EPICO

Electric Pulse Induced Changes in Oxides
## EPICO-2012 Schedule

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**Time relaxation of the resistive retention state of complex oxides (YBCO, LSMO) / metal RRAM devices**

C. Acha

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We have studied relaxation effects of the remnant resistance obtained immediately after the electric-pulse switching process on metal/complex oxide interfaces [(Au, Pt) / (YBCO, LSMO)]. We found that resistance relaxes following a particular stretched exponential law, with a temperature and applied switching power independent exponent. Interestingly and unlike ordinary thermal diffusion processes, we observe that the characteristic relaxation time increases with increasing temperature and applied power. This anomalous dependence of the characteristic time gave us the opportunity of finding an interesting physic process related to the oxygen diffusion on complex oxides, like superconducting cuprates or colossal magnetoresistant manganites.

We claim that the observed behavior, common for both complex oxide interfaces, points to a generic phenomenon that can be understood as due to the diffusion of oxygen ions (or oxygen vacancies) moving on a 2D surface (grain boundaries) with a temperature dependent density of trapping centers.

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**High-temperature dielectrics in CaZrO$_3$-modified Bi$_{1/2}$Na$_{1/2}$TiO$_3$-based lead-free ceramics**

M. Acosta, J. Zang, W. Jo and J. Rödel

*Technische Universität Darmstadt, Darmstadt, Germany*

Ferroelectric ceramic capacitors constitute indispensable components for passive electronics. They are employed for bypassing, coupling, filtering, among others in strongly growing markets. In the meantime, new technological applications require operation in harsh environments in automotive, petrochemical, aero/astronautics and nuclear industries. Many of these applications are subject to high temperatures beyond 200 °C.

In this study, the structural, dielectric and electrical properties of the $(1-x)(0.94\text{Bi}_{1/2}\text{Na}_{1/2}\text{TiO}_3+0.06\text{BaTiO}_3)+xC\text{aZrO}_3$ and $(1-x)(0.82(0.94\text{Bi}_{1/2}\text{Na}_{1/2}\text{TiO}_3+0.06\text{BaTiO}_3)+0.18\text{K}_{0.5}\text{Na}_{0.5}\text{NbO}_3)+xC\text{aZrO}_3$ systems with $x=0.05, 0.1, 0.15$ and 0.2 are reported for temperatures ranging from -150 to 500 °C. These cost effective materials resulted in excellency in temperature stability. They present at least ~ 400 °C of operational windows within 15 % of variation in their $\varepsilon_r$ with average values ranging from ~ 470 up to ~ 2300. Moreover, the $\tan(\delta)$ are below ~ 10% and RC constants range from ~ 0.03 up to ~ 4 s at 300 °C. A phenomenological description of the relaxor features observed in these materials is also presented, and the dielectric anomalies are rationalized by relaxational behaviors of PNRs in an ergodic relaxor state.
Pattern Classification by Memristive Crossbar Circuits with Ex-situ and In-situ Training

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The development of artificial neural networks (ANNs) based on emerging non-volatile memory, such as metal oxide memristors, has attracted an increasing interest recently. In the simplest form of such ANNs, the neurons are implemented with conventional (complementary metal-oxide-semiconductor) technology and interconnected by memristors functioning as artificial synapses. While there are number of recent proof-of-concept demonstration for synaptic operation by single memristive devices, demonstration of even simple functionality memristor-based ANN remains challenging and have yet to be reported. Here, we demonstrate pattern classification by a single-layer perceptron network implemented with hybrid crossbar circuits. 20 synaptic weights, which are realized by Pt/TiO$_2$/Pt memristive devices with sub-20-nm-scale active region are successfully trained by ex-situ and in-situ method. In the first case, the appropriate conductance of each memristor is calculated on the precursor software-based network and then imported sequentially to the crossbar circuits using variation-tolerant programming algorithm. In the second case, the weights are adjusted in parallel following perceptron learning rule by applying voltage pulses from pre-synaptic and post-synaptic neurons. Both ex-situ and in-situ methods work satisfactory despite significant variations in switching behavior of memristive devices as well as half-select and leakage problems in crossbar circuits. The demonstrated results give hope for much anticipated efficient implementation of ANNs and pave the way towards extremely dense and high performance information processing systems.

Insulator to metal transitions and resistive switching in the narrow gap Mott Insulator AM$_4$Q$_8$ (A=Ga,Ge; M=V, Nb, Ta; Q=S, Se)

