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THE INFLUENCE OF TECHNOLOGY AND SWITCHING PARAMETERS ON RESISTIVE SWITCHING BEHAVIOR OF $Pt/HfO_2/TiN$ MIM STRUCTURES

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Abstract. *Resistive switching (RS) effects in $Pt/HfO_2/TiN$ metal-insulator-metal (MIM) capacitors have been investigated in dependence on the TiN bottom electrode engineering, deposition process, switching conditions and dielectric thickness. It is found that RS ratio depends strongly on the amount of oxygen introduced on TiN surface during interface engineering. In some structures a full recovery of conductive filament is observed within more than 100 switching cycles. RS effects are discussed in terms of different energy needed to dissociate O ions in structures with different TiN electrode treatment.*

Key words: *resistive switching; Pt/HfO2/TiN structures; interface engineering; atomic layer deposition.*

1. INTRODUCTION

Among the new types of non-volatile memories (NVM) the resistive switching memories (RRAM) have attracted a great interest because of their simple structure, long retention time, small size, fast switching speed and non-destructive readout [1-4]. Resistive switching (RS) is a phenomenon in which the resistance of material changes under application of electric field or current. RS devices can be switched between low resistive state (LRS) and high resistive state (HRS) over many cycles. Two kinds of RS effect have been recognized – unipolar, where the switching does not depend on polarity of the applied voltage; and bipolar, where the set to LRS occurs in one polarity and the reset to HRS in the reversed polarity. Bipolar switching is usually preferred because of better uniformity, faster switching speed and better control [5]. To obtain RS effect an initial electroforming step is needed, which causes the initially insulating structure to change into a higher conductive state. In fact, electroforming is a current-limited electric breakdown, which in its nature is a kind of "soft" or "arrested" breakdown [6]. The polarity of the forming process as well as compliance current (CC) should be carefully optimized in order to bring the structure to a

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state where RS could be observed. The typical bipolar RS loop is presented in Fig.1. After the forming step the structure is in LRS (ON state). By sweeping the voltage (usually in polarity opposite to forming process), at a certain voltage, V_{reset}, the structure goes to HRS (OFF state). This is the reset process. Then, by sweeping of voltage in the reversed polarity, at a certain voltage V_{set} the structure is brought to LRS – set process. The switching between ON and OFF states is reversible and could be performed over many cycles. RS effect has been observed in various transition metal oxides, but the origin of RS is still unclear. It is generally accepted that the RS is due to formation and rupture of nano-sized conductive filament(s) (CF) through the insulating layer which constitutes the two stable resistive states (ON and OFF states). Formation of localized CFs has been observed in various metal-oxide resistive switching devices by conductive atomic force microscopy [7, 8]. Thermal, electrical or ion-migration-induced mechanisms could control RS [9-11]. For example, there are evidences that the oxygen vacancies play a crucial role in formation and rupture of the conductive filaments in transition metal oxides. Therefore, the effects depend strongly on the dielectric material and the method of its deposition. On the other hand, all the above mentioned mechanisms could be substantially influenced by the metal electrodes and evidence for interface (i.e. metal/dielectric) effects are more frequently reported than bulk switching effects. In other words RS phenomenon is not an intrinsic property of the oxide itself, but a property of both oxide and electrode(s)/ oxide interface(s) [1,4,9]. Metal-insulator-metal (MIM) structures with HfO_2 as insulating film are intensively studied recently due to CMOS compatibility and excellent switching properties [12-14]. TiN and Pt electrodes are often employed to provide bipolar RS. TiN can be easily oxidized during the dielectric growth and acts as an oxygen reservoir due to its high affinity to oxygen while Pt serves as inert electrode [3]. Different modifications of Pt/HfO_2/TiN structure by introducing different cap layers at metal/dielectric interfaces or doping of HfO_2 with different atoms have been suggested in order to optimize RS behavior [3,12,15,16]. In this work, we extend our previous investigation [17] on RS effects in Pt/HfO_2/TiN MIM capacitors and shed more light on the influence of technology and measurement conditions on the RS properties of these structures. A special attention is focused on the enhancement of RS properties by O_3 treatment of TiN bottom electrode.

