Out-of-Plane Resistance Switching of 2D Bi$_2$O$_2$Se at the Nanoscale

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Two-dimensional (2D) bismuth oxyxelenede (Bi$_2$O$_2$Se) with high electron mobility shows great potential for nanoelectronics. Although the in-plane properties of Bi$_2$O$_2$Se have been widely studied, its out-of-plane electrical transport behavior remains elusive, despite its importance in fabricating devices with new functionality and high integration density. Here, the out-of-plane electrical properties of 2D Bi$_2$O$_2$Se at nanoscale are revealed by conductive atomic force microscope. This work finds that hillocks with tunable heights and sizes are formed on Bi$_2$O$_2$Se after applying a vertical electric field. Intriguingly, such hillocks are conductive in the vertical direction, resulting in a previously unknown out-of-plane resistance switching in thick Bi$_2$O$_2$Se flakes while ohmic conductive characteristic in thin ones. Furthermore, the transformation is observed from bipolar to stable unipolar conduction in thick flakes while ohmic conductive characteristic in thin ones. Additionally, the Bi$_2$O$_2$Se-based photodetectors with decent performance in terms of on/off ratio ($\approx 10^5$), photodetectivity ($3.4 \times 10^{15}$ Jones), broadband detection (360-1800 nm), and photoresponse time ($\approx 1$ ps) have been achieved. The above recent achievements suggest that the devices based on Bi$_2$O$_2$Se are important components in future integrated 2D electronics and optoelectronics. Note that all these applications are based on the charge transport in the horizontal direction of Bi$_2$O$_2$Se at micrometric scale. It is therefore intriguing to exploit the out-of-plane transport behavior of 2D Bi$_2$O$_2$Se to extend its functionality.

1. Introduction

Two-dimensional (2D) semiconducting bismuth oxyxelenede (Bi$_2$O$_2$Se) with sizeable bandgap, high electron mobility, and excellent air stability is promising in high-performance nanoelectronics.\cite{1-4} For example, Bi$_2$O$_2$Se has been used to fabricate three-terminal memristors with the combination of short-term and long-term plasticity to realize neuromorphic functions.\cite{7} In another work, gas sensors based on 2D Bi$_2$O$_2$Se show high selectivity and ultralow oxygen detection limit of 0.25 ppm at ambient temperature because of the easy trap of oxygen by the Se vacancies on the surface of Bi$_2$O$_2$Se.\cite{8} Furthermore, controlled thermal oxidation of Bi$_2$O$_2$Se is used to directly construct dielectric/semiconductor (Bi$_2$SeO$_5$/Bi$_2$O$_2$Se) structures like in the case of silicon (SiO$_2$/Si). Because the dielectric constant of the Bi$_2$SeO$_3$ layer is as high as $\approx 21$, such Bi$_2$SeO$_3$/Bi$_2$O$_2$Se structures are promising for fabricating high performance electronics.\cite{2} Additionally, the Bi$_2$O$_2$Se-based photodetectors with decent performance in terms of on/off ratio ($\approx 10^5$)\cite{9} photodetectivity ($3.4 \times 10^{15}$ Jones)\cite{9} broadband detection (360-1800 nm)\cite{10} and photoresponse time ($\approx 1$ ps)\cite{8} have been achieved. The above recent achievements suggest that the devices based on Bi$_2$O$_2$Se are important components in future integrated 2D electronics and optoelectronics. Note that all these applications are based on the charge transport in the horizontal direction of Bi$_2$O$_2$Se at micrometric scale. It is therefore intriguing to exploit the out-of-plane transport behavior of 2D Bi$_2$O$_2$Se to extend its functionality.

