acted as anode is deposited on the side of the MAPbCl\(_3\) substrate while the sliver electrode acted as cathode is deposited on the side of the epitaxial layer. The structure of this device is shown in Figure 1D.

**Characterization and Measurements**

X-ray diffraction (XRD) patterns were taken by XTRA (Switzerland). Photoluminescence (PL) spectra were measured by UV-vis spectroscopy (Lab Tech Bluestar, USA). I-V curve was measured by a Keithley 4200 semiconductor analyzer. QE was measured by Zolix tunable 500-W arc lamplight. A 355-nm pulsed Nd: YAG laser with 6-ns pulse width at 10 Hz as the illumination source and an Agilent oscilloscope were used to measure the carrier mobility.

**RESULTS AND DISCUSSION**

Firstly, we study the optical properties of the pristine MAPbCl\(_3\) substrate, MAPbCl\(_3\) with Bi-doped MAPbCl\(_3\) epitaxial layer, and Bi-doped MAPbCl\(_3\) single crystal, the PL spectra and absorption are shown in Figures 2A,B. The absorption edge of pristine MAPbCl\(_3\) single crystal was located at 430 nm, and the PL peak is obtained at 436 nm with a narrow FWHM of 14.9 nm confirming the high quality of the single crystal [7]. Doping into single crystal would cause a larger increase of the lattice defect which is the reason why the PL of the Bi-doped MAPbCl\(_3\) has a broadening effect [29]. The absorption of Bi-doped MAPbCl\(_3\) single crystal is in good agreement with results that were previously reported [30]. The PL of MAPbCl\(_3\) with Bi-doped MAPbCl\(_3\) epitaxial layer is the stack of the pristine MAPbCl\(_3\) and Bi-doped MAPbCl\(_3\) single crystal, which indicated the successful growth of the Bi-doped MAPbCl\(_3\) epitaxial layer. The absorption moves from 430 to 480 nm after epitaxial growth which approaches the Bi-doped MAPbCl\(_3\) single crystal.

Figures 2C,D provide XRD spectra of pristine MAPbCl\(_3\) single crystal, MAPbCl\(_3\) with epitaxial layer, and Bi-doped MAPbCl\(_3\). To (100) planes, the strongest peaks of MAPbCl\(_3\) are observed at \( 20 = 31.48^\circ, 48.00^\circ, 65.68^\circ \), while the strongest peaks of Bi-doped MAPbCl\(_3\) at \( 20 = 31.50^\circ, 48.02^\circ, 65.70^\circ \) indicating no phase change after doping Bi\(^{3+}\). Besides, Figure 2D illustrates the FWHM of pristine MAPbCl\(_3\) single crystal with the epitaxial layer is 0.113\(^\circ\) which confirms the epitaxial layer remains single crystalline. The stagger XRD peak of MAPbCl\(_3\) substrate and epitaxial layer causes the FWHM of photo-diode to be widened. The lattice constants could be calculated from the XRD spectra by Bragg law (\( 2dsin\theta = n\lambda \), where \( d \) is the distance between two atoms, \( \theta \) is the angle of crystal plane and X-ray, \( n \) is constant, and \( \lambda \) is the wavelength of the X-ray), the lattice constants of MAPbCl\(_3\) and Bi-doped MAPbCl\(_3\) are 5.67902 Å and 5.67550 Å, respectively, the lattice constants of MAPbCl\(_3\) is same as previously reported [31]. After doping Bi\(^{3+}\), the lattice constant is smaller than pristine MAPbCl\(_3\) single crystal indicating the Bi\(^{3+}\) ions are possibly substituting Pb\(^{2+}\) ions in the crystal. The mismatch rate (mismatch rate can be calculated by \( \gamma = (a_1 - a_2)/(a_1 + a_2) \), where \( a_i \) is the lattice constants of the...
crystal) of MAPbCl$_3$ and Bi-doped MAPbCl$_3$ single crystal is 0.3%, suggesting that their lattice could match well.

