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Positive-to-negative subthreshold swing of a MOSFET tuned by the ferroelectric switching dynamics of BiFeO₃

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Abstract

Ferroelectricity can reduce the subthreshold swing (SS) of metal-oxide-semiconductor field-effect transistors (MOSFETs) to below the room-temperature Boltzmann limit of ~60 mV/dec and provides an important strategy to achieve a steeper SS. Surprisingly, by carefully tuning the polarization switching dynamics of BiFeO₃ ferroelectric capacitors the SS of a commercial power MOSFET can even be tuned to zero or a negative value, i.e., the drain current increases with a constant or decreasing gate voltage. In particular, in addition to the positive SS of lower than 60 mV/dec, the zero and negative SS can be established with a drain current spanning for over seven orders of magnitude. These intriguing phenomena are explained by the ferroelectric polarization switching dynamics, which change the charge redistributions and accordingly affect the voltage drops across the ferroelectric capacitor and MOSFET. This study provides deep insights into understanding the steep SS in ferroelectric MOSFETs, which could be promising for designing advanced MOSFETs with an ultralow and tunable SS.

Introduction

Ferroelectric materials show high potential for use in various applications, such as nonvolatile memories^{1–4}, photoelectric detectors^{5–8}, and neuromorphic computing^{9,10}. Recently, it was reported that ferroelectricity can be used to reduce the subthreshold swing (*SS*) of metal-oxide-semiconductor field-effect transistors (MOSFETs) to below 60 mV/dec at room temperature and break the "Boltzmann Tyranny", which may be beneficial to lowering power consumption^{11–14}. For instance, *SS* values less than 60 mV/dec have been achieved, such as in MoS₂/Al₂O₃/Hf_{0.5}Zr_{0.5}O₂-based¹⁴, MoS₂/HfO₂/LiNbO₃-based¹⁵, and PbZr_{1-x}Ti_xO₃-based¹⁶ ferroelectric MOSFETs.

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To understand the important effect of reducing the SS, several explanations have been proposed from different aspects. One is based on the concept of a negative capacitance effect, i.e., the ferroelectric material can serve as a negative capacitor^{17,18}. This is related to the double-well landscape of the free energy in a ferroelectric material, which inevitably leads to a region of negative capacitance when ferroelectricity switches from one polarization state to the other $^{19-22}$. As a result, with a ferroelectric negative capacitor connected to the gate terminal, the channel surface potential of a MOSFET can be amplified to be even higher than the gate potential under capacitance matching conditions^{12,17}. Hence, the ferroelectric MOSFET can be operated with an SS < 60 mV/dec at room temperature^{11,12,14}. In addition to the steep SS, many distinctive phenomena, including a voltage drop across the ferroelectric capacitor with charges flowing, an "S"-shaped relation between polarization (P) and voltage (V), and a stepup conversion of internal voltage, have been reported by using various ferroelectric materials, and these effects

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can also be attributed to the negative capacitance $^{22-26}$. On the other hand, some researchers found that it may not be necessary to introduce the concept of negative capacitance and proposed that an SS less than 60 mV/dec is related to the ferroelectric polarization reversal characteristics^{16,27,28}. For example, as reported by Hwang et al., the voltage drop with flowing charges and the "S"-shaped *P-V* curve can be explained by a reversing ferroelectric domain, of which the charge effect cannot be fully compensated by the supplied charges from the external charge source²⁷. Very recently, Li et al. observed stepwise jumps in the internal potential of a metal/ferroelectric/metal/paraelectric/metal-stacked system near the coercive voltage of the ferroelectric layer; furthermore, they experimentally confirmed the relationship of these jumps with the steep SS of the MOSFET. On this basis, a concise model was raised from the aspect of successive polarized domain flipping and depolarization feedbacks^{16,28}.

Regardless of whether the steep *SS* is due to the negative capacitance, at least ferroelectric polarization reversal can reduce the *SS* of a ferroelectric MOSFET. Considering that the ferroelectric switching dynamics can be controlled by voltage sweeping processes, it will be very interesting to investigate the tunability of *SS* and the corresponding dynamic characteristics in ferroelectric MOSFET devices. Not only can we obtain insights into the ferroelectric polarization switching dynamics affecting the *SS*, but an ultralow and tunable *SS* in MOSFETs may also be achieved, which is important for designing advanced electronic devices.

