

Article

CH₃NH₃PbBr₃ Perovskite Single-Crystal X-Ray Photon-Counting Detection Based on Multi-Layer Electrodes

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Highlights

What are the main findings?

- Au/Pt/Ti multi-layer is a good choice for electrodes for CH₃NH₃PbBr₃ perovskite single crystals.
- Low-noise CH₃NH₃PbBr₃ perovskite single crystals could be applied in X-ray photon-counting detection and imaging.

What are the implications of the main findings?

- Scientists and engineers could try multi-layer electrodes for CH₃NH₃PbBr₃ perovskite single crystals.
- More lead halide perovskite single crystals should be considered in X-ray photon counting detectors.

Abstract

CH₃NH₃PbBr₃ (MAPbBr₃) single crystals have shown great potential in X/γ-ray detection. However, stable electrodes for MAPbBr₃ single crystals still remain challenging. In this work, multi-layer electrodes including Au, Au/Ti and Au/Pt/Ti are investigated. Through I-V characterization, Au/Pt/Ti shows Ohmic contact behavior and the lowest dark current. The potential contact is also confirmed by the Kelvin force probe. Based on these low-noise electrodes, 59.5 keV monochromatic X-ray photon-counting detection and imaging is demonstrated. This work provides useful information for electrode design in lead halide perovskite-based optoelectronic devices.

Keywords: CH₃NH₃PbBr₃ single crystal; multi-layer electrode; X-ray photon counting detection



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1. Introduction

X-ray detection technologies play a critical role in a wide range of applications, including medical imaging, industrial non-destructive testing, and security checks [1–3]. Among various detection modalities, photon-counting detection has emerged as a next-generation

paradigm due to its superior capabilities in energy resolution, contrast-to-noise ratio, and reduced radiation dose compared to conventional energy-integrating systems [4–9]. The success of photon-counting detection, however, heavily relies on the availability of high-performance semiconductor materials that exhibit both efficient X-ray absorption and excellent charge transport properties [10].

In recent years, lead halide perovskites have garnered significant attention as promising candidates for X-ray detection, owing to their exceptional optoelectronic properties, including high atomic numbers, large mobility-lifetime ($\mu\tau$) products, and low-cost solution-processability [11–14]. Among kinds of lead halide perovskites, methylammonium lead bromide (MAPbBr₃, MA=CH₃NH₃) stands out as a particularly attractive material for X-ray photon counting applications, as it combines strong X-ray attenuation capability with high resistivity and quick growth speed from solution precursor [15–17]. These attributes have enabled MAPbBr₃ single-crystal-based X-ray detectors to achieve impressive sensitivity and the lowest detection limits, positioning them as viable alternatives to conventional materials such as amorphous selenium, cadmium zinc telluride, and silicon [18–21]. In previous work, a CsPbBr₃ single crystal was used as a high flux X-ray photon counting detector with a count rate of 263 kcps [9]. Further, the MAPbI₃ single crystal was applied in X-ray photon counting detectors with the extremely low noise-equivalent dose of 90 pGy_{air} [5].

Despite these advances, the application of MAPbBr₃ single crystals in X-ray photon-counting detection remains challenging, primarily due to issues related to device stability, high dark current, and charge trapping under high electric fields [22–24]. The electrode configuration plays a crucial role in governing charge collection efficiency and suppressing dark current in perovskite X-ray detectors. Conventional single-layer metal electrodes such as Au, Ag, Ti and Ga often suffer from inefficient charge extraction, interfacial reactions with the MAPbBr₃ single crystals, and increased leakage currents, limiting the detector's ability to operate stably under sustained high bias and X-ray irradiation [25–28]. While previous studies have largely focused on optimizing the active layer composition and interface engineering, relatively little attention has been paid to the rational design of multi-layer electrode architectures tailored for photon counting mode, particularly in the context of MAPbBr₃ single crystal-based devices [5,29].

In previous reports, multi-layer electrodes including MoOx/Cu/Ag/MoOx, IZrO/IZO, AZO/Au/AZO, AZO/Cu/Ag/AZO and MoOX/Cr/Al were used as low-resistivity and high-transmittance electrodes applied in solar cell [30–34]. However, these electrodes are not stable under high bias when in contact with the MAPbBr₃ single crystal.

