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Surface engineering of SnO₂ for improved perovskite/SnO₂ photodetectors

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Lead halide perovskite (LHP) heterojunctions have proven to be promising for achieving low-cost and efficient photodetection. However, the remarkable interfacial non-radiative recombination severely deteriorated the performance of the resulting devices. Herein, surface engineering of SnO₂ with ammonium tetrathiotungstate ((NH₄)₂WS₄) has been proposed to effectively passivate the interfacial defects at the LHP CH₃NH₃PbI₃/SnO₂ interface to fabricate high-performance photodetectors (PDs). Experimental and theoretical studies showed that the surface engineering with (NH₄)₂WS₄ resulted in S substituting for oxygen lattice of SnO₂, which passivated the surface oxygen vacancies of SnO₂. Further studies have shown that the S atoms at the surface of SnO₂ further suppressed the I vacancies and Pb vacancies at the bottom surface of CH₃NH₃PbI₃. Finally, improved CH₃NH₃PbI₃/SnO₂ PDs with the responsivity and detectivity of 0.41 A W⁻¹ and 5 × 10¹² Jones, respectively, at zero bias, have been demonstrated. The results presented in this work provide promising pathways to effectively passivate the interfacial defects of LHP/SnO₂ heterojunctions for achieving efficient photodetection in the future.

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Introduction

Photodetectors (PDs), which can directly convert photons into electrical charge, are a fundamental component of various modern photodetection and imaging systems, such as digital imaging and hyperspectral sensing.^{1–5} Recently, halide lead perovskites (LHPs) (ABX₃, where A is CH₃NH₃⁺ (MA⁺), HC(NH₂)₂⁺ (FA⁺); B is the Pb²⁺; X is the Cl⁻, Br⁻, and I⁻) have attracted tremendous attention in the optoelectronics community because of their superior optoelectrical properties including high absorption coefficient, long charge carrier diffusion length, and long charge carrier lifetime.⁶ Benefiting from these advantages, LHPs have been regarded as potential candidates for realizing low-cost and efficient light harvesting and photodetection.⁷ Notably, various kinds of LHP PDs, including narrowband, broadband, and wavelength-selective PDs, have been fabricated recently.^{8,9} Benefiting from various advantages, such as a combination of optoelectrical properties of both semiconductors and being free from the need for controlled n- and p-type doping of specific materials,^{10–13} LHP heterojunctions fabricated by preparing LHP and a different

semiconductor in sequence have been considered as essential building blocks for fabricating efficient and cost-effective self-powered PDs.^{14,15}

Recently, the rapid progresses of power conversion efficiency of LHP solar cells have motivated the explorations of advanced carrier transport layers.¹⁶ It is worth noting that wide bandgap semiconductor tin oxide (SnO₂) has been widely employed to fabricate efficient LHP solar cells recently, benefiting from its high electron mobility, deep conduction band, and low-temperature processabilities.¹⁷ Meanwhile, the construction of efficient PDs based on various LHP/SnO₂ heterojunctions has been further explored.¹⁸ Similar to light harvesting, efficient photodetection can be readily achieved through a combination of the efficient absorption of photons and the efficient collection of excess carriers. Benefiting from ion doping-assisted suppression of point defects and seed layer-assisted nucleation-induced suppression of grain defects, the bulk defects of LHP thin films have been remarkably suppressed.^{19,20} However, the interfacial defects induced non-radiative recombination have severely lowered the efficiency of carrier collection, greatly limiting the performance of LHP/SnO₂ heterojunction PDs.^{21,22} In general, the interfacial defects consist of the surface defects of the buried layer and the deposited layer. In the case of the LHP/SnO₂ heterojunctions, the SnO₂ thin films prepared with various techniques have suffered from the natural surface oxygen vacancies (V_{OS}).²² In addition, halide vacancies and lead vacancies have been evidenced at the bottom surface of LHPs owing to the weak bonding between LHPs and SnO₂.²³ It has been widely reported

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that both surface defects deteriorated the performance of LHP/SnO₂ heterojunction devices.²⁴ Very recently, a great amount of effort has been devoted to suppressing the interfacial defects for fabricating improved LHP/SnO₂ heterojunction PDs.²⁵ However, the existing literatures mostly focused on either passivating the surface V_{OS} of SnO₂ thin films or suppressing the halide vacancies and lead vacancies at the bottom surface of LHPs.^{26,27} For example, inorganic dielectric layers have been employed to passivate the surface V_{OS} of SnO₂ thin films.²⁸ Besides, various organic groups have been explored, but mainly suppress the halide vacancies and lead vacancies at the bottom surface of LHP thin films.²⁹ Practically, effective suppression of surface defects of both materials is fundamental for improving the performance of LHP/SnO₂ heterojunction PDs, while the related studies have seldom been reported so far.

