Electric modulation of conduction in MAPbBr₃ single crystals

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Abstract: The resistive switching (RS) mechanism of hybrid organic–inorganic perovskites has not been clearly understood until now. A switchable diode-like RS behavior in MAPbBr₃ single crystals using Au (or Pt) symmetric electrodes is reported. Both the high resistance state (HRS) and low resistance state (LRS) are electrode-area dependent and light responsive. We propose an electric-field-driven inner p–n junction accompanied by a trap-controlled space-charge-limited conduction (SCLC) conduction mechanism to explain this switchable diode-like RS behavior in MAPbBr₃ single crystals.

Keywords: resistive switching (RS); rectifying behavior; hybrid organic–inorganic perovskites; p–n structure

1 Introduction

Hybrid organic–inorganic perovskites (HOIPs) have been dynamically investigated for the potential applications in solar cells [1], light-emitting diodes [2,3], photodetectors [2,3], lasers [4], and transistors [5]. For perovskite solar cells, a large electrical hysteresis in current–voltage (*I*–*V*) curves has been frequently reported, which is generally attributed to ferroelectricity [6], charge trapping [7], or ion migration [8]. Although the large hysteresis needs to be eliminated in optoelectronic devices, this phenomenon inspired the observation of resistive switching (RS) behavior in hybrid perovskite films [9–16], which is exploited for multilevel data storage.

RS phenomena have attracted a great deal of attention

in the past decades. Generally, the RS devices with a capacitor-like structure could be switched by an external bias to two different resistance states: a high resistance state (HRS) and a low resistance state (LRS). A wide variety of inorganic, organic, and organic–inorganic hybrid materials have been found to display RS features by far [17,18]. Yoo *et al.* [9] reported the RS behavior for the first time in MAPbI_{3–x}Cl_x (MA is shorted for CH₃NH₃⁺) perovskite films, and proposed a mechanism based on the trap-controlled space-charge-limited conduction (SCLC). Recently, interface-type RS was observed in MAPbBr₃ thick films (1 mm) by Guan *et al.* [10]. They proposed that the switching mechanism could be due to the migration of MA vacancies and the modification of the interfacial Schottky barrier. The filamentary-type RS has also been observed in several other studies on MAPbI₃-based thin films (100–400 nm), where the conducting filaments were attributed to the migration of anion defects [11–13] or Ag ions from the

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redox reactions of Ag electrode [14,15]. Moreover, the RS behavior of HOIPs could be strongly influenced by illumination. Zhou *et al.* [16] found that the set voltage could be greatly lowered under light illumination in MAPbI₃-based RS devices. However, in Ref. [19], we found no meaningful RS behavior in an individual MAPbI₃ nanowire unless under light illumination. These results clearly indicate that many open questions regarding RS phenomena and switching mechanism in HOIP-based RS devices remain. It is also worth noting that a rather high ON/OFF ($> 10^9$) ratio was observed in quasi-2D halide perovskite-based devices with excellent endurance by Kim *et al.* [20,21], which implies the huge potential commercial applications of HOIP-based RS memories.

In this work, MAPbBr₃ single crystal was chosen for the conduction study, because MAPbBr₃ is non-polar (centrosymmetric) [22] and therefore ferroelectricity could be excluded, and the influence of grain boundaries can also be ignored. Compared with polycrystalline thin films, MAPbX₃ single crystals present lower trap density and much better environmental stability, which can be considered as an ideal platform for investigating their intrinsic physical properties. The electric-field-driven resistance switching accompany by a diode-like behavior was produced in the Au/MAPbBr₃/Au structure. The diode-like switchable effect can be explained by the formation of reversible p–n junctions induced by ion immigration in the crystals. The area-proportional conduction indicates an interface-type RS behavior, which is related to the charge trapping/detrapping process at the interface.

2 Experimental

The MAPbBr₃ single crystals were prepared from solution by inverse temperature crystallization (ITC) method [23]. The raw materials, methylammonium bromide (CH₃NH₃Br, 99.5%), lead bromide (PbBr₂, 99.9%), N, N-dimethylformamide (DMF, 99%), and dimethylsulfoxide (DMSO, 99%) were used as received. CH₃NH₃Br and PbBr₂ with a molar ratio of 1:1 were dissolved in DMSO–DMF (7:3 by volume) to form a solution, and MAPbBr₃ was crystallized at the temperature from 60 to 100 °C with a heating rate of 0.2–0.5 °C/h.