To address these limitations, we propose the implementation of multi-layer electrode structures employing Au/MAPbBr₃/Au, Au/Ti/MAPbBr₃/Ti/Au and Au/Pt/TiMAPbBr₃/Ti/Pt/Au configurations. These multi-layer architectures are designed to combine the advantages of each metal layer: Ti serves as an adhesion layer, where Ti-N bonds are formed at the interface of MAPbBr₃/Ti [35]; Pt functions as an isolating layer to mitigate interfacial reactions and ion migration; while Au ensures high conductivity. By systematically comparing the performance of MAPbBr₃ X-ray detectors with single-layer and multi-layer electrodes, we investigate the influence of electrode architecture on 59.5 keV photon-counting detection and imaging. In this work, low-activity ²⁴¹Am radioactive gamma-ray sources were used as a low-dose monochromatic X-ray source.

Our results demonstrated that the multi-layer electrode strategy, particularly the Au/Pt/Ti/MAPbBr₃/Ti/Pt/Au configuration, significantly enhances both the detection performance and long-term stability of MAPbBr₃-based photon counting X-ray detectors. The improved charge extraction, suppressed dark current, and mitigated interfacial degradation achieved through this rational electrode design offer a promising pathway toward practical and reliable perovskite-based photon-counting X-ray detection systems.

2. Materials and Methods

Materials: Lead bromide (PbBr_2 ; 99%) and methylammonium bromide (MABr , 99%) were purchased from Sigma Aldrich, St. Louis, MO, USA. Dimethyl sulfoxide (DMSO, 99.9%) and dimethylformamide (DMF, 99.9%) were obtained from Aladdin. Metallic gold was purchased from Chinese reagent, China. All the commercial products were used as received.

Growth of MAPbBr_3 perovskite single crystals. High-quality MAPbBr_3 perovskite single crystals were grown by inverse temperature crystallization [36–38]. Specifically, 1 M MABr and 1 M PbBr_2 were dissolved in DMF solution. The solutions were filtered through a PTFE filter with a $0.22\ \mu\text{m}$ pore size. The filtrate was then transferred to a culture dish, which was placed on a programmable heating station (IKA-RET control-visc), and the growth temperature was 65 to 85 °C with a rate of $1\ ^\circ\text{C h}^{-1}$. For the metal electrodes, electrodes were deposited on the face of MAPbBr_3 perovskite single crystals by a metal mask under a vacuum of 6×10^{-4} Pa.

Characterization of the MAPbBr_3 perovskite single crystals. X-ray diffraction (XRD) patterns were obtained using an X'TRA system with a Cu target (Thermo Fisher Scientific, Ecublens, Switzerland). Optical absorption spectra were measured by UV-vis spectroscopy range from 300 to 2000 nm (Lab Tech Bluestar, Ortenberg, Germany). The ultraviolet photoelectron spectroscopy (UPS) was obtained using a PHI 5000 VersaProbe (ULVAC-PHI, Chigasaki, Japan). The PL and PL decay processes were measured using a SpectraMax instrument (Molecular Devices, San Jose, CA, USA). The Kelvin force probe microscopy was used with a Dimension Icon (Bruker, Herzogenrath, Germany). For the response time measurement, ^{241}Am was used as the radioactive source, a high voltage bias (0–50V) was applied to the device, and the signal was input into a pre-amplifier 142PC (ORTEC, Oak Ridge, TN, USA). The response to each alpha particle was traced using a Keysight oscilloscope DMOX4054A (Keysight Technologies, Santa Rosa, CA USA). Characterization of current–voltage was measured using a Keithley 4200SC semiconductor analyzer (Keithley, Solon, OH, USA).

Set-up of the X-ray photon-counting read-out circuits. The pre-amplifier 142PC (ORTEC, Oak Ridge, TN, USA). The shaping amplifier and baseline restorer (CREMAT, West Newton, MA, USA). The high-voltage and low-voltage DC sources (DONGWEN HIGH VOLTAGE, Tianjing, China). And the multi-channel analyzer (AP Techno, Hachimantai, Japan).