Herein, surface engineering of SnO₂ with ammonium tetrathiotungstate ((NH₄)₂WS₄) has been proposed to realize an effective suppression of surface V_{OS} of SnO₂ and iodine vacancies (V_I) and lead vacancies (V_{Pb}) at the bottom surface of MAPbI₃. It has been shown that the surface V_{OS} of SnO₂ were effectively passivated *via* the sulfur (S) substitution for oxygen (O) lattice. In addition, the S atoms at the surface of SnO₂ further suppressed the V_I and V_{Pb} defects at the bottom surface of MAPbI₃. Collectively, performance-improved MAPbI₃/SnO₂ PDs with the responsivity and detectivity as high as 0.41 A W⁻¹ and 5 × 10¹² Jones, respectively, at the zero bias, have been demonstrated.

Results and discussions

The MAPbI₃/SnO₂ PDs were fabricated by spin-coating SnO₂ and MAPbI₃ thin films in sequence on an ITO substrate (Fig. 1a) (see experiments in SI for details). The treatment of surface engineering of SnO₂ was realized by adding (NH₄)₂WS₄ into SnO₂ aqueous solution, where the surface V_{OS} of SnO₂ were passivated through the S substitution during stirring. The morphological and structural properties of samples were characterized by scanning electron microscopy (SEM) and X-ray diffraction (XRD) techniques. The suppression of surface V_O defects in SnO₂ and V_{Pb} of MAPbI₃ has been verified through steady-state and transient photoluminescence (PL), X-ray photoelectron spectroscopy (XPS) characterizations, and density functional theory (DFT) calculations.

Structure and elements analysis have confirmed the crystallization of SnO₂ after spin-coating of pure SnO₂ and SnO₂/(NH₄)₂WS₄ aqueous solutions (Fig. S1–S2, SI). Fig. 1b and c show the typical SEM images of SnO₂ and S-SnO₂ thin films. It shows that the S-SnO₂ thin films exhibit an improved morphology in terms of improved compactness and uniformity, while the SnO₂ thin films show a rough surface with notable gaps. This suggests that the (NH₄)₂WS₄ additive benefits the preparation of high-quality SnO₂ thin films from nanoparticles. Due to the small thickness and limited resolution of the SEM technique, the thickness of SnO₂ and S-SnO₂ thin films was not obtained, but has been estimated as ~10 nm from the previous reports.³⁰ Notably, the compact and uniform morphology of S-SnO₂ thin films exhibits a reduction of carrier scattering effect.

As revealed from current–voltage (*I*–*V*) measurements (Fig. S3, SI), the S-SnO₂ thin films feature a reduced resistivity. As a result, the carrier collection at the MAPbI₃/SnO₂ interfaces is expected to be improved.

Fig. 1d–g present the SEM images of MAPbI₃ thin films spin-coated on SnO₂ and S-SnO₂ thin films. As shown, both samples exhibit a similar morphology with comparable grain size, compactness, and thickness. Specifically, the grain sizes and thickness of both samples were characterized as around 450 ± 32 nm (Fig. 1d and e) and 230 nm (Fig. 1f and g), respectively. This suggests that surface engineering of SnO₂ with (NH₄)₂WS₄ seems not to influence the growth processes of MAPbI₃ thin films.

Structural and optical properties measurements were carried out to further compare the quality of MAPbI₃ thin films prepared on SnO₂ and S-SnO₂ thin films, and the results are shown in Fig. 2. It shows that both MAPbI₃ thin films exhibit similar XRD patterns (Fig. 2a), which can be indexed into the orthorhombic phase. Careful analysis shows that both MAPbI₃ thin films exhibit similar crystalline quality from the XRD peaks' positions and full width at half maximums (FWHMs, Table S1, SI). Such a claim can be supported from small differences in their steady and transient PL spectra (Fig. 2b). As shown, the PL intensity and carrier recombination processes of both MAPbI₃ thin films are almost same. Collectively, surface engineering with the (NH₄)₂WS₄ has improved the morphology of SnO₂ thin films, while it has neglected roles in subsequent crystallization of MAPbI₃ thin films.