An X-ray diffractometer (XRD, Empyrean, PANalytical B.V., the Netherlands) was used for phase identification at room temperature. The absorption spectra were measured

in the wavelength range of 400–800 nm using a UV spectrometer (SpectraPro-275, Acton Research Corporation, China). Au (100 nm) was used as the electrode for electrical characteristics. The top-electrode has two different sizes with 6 mm × 6 mm and 6 mm × 3 mm, and the bottom electrode has a size of 6 mm × 7 mm. The *I*–*V* measurements were carried out using a semiconductor analyzer (FS380, Platform Design Automation, Inc., China) assisted with a probe station (Lakeshore TTPX, USA). To avoid the influence of the environmental gases and moisture [24], the samples were kept in a vacuum chamber for electrical characteristics with a pressure of 10^{−4} mbar.

3 Results and discussion

The inset of Fig. 1(a) shows an optical image of an MAPbBr₃ single crystal with dimensions of 6 mm × 7 mm × 2 mm. Figure 1(a) presents the typical XRD 2θ scan data of the MAPbBr₃ single crystal at room temperature. The crystal adopts a pure cubic phase with good crystallization and high quality, which could be further confirmed by the surface and cross-section scanning electron microscopy (SEM) images (Fig. S1 in the Electronic Supplementary Material (ESM)). The element mapping results (Fig. S2 in the ESM) reveal that the partitioning and segregation of Br or Pb are absent in the crystals. The absorbance of MAPbBr₃ (Fig. 1(b)) shows a clear band edge cutoff at 568 nm with no excitonic signature, matching the values of the band edge cutoff and the photoluminescence peak position reported earlier for the same single crystals grown using ITC process [23]. The optical band gap of MAPbBr₃ could be determined to be 2.17 eV by extrapolating the linear region of the absorption edge to the energy-axis intercept, which is similar to the report in Ref. [22].

Figure 2 shows the dark current of the Au/MAPbBr₃/Au structure with different scanning rates and directions in two different scales. The arrows in Fig. 2 depict the bias scanning directions. The sequence of voltage sweep is 0 → +80 V → 0 → −80 V → 0, where the voltage is termed as positive when the top-electrode is positively biased. A clear switchable diode-like RS behavior is observed in Fig. 2, which is quite different from Refs. [9–16] on HOIP thin film devices. In most of these films, only RS but no diode-like behavior was mentioned. The diode direction can be switched during

Fig. 1 (a) XRD and (b) absorption spectra of MAPbBr₃ single crystals. Inset in (a): image of one of the measured crystals grown from solution; inset in (b): absorbance versus photon energy and the determined band gap E_g , where α is the absorption coefficient, h is Planck constant, and ν is the frequency (a proportion of light velocity to wavelength, $\nu = c/\lambda$).

Fig. 2 I – V curves of an Au/MAPbBr₃/Au device in (a) log-scale and (b) normal-scale in the dark with different scanning rates. All the measurements were conducted at room temperature in a vacuum chamber.

the sweep process in the dark. During the scan path 2, the device switches to the LRS. When the scan direction reverses in the path 3, the current abruptly decreases and the device switches to HRS. The ON/OFF ratio of the dark current is ~ 54 at a bias of +10 V. Figure S3 in the ESM displays the I – V curves of an Au/MAPbBr₃/Au device in the dark with different measurement voltages. It is interesting to find that the switchable diode-like RS is observed even under a low voltage at 1 V. The I – V curves show clearly variation in the “set” voltages without an evident electroforming process.

We now focus on the possible origin of the switchable diode-like RS. The switchable diode-like RS generally occurs in ferroelectric devices [25], where the barrier height at the ferro/electrode interface could be controlled by the switchable ferroelectric polarization. Actually, the switchable diode behavior has been observed in Au/MAPbI₃/ITO thin film devices, which results in a giant switchable photovoltaic effect under

illumination [26]. According to the proposed mechanism in Refs. [26,27], since MAPbBr₃ is non-ferroelectric, the switchable diode can be interpreted in terms of the defect electromigration process, as shown in Fig. 3. Many types of point defects have been reported in the HOIPs, such as positively-charged Br vacancy ($V_{\text{Br}}^{\bullet}$), negatively-charged Pb, and MA vacancies (V_{Pb}'' and V_{MA}'). $V_{\text{Br}}^{\bullet}$ can lead to n-type doping, whereas V_{Pb}'' and V_{MA}' result in p-type doping [28]. Provided that the defects are mobile charges, they can move through the sample under electric fields to find a new thermodynamic equilibrium. For instance, V_{MA}' can move and pile up near the bottom surface for negative bias conditions. Thus, the top surface region acquires n-type carriers, whereas the region near the bottom electrode becomes p-type. Yuan *et al.* [29] have reported a direct observation of MA⁺ ion and V_{MA}' migration under a bias field. The reversible inner p–i–n structure induced by ion drift has also been confirmed directly in MAPbI₃ thin films [26].

