Electronic Supplementary Information

Metallic filamentary conduction in valence change-based resistive

switching devices: the case of TaO_x thin film with $x \sim 1$

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1. Composition of the TaO_x thin films

X-ray reflectometry (XRR) was used to measure the thickness and density of the sputtered thin films. We determined the composition of the TaO_x films by measuring the Rutherford Backscattering Spectrometry (RBS) in a set of calibration samples. The RBS measurements were made at grazing (78° or 80°) angle and 45°. Elastic Recoil Detection Analysis (ERDA) spectra were collected at 24° forward angle. The RBS detector is located at 160° in the Cornell geometry. Spectra were collected for 2 MeV ⁴He⁺. The analysis methods used are described elsewhere.^{1–3} The TaO_x films used for the RBS measurements had a thickness of approximately 50 nm but were not the same as the ones used in the XRD study due to a special structure used to optimize the oxygen signal from the TaO_x layer in the RBS spectra. The film density as determined from XRR data was correlated with the composition as determined by RBS. After an initial calibration, the composition of the measured films was determined based on the density values measured by XRR.



Fig. S1. TaO_x films' density as determined by the XRR plotted against the x index in TaO_x obtained by means of the RBS. The circle data points represent the mean value of the density, while the error bars indicate the highest deviation from the mean value. The red line is the least squares fit to the experimental data. For comparison, the data published by Waterhouse et al.⁴ for films with thicknesses between 140 nm and 620 nm is also included. The inset shows the layer stack used in the samples measured with RBS to determine the composition.

Fig. S1 shows the calibration line (linear fit in red) used to determine the composition of the TaO_x films. The composition in terms of the x index was obtained from RBS measurements performed on a set of calibration samples. Using the same sputtering processes as used for these calibration samples, we deposited five samples for each process and measured the XRR for all these samples. From the data we extracted the films' density. In the graph of Fig. S1, the mean density is represented with the error bar representing the highest deviation from the mean density value. The x index in TaO_x is then calculated from the experimentally obtained density (ρ_V) via the formula:

$$x = \frac{(14.50 \pm 0.34) - \rho_V}{2.96 \pm 0.18}.$$
 (S1)

The obtained data and fit agree well with the density vs. composition data from the work of Waterhouse *et al.*, also shown in the plot of Fig. S1.⁴ The inset of Fig. S1 shows the layer stack used in the RBS measurements, chosen to maximize the relative oxygen signal stemming from the TaO_x layer.

2. X-ray diffraction

Fig. S2 shows the results of the grazing incidence XRD measurements performed on TaO_x films with different x values, including stoichiometric Ta_2O_5 and pure Ta films.

The Ta₂O₅ film shows a broad signal at small angles with no clear peaks from $2\theta = 15^{\circ}$ to $2\theta = 40^{\circ}$ and another one at higher angles centered around $2\theta = 54^{\circ}$. The absence of clear peaks is characteristic of amorphous materials. Ta₂O₅ typically requires temperatures above 650°C to crystallize,^{5,6} so amorphous Ta₂O₅ is expected from sputtering on top of a SiO₂ substrate at room temperature. The broad features observed are in good agreement with previously published data for amorphous Ta₂O₅.^{5,6}

The sputtered Ta film shows a diffraction pattern characteristic of the tetragonal β -Ta phase, which is normally observed for magnetron sputtered thin Ta films grown on top of an amorphous oxide.^{7,8} The diffraction pattern shows no evidence of the cubic α -Ta phase, the stable phase of bulk Ta when crystallized under equilibrium conditions.⁸ The visible diffraction peaks corresponding to the β -Ta phase are also indexed in Fig. S2.