To verify the suppression of surface V_{OS}, XPS measurements on the SnO₂ thin films were performed. Notably, the XPS method has shown strong effectiveness in analyzing surface chemical composition.^{31,32} Fig. 3a illustrates the core-level spectra of O 1s of SnO₂ and S-SnO₂ thin films. Both spectra can be fitted into three peaks (peak I, II, and III), which correspond to various states of O at the SnO₂ surface. As can be seen, the binding energies are observed at 530.5 eV, 531.3 eV, and 532.0 eV, which can be attributed to lattice O, V_O, and adsorbed oxhydryl (OH⁻), respectively.³³ Notably, the concentration of various components can be semiquantitatively evaluated from the relative intensities of corresponding XPS peaks.³³ Accordingly, it can be concluded that S-SnO₂ thin films show a reduced surface V_O from the decreased intensity of peak II compared to SnO₂ thin films. Further observations suggest that the reduction of surface V_{OS} is probably caused by the S substituting for O in S-SnO₂ thin films. On the one hand, a remarkable peak can be observed in S 2p XPS spectrum (Fig. 3b), which verifies the successful doping of S in S-SnO₂ thin films. On the other hand, the binding energies of Sn 3d in S-SnO₂ thin films redshift by around 0.3 eV compared to those in SnO₂ thin films (Fig. 3c). This is attributed to the increased electron density around Sn atoms,³⁴ which well correlates to the formation of the Sn–S bond in the current situation.

The above observations indicate that interface engineering with (NH₄)₂WS₄ resulted in a doping of S atoms, which suppressed the surface V_{OS} in S-SnO₂ thin films. However, it is difficult to reveal the doping processes at this stage, but can be expected as follows:



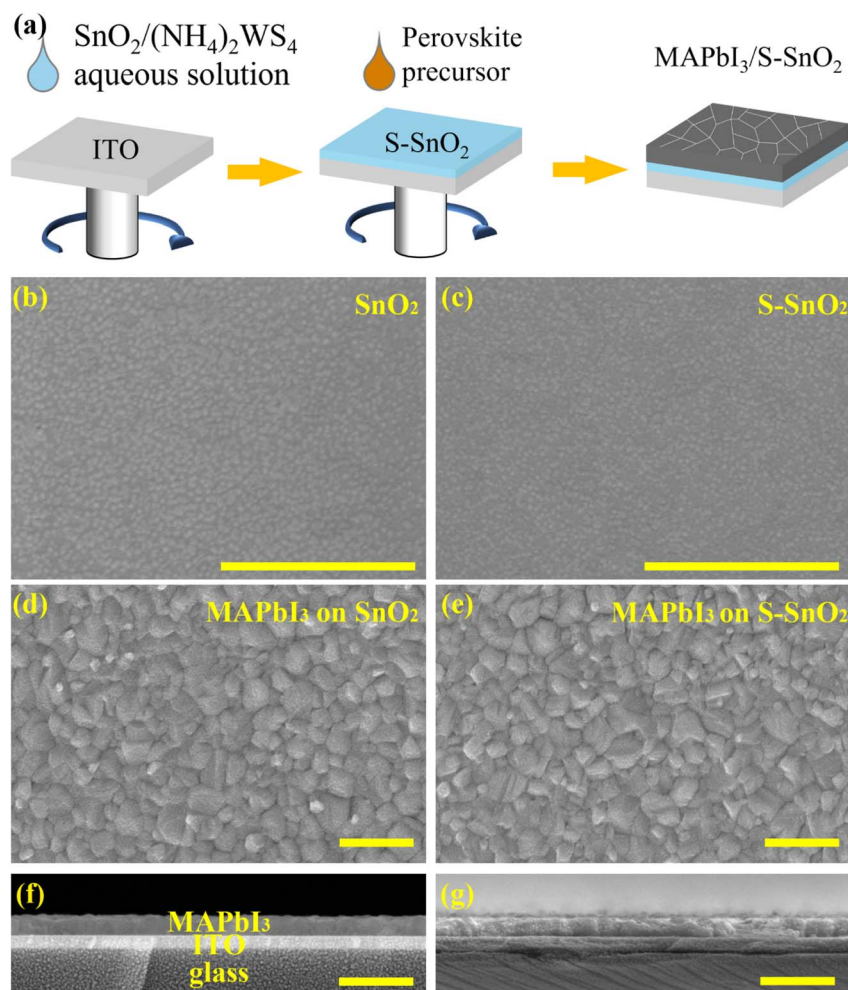
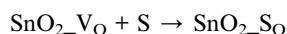