In principle, a ferroelectric material with a large polarization and controllable ferroelectric switching dynamics will be a good candidate for tuning the SS of ferroelectric MOSFETs. According to our previous studies^{29,30}, BiFeO₃ films (BFO) have a large polarization (>60 $\mu C/cm^2)^{31-33}$ and relatively clear ferroelectric switching dynamics. Therefore, in this work, we use an Au/BFO/La_{0.6}Sr_{0.4}MnO₃ (LSMO) ferroelectric capacitor connected in series with a commercial power MOSFET to tune its SS by varying the voltage sweeping sequences. Notably, the SS can be tuned to below 60 mV/dec at room temperature. In particular, an SS = 0and even a negative SS can be realized with a drain current spanning over seven orders of magnitude. These results are highly related to the ferroelectric polarization switching dynamics, which redistribute the charges and accordingly affect the voltage drops across the ferroelectric capacitor and MOSFET.

Materials and methods

Sample preparation

The LSMO layer was grown on a (001)-oriented SrTiO₃ (STO) single-crystal substrate using a DC magnetron sputtering technique with a power of 40 W, a deposition temperature of 750 °C and an argon/oxygen (1:1) pressure

of 4 Pa. For the BFO ferroelectric layer, an RF magnetron sputtering technique was used with a power of 40 W, a deposition temperature of 680 °C and an argon/oxygen (1:1) pressure of 4 Pa. After deposition, the heterostructure was slowly cooled at a rate of 5 °C/min to 250 °C under 10 Pa of oxygen pressure and then naturally cooled to room temperature. The top Au electrodes (300 μ m in diameter) were deposited with a shadow mask.

Characterization

The film thicknesses were measured by cross-sectional scanning electron microscopy (SEM) (SU8200, Hitachi, Japan). The heterostructures were characterized by X-ray diffraction (XRD) using a Cu K α 1 radiation source (λ = 1.540598 Å) (X'Pert Pro diffractometer, Philips Analytical, Netherlands) as well as with high-angle annular dark field scanning transmission electron microscopy (HAADF-STEM) (JEM-ARM200F, JEOL, Japan). The surface morphology was investigated by atomic force microscopy (AFM) (MFP-3D, Asylum Research, USA). The ferroelectric characteristics were investigated by a ferroelectric tester (Precision Premier II, Radiant Technologies, USA). The transport measurements of the ferroelectric MOS-FETs were obtained with a semiconductor parameter analyzer (4200A-SCS, Keithley, USA).

Results and discussion

Structural and ferroelectric characterization

Figure 1a shows the XRD pattern of the BFO (~200 nm)/LSMO (~50 nm) heterostructure grown on an STO single-crystal substrate (see Supplementary Fig. S1a) through a magnetron sputtering technique. Only (00 l)diffraction peaks appear, demonstrating the epitaxial nature of the rhombohedral-like BFO and LSMO lavers grown on the (001)-oriented STO substrate³⁴. Due to bulk BFO (~3.96 Å) having a larger in-plane lattice parameter than STO (~3.905 Å) and LSMO (~3.87 Å)^{35–37}, the BFO film demonstrates an in-plane compressive strain from the lattice mismatch. Thus, the out-of-plane lattice parameter of the BFO film (~4.01 Å, as determined from the XRD data), is larger than that of bulk BFO $(\sim 3.96 \text{ Å})^{35}$. The AFM results show that the material has a smooth surface with a small root mean square roughness of ~0.67 nm (see Supplementary Fig. S1b). Figure 1b shows the cross-sectional HAADF-STEM image. As the magnified image depicted in the inset of Fig. 1b, the visible ferroelectric displacements of the Fe ions projected along the [001] and [010] directions are ~20.4 pm and ~17.6 pm, respectively.