For the TaO_x films with compositions between Ta₂O₅ and Ta, the diffraction peaks tend to get broader and eventually disappear in the noise with increasing oxygen content. The TaO_x film with $x \sim 1$ shows a diffraction pattern that is very similar to the one observed for the Ta

film, with the difference of exhibiting broader peaks shifted to lower angles. As the oxygen content is further increased to x ~ 1.7, the features observed for the films with x ~ 1 decrease in intensity, and a small-angle contribution most likely associated with amorphous Ta_2O_5 becomes more prominent. However, the diffraction pattern of the TaO_x films cannot be explained solely by a combination of the diffractograms of the pure Ta and Ta_2O_5 samples. The presence of suboxide phases could be one explanation, however, the Ta-O system is reported to have only two stable phases: Ta_2O_5 and the Ta(O) solid solution, *i.e.*, Ta with dissolved oxygen.⁹ Indeed, the oxygen can occupy interstitial positions in the β -Ta lattice.^{4,10} The incorporation of oxygen should lead to an expansion of the unit cell and would thus explain the observed decrease in the Bragg angle of the diffraction peaks for the TaO_x films due to an increase of the interplanar distances. The broadening of the peaks is associated with a decrease of the crystallite size.



Fig. S2. Grazing incidence X-ray diffractograms of TaO_x samples with different compositions (x), including Ta (top) and Ta_2O_5 (bottom). The film density as determined by the analysis of the XRR data is also displayed on the right. The green arrows show the probable evolution of the main diffraction peaks ascribed to the β -Ta structure with increasing x values. The characteristic CuK_a radiation with $\lambda = 1.5406$ Å was used in the measurements.

3. In-situ XPS of the TaO_x film with x \sim 1

To obtain further information on the chemical composition of the TaO_x layers, we performed X-ray photoemission spectroscopy (XPS) measurements. For this purpose, a similar sputtering process was implemented in a different sputter cluster tool in order to enable *in*-

situ XPS measurements of the TaO_x films. The measured Ta4f signal is shown in Fig. S3a. A simulation with contributions from three different oxidation states is able to provide a good fit to the experimental data. The data shows a strong contribution from the metallic peak associated with Ta4f 0+, and also a contribution from the Ta4f 5+ state, which is associated with the stoichiometric oxide Ta_2O_5 . Besides these two contributions, a third one, associated with the Ta4f 2+, is also used in the fit. The latter contribution could be associated with a TaO phase which, however, is thermodynamically unstable.⁹ The Ta4f 2+ contribution could also arise from the interfaces between the Ta and Ta₂O₅ phases, where the bonding should differ from that in pure Ta and Ta₂O₅ clusters.

When comparing the *in-situ* measurements with the *ex-situ* data obtained on a sample grown in the same conditions as the samples shown in the main text (Fig. S3b), it is possible to conclude that the oxidation on the surface is responsible for the increase in the relative contribution of the signal arising from the Ta4f 5+ oxidation state. However, it is still possible to observe the contribution from the Ta4f 0+ oxidation state in the *ex-situ* sample, corroborating the existence of Ta and Ta₂O₅ concluded from the analysis of the XPS performed on the *in-situ* sample.



Fig. S3. (a) XPS data for an *in-situ* sputtered TaO_x film with $x \sim 1$. A fit to the data is shown in red, with the individual fitting contributions shown. (b) Comparison of the XPS measurement performed on an *in-situ* sample and an *ex-situ* sample, grown in the same conditions as the samples shown in the main text.

4. Resistivity vs. temperature for the TaO_x films with x = 1.3



Fig. S4. Resistivity as a function of temperature from 2 K up to 300 K for a TaO_x film with x = 1.3. The data can be fitted with the same transport mechanisms used for the analysis of the data retrieved for the film with x = 1 (shown in more detail in ref. 11).

5. Temperature dependence of the resistivity of the substoichiometric TaO_x thin films



Fig. S5. Resistivity vs. temperature for three thin TaO_x films with x = 1.0, 1.3 and 1.5 and the Ta film (all samples were fabricated in the van der Pauw structure shown in the inset of Fig. 2(a) of the main text). The resistivity is displayed in a logarithmic scale. The inset plot shows the data for the film with x = 1 in a linear scale of the resistivity, evidencing the change in the P vs. T behaviour at around 50 K that can also be observed for the more oxidized films with x = 1.3 and x = 1.5.