Fig. 1 Morphology of SnO_2 and MAPbI_3 thin films. (a) Scheme of experimental details for preparing $\text{MAPbI}_3/\text{S-SnO}_2$ heterojunctions. SEM images of (b) SnO_2 , (c) S-SnO_2 , (d and f) MAPbI_3 thin films grown on SnO_2 , and (e and g) MAPbI_3 thin films grown on S-SnO_2 .



where $\text{SnO}_2\text{-V}_\text{O}$ is the SnO_2 with a surface V_O , $\text{SnO}_2\text{-S}_\text{O}$ is the SnO_2 with S substituting for O lattice. Herein, to better understand the mechanisms of suppression of surface $\text{V}_\text{O}\text{S}$ by S doping, DFT calculations were carried out. As reported before,

the probability of S substitution for surface O lattice (S_O) can be revealed from their formation energies (ΔE),³⁵ which can be calculated from the eqn (1):

$$\Delta E(\text{SnO}_2\text{-S}_\text{O}) = E(\text{SnO}_2\text{-S}_\text{O}) - [E(\text{SnO}_2\text{-V}_\text{O}) + E(\text{S})] \quad (1)$$

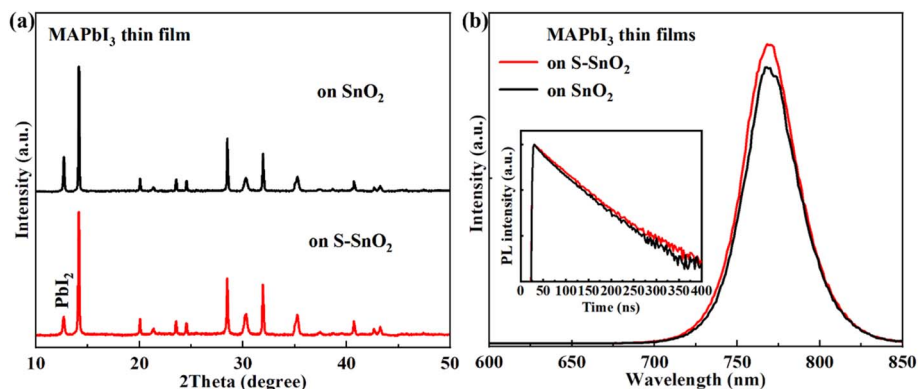


Fig. 2 Structural and optical properties of MAPbI_3 thin films grown on SnO_2 and S-SnO_2 . (a) XRD and (b) PL. The inset in (b) shows the TRPL.



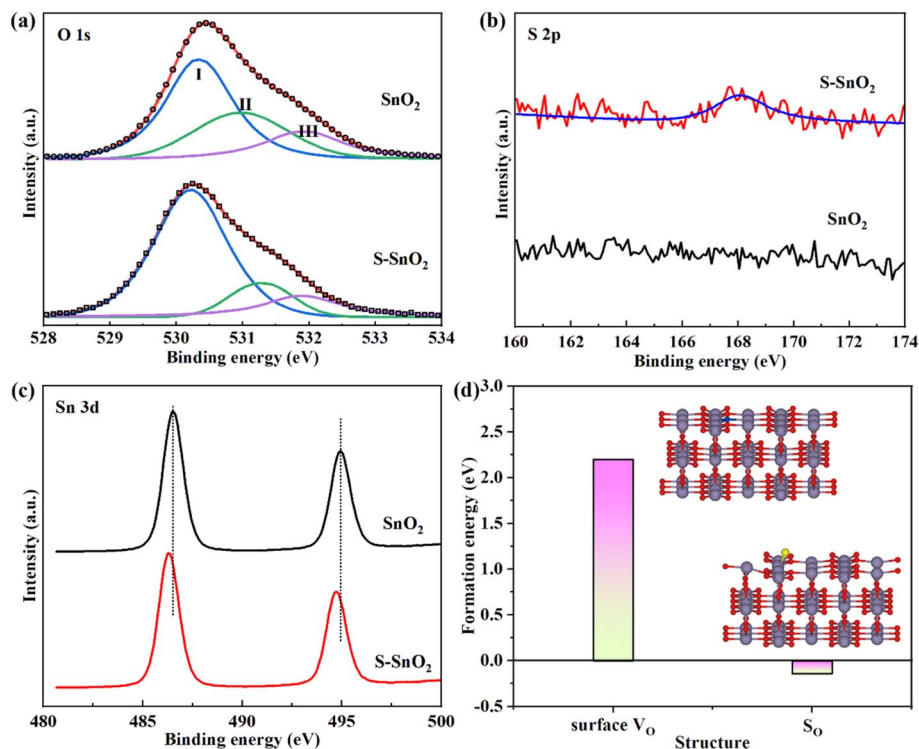


Fig. 3 Passivation of surface V_O s of SnO_2 by S atoms. (a–c) XPS data of (a) O, (b) S, and (c) Sn atoms. (d) Formation energies of surface V_O s and S substituting for O of SnO_2 . The inset in (d) indicates the corresponding supercells, where red, gray, blue, and yellow balls are O, Sn, V_O , and S, respectively.