Besides microscale charge transport, the nanoscale electrical properties of 2D materials play a significant role in the performance and/or mechanism of related microscopic or macroscopic electronic devices.\cite{11} Conductive atomic force microscope (CAFM) is a useful tool with high spatial resolution to study the electrical properties of 2D materials at nanoscale.\cite{12} Along this direction, the performance of 2D dielectric layers has been recently revealed by CAFM technique. For example, Hattori et al. have shown that 2D h-BN is subjected to a layer-by-layer breakdown with a medium dielectric strength of $\approx 12$ MV cm$^{-1}$\cite{13} while 2D CaF$_2$ possesses a high dielectric strength of $\approx 278$ MV cm$^{-1}$ owing to its cubic lattice structure.\cite{14} Researchers also used CAFM to reveal the vertical electronic properties of nanoscale domains and domain boundaries in strong coupled systems such as twisted bilayers of graphene\cite{15} and transition metal dichalcogenides.\cite{16,17} Moreover, CAFM was used to monitor the formation of conductive filaments in memristors based on 2D materials to reveal the operating mechanism. For example, the resistance switching generated in 2D TiO$_2$ relies on the electromigration of the internal oxygen vacancies in the vertical direction\cite{18,19} while the memory behaviors of 2D h-BN stems from the defects generated during the synthesis process.\cite{20,21} Although some intrinsic physical properties of 2D Bi$_2$O$_2$Se, such as mechanical properties\cite{22} and ferroelectricity and piezoelectricity,\cite{23} have been uncovered by scanning probe techniques very recently, its out-of-plane...
electrical properties remains elusive, despite its significance in developing devices with new functionality and meeting the requirements of high integration density and compatibility.[24,25]

Herein, we report unique out-of-plane electrical properties of 2D Bi$_2$O$_2$Se. We find that hillocks are formed on 2D Bi$_2$O$_2$Se after applying vertical electric fields. The heights and widths of the hillocks and accordingly the electrical performance of the 2D Bi$_2$O$_2$Se can be controlled by the amplitude, cycle numbers of the electric field, and thickness of materials. Moreover, nanoscale conductive pathways form at the hillock locations, making the initially insulating Bi$_2$O$_2$Se exhibit out-of-plane resistive switching function and ohmic feature in thick and thin flakes, respectively. Such resistance switching devices show transformation from bipolar to stable unipolar conduction window, enabling its use as a selector in vertical devices. This study not only uncovers the out-of-plane electrical transport behavior of 2D Bi$_2$O$_2$Se for the first time, but also opens a door to explore its use in nanoelectronics with new functions.

2D Bi$_2$O$_2$Se were grown by vapor deposition method[9] and were transferred onto highly conductive substrate made of Au/Cr coated SiO$_2$/Si wafer (hereinafter referred to as Au substrate for short).[22] The as-synthesized flakes are rectangular single crystals with different thicknesses (Figure 1a) with high quality, as indicated by the A$_{1g}$ mode ($\approx$159 cm$^{-1}$) of Raman spectrum (blue curve in Figure S1, Supporting Information). The samples have uniform structure and composition, as shown by optical microscope (Figure 1b) and the intensity distribution of the A$_{1g}$ peak in corresponding Raman map (Figure 1c). After transfer onto the target substrate, Bi$_2$O$_2$Se flakes still keep the initially rectangular shape (Figure 1d). The high-magnification optical microscope image (Figure 1e) and corresponding Raman map of the A$_{1g}$ peak intensity (Figure 1f) of the transferred Bi$_2$O$_2$Se flakes jointly show its good uniformity and similar quality with the as-grown ones. High-resolution transmission electron microscope (TEM) image (Figure S2, Supporting Information) shows that rare defects can be observed, further demonstrating the high quality of the Bi$_2$O$_2$Se flake at nanoscale. In addition, X-ray photoelectron spectroscopy (XPS) spectra indicate the chemical composition of Bi$_2$O$_2$Se flakes (Figure 1g-i), which is consistent to the results reported previously.[9,22] These characterization results above together show the high quality and structural integrity of Bi$_2$O$_2$Se flakes after transferring on conductive Au substrate, providing basis for subsequent electrical studies.