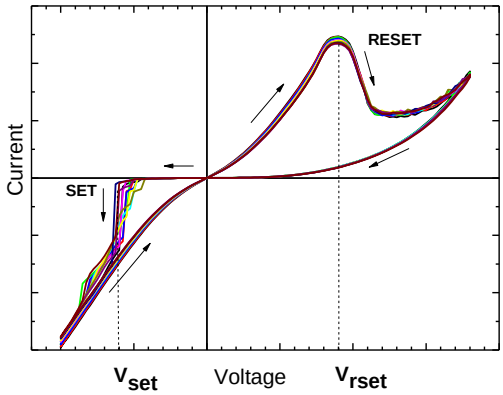

Fig. 1 Typical bipolar resistive switching loop presenting SET and RESET processes.

2. SAMPLE PREPARATION

MIM structures with active HfO_2 layer were used to investigate RS effects. HfO_2 thin (d=5-13 nm) films were grown by plasma or ozone assisted atomic layer deposition (ALD) at 300 °C in Beneq TFS 200 equipment using tetrakis (ethyl methylamino)hafnium as precursor. TiN bottom electrode (BE) was reactively sputtered in Ar/N_2 plasma at temperature of 200 °C. Thickness of the layer was 70 nm and resistivity about 200 μΩcm. Pt top electrodes (TE) (30 nm) were evaporated at room temperature through a shadow mask and capped by 30 nm thick Au. The test MIM structures are presented schematically in Fig.2. For some samples TiN BE was subjected to different number (5-40 cycles) of O_3 treatment before deposition of HfO_2. In another set of samples HfO_2 was deposited on top of very thin (1-1.5 nm) TiO_2, prepared also by ALD by using titanium isopropoxide as a precursor. X-ray diffraction spectra revealed that HfO_2 is amorphous [18].

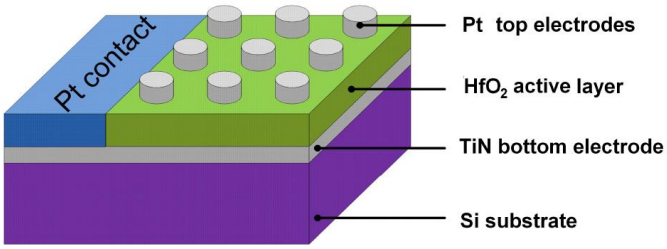

Fig. 2 Experimental MIM structures with HfO_2 as an active layer and TiN and Pt as bottom and top electrodes, respectively.

3.RESULTS AND DISCUSSION

3.1. Dependence of RS effect on ALD process and bottom electrode engineering

Figure 3 shows I-V curves and endurance characteristics of MIM structures with HfO_2 deposited by ozone assisted ALD. The first result to be mentioned is that the O_3 treatment of TiN decreases the forming voltage V_{form}, which is about -4.5 V for samples without O_3 treatment (Fig. 3a) and for 5 cy O_3 samples and decreases to about -2 V for 20 cy O_3 sample. In addition, the initial leakage current before forming increases by several orders of magnitude with increasing O_3 treatment. It is also seen that the character of RS switching as well as the ON/OFF ratio are strongly affected by the ozone treatment of TiN electrode. In samples without O_3-treatment (Fig. 3a) relatively weak abrupt RS is observed. The endurance characteristics measured during 100 switching cycles (Fig. 3b) reveal that RS is not very stable - the ON/OFF ratio is about 10 and it decreases progressively. In addition both set and reset processes occur at very low voltage (< 1 V) (Fig.3a). Although the low V_{set} and V_{reset} are generally desirable to ensure low power consumption, they should be well resolved from the readout voltage which is usually about 0.2 – 0.3 V. Only 5 cy of O_3-treatment (not shown) is enough to significantly change the RS behavior – a gradual reset followed by a weak abrupt reset process is observed; some very strong abrupt reset events have been also registered. The RS ratio is increased and is about 40-60. With increasing the number of O_3-treatment cycles (Fig. 3c,d) this ratio is further increased and is significantly

