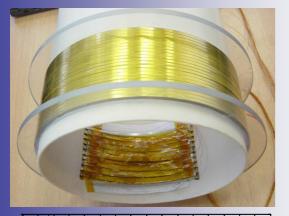
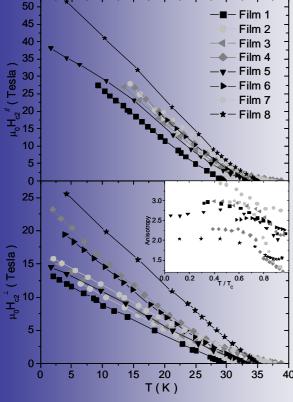


## Superconductivity & technological transfer

Superconducting fault current limiter fabricated at LAMIA for CESI SpA

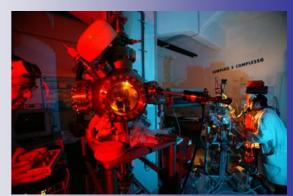




Superconducting resonator fabricated at LAMIA for ESAOTE SpA



Thin films of superconducting materials - MgB<sub>2</sub> with extremely high upper critical fields

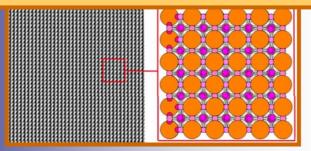


Spin-off company for MgB<sub>2</sub>:
Columbus Superconductors srl:
Km length MgB<sub>2</sub> superconducting tapes and wires

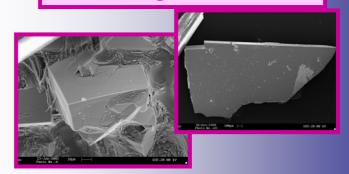


# Materials processing

Synthesis of superconducting oxides Structural refinement and HRTEM analysis



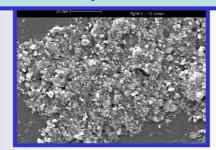
Single crystal growth of superconducting and magnetic oxides

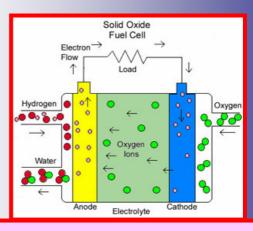


HPHT Combustion synthesis of nitrides



Mechanical alloying of metallic and ceramic nanopowders

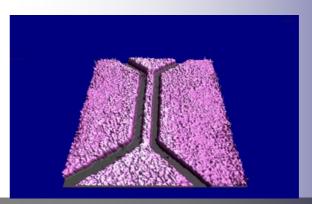




Synthesis of intermetallic alloys for hydrogen storage and ceramics for fuel cells

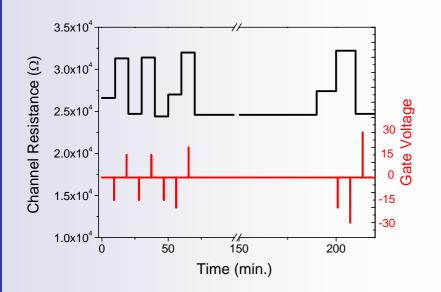
### Oxide Electronics: new materials and nanodevices

Field Effect Nanotransistors on Functional Oxides

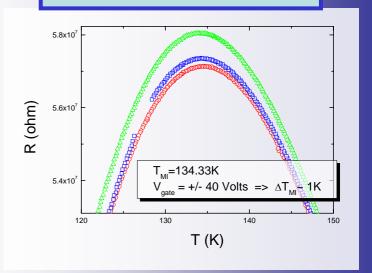


Channel dim: 0.7  $\mu$ m × 8  $\mu$ m

#### Ferroelectric Memories



## Colossal Magnetoresistance based devices



## Outline

Transition metal oxides

· Field effect geometries

Wurzite

Traditional Stacked
Back Gate
Side Gate

Ferroelectric Local (A

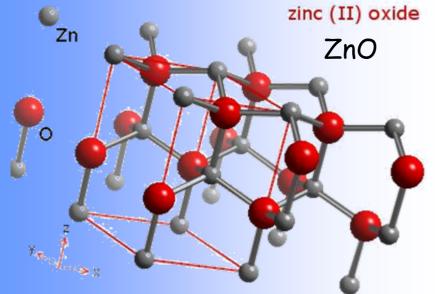
• Experiments Manganites

Superconductor

Oxide Semiconductor

#### Transition Metal oxides

#### Wurzite

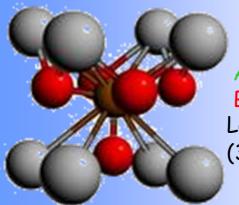


#### Hexagonal

Lattice parameters a=3.24 Å, c=5.19 Å

- Transparent semiconductor
- High mobility
- · Wide-band gap
- Doped with magnetic ions (Co, Mn...): dilute magnetic semiconductors