The ferroelectricity of BFO is also confirmed by the *P*-*V* hysteresis loops, as presented in Fig. 1c. The polarization of the BFO ferroelectric capacitor is ~60 μ C/cm², which is consistent with previous reports^{6,31}. With an increasing measuring frequency, the ferroelectric coercive voltage



increases. This is essentially related to the kinetics of ferroelectric switching that at a higher frequency (i.e., with a shorter time for ferroelectric reversal), a larger voltage is needed to switch the polarization^{38,39}. Then, the ferroelectric polarization switching dynamics were investigated using the positive-up-negative-down (PUND) polarization measurement mode 29,30 (see the details in Supplementary Fig. S2a), as shown in Fig. 1d. Here, the evolution of the ferroelectric up-to-down polarization switching in regard to the pulse width was tested at various voltage amplitudes. As expected, to achieve the same amount of ferroelectric polarization reversal, a longer pulse width is required for a lower pulse voltage^{3,40,41}. In addition, a nucleation-limited-switching (NLS) model (see Supplementary Fig. S2b-d) can be used to describe the ferroelectric switching dynamics in Fig. 1d, as indicated by the solid lines^{3,40,41}. Finally, the activation electric field of ~0.20 V/nm for ferroelectric switching is similar to that in an earlier report²⁹.

Ultralow SS of a MOSFET in series with a BFO ferroelectric capacitor

To investigate the ferroelectric polarization reversalaffected transfer characteristics of a MOSFET, a BFO ferroelectric capacitor was connected in series with the gate terminal of a commercial power MOSFET, as schematically presented in Fig. 2a. The gate voltage $V_{\rm G}$ was applied to the top Au electrode of the BFO ferroelectric capacitor, and a drain voltage $V_{\rm DS}$ of ~10 V was applied to the drain terminal of the MOSFET. The internal voltage V_{int} at the gate terminal of the MOSFET was measured. By sweeping $V_{\rm G}$ from -5 to +10 V (forward) and then back to -5 V (reverse) with a step of 0.5 mV, the drain current I_{DS} vs. V_G curves of the MOSFET with and without a connected BFO capacitor were tested, as illustrated in Fig. 2b. Regarding the standalone MOSFET, $I_{\rm DS}$ switches between the OFF and ON states when $V_{\rm G}$ changes between 1.4 and 4.0 V. In contrast, with the ferroelectric capacitor connected, the drain current I_{DS} switches more abruptly from the OFF to the ON state ($V_{\rm G}$ changes from 4.8 to 4.9 V) and from the ON to the OFF state ($V_{\rm G}$ changes from -1 to -1.05 V) in the switching regions. Here, the drain current of the ferroelectric MOSFET is very small in the OFF state, which is comparable with that of the standalone MOSFET. There is a small dip in $I_{\rm DS}$ in the reverse voltage sweep in the OFF state, similar to earlier reports^{42–44}, which may be related to the ferroelectric switching current. In addition, it is



noted that the $I_{\rm DS}-V_{\rm G}$ curve does not overlap for the forward and reverse voltage sweeps. This is because a certain voltage is needed to switch the ferroelectric polarization (see Fig. 1c, d), especially for the rather thick (~200 nm) BFO ferroelectric film. In fact, the voltage hysteresis can be reduced by using a thinner ferroelectric film^{42,45}, as demonstrated by the ferroelectric MOSFET based on a thinner ~150 nm-thick BFO film in Supplementary Figs. S3–S6.

The steepness of switching from the OFF to the ON state or the ON to the OFF state in the I_{DS} vs. V_{G} curves in Fig. 2b can be quantified by the subthreshold swing, *SS*, which is defined as the change in V_{G} required to tune the I_{DS} by an order of magnitude^{12,17}:

$$SS = \frac{\partial V_{\rm G}}{\partial \log_{10} I_{\rm DS}}.$$
(1)

As shown in Fig. 2c, the calculated *SS* of the standalone MOSFET is ~260 mV/dec for over seven orders of magnitude of $I_{\rm DS}$. With the BFO capacitor connected, the *SS* of the MOSFET in the series system is greatly reduced in the switching regions. The minimum *SS* values are as low as ~1.69 mV/dec and ~0.76 mV/dec for the forward and reverse voltage sweeps, respectively. This record-low *SS* (thus far) obtained by using a common sweeping voltage measurement mode^{14,15} is well below the room-

temperature Boltzmann thermodynamics limit of ~60 mV/dec. With the reduced *SS*, the power consumption for turning on the BFO capacitor-connected MOS-FET is lower than that of the standalone MOSFET (Supplementary S4, Table S1). To understand the ultralow *SS* in the series circuit, the *V*_{int} at the gate terminal of the MOSFET was directly measured, as shown in Fig. 2d. Clearly, the internal voltage gains $dV_{int}/dV_G > 1$ in the switching regions experimentally demonstrate voltage amplification (i.e., the internal voltage amplification effect^{25,46–48}), as shown in Supplementary Fig. S7. As a consequence, owing to the amplified *V*_{int}, a steeper *SS* than that of a standalone MOSFET is established^{25,46–48}.