After transferring 2D Bi$_2$O$_2$Se onto conductive Au substrate (Figure 2a), CAFM is used to study its out-of-plane transport behavior. Figure 2b displays the CAFM setup where the Au substrate is one of the electrodes, and AFM cantilever serves as the other one to apply electric field on Bi$_2$O$_2$Se samples. Prior to CAFM measurements, AFM scanning was performed to image the microscopic morphology and measure the thickness of the selected Bi$_2$O$_2$Se flake (Figure S3, Supporting Information). Afterwards, three random positions on the flake, as marked by dotted circles with different colors in Figure 2c, were chosen and cyclic triangular wave electric field with different amplitudes was applied in the vertical direction by the CAFM tip. Interestingly, we found that a hillock with a lateral size of no more than 200 nm formed at the local place where the tip and Bi$_2$O$_2$Se flake contacted under the cyclic voltage with an amplitude of 10 V (red dotted in Figure 2c,d). Similar phenomenon, i.e., the formation of a hillock after applying electric field, was observed at the position that was stimulated by the electric field with the same cycle number but a lower amplitude of 5 V (green dotted in Figure 2c,e). The height of this hillock is lower than that formed under the 10 V sweeping voltage. However, for the position simulated by the electric field with the amplitude of 2 V, the position keeps flat without the formation of a rough structure (blue dotted in Figure 2c,f). It is further analyzed that the heights of the hillocks formed under 30 cycles of the sweeping voltage with the amplitude of 10 and 5 V are 18.8 and 4.5 nm, respectively (Figure 2g), which demonstrate a positive correlation between the heights of hillocks and the amplitude of the electric fields. In order to trace the formation process of the hillocks, the height at the same position was measured after each cyclic application of electric field with the amplitude of 10 V. We found that the hillock forms ($\approx$2.5 nm) since the first sweep of the applied electric field, and the height increases with increasing cycle number, as exhibited in Figure 2h. Then the height of the hillock keeps stable as $\approx$19 nm after five sweeps of the electric field (Figure 2i). Cross-sectional high-angle annular dark-field (HAADF) imaging and energy dispersive spectrometer (EDS) analysis (Figure S4, Supporting Information) were performed to clarify that the content of oxygen on the surface of the hillock is higher than that at the internal areas. Accordingly, it is reasonable to deduce that the region where electric field was applied was oxidized in air, causing the increase of local layer spacing[20] and formation of hillock on Bi$_2$O$_2$Se. In contrast, the mechanism of similar phenomena in other 2D materials such as multilayer h-BN was proposed as its breakdown under strong electric field.[20] Taken together, the above results show that protruded structure can be constructed at predefined positions on Bi$_2$O$_2$Se flakes via the precise location of the CAFM tip. In addition, the height of the hillock is facilely tuned by modifying the amplitude and cycle number of the applied electric field.

Electric field in the vertical direction not only triggers the formation of hillocks on Bi$_2$O$_2$Se, but also generates out-of-plane conduction. As marked by the red dotted rectangular in Figure 3a, the “forming zone” was formed during the AFM scanning process that the probe along with the application of 5 V constant electric field initiates the formation of hillocks, while the rest part of the Bi$_2$O$_2$Se flake does not exhibit morphology changes because the electric field is removed (Figure S5a, Supporting Information). Importantly, after the forming process, the hillock positions became more conducive that nanoscale conductive pathways in the vertical direction were identified and marked by the yellow dotted rectangular in the corresponding CAFM map (Figure 3b). The current of the nanoscale conductive paths was about 1 nA under the bias of 2 V (top in Figure 3c), while the rest area is very resistive with current of $\approx$1 pA (bottom in Figure 3c). The comparison is more obvious in corresponding log-coordinated graphs (Figure S5b, Supporting Information). As shown in Figure 3d, our AFM studies show that 70-nm-thick Bi$_2$O$_2$Se flake is at insulating state originally (blue plots). Then it becomes conductive (green plots) under the application of cyclic triangular
wave electric field with the amplitude of 10 V, which demonstrates the forming process of conductive channels. With the increasing cycle number to 30\(^{th}\), the \(I-V\) plots with two conduction windows under both positive and negative electric field of this flake (red plots) clarifies out-of-plane resistance switching of the 70-nm-thick Bi\(_2\)O\(_2\)Se. Both the formation of hillock (Figure S6a,b, Supporting Information) and resistance switching phenomenon (Figure S6c, Supporting Information) are observed in Au/Bi\(_2\)O\(_2\)Se/Au architecture, in which an Au-coated AFM probe was used instead of doped-diamond one, to apply cyclic electric field. Different resistance switching phenomena were observed when cyclic electric fields with different frequencies were applied on the same Bi\(_2\)O\(_2\)Se flake. As shown in Figure S7 (Supporting Information), Bi\(_2\)O\(_2\)Se shows a wider conduction window under the positive electric field with a lower frequency, while the resistance switching performance keeps stable under the opposite electric field with changing frequency. After the forming process, the resistance switching can operate under a lower electric field (Figure S8, Supporting Information).