3. Results

As shown in Figure 1a, the energy band of the MAPbBr_3 single crystal was investigated. The band gap was fitted to 2.1 eV by the Talc plot (Figure S1). And the valence band maximum was measured at 5.7 eV by UPS (Figure S2). And the work function of Au, Pt, and Ti were chosen to be 5.1 eV, 5.3 eV and 4.3 eV from published work, respectively. Figure 1b shows the structure of detectors made of MAPbBr_3 single crystals with different electrodes, including $\text{Au}/\text{MAPbBr}_3/\text{Au}$, $\text{Au}/\text{Ti}/\text{MAPbBr}_3/\text{Ti}/\text{Au}$ and $\text{Au}/\text{Pt}/\text{Ti}/\text{MAPbBr}_3/\text{Ti}/\text{Pt}/\text{Au}$. An interdigitated electrode was chosen as shown in Figure 1c; the thickness of the three types of electrodes was 200 nm, and the area of the active area was $25\ \text{mm}^2$. Here, ^{241}Am radioactive sources, which emit 59.5 keV photons, were used as a monochromatic X-ray source. As shown in Figure 1d, the 59.5 keV monochromatic X-ray photons would incident on the detectors, while high voltage bias and ground would connect to each electrode, respectively.

Figure 1e shows the current density–voltage characterization of detectors with different electrode structures. The $\text{Au}/\text{Pt}/\text{Ti}$ multi-layer electrodes show nearly linear behavior, while the Au electrode or Au/Ti electrode shows back-to-back Schottky behavior. And the I-V characterization with different temperatures is shown in Figure S3. The $\text{Au}/\text{Pt}/\text{Ti}$ multi-layer seems to form a more Ohmic contact with the MAPbBr_3 single crystal. Further, the dark

current density under -50 V bias was investigated in Figure 1f; the Au/Pt/Ti multi-layer device shows the lowest dark current of 107 nA cm^{-2} , which was much lower than the Au/Ti device and the Au device of 372 nA cm^{-2} and $227\sim 445 \text{ nA cm}^{-2}$, respectively. For MAPbBr₃ single crystals with only Ti and Pt, the I-V characterization is shown in Figure S4.