To investigate the effect of polarization switching on the *SS*, the voltage V_{FE} across the BFO ferroelectric capacitor was calculated by:

$$V_{\rm FE} = V_{\rm G} - V_{\rm int}.$$
 (2)

The result is shown in Fig. 2e. Clearly, for both the forward and reverse sweeps, the absolute amplitude of $V_{\rm FE}$ first increases to a high voltage level and then decreases sharply around the switching points at $V_{\rm G}$ values of ~4.8 V and -1.0 V and then increases back again with a further increase in $V_{\rm G}$ to the positive and negative maximums. Considering the series circuit containing the BFO ferroelectric capacitor and MOSFET, the sudden



variations in V_{int} and V_{FE} at the switching points can be understood based on ferroelectric switching-induced charge redistributions^{16,49}. During the forward branch measurement, the ferroelectric polarization up-to-down switching around a $V_{\rm G}$ value of ~4.8 V requires more negative screening charges at the bottom electrode of the ferroelectric capacitor, which come from the gate terminal of the MOSFET. This charge redistribution will result in more positive charge accumulation at the gate terminal of the MOSFET, thus leading to an increase in V_{int} and a drop in V_{FE} . Similarly, an abrupt drop in V_{int} and an increase in $V_{\rm FE}$ can be observed around a $V_{\rm G}$ value of \sim -1.0 V when the ferroelectric polarization switches from down to up. Consequently, the stepup conversion of V_{int} and the corresponding reduction of SS can be understood in terms of charge redistributions mediated by ferroelectric switching. It should be noted that the amplified V_{int} and reduced SS in the ferroelectric MOSFETs may also be explained in terms of the negative capacitance effect¹⁹⁻²², as discussed in Supplementary S6. The underlying mechanism of the reduced SS and amplified $V_{\rm int}$ by ferroelectric switching has drawn considerable interest from both experimental and theoretical researchers^{12,16,50–52}, and further in-depth investigations are necessary to reveal its comprehensive physical process. Other mechanisms for the reduction of the SS, such as filament forming/rupture in BFO⁵³, can be excluded in our work. If filament forming/rupture⁵³ occurs in BFO, an amplified V_{int} and reduced SS will appear as a result. However, if the conducting filament forms in a ferroelectric layer, the ferroelectric polarization can hardly be reversed because of the conductive filaments connecting both electrodes^{54,55}. Regarding our ferroelectric MOS-FET, the ferroelectric hysteresis of the BFO capacitor can still be measured after the characterization of the steep SS.

The sudden drop in V_{FE} at the switching points suggests the famous "S"-shaped *P*-*V* loop of the ferroelectric capacitor. By directly applying the voltage waveform of

 $V_{\rm FE}$ in Fig. 2e to the standalone BFO ferroelectric capacitor through a ferroelectric analyzer, the "S"-shaped-like $P-V_{\rm FE}$ loop can be experimentally obtained (Fig. 2f), which is similar to the experimental results in the literature^{26,56,57}. This corresponds to the real-time ferroelectric domain switching process during the measurement of the $I_{\rm DS}$ vs. $V_{\rm G}$ curves. As the $V_{\rm FE}$ is close to the coercive voltage, the polarization begins switching, and as long as polarization switching occurs, even when $V_{\rm FE}$ decreases in certain switching regions, P keeps increasing, possibly because less energy is needed for further switching⁵⁸. In addition, the measured $P-V_{\rm FE}$ loop in Fig. 2f shows that the maximum $V_{\rm FE}$ values on the positive and negative voltage sides are ~ 3.0 and ~ -4.4 V, respectively. Because the SS in MOSFETs can be efficiently tuned by ferroelectric polarization switching dynamics, the forward and reverse voltage sweeps demonstrate different polarization switching processes (see Fig. 1d and Supplementary Fig. S2), resulting in the different SS values in Fig. 2c.