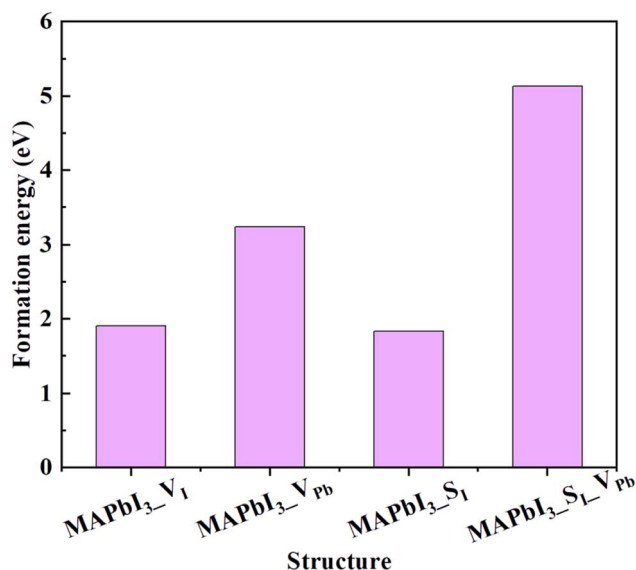


Fig. 4 Formation energies of V_I , V_{Pb} , S substituting for I, and S substituting for I with a V_{Pb} nearby of $MAPbI_3$. The corresponding supercells are depicted in Fig. S4 (SI). The formation energies were calculated using formulas similar to eqn (1).

where $E(SnO_2-S_O)$, $E(SnO_2-V_O)$, and $E(S)$ are the total energies of the supercell with S substitution for surface O and that with a surface V_O , and the energy of individual S atom, respectively. The formation energy of SnO_2-V_O structure ($\Delta E(SnO_2-V_O)$) was

calculated with a similar scenario. Fig. 3d depicts the formation energies of SnO_2 with a surface V_O and S substitution for surface O. It can be seen that the formation energy of surface V_O is as high as 2.198 eV. This suggests that the high-density surface V_O (Fig. 3a) cannot be related to thermodynamic effects, but might be attributed to mechanical effects between the nanoparticles or hydrogen bonding in the solvent environment. Notably, the formation energy of S substitution for surface O was calculated as low as -0.141 eV. This shows that the S_O structure is thermally preferred, which demonstrates the effective suppression of surface V_O s of SnO_2 through the surface engineering with the $(NH_4)_2WS_4$.

It has been noticed that S atoms on the SnO_2 surface showed a strong electrostatic interaction with Pb ions of $MAPbI_3$ compared to O of SnO_2 .³⁶ In this case, the interfacial S atoms are expected to fill the V_I and further suppress the formation of V_{Pb} at the bottom surface of $MAPbI_3$ thin films. Since the small penetration depth of X-ray, the local chemical states of Pb and I atoms at the $MAPbI_3/SnO_2$ interface cannot be experimentally explored from XPS techniques. To demonstrate the hypotheses, the formation energies of $MAPbI_3$ with a V_I ($MAPbI_3-V_I$), $MAPbI_3$ with a V_{Pb} ($MAPbI_3-V_{Pb}$), $MAPbI_3$ with a S atom substituting for I lattice ($MAPbI_3-S_I$), and $MAPbI_3-S_I$ with a V_{Pb} nearby S ($MAPbI_3-S_I-V_{Pb}$) (Fig. S4, SI) were calculated, and the results are provided in Fig. 4. It shows that the $MAPbI_3-S_I$ structure shows a reduced formation energy (1.84 eV) compared to $MAPbI_3-V_I$ (1.91 eV), and the $MAPbI_3-S_I-V_{Pb}$ exhibits a reduced formation energy (3.25 eV) compared to $MAPbI_3-V_{Pb}$