higher than 100 (Fig.3d). The typical reset process in this kind of samples is the gradual reset followed by a strong abrupt reset (Fig. 3c). As is seen, V_{set} = -1 - -2 V and V_{reset} = 1-2 V, i.e. both they well resolved with respect to the read-out voltage and are low enough to ensure low power consumption. All these observations indicate that O_3 treatment introduces some structural changes which enhance the RS behavior of structures. The most plausible hypothesis is that O_3 treatment oxidizes the TiN surface; a thin TiON film is formed and the thickness of this layer increases with increasing the number of O_3 cycles. This layer could influence the initial current and V_{form} by two ways: 1) it is a defect-rich layer which gives rise to defect-assisted transport mechanisms, hence to increased leakage current. It is very likely that this layer is conductive, i.e. it is always in LRS [19]; 2) this layer modifies the barrier at HfO_2/TiN interface. Most probably both factors play a role, but the decreased V_{form} and increased leakage current when increasing O_3 treatment give evidence that the defect-related mechanism is a dominant one. It is established in other works [20] that V_{form} is linearly dependent on the thickness of oxide film, hence thinner oxides are required to reduce V_{form}. The present work shows that it is possible to reduce V_{form} not only by reducing the oxide thickness, but also by modifying the oxide/metal interface.

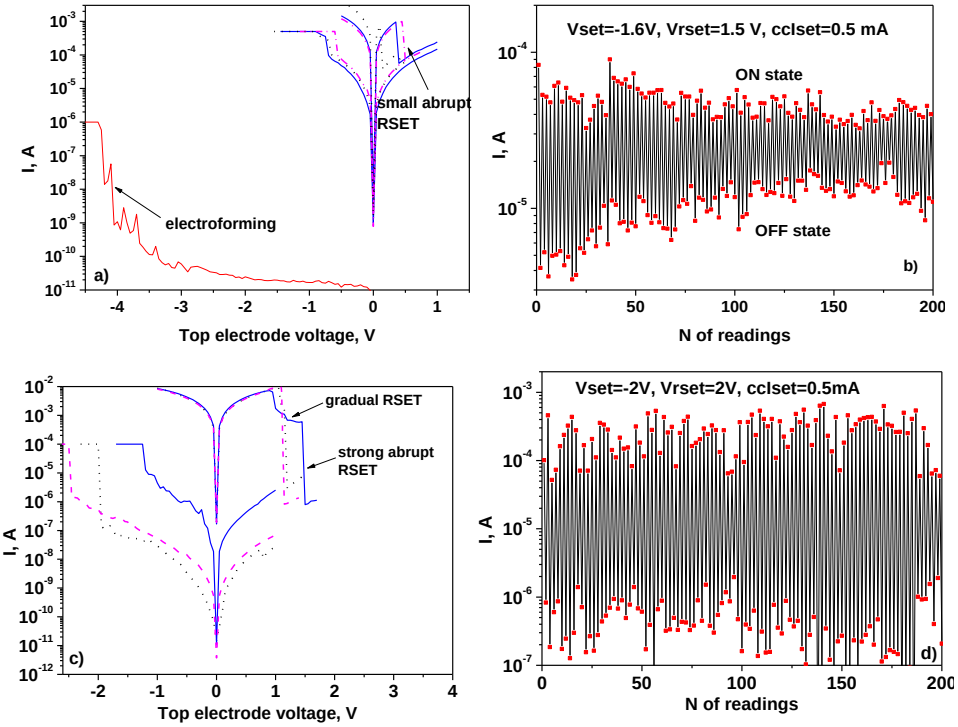

Fig. 3 Resistive switching in: Pt/HfO_2/TiN structures without O_3 treatment of TiN electrode a) typical I-V RS loops, b) endurance characteristics (ON and OFF current levels) within 100 switching cycles; and Pt/HfO_2/TiN structures with 10 cy O_3 treatment of TiN electrode c) typical I-V RS loops, d) endurance characteristics. HfO_2 is deposited by ozone assisted ALD.