Apart from the cycle number, the amplitude of the sweeping electric field also contributes to the out-of-plane resistance...
switching phenomenon of Bi$_2$O$_2$Se. Specifically, resistance switching forms under 30-cycle sweep of the electric field with the amplitude of 10 V (red plots in Figure 3e). Although the periodic voltage with the amplitude of 5 V makes the Bi$_2$O$_2$Se flake conductive at the 30th cycle (blue plots in Figure 3e), conduction windows are difficult to form in this case. Additionally, no electrical responses in the vertical direction were collected under a 2 V electric field (gray plots in Figure 3e). By combination with the morphological analyses in Figure 2, we conclude that the formation of conduction windows is related to the height of hillocks. The thickness of Bi$_2$O$_2$Se flake is another factor that imposes effects on its out-of-plane electrical features. The transformation from insulating to conductive state combined with the formation of hillocks (Figure S9, Supporting Information) was also monitored in a thinner Bi$_2$O$_2$Se flake with a thickness of 8 nm under 10 V sweeping electric field. Different from the thick sample, the I–V plots of this 8-nm-thick flake show ohmic characteristics instead of resistance switching after the application of 20 cycles of the voltage suppress, which stems from the electrically hard breakdown of the thin sample (Figure 3f). The I–V plots during the same cycle of various Bi$_2$O$_2$Se flakes with different thicknesses were further analyzed and the results show that conductive channels form under a lower electric field strength for thinner sample (Figure 3g). Therefore, the out-of-plane nanoscale electrical properties of 2D Bi$_2$O$_2$Se show a unique thickness-dependent behavior, i.e., thick flakes show resistance switching phenomenon while thin ones show linear I–V curves.