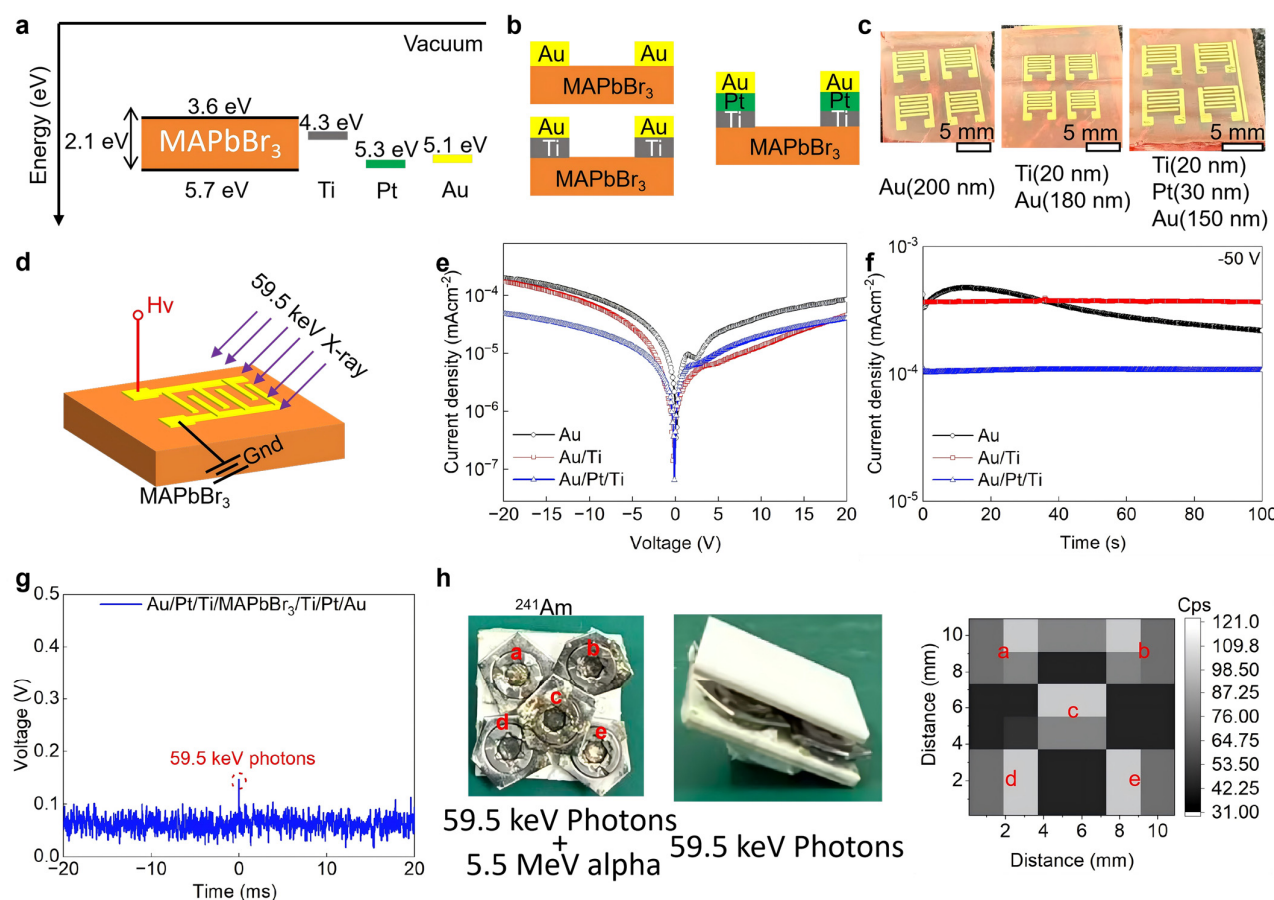


Figure 1. (a) Energy band diagram of MAPbBr₃ single crystal and different metals. (b) Device structure. (c) Optical photo of devices with different electrodes. (d) Diagram of the device under bias and 59.5 keV radiation. (e) I-V characterization of MAPbBr₃ single crystals with different electrodes. (f) I-T characterization of MAPbBr₃ single crystals with different electrodes under -50 V. (g) 59.5 keV photon response of Au/Pt/Ti/MAPbBr₃/Ti/Pt/Au. (h) Image of five ²⁴¹Am source, (a–e) represent the five different locations of ²⁴¹AM source.

For the Ti/MAPbBr₃ single crystal interface, Wenqing Zhang and co-author have demonstrated that Ti–N bonds formed at the interface of Ti/MAPbBr₃ single crystal effectively inhibit the electrochemical reaction and ultimately improve the operating stability under a high electric field [35]. However, the Ti electrode was easily oxidized in air. In addition, a Ti/Au electrode would cause significant inter-layer diffusion between Au and Ti, which could lead to device degradation. Thus, inserting a 20 nm thickness Pt layer could effectively prevent Ti segregation [39].

Owing to the low and stable dark current, the Au/Pt/Ti multi-layer device was used to detect 59.5 keV photons. As shown in Figure 1g, a clear potential pulse induced by a 59.5 keV photon can be observed. In contrast, for the Au/Ti multi-layer device and the Au-only device, the noise levels were too high to discriminate the useful signal (Figure S5). The response to alpha particles is presented in Figure S6.

Furthermore, five ²⁴¹Am radioactive sources were used as five point-like X-ray sources. A 1 mm thick Al₂O₃ ceramic sheet was placed to filter out alpha particles, allowing only

59.5 keV photons to pass through, as illustrated in Figure 1h. By analyzing the count rate (counts per second) of the Au/Pt/Ti multi-layer device at different positions, we successfully obtained an X-ray photon counting image.