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The AM$_4$Q$_8$ (A = Ga, Ge; M = V, Nb, Ta, Mo; Q = S, Se) compounds represent a family of narrow gap Mott insulators with very interesting electronic properties. These compounds exhibit a lacunar spinel structure with tetrahedral transition metal clusters M$_4$.$^1$ Compared to most other inorganic Mott insulators, the AM$_4$Q$_8$ compounds show very small Mott-Hubbard gaps (0.1-0.3 eV) as the electronic repulsion occurs on the scale of these tetrahedral clusters and not on the scale of single atoms.$^2$ A direct consequence of this low gap value is the high sensitivity of these compounds to external perturbations such as pressure, doping and electric field. For example, a bandwidth-control Insulator to Metal Transition (IMT) and superconductivity appear in GaTa$_4$S$_8$ and GaNb$_4$S$_8$ under external pressure.$^2,3$ On the other hand, the substitution of V per Ti or Ga per Ge in GaV$_x$S$_8$ leads to doping control IMT with the emergence of a half ferromagnetic metal.$^4,5$ or of negative colossal magnetoresistance.$^6$
We have recently discovered that the AM$_4$Q$_8$ compounds are also sensible to another perturbation, namely the electric field. Application of short electrical pulses on these compounds induces indeed a new phenomenon of volatile or nonvolatile resistive switching (RS) $^{7,8,9}$. The volatile transition appears above threshold electric fields of a few kV/cm, while for higher electric fields, the resistive switching becomes non-volatile. The application of successive very short electric pulses enables to go back and forth between the high and low resistance states. All our results indicate that the resistive switching discovered in the AM$_4$Q$_8$ compounds does not match with any previously described mechanisms $^{10}$. Conversely, our recent work shows that this RS is related to a purely electronic mechanism $^{11}$ and to an IMT at the nanoscale $^7$. Finally, it is possible to deposit a thin layer of GaV$_4$S$_8$ $^{12}$and to retrieve the reversible resistive switching on a metal-insulator-metal (MIM) device which proves the potential of this new class of Mott-memories for applications $^{13}$.

References
11. V. Guiot et al. submitted (2012)

Nanoscale analysis of redox-processes in resistive switching complex oxide devices

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Although there exists a general consensus that bipolar resistive switching in transition metal oxides is in most cases connected with a redox-process, the details of the underlying physical mechanism are only poorly understood up to now. One of the obstacles for its further elucidation is that the net changes of structure, stoichiometry and valence state during electroforming and switching are very small and occur primarily at the electrode interface or within nanoscale filaments. Besides electron microscopy and spectroscopy, different Synchrotron radiation based spectroscopy and scattering techniques have been employed so far in order to detect local redox-processes and/or filament formation caused by the electrical treatment of resistive switching oxide devices. Within this talk, an overview about different experimental approaches, ranging from surface sensitive techniques to bulk sensitive techniques, will be given.

In particular, for epitaxial SrTiO$_3$ thin film devices, by the combination of scanning force microscopy with photoelectron spectromicroscopy and X-ray absorption spectroscopy, we were able to prove that electroforming goes along with a homogenous as well a local formation of oxygen vacancies $^{[1]}$, $^{[2]}$. Moreover, significant changes are detected in the chemical state and the relative stoichiometry of the cation sublattice during electroforming demonstrating the formation of new phases $^{[2]}$. 

References

Furthermore, we have elucidated the details of the electronic structure of resistive switching Pt/Ti/PCMO/SrRuO$_3$ devices and the influence of electroforming by combining hard X-ray photoelectron spectroscopy with transmission electron microscopy- and spectroscopy. By supplementing theses investigations with impedance spectroscopy, we were able to convolute the influence of the different interface contributions to the electrical properties of different resistive states. These investigations indicate that the PCMO undergoes minor changes during electroforming and the main chemical changes in the device structure occur in the TiO$_x$ interface layer [3].


**Shock wave mechanism for bipolar resistive switching**

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Many recently discovered systems displaying resistive switching phenomena have been widely studied as potential basis of future electronic memory devices. The hysteresis cycles observed in several such transition-metal oxide devices show a universal feature related to an abrupt onset of resistance switching. Here, we present an analytic analysis of a recently proposed phenomenological model$^1$, via first principle derivation of an appropriate non-linear diffusion equation describing the rapid oxygen vacancy migration under strong time-dependent external electric fields. The non-linearity effect, which reflects the vacancy concentration dependence of the local resistivity, can be related to the modified Burger’s equation describing shock waves. We show that the sudden resistance drop observed in the numerical solution of the model occurs exactly when the vacancy shock wave front reaches the interface between the highly resistive Schottky barrier and the bulk. We argue that the magnitude of the relevant nonlinear term is maximal for materials in the close vicinity of the metal-insulator transition; this insight may facilitate the optimization of device performance, possibly leading to a new class of memristor devices.


**Resistive Switching in multilevel TiO$_2$ memory devices**

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In the present work, we study Au / TiO$_2$ / metal (Cu and Al) junctions in a crossbar geometry, which exhibit resistive switching characteristics. Samples were grown by sputtering TiO$_2$ and miniaturized by means of standard optical lithography. We tested the behaviour of the physical system after electroforming with both polarities.
In devices with Cu top electrode we obtain evidence of a cooper metallic filament for negative electroforming, while for positive electroforming the filament is presumably Magnelli phase based. From the comparison of both states we discuss the role of the active defects involved in the oxide matrix, which are responsible for the multilevel observed states. In devices with Al top electrode, after the negative electroforming process we observe the presence of micrometer sized “craters”. We study the effect of changing the current compliance $I_{cc}$ to control the area covered by these craters. We suggest that the change in the affected area (due to the different $I_{cc}$'s) is related to the number of formed filaments at the oxide matrix. Additionally we reproduce the experimental results through a realistic model that includes as a main ingredient the oxygen vacancies diffusion under applied electric field. The associated vacancy profiles further unveil the prominent role of the effective electric field acting at the interfaces. These simulations allow to disentangle the microscopic mechanisms behind the resistive switching in metal-transition metal oxide interfaces.

**Asymmetric pulsing for reliable operation of titanium/manganite memristors**

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We present a pulsing protocol that significantly increases the endurance of a titanium-manganite interface used as a binary memory cell. The core of this protocol is an algorithm that searches for the proper values for the set and reset pulses, cancelling the drift in the resistance values. A set of experiments show the drift-free operation for more than $10^5$ switching cycles, as well as the detrimental effect by changing the amplitude of pulses indicated by the protocol. We reproduced the results with a numerical model, which provides information on the dynamics of the oxygen vacancies during the switching cycles.