6. Ta₂O₅-based resistive switching devices

6.1 Device fabrication

The substrate, preparation and general sputtering conditions for the fabrication of the $Ta/Ta_2O_5/Pt$ resistive switching devices were the same as the ones described in the main text. In the devices, however, a 10 to 15 nm thick TiO_x adhesion layer was sputtered on top of the thermally oxidized Si wafers, prior to the sputtering of the Pt bottom electrodes. Afterwards, a 5-nm-thick Ta_2O_5 layer was sputtered, and finally the remaining top metal layers were deposited: first a 15-nm-thick Ta layer, and then a second Pt layer. The latter is used to cap the Ta top electrode to avoid its oxidation when exposed to the air. We fabricated two series of samples, differing in the thickness of the Pt layers. For series 1 the thickness of both Pt layers is 20 nm, while for series 2 it is 30 nm. The samples were patterned, using conventional UV-lithography and liftoff, into cross-point devices with a square-shaped crossing with lateral sizes of 5, 10 and 25 µm, as schematically depicted in Fig. S6.



Fig. S6. Schematic representations of the Ta_2O_5 -based crossbar ReRAM devices used in this study, showing the geometry and layer stacks. Reprinted from C. M. M. Rosário, B. Thöner, A. Schönhals, S. Menzel, M. Wuttig, R. Waser, N. A. Sobolev and D. J. Wouters, *Appl. Phys. Lett.*, 2018, 112, 213504, with the permission of AIP Publishing.

6.2 Resistive switching

The *I-V* characteristics measured on our $Ta/Ta_2O_5/Pt$ devices show the bipolar RS typical of VCM-based systems. Two examples of the quasi-static current-voltage (*I-V*) characteristics measured at room temperature are presented in Fig. S7a and S7b. The devices were self-complied by the series resistance of the crossbar metal lines, so that no current compliance was applied during switching. However, we could control the series resistance by changing the

thickness of the Pt electrodes used. For 20 nm of Pt (series 1), the series resistance is around 500 Ω for the 5 × 5 μ m² devices, but for 30 nm of Pt (series 2), the series resistance drops to approximately 300 Ω for devices with the same area. The devices are initialized with an electroforming step accomplished at approximately + 2 V, after which the devices switch between the LRS and the HRS. The applied voltage was normally limited between – 1.5 V and + 1 V. For devices from series 1, the LRS ranged from 250 Ω to 3 k Ω , while the HRS varied from 5 k Ω to 40 k Ω . For devices in series 2, a lower resistance in LRS can be achieved due to a higher current through the device. The real ("intrinsic") switching voltage across the device was calculated by subtracting the voltage drop over the series resistance¹² yielding the switching curve shown in green in Fig. S7a. For the case illustrated in Fig. S7b, the "intrinsic" switching curve was directly measured using the 4-wire configuration depicted in Fig. S7b, while the black curve shows the same measurement in a 2-wire configuration. The *I-V* characteristics are typically linear in the LRS and nonlinear in the HRS. The linearity of the LRS is also observed at 2 K.

6.3 Temperature dependence of the resistance in LRS

For the devices in series 1, the LRS resistance normally shows the temperature dependence depicted in Fig. S7c. The resistance decreases with increasing temperature, exhibiting a negative temperature coefficient of the resistance (TCR). The resistance changes by less than 10% from 300 K to 2 K. The fitting of the data leads to two different regimes (see the insets of Fig. S7c): (i) high-temperature regime, at 50 K < T < 300 K, where the best fit to the data is obtained with the \sqrt{T} dependence of the conductance; (i) low-temperature regime, T < 50 K, where the exp $(1/\sqrt{T})$ dependence of the resistance fits well the data.¹¹ For other devices operating in the same conditions, the low-temperature regime is less clearly observed, or perhaps shifted to a lower temperature, but most of them show the high-temperature regime.

For the series 2 devices, the temperature dependence of the LRS resistance can be quite different. As can be seen in Fig. S7d, the TCR can be positive, *i.e.*, the resistance increases with rising temperature. Interestingly, the positive TCR is also observed for devices in both series that suffered a hard breakdown, which means it is clearly a product of high currents through the conductive filament and possible high temperatures achieved via Joule heating. The

resistance changes less than 10% in the whole temperature range. At low temperatures there is again a change of the *R* vs. *T* behavior with an upturn of the resistance, similar to the change observed for the devices with negative TCR. For the device shown in Fig.s S7b and S7d, the