Next, we study the polarity of the resistance switching behavior of Bi$_2$O$_2$Se. Essentially, the mode of out-of-plane resistance switching in Bi$_2$O$_2$Se flakes experiences a change during the sweep of cyclic, which is indicated by three I–V curves collected sequentially with CAFM. To start with, the I–V curve shown in Figure 4a claims the bipolar conductive behavior of Bi$_2$O$_2$Se after the formation of conductive paths in the vertical direction, which is consistent with most of the memory devices based on 2D materials.[26] Under the 0 to 10 V forward sweep (Step 1), the flake was at high-resistance (HR)
state, and then switched to low-resistance (LR) state during the sweep from 10 to 0 V (Step 2). Afterwards, the LR state was maintained under the negative electric field swept from 0 to −10 V (Step 3) and restored to HR under the −10 to 0 V sweep (Step 4). During the next cycle, a sudden transformation from HR to LR state was observed under the forward sweep of the positive voltage (Step 2 in Figure 4b). Subsequently, the electrical properties still comply with LR (0 to −10 V, Step 4) to HR (−10 to 0 V, Step 5) switching under the sweep of negative voltage. From then on, the $I$–$V$ curves exhibit alternative switching between LR and HR state (Figure 4c), demonstrating a unipolar conductive behavior which is converted from initial bipolar resistance switching. The alternation between resistance states is also found in the in-plane and out-of-plane memory devices based on other 2D materials. The cyclic CAFM measurements in Figure 4d further point out the high stability of the unipolar resistance switching of Bi$_2$O$_2$Se in the vertical direction. In comparison to other 2D oxyselenides with out-of-plane unipolar resistance switching behavior such as HfSe$_2$O$_x$, Bi$_2$O$_2$Se possesses lower set/reset voltage. It is noticeable that similar morphological change and resistance switching phenomenon were observed under the inverse...
electric field (0 V → −10 V → 10 V → 0 V). Specifically, the inverse cyclic electric field activated the formation of a hillock (Figure S10a, Supporting Information), as well as the unipolar resistance switching performance (Figure S10b, Supporting Information). The transformation from bipolar to stable unipolar resistance switching behavior may be dependent on the oxidation of Bi₂O₂Se to produce many more Se vacancies as well as Joule heat. Specifically, the migration of the Se vacancies is dominantly controlled by the electric field in the case of temporary bipolar resistance switching. Regarding of the stable unipolar behavior, with the increasing swept cycles of the electric field, the oxidation of Bi₂O₂Se produces much more Se vacancies, the movement of which increases the current and produce more Joule heat at this nanoscale region. The generation of local Joule heat may break parts of the current paths that switch the sample from LR to HR state with the decreasing positive electric field. Afterwards, the accumulated Se vacancies at the bottom would diffuse to their original position, during the process of which the conductive paths form again to show LR and produce Joule heat. These processes rely on the dynamic equilibrium between the formation of conductive paths and the production of Joule heat, which is similar to the unipolar resistance switching behavior of 2D TiO₂ based on the migration of O vacancies.[8] Regarding the stable unipolar resistance switching behavior, many more Se vacancies induced by the oxidation of Bi₂O₂Se lead to the electric field and the Joule heat dominated migration of Se vacancies. With the combination of EDS map shown in Figure S4 (Supporting Information) and the above discussions, the temperature of the conductive region is dramatically increased to trigger local oxidation and interlayered expansion that inspires the formation of hillock. Accordingly, in Figure 2i, five cycles of the swept voltage lead to the oxidation of Bi₂O₂Se and its interlayered expansion to the maximum extent that the height of the hillock keeps stable. The stable value of the height of the hillock is determined by the thickness of Bi₂O₂Se and the amplitude of the applied electric field.

In order to further verify this hypothesis, two random positions of a flake were selected to apply the same cyclic electric field in air and N₂ gas, respectively. In air, we found a hillock formed at the place to make resistance switching happen. As a sharp contrast, in N₂, no morphology change on Bi₂O₂Se was observed and it kept electrically insulating (Figure S11, Supporting Information). In consequence, the existence of O₂ is responsible for the morphological changes and resistance switching of Bi₂O₂Se under electric field. The resistance switching behaviors of Bi₂O₂Se is relevant to the formation of hillock, which is distinguished from that in 2D dielectric layers such as h-BN. In h-BN, the resistance switching is associated with the migration of metal atoms from the electrodes through its intrinsic defects to form conductive filaments. The formation of hillock is attributed to the soft breakdown of h-BN under a high electric field.[20] The out-of-plane resistance switching assures Bi₂O₂Se for applications in future nanoelectronics with new functionality, high integration, and high compatibility. In addition, crossbar electronic devices are subjected to the sneak current in the vertical direction, which can be mitigated by the addition of selector device with unilateral electrical conduction between two layers of electrodes.[30] Therefore, owing to the out-of-plane unipolar conduction behavior, Bi₂O₂Se is suitable for applications in vertically oriented devices as a functional material as well as a selector.