**Modulation of Forming Voltage in Reactively Sputtered Titania Resistive Switches**

Brian Hoskins

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Metal oxides are typically grown as close to stoichiometric as possible for use as dielectrics. Consequently, there exists comparatively little experience in the growth of reduced metal oxides. Since oxygen vacancies have been implicated in the switching behavior of these films, introducing them into the virgin state should yield desirable results. In this unpublished work, the processing conditions leading to the growth of reduced films by reactive sputtering of Titania are discussed. Hysteresis free operation of the growth conditions is demonstrated yielding continuous control of the film stoichiometry. It is then shown that sandwich structures of Pt/100-nm TiO$_2$$_x$/Pt can achieve forming voltages below 2.5 volts, suggesting a new pathway whereby forming free devices can be realized. Issues concerning the limits of processability are discussed as well as means of surmounting them.

This work is supported by AFOSR and NSF.
Resistive Switching Mechanism of Single-Crystalline Oxide-based Schottky Junctions

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Electric-field- and/or current-induced resistive switching (RS) phenomena in metal oxides have attracted considerable research interest due to their potential use in nonvolatile memory device applications. Intensive investigations have revealed that coupled electron–ion dynamics plays a key role the RS mechanism. In thin films, structural defects (e.g., grain boundaries) and chemical inhomogeneity can provide fast electronic and ionic transport path, and hence very sophisticated nano-scale characterization is essential in unveiling the switching mechanism of a specific sample [1,2]. Single crystals, on the other hand, have negligible crystalline disorder and compositional variation. Thus, a metal/single crystal junction can be an ideal model system to investigate the RS behaviors dominated by interface-effects [3-6].

We investigated transport and RS behaviors of rectifying junctions consisting of Pt electrodes and Nb-doped SrTiO$_3$ (STO) single crystals. Current-voltage (I-V) and capacitance(C)-V characteristics were analyzed using the thermionic emission model and the Schottky barrier height (SBH) for the junctions, exhibiting notable difference depending on the resistance state, was extracted [3]. Internal photoemission spectroscopy (IPES) measurements, as comparative means to the conventional electrical measurements, were also performed for the junctions. The IPES results indicated invariance of the mean barrier height. Tunneling effects and an inhomogeneous barrier model could be proposed to resolve the apparent discrepancy [4]. We also obtained and analyzed the admittance spectra of the Pt/STO junctions at different resistance states in air and vacuum. The analyses showed that the carrier lifetime at the traps was largely varied depending on the resistance state [5]. The ambient dependence suggested that the charges at the trap states were affected by the oxygen adsorption/desorption at the surface. Regarding the surface adsorption and related transport characteristics, we investigated TiO$_2$ single crystals using scanning probe microscopy [6,7].

We have investigated electric-field-induced transport behaviors of SrTiO$_3$ and TiO$_2$ single crystals, as representative metal oxide materials showing RS. Photocurrent and scanning probe microscopy measurements, as well as the conventional electrical characterizations, clearly revealed the electric-field-induced influences on our samples. Further understanding and careful analyses of the single crystalline samples are expected to give us valuable insights to unveil underlying RS mechanism.

References
Talks

Overview of novel back-end memories

Jorge Kittl
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Non-volatile Storage: Opportunities for Business and Science

Gilberto Medeiros-Ribeiro
Centro de Eletronica Avançada/CEITEC-AS, Porto Alegre, RS, Brazil, and
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A universal memory, that could replace existing technologies at many hierarchical levels, is a highly sought goal that would significantly change the landscape of computer architecture. Solid state, non-volatile storage without moving parts can be embodied in a variety of forms, and there is currently an intense race among companies, universities and laboratories to make such concept a reality. Here I will cover some recent developments in the implementation of non-volatile memories from a fabrication standpoint as well as from a fundamental physics. From ionic motion to single point contact, these systems offer a unique opportunity to create not only a new class of memory, but a laboratory to study coupled ionic-electronic transport in a controlled manner.

Compact modeling of filamentary conduction in resistive switching devices

E. Miranda
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Within the wide variety of physical mechanisms governing the resistive switching mechanism in oxide films, filamentary conduction across the dielectric layer is one of the most invoked. Because of the localized nature of electron transport, the phenomenon often exhibits quantization effects that are consistent with the Landauer formulation for mesoscopic conducting systems. Starting from this idea a number of compact models for the reversible behavior of the current-voltage characteristics can be developed. The connection with memristive devices will be also discussed.

Dirac Cone systems

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In 2004, a new electronic system has emerged in solid state physics: massless-Dirac-electron band structure systems, where graphene is the best example. Since then, research on graphene has grown exponentially. The 2010 Nobel prize to A. Geim and V. Novoselov for possible future applications on graphene shows that this subject is not only important for a close physics community but it is also relevant to the industrial community.