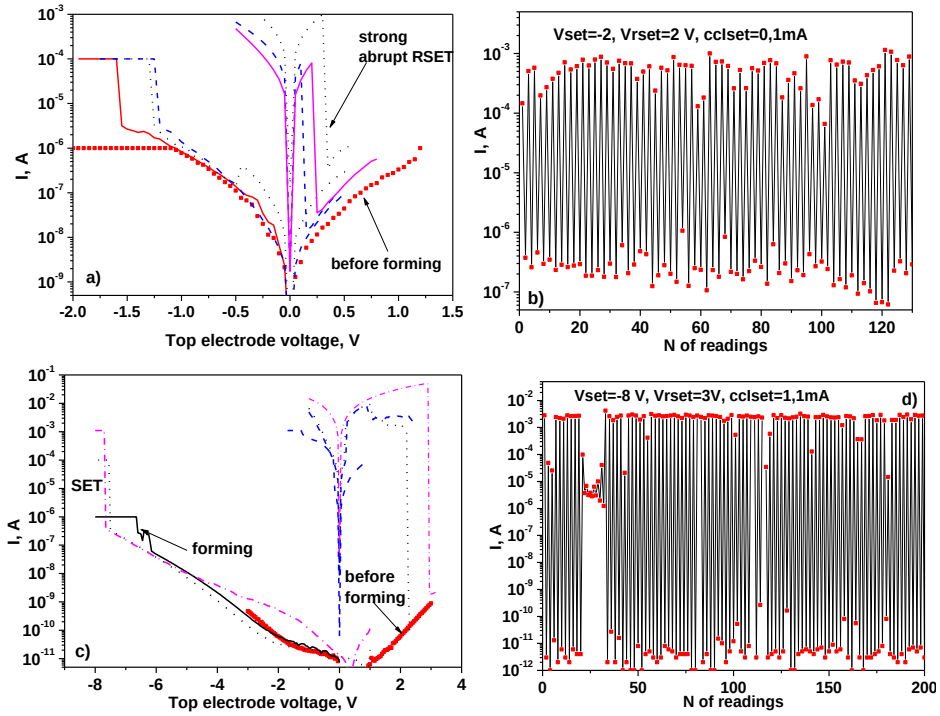

Fig. 4 Resistive switching in: Pt/HfO_2/TiN structures with 20 cy of O_3 treatment of TiN electrode, a) typical I-V RS loops, b) endurance characteristics; and Pt/HfO_2/TiN structures with ultrathin TiO_2 deposited on TiN c) typical I-V RS loops, d) endurance characteristics. HfO_2 is deposited by plasma assisted ALD.

In Fig. 4a,b the RS characteristics of HfO_2 deposited by plasma-enhanced ALD and 20 cy of O_3 treatment of TiN are shown. In this sample only strong abrupt switching events have been observed and an ON/OFF ratio of nearly 1000 is achieved. Unlike the typical RS phenomenon where OFF current is significantly higher than the current before electroforming process, in this case the current resets almost to its initial level. As the forming process is in fact controlled soft or arrested breakdown (BD), this result reveals that in this kind of structures it is possible to achieve a full recovery of BD. This phenomenon is also observed in samples with plasma-enhanced ALD HfO_2 and ultrathin TiO_2 deposited in-between TiN and HfO_2 (Fig. 4c,d). In this case the current before forming is significantly lower compared to all previously discussed samples. In addition, much higher V_{form} in the order of -6 V is observed. These results indicate for a better dielectric quality of HfO_2/TiO_2 stacks. As the difference to sample presented in Fig. 4a,b is the interfacial layer, these results support the assumption for a defect-rich TiON layer formed during O_3 treatment of TiN. It should be noted that a full recovery of BD has been achieved within more than 100 switching cycles even in the structure with ultra-thin TiO_2 and due to the very low initial leakage current, an extremely high RS ratio of about eight orders of magnitude has been measured (Fig. 4d). It should be mentioned that the similar