Further, the charge mobility of the photo-diode is investigated by the TOF method. After the optical signal is received, the photo-induced holes could immediately be collected under forward bias, while electrons have to drift through the whole device to be collected, the electrons mobility could be calculated through the drift time. Similarly, under the reverse voltage, the holes’ mobility could be calculated in the same way. A 355-nm pulsed Nd: YAG laser with 6-ns pulse width at 10 Hz is used as the illumination source, the direction of the light is shown in Figure 3A. The average electron mobility was measured as shown in Figure 3B. And the average hole mobility was measured as shown in Figure 3C. The carrier mobility can be calculated according to:

$$T_{\text{tof}} = \frac{I^2}{\mu} \times \frac{1}{V_s}$$

where $\mu$ is the carrier mobility, $I$ is the thickness of the photo-diode, $V_s$ is the bias voltage between two electrodes, and $T_{\text{tof}}$ is the transmit time [32, 33]. The fitting plot of carrier mobility vs. reciprocal of the bias voltage is shown in the insets, the result shows that the average holes mobility is $55.06 \pm 0.61 \text{ cm}^2\text{V}^{-1}\text{s}^{-1}$ is faster than UV detector based on MAPbCl$_3$ single crystal (26 cm$^2$V$^{-1}$s$^{-1}$) reported [16], the reason is Bi-Doped MaPbCl$_3$ has a high conductivity. And the average electrons mobility is $40.01 \pm 1.30 \text{ cm}^2\text{V}^{-1}\text{s}^{-1}$.

Figure 4A illustrates the EQE of the photo-diode under $-20\text{V}$, the EQE peak locates at 398 nm which prove the device can be applied in UV photo-detection. EQE is calculated by:

$$\text{EQE} = \frac{h \nu}{e} \times \frac{I_{\text{Light}} - I_{\text{Dark}}}{P \times S} \times 100\%$$

where $I_{\text{Light}}$ is the photocurrent (A), $I_{\text{Dark}}$ is the dark current (A), $h$ is Plank’s constant, $\nu$ is the frequency of incident light, $S$ is the area being illuminated, and $P$ is the incident illumination power density ($\text{W/cm}^2$), and $e$ is the element charge [34].

To further study the optoelectronic performance of this photo-diode, an LED with an emission peak of 393 nm and the power intensity of the LED is 20 mWcm$^{-2}$, and the current-voltage characterization of the device in the dark and under illumination is shown in Figure 4B, respectively. The photocurrent under reverse bias is much larger than the photocurrent under forward bias. Because of the built-in electric field, holes are hard to transfer from MAPbCl$_3$ substrate to Bi-MAPbCl$_3$ epitaxial layer and electrons are hard to transfer from Bi-MAPbCl$_3$ epitaxial layer to MAPbCl$_3$ substrate when the device is under reverse bias and in a dark environment, and the homojunction restrains the dark current further. Thanks to the highly crystallized epitaxial layer, the dark current of the photo-diode is $-2.15 \text{ \mu Acm}^{-2}$ at $-20 \text{V}$, while its photocurrent reaches approximately $-82.69 \text{ \mu Acm}^{-2}$ under illumination. To analyze the response ability of the photo-diode to UV radiation, the responsivity (R) and detectivity (D) of the photo-diode was calculated as follow [35, 36]:

$$R = \frac{I_{\text{Light}} - I_{\text{Dark}}}{P \times S}$$

$$D = \frac{R}{\sqrt{2eJ_{\text{Dark}}}}$$
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**FIGURE 2 |** (A) PL spectra of pristine MAPbCl$_3$ substrate, MAPbCl$_3$ with epitaxial layer, and Bi-doped MAPbCl$_3$. (B) Absorption of pristine MAPbCl$_3$ substrate, MAPbCl$_3$ with epitaxial layer, and Bi-doped MAPbCl$_3$. (C) XRD spectra of the pristine MAPbCl$_3$ substrate, MAPbCl$_3$ with Bi-doped MAPbCl$_3$ epitaxial layer, and Bi-doped MAPbCl$_3$ single crystal. (D) XRD spectra from 47.5° to 48.5° of the three kinds of PSCs.