In this talk, I will first introduce graphene and address some of its possible future applications. Then, I will present Dirac cone physics on graphene and some research I have performed in order to answer a debated issue: why Dirac carriers have a limited mobility on graphene? Finally, I will address Dirac cone physics in another system: α-(BEDT-TTF)2I3 where the Dirac carrier properties prove to be at least as good as the “state of the art” suspended graphene or graphene on Boron-Nitride devices. In this system, a
coexistence of mass-less and massive fermions occurs. Such coexistence of two carrier types represent a very unusual situation in condensed-matter physics, namely because of a very low Fermi temperature (of the order of 1 Kelvin or 100µeV). Furthermore, it allows us to perform an original study of the interplay between Dirac and massive carriers in view of electronic correlations that are expected to be relevant in this organic compound in contrast to graphene.

Stochastic resistive switching

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Strukov et al. [1] presented a simple nonlinear dynamical model of memristors which reproduces well the observed behavior of actual devices. They devised a one-dimensional model of the sample, by dividing it into two well defined regions with different resistivities. The boundary between these regions is governed by a first order differential equation such that there is a nonlinear dependence of the velocity on the externally applied field and the intrinsic physical characteristics. The total resistance of the memristor is thus the in-series resistance of the two regions.

Recently, Stotland and Di Ventra [2] presented simulations based on this model which take into account the interaction of internal (inherent to the sample) noise with the nonlinearity of the memristor. In particular, they studied the effect of additive white Gaussian noise in the first order equation of the aforementioned model. They showed that there is an optimal noise intensity for which the contrast between low- and high-resistance values is maximized. They also suggested that externally applied noise should have a similar effect on memristors.

In this work, we extend the results in Stotland and Di Ventra. On the one hand, we study the influence of external noise and show that, contrary to what it was suggested, it does not enhance the contrast between low- and high-resistance values. On the other hand, we present experiments conducted on a manganite sample showing that the addition of external white Gaussian noise to a small amplitude driving signal yields a contrast ratio between low- and high-resistance states, comparable to that obtained by the application of a large amplitude noiseless signal. Qualitatively, it appears that the noise helps small signals to overcome some threshold values beyond which a drastic change in the memristor behavior is observed. The simple model in Struko et al. does not include this type of threshold behavior.

Pickett et al. [3] presented a model of the behavior of a memristor consisting of a resistor, corresponding to the bulk resistance, in series with an electron tunnel barrier formed at the interface between a metallic contact and the sample material, such that its width can be changed by an external field. A numerical simplification of Pickett's model introduced by Kvatinsky et al. [4] shows three distinct dynamic regimes depending on the input current, i.e., there is a thresholding behavior similar to that observed in the experiments. Indeed, we found a very good agreement between numerical simulations based on Kvatinsky's model and our experimental results. Encouraged by this agreement, we present simulation results aimed at further characterizing the role of noise in resistive switching.

References


Electric Pulse Induced Changes in Oxides/EPICO2012 CAC, BsAs, 12-14 December, 2012
X-rays induced discharge of programmed NROM

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We study NROM (nitride read-only memories) memory cells with an ONO (oxide/nitride/oxide) structure as insulator layer. Two different bits can be programmed in these devices depending on the way in which the channel is polarized. We analyzed X-rays induced discharge of stored charge at nitride. Different sources of X-rays were tested: tungsten (10keV), chromium (5keV) and silver (20keV). Various NROM were irradiated simultaneously to test the correlation between discharge events in neighboring devices. Stress on-line measurements were performed. Discharge was observed in all cases (different sources), even in non-programmed NROM. Degradation velocity seems to follow an inverse law with the source energy probably linked with the probability of interaction in each case.

Voltage-Induced Metal-Insulator Transition in Vanadium Oxides: The role of Joule Heating

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Many compounds of the vanadium oxide family undergo a metal-insulator transition (MIT) at temperatures ranging from 68 to 430 K. Two examples are VO₂ and V₂O₃. While VO₂ undergoes a 5-order of magnitude change in the resistance at 340 K from a monoclinic insulator to a rutile metal, in V₂O₃ the MIT occurs at 150 K from a monoclinic insulator to a rhombohedral metal, accompanied by an antiferromagnetic to paramagnetic transition. In addition to temperature, the MIT can be induced by pressure, light irradiation among others. Of special interest is the voltage-induced MIT, which remains controversial as it may originate from an electric breakdown or local heating. Here we will present a variety of measurements of electric transport, local-temperature and Low-Temperature SEM (LTSEM) on VO₂ and V₂O₃ thin films devices. Our results suggest that the electrical breakdown at macroscopic scale is caused by the formation of electro-thermal domains (ETDs). On the contrary, it appears that at nanoscale the electrical breakdown cause the MIT of single domains. These results will shed light on the switching mechanism in the vanadium oxide family. Furthermore, the nonlinear dynamic response of the MIT at sub-micron scale reveals that the switching dynamics can be related to a self-organized criticality process.

Crossover regime in manganite based resistive switching memory devices

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Here we report on the preparation and electrical characterization of thin films of the canonical La\textsubscript{0.66}Ca\textsubscript{0.33}MnO\textsubscript{3} manganite. Films were prepared by pulsed laser deposition on top of highly doped (conductive) Si. X-Ray Diffraction spectra showed that the films resulted single phase and polycrystalline. Resistive switching properties were probed by means of dynamic pulsed I-V curves and remanent hysteresis switching loops. Different growth conditions (namely the partial oxygen pressure during deposition), film thickness and metallic top electrodes (Ag, Cu, Al) were studied. In all samples we observed bipolar resistive switching with abrupt SET and RESET transitions. Systematic studies of the evolution of the $R_{\text{HIGH}}$, $R_{\text{LOW}}$ and their ratio as a function of the SET compliance current (CC) were performed. We found that for low CCs, $R_{\text{HIGH}}$ remains constant while $R_{\text{LOW}}$ decreases, being increased in this way the $R_{\text{HIGH}}/R_{\text{LOW}}$ ratio (values up to $\sim$1000 were observed). For higher CCs, $R_{\text{HIGH}}$ abruptly drops leading to a decrease of the ratio. Based on the analysis of the I-V curves and the behavior of $R_{\text{LOW}}$ as a function of the electrode area, we propose a crossover mechanism from an interface related resistive switching effect for low CCs to a filamentary regime for higher CCs.