values of OFF current after strong reset and the initial current before electroforming are due rather to a full annihilation of the filamentary path than to any area effect arising from the relatively large top electrode (10^{-4} cm^2). The following observations support this conclusion: 1) the effect is observed in samples with quite different initial current values (~10^{-7} A at 1 V for sample presented in Fig. 4a, and <10^{-11} A at 1 V for sample presented in Fig. 4c); 2) when the sample undergoes only a gradual reset and is not allowed to go to a strong abrupt reset (see the dash line in Fig. 4c), the OFF current is several orders of magnitude higher than the initial current (i.e. in this case only a partial annihilation of conductive filament occurs); 3) V_{set} following strong abrupt reset process is similar (and even slightly higher) than the forming voltage (Fig. 4c). It should be mentioned also that the gradual reset is a preferred mode as it provides more controllable and stable RS process. The extremely large RS ratio obtained in some of the structures could hardly be implemented in RS devices. The observed phenomenon, however, could give valuable information about the BD process in these structures and the possibility to control it. This finding could be also very useful for manufacturing of structures with increased immunity to BD, i.e. structures in which multiple self-healing of BD could be obtained.

The presented results reveal that there exist two kinds of reset processes – gradual reset which is attributed to gradual annihilation of O vacancies only in a narrow region at TiN electrode [21, 22] and a strong abrupt reset process, in which a substantial part of and in some cases the whole conductive filament is erased. The results give evidence that the extent of the strong reset depends strongly on the amount of oxygen introduced to TiN bottom electrode interface by O_3 treatment – the more O_3 cycles performed, the stronger the abrupt reset is. The most widely accepted model explaining RS effect involves breaking of metal-oxygen bonds in dielectric layer during the electroforming step, dissociation of O ions and formation of oxygen vacancies. As a result a localized path with increased conductivity (conductive filament) is formed between the electrodes. This filament is characterized with increased density of O vacancies and is essentially metallic in nature [22]. The dissociation of O ions and formation of O vacancies are driven by the electric field and elevated temperature. It should be mentioned that the formation energies of O vacancies are quite different for different materials and even for the same material having different structure (e.g., amorphous or crystalline) [23], which on its turn defines different RS behavior. The dissociated O ions diffuse out of the conductive filament and some of them are stored at the anode. The activation energy of O ion diffusion is 0.3 eV, while that of O vacancy diffusion is about 1.2 eV and 0.7 eV for single and double ionized vacancies, respectively, i.e. the diffusion of O ions is more effective [22]. This implies that the process following electroforming is rather diffusion of O ions than diffusion of O vacancies. In a reset process under opposite bias polarity, the stored O ions at the electrode can be moved back to the RS switching layer where they annihilate some of the O vacancies, thus causing a rupture of conductive filament. Therefore, it is reasonable to think that the two kinds of reset processes stem from O ions dissociated from different bonds. We suggest that O ions released in HfO_2 during the forming process are swept to TiN electrode and form weak bonds there. Most likely these are O ions which take part in gradual reset process. Oxygen introduced by O_3 treatment is bounded more strongly in TiN by forming TiON and/or TiO_x. More energy (i.e. stronger electric field) is required to break these bonds and to release O ions, which under positive bias drift toward anode and recombine with the O vacancies in the conductive filament. Therefore, due to the easy oxidation of TiN, this electrode serves