#### Perovskite



 $ABO_3$ 

A: Alkaline Earth B: Transition Metal Lattice Parameter  $(3.9 \pm 0.1)$ Å

#### (Pseudo)cubic

Many physical properties depending on cations

- · High Tc superconductivity
- Ferroelectricity
- Ferromagnetism, Colossal

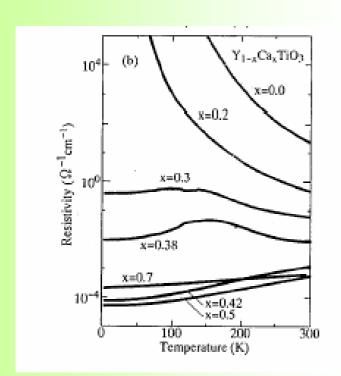
Magnetoresistance

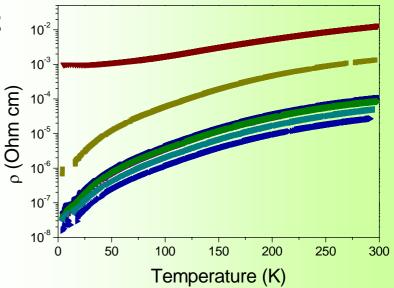
Semiconductors, Insulators

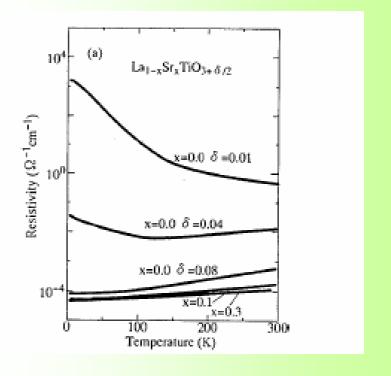
Dielectrics and semiconductors:

SrTiO<sub>3</sub>, LaVO<sub>3</sub>, ZnO

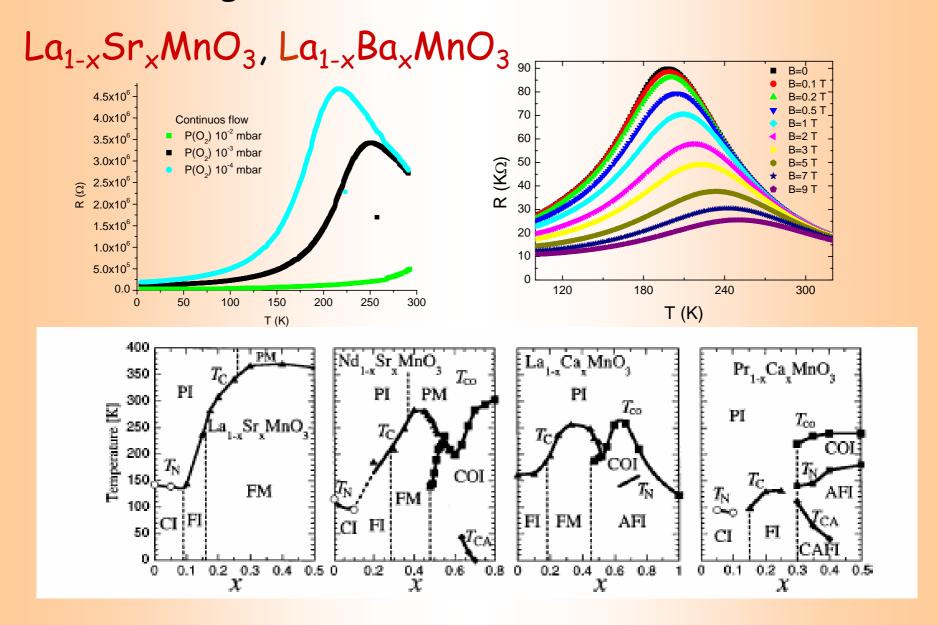
M-I transition in titanates





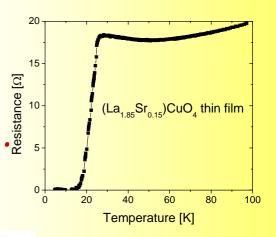


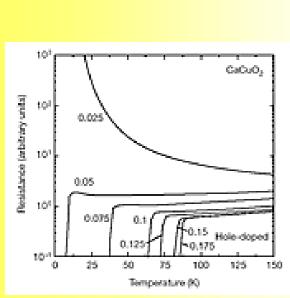
## Colossal magnetoresistance:

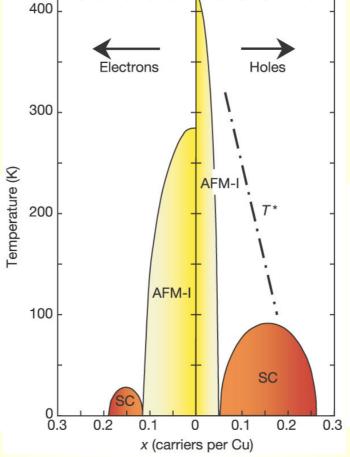


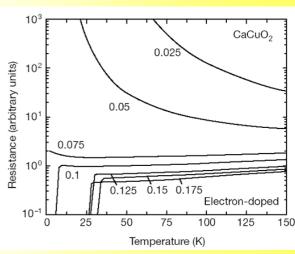
## Superconductivity:

YBCO, LaSCO, infinite layers....









#### Properties are controlled by:

n Band filling factor  $\rightarrow$  proportional to the charge

W Band width  $\rightarrow$  proportional to d and p orbital overlapping

How to change n and W?

BANDWIDTH CONTROL

Cell deformation



Orbital overlapping

Isovalent atomic substitution (different radius)

Pressure

FILLING CONTROL

Fermi level variation



Eterovalent atomic substitution
Field effect

Problem: the phase diagram depends not only on the carrier concentration but also on the lattice structure; chemical doping affects both carrier concentration and lattice structure and it is very difficult to discriminate between these two contributions. Field effect tunes the carrier concentration only, thereby it is a powerful tool to study this correlated system

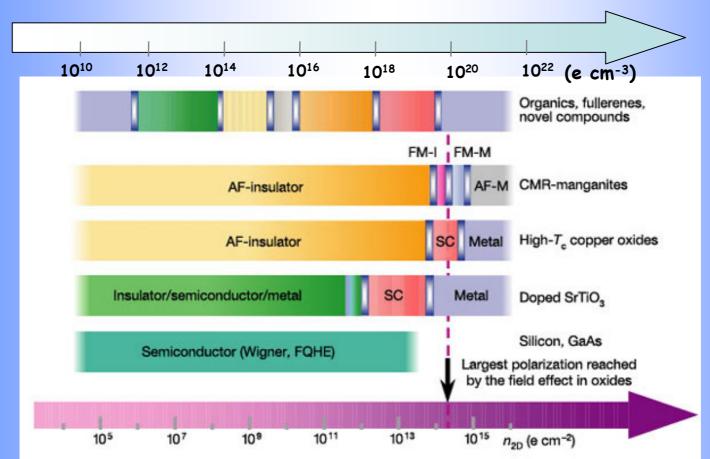


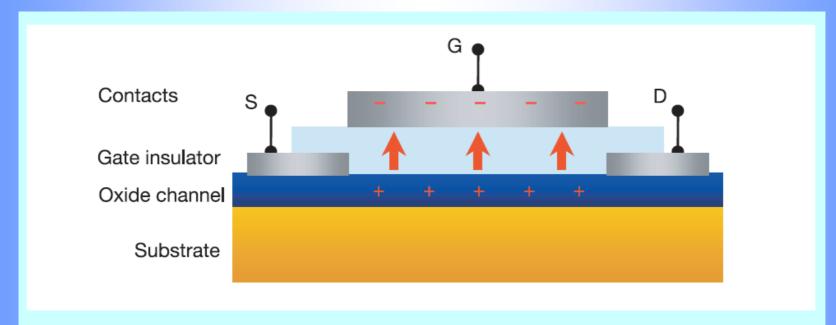
Illustration of the zero-temperature behaviour of various correlated materials as a function of charge density. Silicon is shown as a reference. The examples for high- $T_c$  superconductors and for colossal magnetoresistive (CMR) manganites reflect  $YBa_2Cu_3O_{7-}$  and  $(La,Sr)MnO_3$ , respectively.