Fig. S7. (a) Quasi-static current-voltage (*I-V*) characteristics of a device with a series resistance of ca. 500 Ω and maximum current through the filament of 1.5 mA. The black lines show the measured data, while the green lines show the "intrinsic" switching curve, calculated by subtracting the voltage drop over the series resistance. Reprinted from [FULL CITATION], with the permission of AIP Publishing. (b) Same as (a) for a device with a series resistance of ca. 300 Ω and maximum current through the filament of 2.2 mA. The green curve was measured in a 4-wire configuration, thus showing the "intrinsic" switching curve (without the voltage drop over the series resistance). (c-d) Temperature dependence of the resistance in the LRS for the device shown in (a) and (b), respectively. (a) and (c) are reprinted from C. M. M. Rosário, B. Thöner, A. Schönhals, S. Menzel, M. Wuttig, R. Waser, N. A. Sobolev and D. J. Wouters, Appl. Phys. Lett., 2018, 112, 213504, with the permission of AIP Publishing.

TCR is of approximately + 3×10^{-5} K⁻¹.

7. Reduced activation energy

The reduced activation energy is defined as:¹³

$$w = \frac{d\ln\sigma}{d\ln T} = -\frac{d\ln\rho}{d\ln T}.$$
 (S2)

The numerical differentiation needed to calculate w from the measured temperature dependence resistivity data enhances the noise of the measurement, and thus can be problematic. To minimize this problem, a centered moving average with an odd number of points n was applied to the resistivity data, and then the reduced activation energy was obtained by calculating the divided differences between adjacent points after the averaging procedure. n varied between 10 and 15 for the used data. An additional moving average filtering was used after the numerical differentiation, while keeping the same temperature dependence observed prior to the smoothing procedure in order to make the dependence clearer.

8. Magnetoresistance

8.1 Magnetoresistance vs. temperature



Fig. S8. (a) Decay with temperature of the MR at B = 9 T of the TaO_x film with x = 1. The inset shows the same data but on a logarithmic *y*-axis evidencing a *quasi*-exponential decay. (b) Same as in (a) but for the Ta film.

The MR shows a *quasi*-exponential decay as the temperature is increased, as can be seen in Fig. S8. The insets of the plots show the MR on a logarithmic scale, evidencing the exponential decay as the temperature is increased.

8.2 TaO_x vs. Ta

Fig. S9 shows the MR measured on the Ta film and the TaO_x film with x = 1 at a temperature of T = 1.8 K and on the film with x = 1.3 at T = 2 K, for comparison.



Fig. S9. MR as a function of the magnetic field at T = 1.8 K for the TaO_x film with x = 1.0 and for the Ta film, and at T = 2 K for the TaO_x film with x = 1.3.

8.3 Resistivity vs temperature at different applied magnetic fields

The effect of the magnetic field on the resistivity can be directly observed in the temperature dependence of the resistivity measured for different magnitudes of the externally applied magnetic field, shown in Fig. S10. The positive MR is translated into a rise of the resistivity with increasing magnetic field. For the TaO_x film with x = 1.0 shown in Fig. S10a, the effect of the magnetic field is visible up to approximately 30 K and leads to a steeper increase of the resistivity with decreasing temperature. For Ta, we clearly observe a pronounced downturn of the resistivity below 10 K, as shown in Fig. S10b. This downturn and the positive, cusp-like shaped MR curve are typical signatures of weak antilocalization. Weak localization (WL) and weak antilocalization (WAL) are quantum interference effects that induce a change in the resistance of a disordered system due to interference of the wavefunctions of the charge carriers with themselves after elastic scattering from static

disorder.¹⁴ With the strong spin-orbit interaction of the heavy Ta atoms, WAL is expected in our Ta-based samples. The application of a magnetic field introduces an additional phase component to the electron waves, so that the interference is modulated and eventually destroyed in a sufficiently high magnetic field. Thus, by applying an external magnetic field to the samples it is possible to destroy the WAL, so that the resistance increases as shown in Fig. S10.



Fig. S10. Temperature dependence of the resistivity for different applied magnetic fields up to 9 T for: (a) TaO_x film with x = 1.0; (b) Ta film.