as oxygen reservoir, providing O ions needed for the erasure of O vacancies in the conductive filament. O_3 treatment of TiN electrode increases the amount of oxygen stored at TiN/HfO_2 interface, thus enhancing the ON/OFF ratio. As already discussed the O_3 treatment most likely results in partial oxidation of TiN and formation of TiON defect-rich (very likely conductive) layer. Unlikely, in the case of ultrathin TiO_2, a nearly stoichiometric layer is formed. Therefore, stronger bonds are formed and more energy is needed to knock-out O ion from these bonds, hence the largest reset voltage is observed. Once the field is high enough to break Ti-O bonds, the amount of dissociated O ions is enough to annihilate the whole CF, thus resulting in full recovery of breakdown. Next, a set voltage in the order of (or even higher than) V_{form} is required to create CF anew. Due to the strong thermodynamic ability of Ti to extract oxygen from HfO_2 the process of full recovery of breakdown could be repeated multiple times.

3.2. Dependence of RS effect on switching conditions

The RS effects are strongly dependent on measurement conditions such as V_{set} and V_{reset} voltages and compliance current during the set process ccI_{set}. Compliance current is usually needed to avoid too high current flowing through the active layer which may bring it to a hard breakdown. The stability of switching and RS ratio can be varied in a wide range by changing the above mentioned parameters of switching process. Fig. 5 shows the difference in RS at different switching conditions for sample presented in Fig. 4b. The following parameters have been used to measure the RS shown in the inset of Fig. 4b: V_{set} = -2 V; V_{rset} = 2 V; ccI_{set} = 0.1 mA. As is seen in Fig. 5a by decreasing V_{reset} to 1.5 V the RS ratio decreases to below 10 and the effect is not very stable. On the other hand the increase of ccI_{set} to 0.5 mA (Fig. 5b) resulted in a more stable effect with well resolved ON and OFF state. In other words, by optimization of switching parameters it is possible to control RS effect and to obtain stable switching characteristics. It also opens-up a way for manufacturing of multilevel RS devices [15, 16], i.e. devices in which ON/OFF ratio could be varied by changing reset voltage or compliance current.

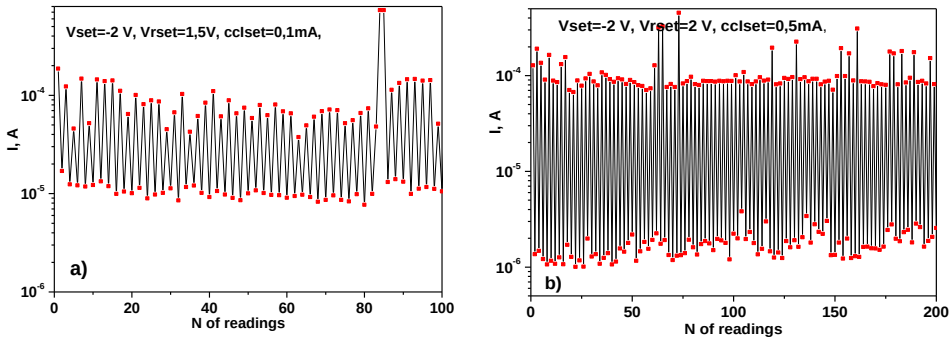

Fig. 5 Dependence of the RS effect in Pt/HfO_2/20 cy O_3/TiN sample on switching parameters: a) decrease of V_{rset}, and b) increase of ccI_{set} with respect to measurement conditions in Fig. 4b.