The rectifying I – V traces (paths 2→3 and 4→1) are attributed to the p–n junction created through the asymmetric distribution of charged defects, which is also believed to induce a RS behavior directly. The LRS can be attributed to the creation of p–n junction. By applying an opposite electric field, the inhomogeneous donor and/or acceptor distribution can be reversed to an evenly distributed insulating state (HRS). For a full sweep cycle (Fig. 2), scan path 1 (HRS) is quite sensitive to the scanning rate, which just reveals the defect electromigration process. Path 1 displays the forming process of the p–n structure from an evenly defect distribution. After the creation of inner p–n junction, the Au/MAPbBr₃/Au structure shows a LRS and its current is then not sensitive to scan speed (path 2 in Figs. 2(a) and 2(b)). The I – V traces of paths 3 and 4 show strongly scan speed-dependent because it corresponds to the establishment of a reverse p–n junction (points c, d, and e in Fig. 3), where the defect electromigration process is time- and voltage-dependent.

Although the defect electromigration approach can be used to describe the switchable diode-like RS in MAPbBr₃ single crystal-based devices, the detailed

charge transport mechanism is still unclear. In the previous reports on HOIP-based RS devices, the switching operations were attributed to the creation and annihilation of conducting filaments [11–13]. However, the filamentary conduction and the inner p–n junction cannot coexist. Furthermore, the filamentary scenario cannot explain the asymmetrical and electrode-area dependent I – V results. Figure 4 displays the RS behavior of an Au/MAPbBr₃/Au device with different sizes of top-electrode. It can be seen that both the HRS and LRS are electrode area-dependent, which could be regarded as a rejection of a filamentary-type RS [30]. Furthermore, as shown in Fig. 2, the hysteresis loops in one sweep cycle obtained under forward and reverse biases are asymmetrical, which further excludes the interface-type RS nature. These asymmetrical loops can be observed more conspicuously in a Pt/MAPbBr₃/Pt device (Fig. 5).

The most common RS process related to electronic mechanisms include Schottky barrier modulation, tunneling at the interface, hopping conduction, and charge trapping/detrapping at the interface or in the bulk [9,30]. It was previously reported that Au and Pt form an ohmic

Fig. 3 Schematic diagram describing the rectifying and RS behavior of electronic states in MAPbBr₃ single crystal. Negatively charged V'_{MA} is focused, and the inclusion of other defects actually does not alter the conclusion. The MA vacancies respond to external electric field E . (a) The MA vacancies display an evenly distributed insulating state. (b) In a negative field (bottom to up), MA vacancies are moving downward and accumulating near the bottom surface and are frozen when the field is turned off. As a result, a state with p-type carriers is realized; the opposite region becomes a n-type conductor. Then the internal p–n junction is formed and the diode-like I – V shape could be found (point b → c). (d) Applying the opposite electric field moves MA vacancies to the original evenly distribution, thus forming an HRS state. (e) An inverted p–n junction will be formed by further increasing the opposite electric field.

Fig. 4 I - V curves of an Au/MAPbBr₃/Au device in the dark with different top-electrode areas.

Fig. 5 HRS of MAPbBr₃ single crystals with Au and Pt electrodes in a log-log scale. Characteristic I - V traces show two different regimes. A linear ohmic regime ($I \propto V$) is followed by the trap-filled regime ($I \propto V^{n>3}$). In the SCLC model, n is an exponent and $n>3$ indicates the trap-filled conduction region.

constant contact with HOIPs [10,31]. Then the interfacial Schottky barrier [10,16] scenario should be excluded in our case. To further understand the conduction mechanism, we replotted the I - V curves of HRS in a log-log scale as shown in Fig. 4. Two regions were evident in the HRS curves. At low voltages, the I - V response was ohmic ($I \propto V$). At high electric fields, the current exhibited a rapid non-linear rise and signaled the transition onto the trap-filled limit (TFL) regime, in which all the available trap states were filled by the injected carriers [32]. The I - V response of the trap-filled SCLC is marked by a steep increase in current ($I \propto V^{n>3}$). The onset voltage V_{TFL} ($V_{\text{TFL}} =$

37 V for Pt electrodes and 39 V for Au electrodes) is typically linearly proportional to the density of trap states as follows [22]:

$$V_{\text{TFL}} = 8en_t d^2 / (9\epsilon\epsilon_0) \quad (1)$$

where e is the elementary charge, n_t is the density of trap states, d is the thickness of the sample, ϵ is the dielectric constant (here we use 25 for MAPbBr₃), and ϵ_0 is the vacuum permittivity. Correspondingly, it could be found that our MAPbBr₃ single crystals have a medium trap density of $\sim 1.5 \times 10^{12} \text{ cm}^{-3}$.