Bipolar resistive switching in oxide-based memory cells

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I shall review the main ingredients and findings of the Voltage Enhanced Oxygen Vacancy (VEOV) model \cite{1,2,3}, originally introduced to explain the microscopic origin of BRS in complex oxide-based memory cells. In particular I will discuss how it can be adapted to account for BRS in binary oxides cells. I will show numerical results that qualitatively reproduce nontrivial resistance hysteresis experiments on Au/TiO\textsubscript{2}/Al cells \cite{4}.

\begin{itemize}
\item \cite{1} M. Rozenberg, M.J. Sánchez, R. Weht, C. Acha, F.G. Marlasca and P. Levy, Phys. Rev. B 81, 115101 (2010).
\item \cite{2} N. Ghenzi, M. J. Sánchez, F. G. Marlasca, P. Levy and M. Rozenberg, J. Appl. Phys. 107, 093719 (2010).
\item \cite{4} N. Ghenzi, M. J. Sánchez and P. Levy, in preparation.
\end{itemize}

Resistive Switching Effect in Conductive Ferroelectric Oxides

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Resistive switching phenomena in metal oxides have been intensively studied in recent years, because of the potential for nonvolatile memory application, i.e. resistance random access memory (ReRAM). The main mechanism of the resistive switching phenomena is a nanionic redox reaction triggered by Joule heating or electrochemical migration of oxygen vacancies. Since chemical alterations of materials are
inevitably induced in both mechanisms, there is concern for the reliability, such as the data retention and endurance. As a solution of this problem, resistive switching based on an electronic mechanism is being considered. Ferroelectric resistive switching effects based on polarization reversal are practically attractive, because polarization reversal does not induce a chemical alteration. We have developed a ferroelectric resistive switching device consisting of a multiferroic BiFeO$_3$ (BFO). The devices showed hysteretic current-voltage (I-V) characteristics, i.e., a resistive switching phenomenon. We also succeeded in resistive switching by pulsed-voltage applications. The devices showed endurance of $>10^5$ cycles and data retention of $>10^5$ s. These results demonstrate promising prospects for application of the ferroelectric resistive switching effect at BFO interfaces to nonvolatile memory.

This work was supported in part by the Japan Society for the Promotion of Science (JSPS) through the “Funding Program for World-Leading Innovative R&D on Science and Technology (FIRST Program)”, initiated by the Council for Science and Technology Policy (CSTP), Grant-in-Aid for Scientific Research (No. 22360280), and Grant-in-Aid for Young Scientists (No. 24760557).


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**Phase control of bistable metal-insulator states in vanadium dioxide**

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Strongly correlated electron system often shows collective changes in electronic states by tiny external stimuli, being a candidate material for switching devices. A typical example is the metal-insulator transition in vanadium dioxide (VO$_2$), which exhibits a huge jump in resistivity from high temperature metallic to low temperature insulating phases at around room temperature. Dimerization of V ions to form spin singlet state opens the gap. Therefore, numbers of studies have been performed to realize switching devices including resistance memories operating at room temperature. The stability of this metal-insulator transition has been examined against electron doping and quantum confinement. The metal-insulator transition temperature in VO$_2$ thin films can be systematically reduced by electron doping with W$^{6+}$ ($V_{1-x}W_xO_2$), and eventually a metallic ground state is realized at around $x = 0.08$. [1] TiO$_2$/V$_{1-x}W_xO_2$ superlattices were fabricated and it was found out that the insulating phase of VO$_2$ is so robust that even 3 unit cells VO$_2$ sample shows metal-insulator transition. The robust lattice-structural transition arises from the strong electron-lattice interaction in highly correlated electron system. [2] These results indicate that the insulating phase is very robust but can be altered to metallic state by carrier injection.

The phase control of the bistable metal-insulator states was also investigated by external stimuli. I will show three examples: electric-field [3], x-ray [4], and hydrostatic pressure induced insulator-metal transitions. The consequences demonstrate a great deal of potential of VO$_2$ for switching application.

Comparative Study of Transport Models for Bipolar Switching in Memristors

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Two granular models of the bipolar resistance switching observed in TiO\textsubscript{2-x}/TiO\textsubscript{2} layered memristor systems are introduced. The first model contains only electron transport across the network of grains, the second also includes the movement of the ions. Both models associate a random energy and a self-charging energy term with each grain and also account for the long range Coulomb interaction. Both models capture the layered structure of the system: a highly conductive TiO\textsubscript{2-x} layer and a thin TiO\textsubscript{2} layer whose conductivity varies greatly with the ion concentration. The electrodes are explicitly modeled. The transport is controlled by a kinetic Monte Carlo Dynamics. We find that the electrons-only model does not reproduce key aspects of the experiments, including the lack of hysteretic current-voltage curves. In contrast, when ionic motions are also included (1) the hysteretic behavior of the current-voltage curves is recovered; (2) the two resistance states are preserved even at zero voltage crossing; and (3) the experimentally observed asymmetry between the positive and negative lobes is also observed. Our studies underline the importance of including the ionic dynamics into the full model of the resistive switching phenomena.
Growth and characterization of memristive manganite thin films