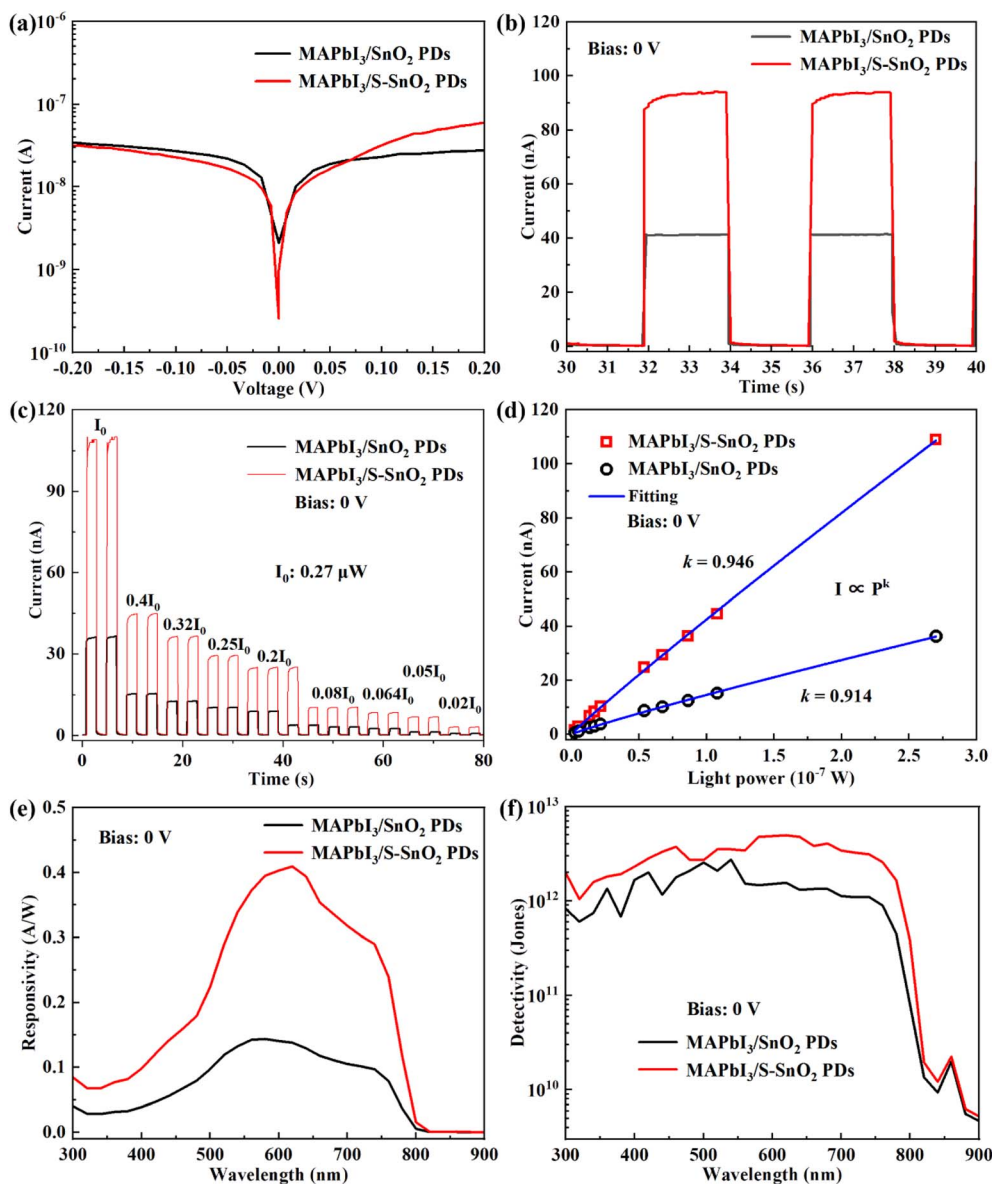


Fig. 5 Performance of MAPbI₃/SnO₂ PDs. (a) I - V curves captured in dark. (b) I - t curves captured under pulsed illumination. (b and c) I - t curves and photocurrent captured under different light powers. Data in d were extracted from c. (e) Responsivity. (f) Detectivity.

(5.14 eV). This suggests that the S atoms at the surface of SnO₂ thin films have remarkable potential to suppress the V_i and V_{pb} defects at the bottom surface of MAPbI₃ thin films.