8.4 Weak antilocalization fit

The MR data shown in the main text was fitted by two different WAL formalisms. The need for the different formalism came from the different dimensionality of the quantum interference effect, determined by comparing the phase breaking length (PBL) L_{ϕ} with the film thickness t, as described in the main text. For the 2D case, a simplified formula obtained by Assaf *et al.*¹⁵ was used. This formula has the quadratic component b that relates not only with the classic cyclotronic term but also with the simplified components of the spin-orbit and elastic terms of the general Hikami-Larkin-Nagaoka model, giving $b = b_c + b_q$, with b_c and b_q being the classical and quantum terms, respectively. The classical term arising from the cyclotronic MR is given by:¹⁵

$$b_c = -\mu_{MR}^{2} G_{0_{j}}$$
(S3)

where μ_{MR} is the MR mobility that can be approximated by the Hall mobility,¹⁵ and $G_0 = \frac{2e^2}{h}$ is the conductance quantum. On the other hand, the quantum term is given by:¹⁵

$$b_q = -\frac{e^2}{24\pi h} \left(\frac{1}{B_{SO} + B_e}\right)^2 + \frac{3e^2}{48\pi h} \left(\frac{1}{\frac{4}{3}B_{SO} + B_\phi}\right)^2$$
(S4)

where the different characteristic fields B_i have the same meaning as in the main text. Considering the measured Hall mobility for the Ta film, the classical term b is much lower than the parameter b obtained from the fits, therefore the quantum term is the dominating one, and $b \approx b_q$. Considering also that the mean free path L_e is much smaller than L_{SO} , we can derive

the expression relating $B_{SO} = \frac{\hbar}{4eL_{SO}^2}$ with the fitting parameters $B_{\phi} = \frac{\hbar}{4eL_{\phi}^2}$ and *b*:

$$B_{SO} \approx \frac{3}{8} \left[\left(B_{\phi}^{2} - 4B_{\phi} + \frac{e^{2}}{4\pi hb} \right)^{1/2} - B_{\phi} \right],$$
(S5)

The fit parameter L_{ϕ} is already shown in the main text in Fig. 6d. The parameters α and L_{SO} resulting from the fit of the 3D WAL formalisms to the data measured on the TaO_x film with x = 1 and on the Ta film are shown in Fig. S11a and b, respectively. For the 2D case, the L_{SO} was determined from the fitting parameter b using Equation (S5) and added to the plot of Fig. S11b. As can be seen in Fig. S11b, the change of the formalism did not create big discontinuities in the temperature dependence of the α and L_{SO} parameters.



Fig. S11. Remaining parameters resultant from the fit using the weak antilocalization formulas presented in the main text. α and L_{so} for: (a) TaO_x film with x = 1. (b) Ta film with the parameter *b*, from which L_{so} is calculated in the inset of the L_{so} vs. temperature plot.

9. Resistivity vs. T for Ta on top of Ta₂O₅

A 15-nm-thick Ta layer was also sputtered on top of a Ta_2O_5 (5 nm)/SiO₂ (430 nm)/Si substrate. This layer shows a positive TCR, in contrast to the 15-nm-thick Ta layer sputtered on top of a SiO₂/Si substrate, shown in the main text. The resistivity measured in the Ta layer on top of Ta_2O_5 in the temperature range from 1.8 K up to 300 K is shown in Fig. S12.



Fig. S12. Resistivity vs. T of a15-nm-thick Ta layer deposited on top of Ta₂O₅ (5 nm) / SiO₂.

10. Contribution from the contact resistance in Ta/Ta₂O₅/Pt devices

The possible parasitic and other contributions that could be present in the resistance measurements of the Ta_2O_5 -bsed ReRAM devices have been discussed in a previous publication.¹¹

Another possible contribution for the temperature dependence of the resistance of the ReRAM devices in the LRS can arise from the interfacial contact resistance. Following Wang et al.¹⁶ we checked the possible contact resistance by measuring a device after breakdown, in order to reduce the possible contribution from the conduction through the conductive filament. The resistance of this device was measured in the temperature range from 2 K up to 300 K. As can be seen in Fig. S13, a positive TCR is measured as well as a very weak temperature dependence. This contribution cannot explain the negative TCR measured for the ReRAM devices we discussed. Care must be taken, however, when the positive TCR is discussed, as the temperature dependence could at least partially stem from this contribution.



Fig. S13. Resistance as a function of temperature from 2 K up to 300 K for a device where a breakdown was induced in order to reduce the contribution from the conductive filament and check the possible temperature dependence of the contact resistance. It shows a positive TCR with a very weak temperature dependence of the resistance.

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