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Resistive switching effects were studied on manganite based devices at room temperature. Thin films of \( \text{La}_{0.66}\text{Ca}_{0.33}\text{MnO}_3 \) were grown using the pulsed laser deposition technique on top of conductive silicon substrates. Top contacts were deposited either by hand (Ag paint) or by sputtering (Cu). The Mn valence (which is correlated with the oxygen content) was probed by means of XPS experiments. Obtained devices were characterized electrically by means of pulsed I-V curves, and the remnant resistance state was measured after each voltage pulse. In all samples we observed bipolar resistive switching with sharp SET and RESET transitions. Typically, around 20 switching cycles could be repeated, with an OFF/ON ratio of 2-3 orders of magnitude. We found different SET and RESET voltages for Ag and Cu top electrodes. In addition, we found a correlation between the amplitude of the ON/OFF ratio and the oxygen content of the films, suggesting that oxygen vacancies play a key role in the switching mechanism. Finally, the electrical response of a Cu/LCMO sample was studied after a controlled damage process (irradiation with 25 MeV oxygen ions with a fluence of 3E10 ions).

Researches on Ferroelectrics in SFB 595 project as of 2011

Matias Acosta, Jürgen Rödel, Han-Joachim Kleebe/ Wolfgang Donnor, Heinz von Seggern, Andreas Klein, Hergen Breitzke, Gerd Buntkowsky, Karsten Albe, Yuri Genenko, Jürgen Rödel

Technische Universität Darmstadt, Darmstadt, Germany

The center of excellence in advanced functional ceramics at the “Technische Universität Darmstadt” develops and characterizes a wide spectrum of functional ceramics. In the core of the center lie eight working groups within the framework of SFB 595 project sponsored by the German Science Foundation and the state-funded Center for Adaptronics. Over the last decade, synergic collaborations among the groups encompassing relevant fields in the Materials Science, i.e., processing, electrical characterization, structural analyses, and theoretical modeling, has proven highly successful with noticeable outcomes. Representative achievements by the eight working groups on the mechanistic studies of the reliability of ferroelectric materials and the development of new lead-free materials are presented.

Electronic transport mechanisms in metal-manganite memristive interfaces

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We studied a \( \text{La}_{0.325}\text{Pr}_{0.300}\text{Ca}_{0.375}\text{MnO}_3\)-Ag memristive interface. We present a pulsing/measuring protocol capable of registering both quasi-static \( i-v \) data while pulsing and non-volatile remnant resistance after pulsing. This protocol allowed distinguishing two different electronic transport mechanisms coexisting in the memristive interface. These two mechanisms appear to be space charge limited current and thermionic emission limited current. We introduce a 2-element electric model that accounts for the obtained results and allows predicting the quasi-static \( i-v \) relation of the interface as a simple function of
the remnant resistance. Each of the elements of the electric model is associated to one of the electronic transport mechanisms found. This electric model may result useful for developing time-domain simulation models of metal-manganite memristive interfaces.

**Optimization of resistive switching performance of memristive metal manganite oxide interfaces by a multipulse protocol**

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Non volatile memory concepts for electronic applications are presently based on resistance change rather than in charge storage. Electric pulse induced resistance switching (RS) was shown to produce useful retention time capability for massive applications. Several basic and applied research teams are presently devoted to the study of transition metal oxides contacted through metal electrodes driven by appealing fast switching and scalability.

Evidence for an oxygen diffusion mechanism by means of electric transport measurements was recently obtained by means of a Hysteresis Switching Loop (HSL) procedure in which pulses of varying amplitude determine the state of the interface, and a small bias is used to test the remnant state. We performed HSL’s in manganite La 3.375Pr 0.325Ca 0.300MnO 3 at various conditions.

We study the characteristics of non common higher resistance states than the usual high and low resistance states accessible through bipolar pulsing. We found that these higher resistance states can be obtained by repeatedly pulsing with a single polarity. The accumulative action of successive pulsing drives the resistance towards saturation, the time constant being a strong function of the pulsing amplitude. These results can be explained by oxygen vacancy detrapping in the interface oxide-metal. In this work we analyze the different vacancies profiles obtained through numerical simulations which give insight in the resistive switching dynamics.

**Resistive Switching in multilevel TiO 2 memory devices**

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Non volatile memory concepts for electronic applications are presently based on resistance change rather than in charge storage. Electric pulse induced resistance switching (RS) was shown to produce useful retention time capability for massive applications. Several basic and applied research teams are presently devoted to the study of transition metal oxides contacted through metal electrodes driven by appealing fast switching and scalability. In the present work, we study Au / TiO 2 / metal (Cu and Al) junctions in a crossbar pattern which exhibit resistive switching characteristics (*). We study its physical-chemical mechanisms and its response in aggressive environments. We perform its electrical characterization,
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both, IV (dynamic measurement) and HSL (remnant measurement) curves. Besides we compare the evolution of the RS response with / without protons and oxygen ions irradiation.

In devices with Cu top electrode we obtain evidence of a copper metallic filament for negative electroforming, while for positive electroforming the filament is presumably TiO$_2$ - Magnelli phase based. From the comparison of both states we discuss the role of the defects involved in the oxide matrix, which are responsible for the multilevel states observed.