After sputtering Au electrodes, MAPbI₃/SnO₂ heterojunction PDs were fabricated (Fig. S5, SI). As shown in Fig. S6 (SI), among various concentrations, engineering SnO₂ NPs with 1.0 mg mL⁻¹ of (NH₄)₂WS₄ produced the optimal MAPbI₃/S-SnO₂ PDs, resulting in the highest photocurrent. Accordingly, the performance of MAPbI₃/S-SnO₂ PDs, prepared with 1.0 mg mL⁻¹ (NH₄)₂WS₄ engineering, is examined in detail below. Fig. 5a illustrates the I - V curves of MAPbI₃/SnO₂ and MAPbI₃/S-SnO₂ PDs captured in the dark. It shows that the MAPbI₃/S-SnO₂ PDs exhibit reduced current under reverse bias and reduced turn-on voltage under forward bias. As shown in Fig. 1 and 2, the MAPbI₃ thin films prepared on SnO₂ and S-SnO₂ exhibit a similar crystalline quality.

Thus, the reduced reverse current and turn-on voltage can be attributed to the suppression of interfacial defects at the MAPbI₃/S-SnO₂ interface, as discussed above.

Fig. 5b shows the current-time (I - t) curves of MAPbI₃/SnO₂ and MAPbI₃/S-SnO₂ PDs at zero bias with 540 nm illumination switched on and off. It can be seen that both devices show excellent on-off switching performance. Notably, the MAPbI₃/S-SnO₂ PDs show an improved photocurrent, which increased by around 2.2 times compared to MAPbI₃/SnO₂ PDs. According to above discussions, this should be caused by the suppressed interfacial non-radiative recombination at MAPbI₃/S-SnO₂ interfaces. Further studies suggest that both devices exhibit nearly a linear response to light power. As shown in Fig. 5c and d, the photocurrents of MAPbI₃/SnO₂ and MAPbI₃/S-SnO₂ PDs nearly scale linearly with light power from 10⁻⁹ W to 10⁻⁷ W,



Table 1 Comparison of photodetection performance of MAPbI₃/SnO₂ heterojunction PDs and related devices reported before

Device	Bias (V)	Responsivity (A/W)	Detectivity (Jones)	Ref.
Spiro-OMeTAD/FAPb(I/Br) ₃ /SnO ₂	1.0	0.0438	3.56×10^{13}	37
Spiro-OMeTAD/MAFAPb(Br/I) ₃ /SnO ₂	1.0	0.0722	4.67×10^{13}	38
SnO ₂ /MAPbI ₃ /MoO ₃	0	0.0509	2.23×10^{12}	39
CsCu ₂ I ₃ /GaN	0	0.10634	9.24×10^{11}	40
MAPbI ₃ /ZnO	7	2.73	1.09×10^{12}	41
C ₆₀ /MAPbI ₃ /GaN	0	0.198	7.96×10^{12}	42
TiO ₂ /MAPbI ₃	-0.8	0.405	2.07×10^{11}	43
PEDOT:PSS/MAPbI ₃ /PTB7-Th:COTIC-4F	-0.5	0.58	1.64×10^{12}	44
MAPbI ₃ /SnO ₂ @r-PMO ₁₁ V	3	0.039	9.3×10^{10}	45
MAPbI ₃ /S-SnO ₂	0	0.41	4.6×10^{12}	This work

suggesting that MAPbI₃/SnO₂ heterojunction PDs can be used to detect the light power.

The performance of MAPbI₃/SnO₂ and MAPbI₃/S-SnO₂ PDs is further studied from the responsivity and detectivity, and the results are shown in Fig. 5c and d. As reported before, the responsivity (R) and detectivity (D^*) can be calculated from eqn (2) and (3) provided below:

$$R = \frac{I_{\text{ph}} - I_{\text{dark}}}{P \times S} \quad (2)$$

$$D^* = \frac{R}{\sqrt{2eI_{\text{dark}}/S}} \quad (3)$$

where I_{ph} and I_{dark} are the photocurrent and dark current, P is the light power density (in W cm^{-2}), S is the device area (the area of the top Au electrode, 0.01 mm^2), e is the electric charge ($1.6 \times 10^{-19} \text{ C}$). As depicted in Fig. 5e and f, the photoresponsivity and detectivity of MAPbI₃/SnO₂ PDs are 0.14 A W^{-1} and 2×10^{12} Jones, respectively, at the zero bias, which increased by around 2.9 times (0.41 A W^{-1}) and 2.3 times (4.6×10^{12} Jones) for MAPbI₃/S-SnO₂ PDs. The improved

photodetection performance of MAPbI₃/S-SnO₂ PDs is mainly attributed to the suppressed interfacial non-radiative recombination, which benefits from the effective interfacial passivation induced by the surface engineering with $(\text{NH}_4)_2\text{WS}_4$ as evidenced above. Table 1 summarizes a comparison of the photodetection performance of the MAPbI₃/S-SnO₂ PDs shown herein and related PDs reported before, highlighting the critical roles of surface engineering with $(\text{NH}_4)_2\text{WS}_4$ in improving the performance of MAPbI₃/SnO₂ heterojunction PDs.