AF, antiferromagnetic; FM, ferromagnetic; I, insulator; M, metal; SC, superconductor; FQHE, fractional quantum Hall effect; Wigner, Wigner crystal.

#### Electric field effect in correlated oxide systems

C. H. Ahn, J.-M. Triscone and J. Mannhart Nature 424, 1015-1018 (28 August 2003) doi: 10.1038/nature01878 Many of interesting physical properties in these material occur at  $10^{19}$ - $10^{21}$  e-/cm<sup>3</sup> (for 100 nm film =>  $10^{14}$  –  $10^{16}$  e/cm<sup>2</sup> => 10 – 1000  $\mu$ C/cm<sup>2</sup>) Very high q value!

#### High polarization Ultrathin films



**Figure 2** Cross-section of a typical sample geometry used for field-effect studies. S, source; G, gate; D, drain.

#### The characteristic width of accumulation or depletion layer is given by the electrostatic screening length

Thomas-Fermi for metal

$$k_{TF} = \sqrt{\frac{3ne^2}{\varepsilon_0 \varepsilon_r E_F}}$$
  $\approx 1 \text{ A}$ 

$$\lambda_{TF} = \frac{2\pi}{k_{TF}} = \sqrt{\frac{4\pi^2 \varepsilon_0 \varepsilon_r E_F}{3ne^2}}$$

Debye for semiconductors  $\lambda_D = \sqrt{\frac{kT\varepsilon_0\varepsilon_S}{e^2n}}$ 

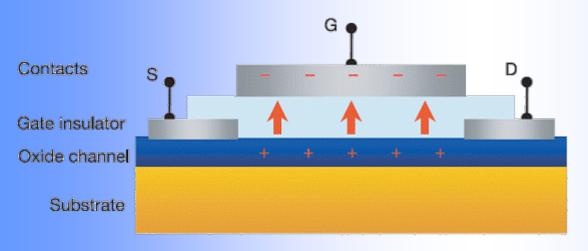
$$\lambda_D = \sqrt{\frac{kT\varepsilon_0\varepsilon_S}{e^2n_{ext}}}$$

≈10 nm

Lower carrier density  $\rightarrow$  larger  $\lambda$ 

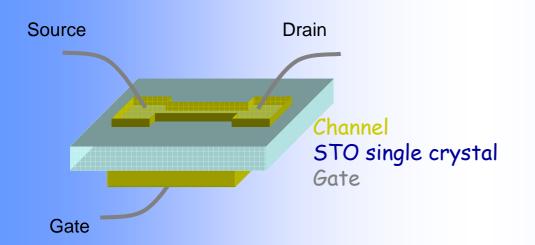
Debye length at room temperature			
$N(e/cm^3)$	$\varepsilon_{\rm r} = 300$	$\varepsilon_{\rm r} = 100$	$\varepsilon_{\rm r} = 10$
$10^{20} (e/\text{cm}^3)$	2.0 nm	1.2 nm	0.38 nm
$10^{19} (e/\text{cm}^3)$	6.5 nm	3.8 nm	1.2 nm
$10^{18} (e/\text{cm}^3)$	20 nm	12 nm	3.8 nm

## Field effect devices: Stacked Geometry



## MIS heterostructure Metal-Insulator-Semiconductor

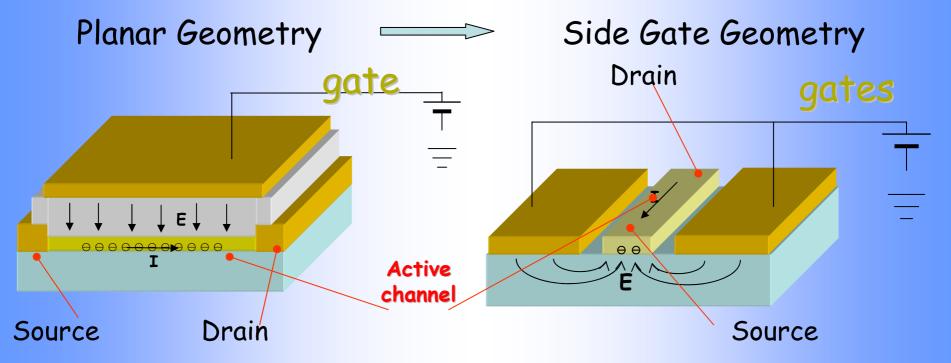
- Growth of the dielectric layer on the channel
- Compatibility problems
- Leakage