In devices with Al top electrode, after the negative electroforming process we observe the presence of micrometer sized “craters”. We study the effect of changing the current compliance $I_{cc}$ to control the area where these craters appear. The resistance of the two resistive states decreases when $I_{cc}$ is increased.

Based on these trends, we suggest that the change in the affected area (due to the different $I_{cc}$’s) is related to the number of formed filaments at the oxide matrix.

Additionally we reproduce the experimental results through a realistic model that includes as a main ingredient the oxygen vacancies diffusion under applied electric field. The associated vacancy profiles further unveil the prominent role of the effective electric field acting at the interfaces. These simulations allow to disentangle the microscopic mechanisms behind the resistive switching in metal-transition metal oxide interfaces.

(*) Samples were grown by sputtering TiO$_2$ and using standard optical lithography. We acknowledge the MEMS group at CAC-CNEA-Argentina and the Microelectronic Group at INTI- Argentina for assistance and fruitful discussions during fabrication.

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Characterization of charge transport in vanadium oxide $V_2O_5$ thin films obtained by RF sputtering deposition and post-annealing.

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$V_2O_5$ vanadium oxide thin films were prepared via RF Magnetron Sputtering deposited at room temperature and then post-annealing on air at different temperatures and characterized via SEM-AFM-XRD-FTIR. Then was tested resistivity and fitted with the charge carrier hopping transport process. The temperature coefficient of resistance obtained was TCR =-3.61%/ºK with sheet resistivity of 104:5MΩ cm$^2$ @ 25ºC.

Micro- and nanoprotyping facilities at INTI

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Sensor technologies and Micro-Electro-Mechanical Systems manufacturing require a wide set of materials and methods, due to the wide range of possible applications: from physical to optical, chemical and biological. Packaging and testing play also a significant role, since they consume the majority of the total fabrication cost.

INTI-CMNB offers several facilities involved with every step in the manufacturing chain: from device nanoprotyping to wafer level testing. We present here an overview of these facilities and example applications.
**Growth and electrical characterization of bismuth ferrite thin films**

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Thin films of the room temperature multiferroic BiFeO\(_3\) were grown by pulsed laser deposition on top of n-type silicon. The growth was performed at 650ºC under an O\(_2\) pressure of 0.12mbar. The thickness (~100nm) was estimated from scanning electron microscopy cross-view. A moderate amount of particulate was found on the surface. X-ray diffraction suggests a BiFeO\(_3\) textured growth, along with the presence of segregated Fe\(_3\)O\(_4\) magnetite phase. Cu top electrodes were deposited by sputtering at room temperature and shaped by standard optical lithography. Resistive switching effects were studied at room temperature. Obtained devices were characterized by means of pulsed I-V curves, and the remnant resistance state was measured after each voltage pulse (Hysteresis Switching Loops, or HSL). We observed bipolar resistive switching, with an OFF/ON ratio of up to 40, depending on the compliance current applied during the SET process. SET and RESET voltages were found at 5V and -5V. We observed squared HSLs, suggesting that only one interface is active (presumably BFO/Si), while the other one remains ohmic.

**Electric voltage effects on silver-mesoporous titania nanocomposite thin film arrays during conductive tip AFM scannings**

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In this work we present the results obtained with conductive-tip atomic force microscopy (CT-AFM) in silver nanoparticles (AgNP) patterns infiltrated in TiO\(_2\) mesoporous thin films (MTF). The TiO\(_2\) MTF infiltrated with AgNP arrays were fabricated by the lithography-assisted photocatalysis method [1]. In these nanostructures, the properties related with the size and the confinement of the Ag-NP and the TiO\(_2\)-Ag interfaces can be explored while the NP remains protected from the environment. CT-AFM results revealed a conduction contrast of more than two orders of magnitude between the current measured inside and outside the infiltrated regions. The dependence of the electric properties with the thickness, the MTF structure and the AgNP loading confirmed that the electric conduction develops tridimensionally within the film [2]. Here it will be displayed that it is possible to modify locally the electric resistance and the topography depending on the applied voltage during the CT-AFM scanning.

Electronic transport in ferromagnetic multilayers deposited by pulsed laser ablation

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Ferromagnetic multilayers with different configurations were deposited by pulsed laser ablation. Spin valves (10nmCoFe\textsubscript{2}/3nmCu/10nmCoFe\textsubscript{2}/20nmCoFe\textsubscript{2}O\textsubscript{4}/Si) and magnetic tunneling junctions (10nmNiFe/5nmZnO/10nmCo/Si) were fabricated and characterized at room temperature without external magnetic field applied.

The spin valve showed two different bias regimes, with a marked decrease in resistance for applied voltages greater than 0.2 V. We suggest that this could be attributed to the creation of conduction paths with increasing voltage, due to a partially oxidized copper layer during deposition.

Resistive switching and hysteresis in the I-V curve were observed in the magnetic tunneling junction. We propose that the switching effect could be explained by a change in the relative magnetization direction between ferromagnetic layers due to the circulation of spin polarized current (spin-transfer torque). This effect is typically observed in spintronic memristors.