3.3. Dependence of RS effect on oxide thickness

Further, the influence of dielectric thickness on RS has been investigated. In this set of samples 40 cy O_3 treatment of TiN has been performed and the thickness of HfO_2 layer has been varied between 5 and 9 nm. We used thinner layers because RRAM devices have to operate at low voltages, hence the thickness of dielectric layer should be decreased to obtain acceptable values for V_{form}, V_{set} and V_{reset}. We have chosen switching conditions resulting in gradual reset process. As discussed above this mode of reset provides more controllable RS process. In Fig. 6 the resistive switching in stacks with HfO_2 thickness of 5.4 and 7.2 nm, respectively is presented. The sample with a 9 nm thick HfO_2 (not shown) exhibited similar behavior. As shown above the ON/OFF ratio depends strongly on measurement conditions. In order to study the influence of the oxide thickness itself and to disregard the influence of measurement conditions, the best results in terms of stable ON/OFF ratio for each sample have been presented in Fig. 6. All three samples show very stable RS for at least 100 switching cycles. Moreover, in the thinnest sample this stable RS is obtained without applying compliance during the set process. In thicker samples RS without compliance current has also been observed, but it has not been stable and RS ratio has varied in a wide range. It is seen (Fig. 6) that the ON/OFF ratio increases with decreasing the HfO_2 thickness and the largest ratio of about 100 is obtained for the thinnest (5.4 nm) sample. For 7.2 and 9 nm thick samples the obtained ratio is about 20 and 10, respectively. It should be noted that the difference in ON/OFF ratio comes from the differences in ON current – it increases with decreasing the thickness. The OFF current is similar in all three samples. These results indicate stronger low resistive state in thinner samples, which is assigned to formation of wider conductive filament that may be a consequence of set process performed without compliance.

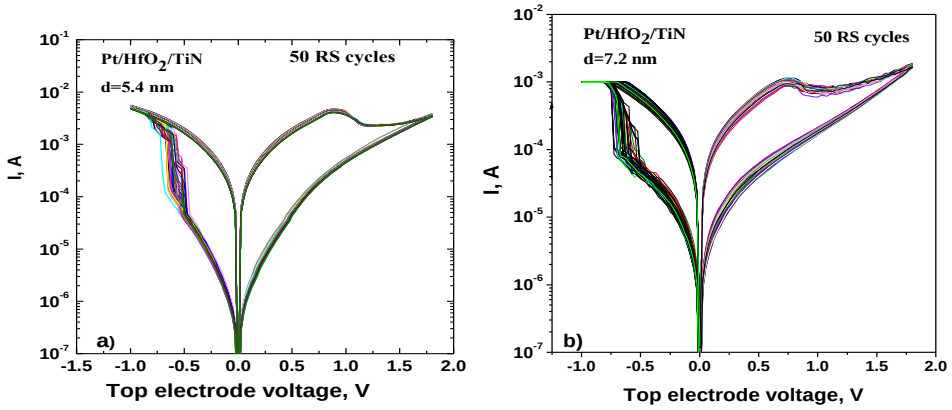

Fig. 6 Stable RS in $Pt/HfO_2/TiN$ structures with 40 cy O_3 treatment of TiN and different HfO_2 thickness: a) 5.4 nm and b) 7.2 nm

4. CONCLUSION

The results presented give evidence that by incorporation of oxygen at HfO_2/TiN interface and the choice of deposition technique it is possible to enable and enhance substantially the resistive switching of structures. The effects depend strongly on the amount of incorporated oxygen; how it is incorporated and what kinds of bonds it forms at the interface. The presence of TiON (TiO_2) ultra thin film plays a crucial role in RS process – it serves both as a reservoir for O ions released from breaking of Hf-O bonds during electroforming process and as a source of additional oxygen, released from dissociation of Ti-O and/or Ti-O-N bonds. All switching parameters (V_{form}, V_{set}, V_{reset}, ON/OFF ratio) could be substantially changed by the surface engineering, hence the amount of O and its bonding in TiO-based film as well as RS layer thickness and switching conditions should be carefully optimized to obtain stable RS effects with controllable switching behavior. By proper engineering even a multiple full recovery of CF (i.e. full healing of "arrested" breakdown) is possible.

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