Zero SS and dynamic characteristics of the ferroelectric MOSFET

By tuning the sweeping process of $V_{\rm G}$ to control the ferroelectric polarization switching dynamics, even sharper ramping of $I_{\rm DS}$ can be achieved, as shown in Fig. 3. According to the ferroelectric switching dynamics in Fig. 1d, the polarization can be continuously switched with an increasing pulse width of the applied voltage. Thus, by sweeping $V_{\rm G}$ to 4.8 V or -1 V and then fixing $V_{\rm G}$ at the switching point of the forward or reverse branch (see the waveforms of $V_{\rm G}$ in Supplementary Fig. S8a), $I_{\rm DS}$ keeps changing until it reaches the ON or the OFF state, as shown in Fig. 3a. Figure 3b indicates the corresponding relationship between the *SS* and $I_{\rm DS}$, and a record-low *SS* down to zero is obtained for over six orders of magnitude of the $I_{\rm DS}$ of both the forward and reverse branches.

To further understand the ferroelectric polarization switching dynamics affecting the I_{DS} vs. V_{G} curves with an



SS = 0, the I_{DS} switching dynamics of the MOSFET with a connected BFO capacitor were investigated by applying square voltage waveforms of V_G (schematically shown in Supplementary Fig. S8b) with different amplitudes, as displayed in Fig. 4. Before each measurement, the BFO capacitor was initialized to the up state. Through the application of a different pulsed $V_{\rm G}$ from 5.5 to 9.5 V, $I_{\rm DS}$ increases gradually from the OFF state to the ON state, as shown in Fig. 4a, and an SS = 0 is established for over seven orders of magnitude of I_{DS} . Figure 4b shows the relationship among the $I_{\rm DS}\text{, }V_{\rm int}$ and time windows from the OFF state to the ON state. Clearly, with increasing $V_{\rm G}$, the $I_{\rm DS}$ changes more quickly from the OFF to the ON state, similar to the BFO ferroelectric switching dynamics in Fig. 1d, which show that at higher voltages, ferroelectric polarization switches more quickly from the up to the down state. By applying a $V_{\rm G}$ as high as 40 V, the ferroelectric MOSFET starts to turn ON with a time delay of 525 ns (see Supplementary Figs. S9-S10), which is longer than that of the standalone MOSFET⁵⁹; this result is similar to earlier reports^{60,61}. These results imply that there is a close relation between the ramping of $I_{\rm DS}$ with an SS = 0 and the ferroelectric polarization switching dynamics.

Furthermore, the evolution of V_{FE} calculated by using Eq. (2) is displayed in Fig. 4c. It is found that when a certain V_{G} is applied, V_{FE} decreases gradually as the applied V_{G} increases. This can be due to continuous ferroelectric polarization switching, which leads to more charge accumulation on the MOSFET and results in an

increase in $V_{\rm int}$ and a decrease in $V_{\rm FE}$ over time. It is worth mentioning that the evolution trend of $V_{\rm FE}$ with a constant $V_{\rm G}$ in BFO-based MOSFETs is different from that of the ferroelectric capacitor in series with a resistor (another classic circuit for investigating the ferroelectric negative capacitance effect). Regarding a system with a resistor-connected circuit, the $V_{\rm FE}$ decreases first over time and then increases back to $\sim V_{G}$. In other words, the stepup conversion of V_{int} (= $V_{\text{G}} - V_{\text{FE}}$) disappears after ferroelectric switching finishes^{23,56,62}. Regarding the system with a MOSFET (comparable to a capacitor) connected with a ferroelectric capacitor, the ferroelectricitytuned SS of the MOSFET can be revealed, and the $V_{\rm FE}$ does not recover after ferroelectric switching is finished. Although the phenomena in both systems are different, the underlying mechanisms for the decreasing $V_{\rm FE}$ are consistently linked with ferroelectric switching. Different from the resistor, the gate of the MOSFET (or capacitor) will accumulate charges that will not disappear when ferroelectric switching finishes; thus, V_{int} across the gate of the MOSFET remains even when $V_{\rm FE}$ does not increase back to $\sim V_{\rm G}$.