Further studies reveal that surface engineering of SnO₂ with $(\text{NH}_4)_2\text{WS}_4$ improved the stability of photodetection. As shown in Fig. 6a and b, the photocurrent of MAPbI₃/SnO₂ PDs decreases to 85% of the initial value under pulsed illumination (Fig. 6a) and 60% under steady illumination (Fig. 6b) over 3600 s. In comparison, the photocurrent slightly decreases (2% and 10%) in MAPbI₃/S-SnO₂ PDs. Generally, the interfacial non-radiative recombination induces substantial thermal effects under illumination,⁴⁶ which account for the inferior stability observed in MAPbI₃/SnO₂ PDs. Thus, the improved stability of photodetection of MAPbI₃/S-SnO₂ mainly benefits from the effective interfacial passivation as discussed above.

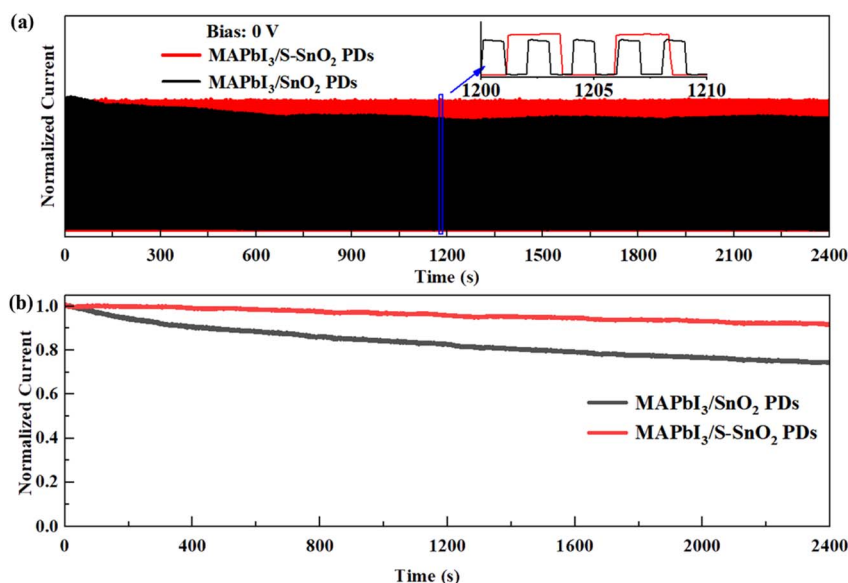


Fig. 6 Stability of photodetection of MAPbI₃/SnO₂ PDs. $I-t$ curves captured (a) under pulsed illumination and (b) steady illumination.



In conclusion, surface engineering of SnO₂ with (NH₄)₂WS₄ has been demonstrated to effectively passivate the interfacial defects to fabricate improved MAPbI₃/SnO₂ PDs. It has been shown that the surface engineering with (NH₄)₂WS₄ resulted in S substituting for O lattice of SnO₂, which passivated the surface V_{OS} of SnO₂. Meanwhile, theoretical studies suggested that the S atoms at the surface of SnO₂ further suppressed the V_I and V_{Pb} at the bottom surface of MAPbI₃. Accordingly, the interfacial non-radiative recombination has been suppressed. On this basis, improved MAPbI₃/SnO₂ PDs have been fabricated, where the responsivity and detectivity of 0.41 A W⁻¹ and 5 × 10¹² Jones at the zero bias have been demonstrated. The results shown in this work pave the way for efficiently passivating the interfacial defects of LHP/SnO₂ heterojunctions in the future.

Conflicts of interest

The authors declare no conflict of interest.

Data availability

The data that support the findings of this study are available from the corresponding authors upon reasonable request.

Supplementary information (SI): experiments, and supporting figures including XRD and XPS results of SnO₂ and S-SnO₂ thin films, *I*-*V* curves of Au/SnO₂ (S-SnO₂)/ITO structures, supercells of MAPbI₃ containing various defects, and a photo image of MAPbI₃/SnO₂ PDs, statistical results of dark current and photocurrent of MAPbI₃/SnO₂ PDs treated with different (NH₄)₂WS₄, and supporting Tables for comparison of crystallinity of MAPbI₃ thin films prepared on SnO₂ and S-SnO₂. See DOI: <https://doi.org/10.1039/d5ra10121b>.

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