According to the above results, it is concluded that the main conduction mechanism in our device should be the trap-controlled SCLC by the charged defects. Besides the inner p-n junction, the empty traps can also lead to a HRS, while if all the traps were filled, the injected carriers can move freely into the perovskite crystals and then lead to a LRS. The next question is the charge trapping/detrapping is at the interface or in the bulk. Figure S4 in the ESM displays the contact resistance and bulk resistance of the Au/MAPbBr₃/Au devices. The calculation results indicate that the contact resistance between the Au electrode and the crystal is about 10 W, which is much smaller than the bulk resistance ($\sim 10^8 \text{ W}$). This suggests that the RS occurs through bulk trap generation, moving and filling in the MAPbBr₃.

Interestingly, the trap moving and filling states can be modulated by light absorption. Figures 6(a)–6(c) show the I - V curves of a Pt/MAPbBr₃/Pt device in the dark, under light illumination and subsequent light off, respectively. For the initial measurement in the dark, the switching loop under reverse bias is severely restricted, which implies a weakened RS. This means the p-n junction was not established and the traps were not fully filled in this sweep process. The speculation could be confirmed by further SCLC analysis as shown in Fig. 6(d). The characteristic I - V traces in the initial dark state show only a linear ohmic regime ($I \propto V$). After light illumination, some of the traps/defects are “activated” and then could be filled by injected carriers (Fig. 6(d)). Figure 7 illustrates the evolution of RS behavior of Au/MAPbBr₃/Au device under light illumination with different intensities. Under the light illumination, both the HRS and LRS currents increase due to the existence of photocurrent. The RS loops became narrower than the dark case, and wore off eventually. The results of light-responsive experiments also exclude the possible of metallic filament mechanism

Fig. 6 I – V curves of a Pt/MAPbBr₃/Pt device: (a) in the dark, (b) under light illumination (1500 lx), and (c) subsequent light off. (d) The so-called HRS (path 3 under reverse bias) of MAPbBr₃ single crystals with Pt electrodes in a log–log scale. Characteristic I – V traces in the initial dark state show only a linear ohmic regime ($I \propto V$), while a trap-filled regime ($I \propto V^{n>3}$) could be found after light illumination.

Fig. 7 I – V curves of an Au/MAPbBr₃/Au device under light illumination with selected power densities.

for the observed RS behavior.

It should be mentioned that the recent results from Hong *et al.* [19] have shown that the switchable diode-like RS behavior can be induced solely by an interface charge trapping/detrapping mechanism. Two back-to-back diodes could be formed due to the change

in the interfacial barrier width (Fig. S5 in the ESM). However, the interface resistance is much smaller than the bulk one in our devices, and the series conduction is governed by the bulk state. It is worth noting that the trap-controlled SCLC could be used to describe not only the conduction of a charge trapping/detrapping process, but also the defects electromigration process [26,33]. The aforementioned switchable diode-like RS behavior can be then explained by the formation of an electric-field-driven p–n junction accompanied by a trap-controlled SCLC in the perovskite. The resistance states (HRS and LRS) depend on the synergy between the inner p–n junction and the charge trapping/detrapping states.

4 Conclusions

In summary, a switchable diode-like RS behavior was observed in MAPbBr₃ single crystals using Au (or Pt) symmetric electrodes. The trap generation, moving, and filling states play a vital role on the resistance

switching. The charged defects migrate under applying a bias and lead to a reversible p–n structure in the crystal. The rectifying I – V traces and the LRS are attributed to the p–n junction created through the asymmetric distribution of charged defects. The inhomogeneous charged trap distribution can be reversed to an evenly distributed insulating state (HRS) by an opposite electric field. Accompanying, charge trapping/detrapping occurs associated with the mobile traps. The RS behavior is then further enhanced by this trap-controlled SCLC conduction process.

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Electronic Supplementary Material

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