The five ^{241}Am radioactive sources emit only approximately 1000 59.5 keV photons per second, resulting in a photon injection rate of just $100\text{ cm}^{-2}\text{ s}^{-1}$. According to the formula $D_{\text{air}} = \varphi \times \left(\frac{\mu_{\text{en}}}{\rho}\right) \times E$, where D_{air} is the dose rate, φ is the photon injection rate, E is the energy of each photon and $\frac{\mu_{\text{en}}}{\rho}$ is the mass energy absorption coefficient, which is $0.0303\text{ cm}^2\text{ g}^{-1}$ for air [32], the calculated dose rate is only 0.03 nGy s^{-1} . This value is far below the lowest detectable dose rate of typical energy-integrating X-ray detectors.

4. Discussion

After multi-layer electrode deposition, we further confirmed the surface property of MAPbBr_3 single crystals. Since the deposition temperature for metal Pt is much higher than that of Au, the high-temperature-vaporized Pt or Au may destroy the surfaces of MAPbBr_3 single crystals. As shown in Figure 2a, the surface property of MAPbBr_3 single crystals was first investigated by X-ray diffraction. The full width half height (FWHM) for 30.080 degrees was 0.2, 0.2 and 0.18 degrees for the Au, Au/Ti and Au/Pt/Ti devices, respectively. The X-ray diffraction shows that the crystallization quality of the three MAPbBr_3 single crystals after electrode deposition was close. Further, the photoluminescence of three MAPbBr_3 single crystals after electrode deposition was investigated in Figure 2b. The peak of the photoluminescence was located at 530 nm, and the photoluminescence intensity was nearly equal. And the time-resolved photoluminescence of 530 nm was further investigated in Figure 2c. Through exponential decay fitting, the surface lifetime was 0.32 ± 0.02 , 0.37 ± 0.02 and $0.35 \pm 0.04\text{ }\mu\text{s}$, and the bulk lifetime was 10.5 ± 3.7 , 10.5 ± 2.3 and $14.2 \pm 4.5\text{ }\mu\text{s}$ for the Au, Au/Ti and Au/Pt/Ti devices, respectively. As a result, the dark current density and noise behavior were attributed to the different electrodes, not to the crystallization quality differences in MAPbBr_3 single crystals.

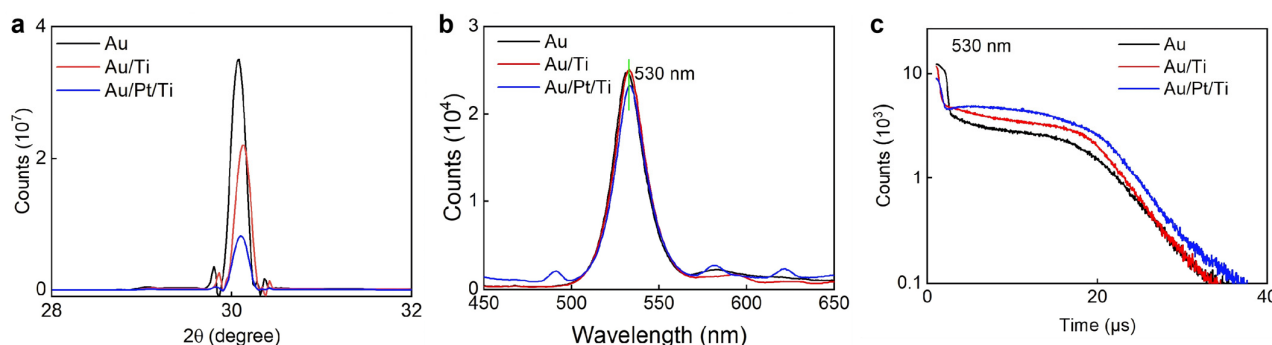


Figure 2. Characterization of MAPbBr_3 single crystals with different electrodes: (a) X-ray diffraction. (b) Photoluminescence under 350 nm. (c) Time-resolved photoluminescence of 530 nm.