Figure 4. Transformation from bipolar to unipolar conduction window in Bi₂O₂Se devices. a–c) Three sequential I–V curves collected under the application of 10 V cyclic triangular electric field. d) 10-cycle measurements of the resistance switching of the same Bi₂O₂Se flake.
3. Conclusion

We have revealed the out-of-plane electrical properties of 2D Bi$_2$O$_2$Se at nanoscale for the first time. We found that hillocks formed on 2D Bi$_2$O$_2$Se under cyclic triangular electric field. The height of these hillocks is controlled by amplitude or cycle number of the cyclic voltage suppress and the thickness of Bi$_2$O$_2$Se flakes. It is noteworthy that the hillock positions contain nanoscale conductive pathways, leading to a unique vertical resistance switching behavior in thick Bi$_2$O$_2$Se flakes while ohmic characteristic in thin ones. In addition, the devices based on thick Bi$_2$O$_2$Se flakes exhibited transformation from bipolar to stable unipolar resistance switching behavior. The work not only discloses novel out-of-plane transport behaviors of 2D Bi$_2$O$_2$Se, but also opens an avenue for the use of 2D materials in highly integrated nanoelectronics with new functionality.

4. Experimental Section

Growth, Transfer, and Characterization of 2D Bi$_2$O$_2$Se: 2D Bi$_2$O$_2$Se samples were synthesized on mica substrate by vapor-solid deposition,[9] and were transferred onto Au/Cr-coated SiO$_2$/Si substrate by the PDMS-mediated[8] or PMMA-assisted method.[8] For preparation of Au/Cr-coated substrate, SiO$_2$/Si substrate was put into the chamber of electron beam evaporation system (TSV-1500, Tianxingda Vacuum Coating Equipment Co., Ltd., China). Then, Cr layer with a thickness of 5 nm was deposited on the SiO$_2$/Si substrate at the speed of 0.2 Å s$^{-1}$, which was followed by the deposition of Au layer with the thickness of 40 nm at the speed of 2 Å s$^{-1}$, in order to obtain conductive Au/ Cr/SiO$_2$/Si substrate.

The surface morphology of Bi$_2$O$_2$Se samples was characterized by optical microscope (Imager A2m, Carl Zeiss, Germany). XPS (ESCALAB 250Xi, Thermo Fisher, USA) was used to reveal the chemical composition of Bi$_2$O$_2$Se flakes. Raman spectrometer (HR8000, Horiba JY, Japan) was used to collect the Raman spectra and mapping of Bi$_2$O$_2$Se. The spot size of the incident laser with the wavelength of 633 nm is 500 nm, and each step of Raman mappings is 1 μm. The cross-section of hillocks on Bi$_2$O$_2$Se was obtained by focused ion beam (FIB, Helios UC, FEI, USA). A high bias of 10 V was applied by the AFM probe during the scanning process to obtain microscale hillocks, which destructed and separated the Bi$_2$O$_2$Se flake from the hillock due to the high strain (Figure S12a,b, Supporting Information).

Subsequently, scanning electron microscope was used to select a Bi$_2$O$_2$Se flake with separated microscale hillocks (Figure S12c, Supporting Information) and FIB was performed to cut a hillock in a certain direction, in order to obtain the cross-section TEM sample, as shown in Figure S13 (Supporting Information). HAADF imaging and EDS analysis were performed to characterize the chemical composition of hillocks on Bi$_2$O$_2$Se with a TEM (Talos, FEI, USA) at an operating voltage of 200 kV.

CAFM Measurement of Out-of-Plane Conductance of Bi$_2$O$_2$Se: CAFM scanning (Cypher ES, Oxford Instruments, USA) was performed to image the morphology and measure the thickness of Bi$_2$O$_2$Se with contact mode. The AFM probe (FM-LC, Adama Ltd., Ireland) with doped diamond coating and the radius of 20 nm was used for morphology imaging, application of cyclic triangular electric field in the vertical direction at the frequency of 1 Hz, and collection of I−V curves in CAFM setup. The compliant current was 10 μA. For obtaining CAFM map of the out-of-plane current, a bias was set and applied by the tip while scanning the morphology of samples. For the measurements in pure N$_2$, which is introduced into the cell for 10 min to eliminate the air, eventually the pressure of N$_2$ maintained at 190 mbar. The frequency of the sweeping voltage is 1 Hz.

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.

Data Availability Statement

Research data are not shared.

Keywords

2D materials, Bi$_2$O$_2$Se, nanoscale electrical property, out-of-plane resistance switching, unipolar conduction window

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