**FIGURE 3 |** (A) Illustration of the optoelectronic experiment. (B) Holes mobility measured by time-of-flight (TOF) method. (C) Electrons mobility measured by the TOF method.
The responsivity is calculated to be 404.7 mA/W, detectivity is calculated to be $4.87 \times 10^{11}$ Jones which is one order of magnitude larger compared to MAPbCl$_3$ film ($6.87 \times 10^{10}$ Jones) [37].

To study the signal noise at low frequency, we investigate 10 Hz temporal response of the photo-diode under different reverse voltage bias from $-10$ to $-50$ V, the Fast Fourier Transform (FFT) method is used to transfer the current vs. time curve to signal current vs. frequency curve. The signal noise from 2 to 18 Hz is calculated and the curve is shown in Figure 4C. The average noise current increases from $2.3 \text{ pAHz}^{-0.5}$ to $17.0 \text{ nAHz}^{-0.5}$ under illumination with bias voltage from $-10$ to $-50$ V.

Figure 4D illustrates the on–off switching properties of the photo-diode with the bias voltage range from $-10$ to $-50$ V. Obviously, the photo-diode could switch rapidly and stably under different bias. The response speed was measured as shown in Figure 4E, the rise time is 176.3 ns and the fall time is 11.6 $\mu$s under the bias of $-150$ V, the fast carrier mobility contributes to the rapid response speed.

The comparison of responsivity and response time between this work and previously reported MAPbCl$_3$ photodetectors is summarized in Table 1. The photo-diode fabricated in this work has a higher responsivity than most UV detectors reported, and the decay time is the fastest.

### TABLE 1 | Comparison between this work and previously reported MAPbCl$_3$ photodetectors.

| UV Detector | Responsivity @light source | Decay time | Reference |
|-------------|----------------------------|------------|-----------|
| MAPbCl$_3$ film | 0.97 A/W @380 nm (2.2 mWcm$^{-2}$) | 220 $\mu$s | [38] |
| | 71 mA/W @398 nm | 20 ms | [37] |
| | Below 1 mA/W @385 nm | 62 ms | [16] |
| MAPbCl$_3$ single crystal | 46.9 mA/W @365 nm | 62 ms | [14] |
| | 0. 60 mA/W @405 nm | 62 ms | [28] |
| | 1.85 A/W @415 nm | 368 $\mu$s | [15] |
| Photo-diode | 404.7 mA/W @393 nm | 11.6 $\mu$s | This work |

**CONCLUSION**

In summary, we have fabricated a UV photo-diode by growing Bi-doped MAPbCl$_3$ epitaxial layer on a pristine MAPbCl$_3$ single crystal substrate. And we calculate the lattice constants of pristine MAPbCl$_3$ and Bi-doped MAPbCl$_3$ single crystal through XRD, which are lattice-matched. Further, we investigate the optoelectronic properties of the photo-diode. At $-20$ V voltage, the dark current is $-2.15 \mu$Acm$^{-2}$ while the photocurrent reaches $-82.69 \mu$Acm$^{-2}$, the on-off ratio reaches nearly 40 under a 393 nm LED with 20 mW/cm$^2$. Moreover, the photo-diode has a responsivity of 404.7 mW/cm$^2$ and detectivity of $4.87 \times 10^{11}$ Jones. The carrier mobility is calculated to be $55.06 \pm 0.61 \text{ cm}^2\text{V}^{-1}\text{s}^{-1}$ by TOF. The rise time and decay time are 176.3 ns and 11.6 $\mu$s, which is faster than the previously reported UV detector based on MAPbCl$_3$ single crystal and ploy-crystal film. This work provides useful information for UV detection and photoelectrical device based on MAPbX$_3$.
DATA AVAILABILITY STATEMENT

The raw data supporting the conclusions of this article will be made available by the authors, without undue reservation.

AUTHOR CONTRIBUTIONS

JZ and YP grew the perovskite single crystals. JZ did the epitaxial experiments. YX and JZ did the measurements. XW and JZ analyzed these results. JZ wrote this manuscript. All authors make comments on the manuscript.

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**Conflict of Interest:** JZ is employed by China Nuclear Power Engineering Co. Ltd., Shenzhen, Guangzhou, China.

The remaining authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

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