To further understand the metal– MAPbBr_3 single crystal contact, here, Kelvin force microscopy (KFM) was used to measure the contact potential of different electrodes. The KFM results for the Au device are shown in Figure 3a; the roughness was 5 ± 1 and $25 \pm 3\text{ nm}$ for the Au area and the MAPbBr_3 single crystal region, respectively. And the contact potential was measured at $0.15 \pm 0.02\text{ V}$, resulting in an energy barrier of 80 mV in the Au– MAPbBr_3 interface. Similarly, for the Au/Ti device, the roughness was 3 ± 1 and $15 \pm 3\text{ nm}$ for the Au/Ti area and MAPbBr_3 single crystal region, respectively. Further, the contact potential was measured at $0.15 \pm 0.02\text{ V}$, resulting in an energy barrier of 100 mV in the Au/Ti– MAPbBr_3 interface. For the Au/Pt/Ti device, the roughness was 3 ± 1 and $15 \pm 3\text{ nm}$ for the Au/Ti area and MAPbBr_3 single crystal region, respectively. What's

interesting is that we can not observe a clear potential difference between Au/Pt/Ti and MAPbBr₃ single crystal. It also explained the Ohmic behavior as shown in the current density–voltage characterization.

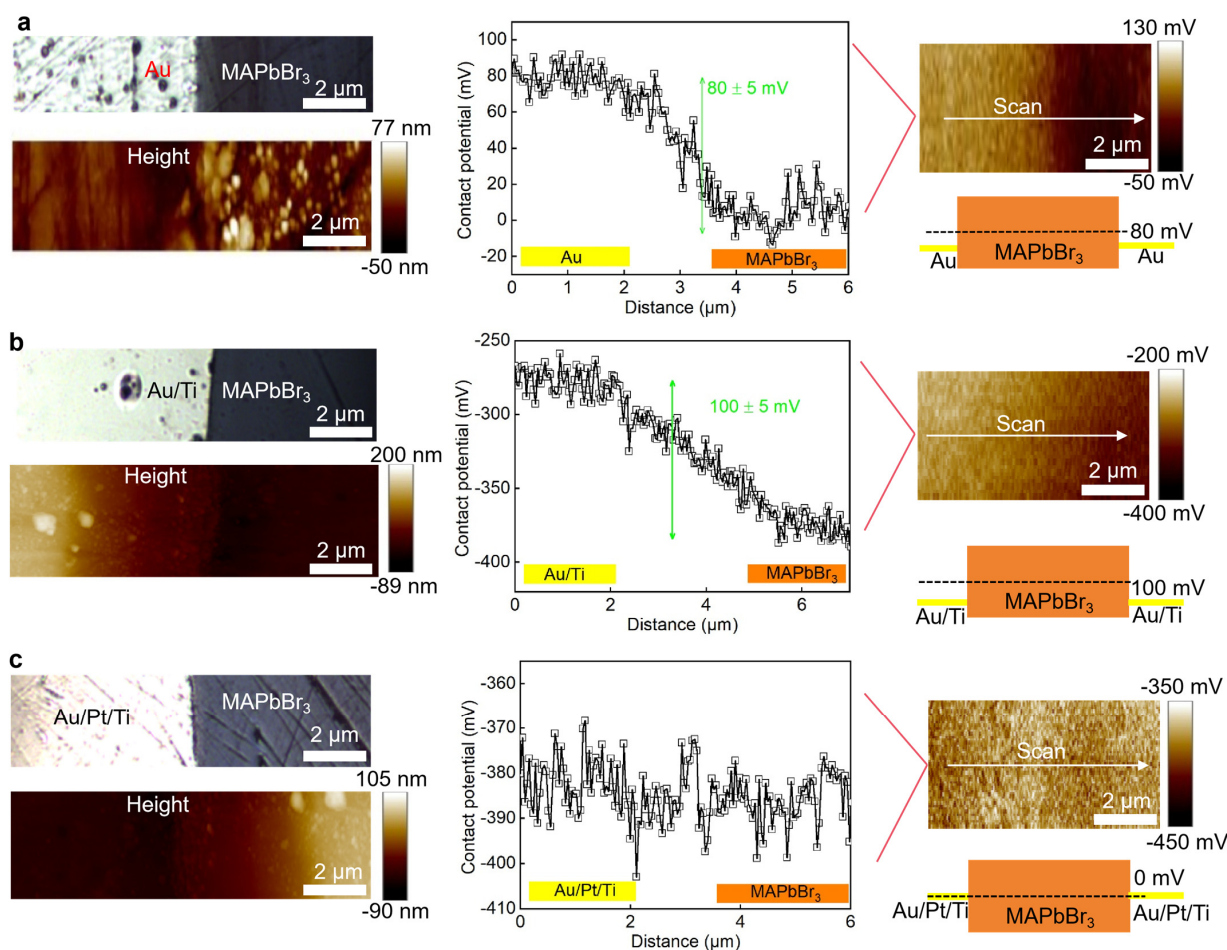


Figure 3. Kelvin force microscopy results: (a) Au/MAPbBr₃. (b) Au/Ti/MAPbBr₃. (c) Au/Pt/Ti/MAPbBr₃.

As shown in Figure 4a, the response speed for the Au/Pt/Ti device, at -50 V bias, the response speed reached 2.9 ± 0.2 μ s, corresponding to the maximum count rate of 34.4 k s^{-1} . As shown in Figure 4b, the counting stability of the Au/Pt/Ti device was 110 ± 15 for 59.5 keV photons for 30 min, demonstrating