#### Back gate geometry

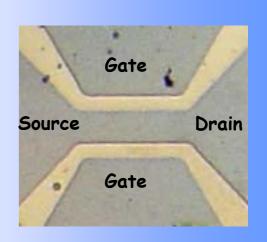
- Very good dielectric properties of the oxide (single x-tal)
  - Very thick→ High voltage

## Side gate devices



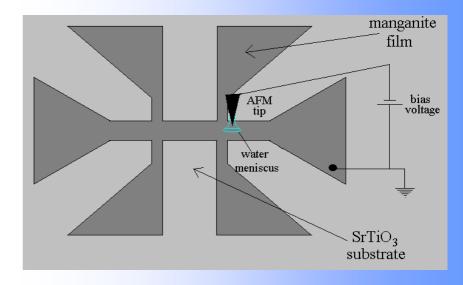
Advantages of the side gate geometry

- Easy: 2-layers structure (film/substrate)
- High quality substrates → Best Dielectric Properties
- Low voltage required
- Possibility to study the surface by Scanning Probe
   Microscopy

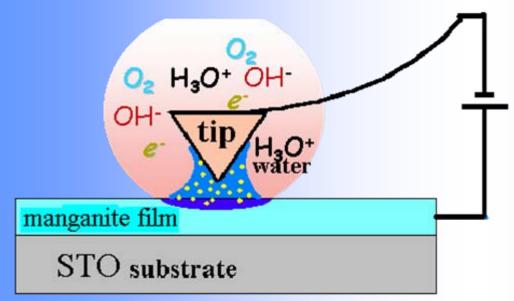


#### Side gate field effect devices fabrication by AFM

First step: patterning by optical lithography and wet etching in 10-20  $\mu$ m wide crossing channels with bond pads

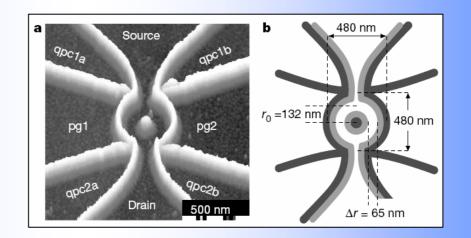


Conducting silicon
W<sub>2</sub>C coated tip
0.12N/m



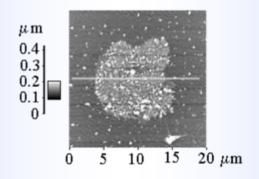
Second step: sub-micron patterning by Atomic Force Microscope anodization: the AFM biased tip triggers a local chemical and morphological transformation.

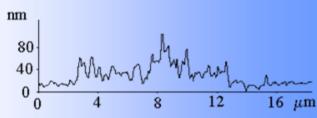
Nanoxidation of silicon J.A.Dagata,et al., Appl. Phys. Lett. **56**, 2001 (1990)  Nanoscale GaAs/AlGaAs heterostructures



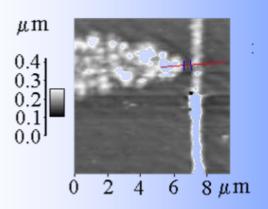
#### Local "anodic oxidation" of oxides:

The modified regions are swollen, porous and electrically insulating

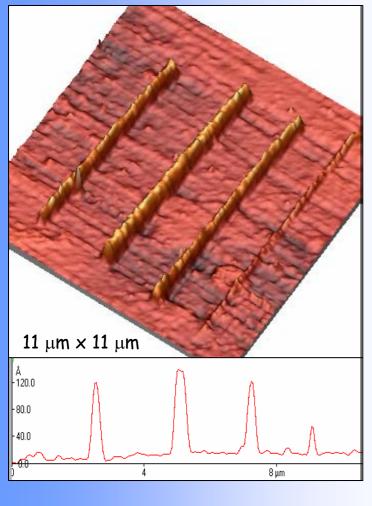




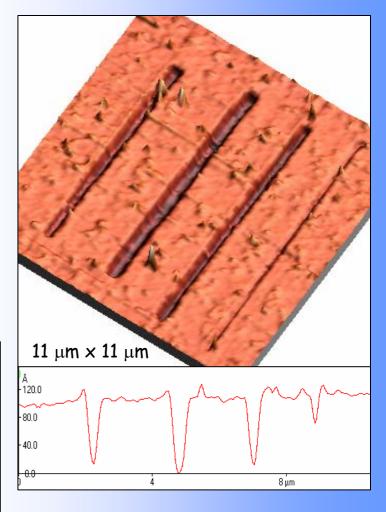
Fabrication of constrictions as narrow as 40 nm.



#### Modified regions are selectively etched in HCl solution

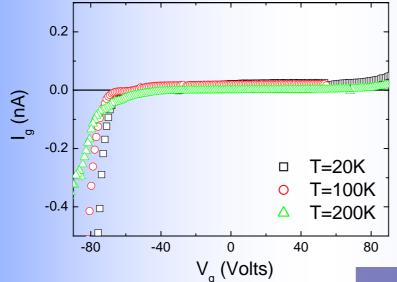


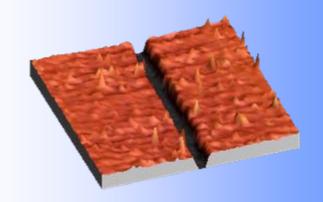




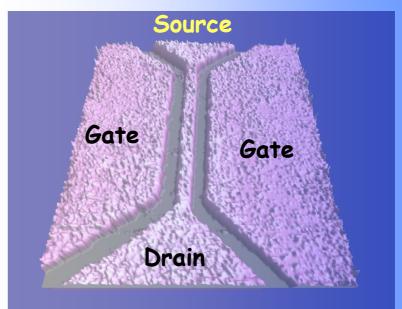
Voltage controlled etching depth !!!

Etched sub-micron wide insulating barriers can sustain applied voltages up to  $\pm 80$  Volts without appreciable leakage (<100pA).