Negative SS tuned by the ferroelectric switching dynamics

From the above results, it can be seen that by varying $V_{\rm G}$, the *SS* can be tuned to zero. In fact, by further manipulating the $V_{\rm G}$ waveform by sweeping $V_{\rm G}$ back by a small voltage $V_{\rm return}$ when the $V_{\rm G}$ is close to the switching points (see the waveform of $V_{\rm G}$ in Supplementary Fig. S5a), the *SS* can even be tuned to be negative, as shown in



Fig. 5a, b. Figure 5c plots the *SS* vs. $I_{\rm DS}$ curves with $V_{\rm return} = -10$ mV. Surprisingly, although $V_{\rm G}$ drops in the region of $V_{\rm return}$ $I_{\rm DS}$ still increases because of the ongoing ferroelectric domain reversal; thus, a negative *SS* occurs for over three orders of magnitude of the $I_{\rm DS}$ of both the forward and reverse branches, as shown in Fig. 5c. It should be noted that a negative *SS* can even be obtained for over seven orders of magnitude of $I_{\rm DS}$ when $V_{\rm return}$ is ~ -100 mV, as shown in Fig. 5d. The discovered negative *SS* in the MOSFETs, which corresponds to the decrease in gate voltage with the increase in internal voltage, provides an interesting method for tuning the drain current of the MOSFET and reduces the gate power consumption; this method may be helpful for understanding the working principles of ferroelectric MOSFETs.

The tunable and even negative *SS* obtained by setting V_{return} , as shown in Fig. 5 and Supplementary Fig. S11, can be understood based on the "S"-shaped *P*- V_{FE} curve observed in Fig. 2f; for example, when V_{FE} decreases in certain switching regions, *P* keeps increasing. This is because even with V_{return} , V_{FE} is still large enough to trigger further ferroelectric switching. Consequently, a polarization reversal-induced ramping of I_{DS} still occurs within the V_{return} region, and a negative *SS* is achieved as a result. The tunable, ultralow, and even negative *SS* not only enhances the functionality and performance of ferroelectric MOSFETs but also demonstrates the

importance of the ferroelectric domain switching dynamics in explaining all the distinctive phenomena that was observed.

Here, a single-crystalline epitaxial BFO film with a large polarization of ~60 μ C/cm² was used to investigate the principle of manipulating the SS by ferroelectric switching. Based on this principle, other ferroelectric materials with smaller polarizations, such as $Hf_{1-x}Zr_xO_2$ $(\sim 30 \,\mu C/cm^2)^{14,63}$ and BaTiO₃ $(\sim 25 \,\mu C/cm^2)^{64}$, could also be used to tune the SS of MOSFETs from positive to negative. Other ferroelectric field-effect transistors based on nanowire FETs, FinFETs, would have similar behavior to the zero or negative SS by tuning the switching dynamics of the ferroelectric materials. In addition, it is worth mentioning that the compatibility of ferroelectric materials with Si substrates is important for their practical application. According to earlier reports^{65–67}, a single-crystalline epitaxial BFO film with a polarization of $\sim 45 \,\mu\text{C/cm}^2$ can be obtained by using singlecrystalline Si substrates. Recently, the use of Si substrates for growing $Hf_{1-x}Zr_xO_2$ offers a very important advantage to be more compatible with current CMOS processes than using a SrTiO₃ substrate, although realizing a single-phase Hf_{1-x}Zr_xO₂ ferroelectric film is still challenging⁶⁸⁻⁷⁰. Therefore, further detailed and systematic investigations are necessary for practically applicable devices.

Conclusion

In summary, by connecting a Au/BFO/LSMO ferroelectric capacitor with the gate terminal of a commercial power MOSFET, the *SS* can be flexibly tuned by manipulating the ferroelectric polarization switching process. In addition to a positive *SS* of lower than 60 mV/dec, an SS = 0 and even a negative *SS* can be obtained for over seven orders of magnitude of the drain current. The results are closely related to the ferroelectric polarization switching dynamics, which affect the charge redistributions and accordingly decrease (increase) the voltage drop on the ferroelectric capacitor (MOSFET). Our work is helpful to understand the steep and tunable *SS* in ferroelectric MOSFETs and provides a possible strategy to further improve the performance of MOSFET devices by designing ferroelectric switching dynamics.

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Author contributions

C.C.L. prepared the samples and performed the measurements. Y.C.W. prepared a thinner ~150 nm-thick BFO film and measured the properties of the corresponding ferroelectric MOSFET. H.Y.S. and Z.L. performed the ferroelectric domain dynamics and the corresponding fittings. C.M. analyzed the HAADF-STEM result. Z.L. conducted the AFM measurements. H.W. performed the XRD and SEM measurements. X.G.L. and Y.W.Y. designed and supervised the experiments. All the authors contributed to the refinement of the manuscript.

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Conflict of interest

The authors declare no competing interests.

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