not bad counting stability. The Au/Pt/Ti device was also very stable. As shown in Figure 4c, the dark current after 6 months (107 nA) maintained almost the same dark current after 3 months (106 nA) stored in ambient. And the long-term irradiation stability was investigated in Figure S7.

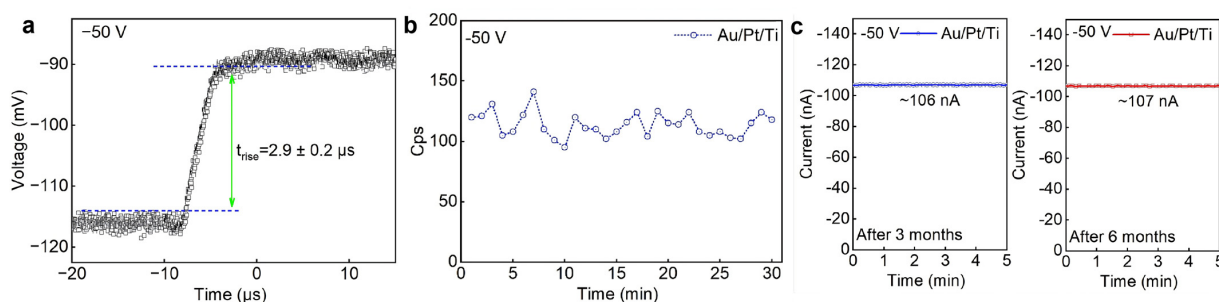


Figure 4. (a) Response time to single 59.5 keV photon. (b) Count per second stability. (c) Long-time stability of Au/Pt/Ti/MAPbBr₃/Ti/Pt/Au.

5. Conclusions

In this work, multi-layer electrodes including Au, Au/Ti and Au/Pt/Ti were investigated. Au and Au/Ti would form energy barriers when in contact with MAPbBr₃ single crystals, while Au/Pt/Ti could form Ohmic contact with MAPbBr₃ single crystals. Au/Pt/Ti multi-layer electrode shows the lowest dark current and noise, which enables 59.5 keV photon-counting detection and imaging. This work provided useful information about the electrode structure for lead halide perovskite-based optoelectronic devices.

Supplementary Materials: The following supporting information can be downloaded at <https://www.mdpi.com/article/10.3390/s26103030/s1>. Figure S1: Absorption curve of MAPbBr₃ single crystal; Figure S2: UPS result of MAPbBr₃ single crystal; Figure S3: Temp-dependent I-V characterization of Au/Pt/Ti device. Figure S4: I-V characterization results of MAPbBr₃ single crystal with only Ti and Pt electrodes; Figure S5: Noise of Au, Au/Ti and Au/Pt/Ti under −50 V bias; Figure S6: Alpha response of Au/Pt/Ti device. Figure S7: X-ray irradiation stability.

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