Experiments on noise assisted resistive switching

A. A. García [1], G. A. Patterson [2], P. I. Fierens [2,3], and D. F. Grosz [2,3]


Recently, Stotland and Di Ventra [1] presented simulations, based on a model of resistive switching by Strukov et al. [2], which take into account the interaction of internal (inherent to the sample) noise with the nonlinearity of the device. They showed that there is an optimal noise intensity for which the contrast between high and low resistance values is maximized. They also suggested that externally applied noise should have a similar effect.

In this work, we present experimental results on the effect of external noise on a sample of La\textsubscript{0.325}Pr\textsubscript{0.3}Ca\textsubscript{0.375}MnO\textsubscript{3}. We first studied the behavior of the sample resistance under the application of short (~ 1 ms) noiseless current pulses and found that only large amplitude pulses (>~ 500 mA) produced a significant resistance change. We then added white Gaussian noise to the current pulses. In this case we found that noisy pulses with a small mean current amplitude induced similar resistance changes as those observed for much larger noiseless pulses.

Simulating Resistive Switching in Manganite Thin films

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We study the resistive switching behaviour in capacitor like structure devices with a transition metal oxide sandwiched between two metallic electrodes using a phenomenological model where the oxygen vacancies migration is enhanced by the local electric field. We assume that the local resistivity has a linear dependence with the vacancy concentration, and that the metal oxide interfaces are the regions were a high resistance develop. The model consists of a single conductive channel within an insulating dielectric, which is represented by a one dimensional resistive network of N links. Upon applying an external voltage, the local voltage drops at the network domains producing a vacancy drift which determines the total resistance of the device.

We explore the parameter space of the model with the aim of optimizing the ratio between High and Low resistance values. We varied the total amount of vacancies, the width of the interface/bulk region and the magnitude of the external electric field in order to simulate the switching response of a manganite thin film. We obtain two quantitatively different regimes, which are related to the amount of vacancies that could reach the bulk region. We discuss these regimes, and compare obtained results with experimental data obtained on LaCaMnO thin films.


Electrical Characterization of MEM’s at the MicroLAB – UNSAM ECyT

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Nowadays, the most advanced storage devices available known are flash memories. The Metal-Insulator-Metal (Co-HfO2-Ti and Al-TiO2-Au) structures we’re working with are based on the Resistive Switching (RS) concept idea. They’re faster, and, hypothetically, more tolerant to E-M radiations than flash memories. Here we explore this new technology for memory devices, by sourcing interleaved DC pulses, both positive as well as negative. Here we present a promising method based on a technology which may be relevant to the field of spatial electronics, particularly telecommunication satellites, being of extreme importance, for instance, to the satellite’s resetting cycle, due to its hypothetically high tolerance to E-M radiations. We manipulate these memories with three kinds of pulses. The basic experience begins with a one-time procedure that involves sourcing a series of Set/Reset pulses to a pristine device, this process being known as Forming process. Then, for these memories we determine, as stated above, three different procedures. A Reset Procedure, a Set Procedure and a Reading Procedure. These are conceived as follows: the Reset procedure, changes the memory state into high resistance; the Set procedure, sets the memory into a low resistance state; and the Reading procedure, which bias the DUT in order to get the resistance value. The pulses are as small as necessary to avoid that the resistive state is changed.
Fatigue effects by accumulative pulse application on bipolar RRAM devices

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We have studied the effects of accumulating electrical pulses of the same polarity on the resistance state of interfaces made by sputtering a metal (Au, Pt) on top of the surface of a cuprate superconductor YBa$_2$Cu$_3$O$_{7-d}$ (YBCO). We show that the evolution of resistance for the high and the low resistance state depends logarithmically on the number of accumulated pulses. We have established a criterion to determine the failure of a device based on the saturation of the relative resistance variation between the high and the low resistance states upon applying pulses of increasing voltage. This criterion is also useful to compare different devices and to determine their optimal working conditions. Our results show the similarity between the physics of the diffusion of oxygen vacancies induced by electrical pulses and the propagation of defects in materials subjected to repeated mechanical stress.

Resistive switching effects in HfO$_2$ based memory devices

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We study capacitor-like structures with HfO$_2$ as the active insulating material, for the implementation of non-volatile memory devices. Arrays of Co/HfO$_2$/Ti devices were fabricated with atomic-layer-deposited HfO$_2$ (20 nm thick) and sputtering of the top (Co) and bottom (Ti) electrodes. Devices were characterized electrically by means of standard and pulsed I-V curves at room temperature. The remnant resistance state was also tested when pulsing.

An hysteretical electric response was observed upon cycling the applied voltage up to +/- 15 V. Obtained I-V curves are “non crossing” type, i.e. two sharp SET transitions (from High Resistance to Low Resistance) are observed at around +/- 5 V. For each polarity, the steep 4-5 order of magnitude switch to a Low Resistance state turns into a High Resistance state upon polarity reversal, suggesting the presence of rectifying junctions. Around a hundred switching cycles could be repeated without device degradation. Besides, the conducting state has a fairly high resistance (around $1 \times 10^{-6}$ A for 10 volts), a prerequisite for low power consumption.

Samples were irradiated with 20 MeV oxygen ions up to a dose of $3 \times 10^{12}$ ions. Their electrical response after this controlled damage process is similar to the one obtained at the virgin state. Other radiation hardness tests are in progress.

We discuss a model based on the oxygen vacancy drift to account for the observed experimental results.
Clean Room facilities at CAC- CNEA


El CAC cuenta con una Sala Limpia de área total: 160 m²; 30 m² clase 1.000; 60 m² clase 10.000, más 70m² de área de servicio.

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