Side gate field effect devices by AFM

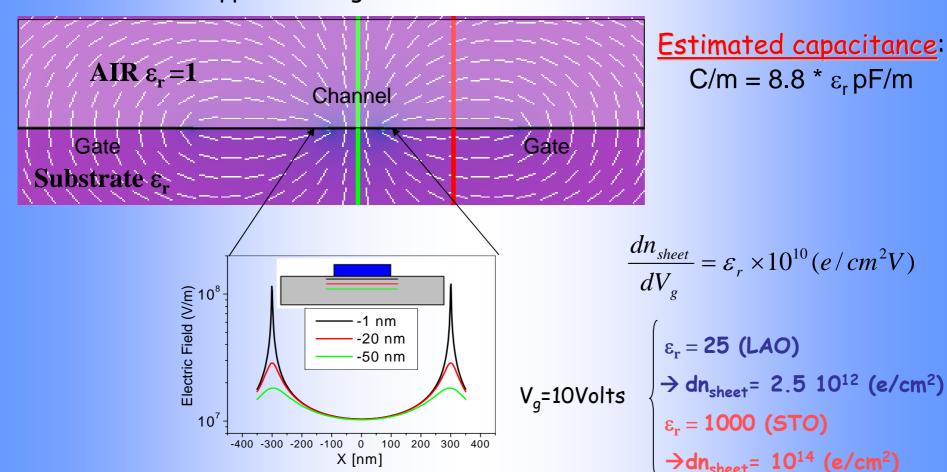


### Electric field profile and capacitance calculation

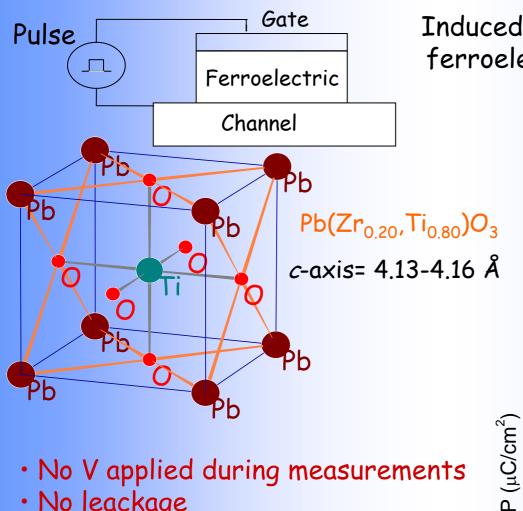
Channel width 600 nm

Gap 1200 nm, Thickness 10 nm

Applied Voltage V = 10V



## Ferroelectric Field effect



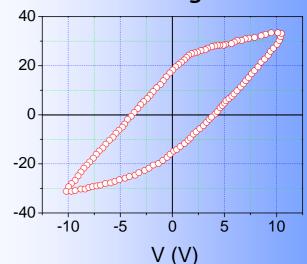
Induced charge proportional to the ferroelectric remnant polarization

> Remnant polarization  $P_{r} \sim 10 \div 60 \, \mu \text{C/cm}^{2}$

Coercive field  $E_c \sim 100 \text{ kV/cm}$ 

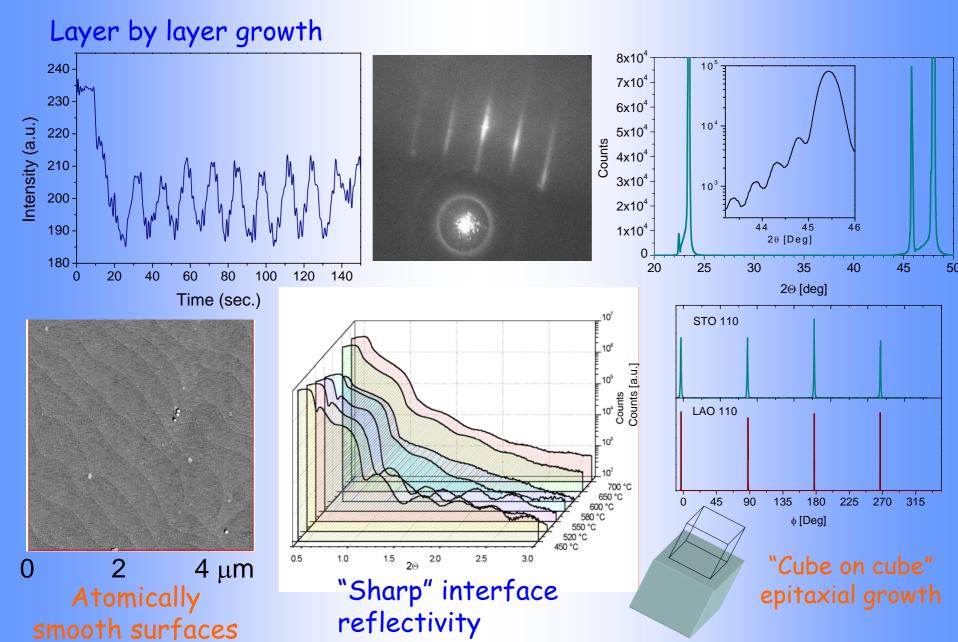
#### Ferroelectric properties:

 $P_r \sim 20 \,\mu\text{C/cm}^2$  $\sigma \sim 10^{14} \text{charges/cm}^2$ 



- No V applied during measurements
- · No leackage
- Only 2 states available

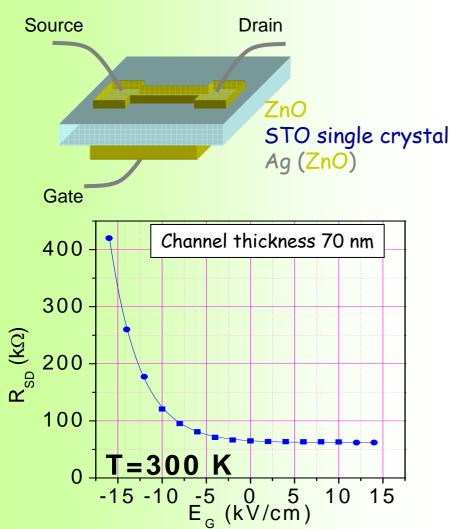
## Perovskite films growth and structural properties

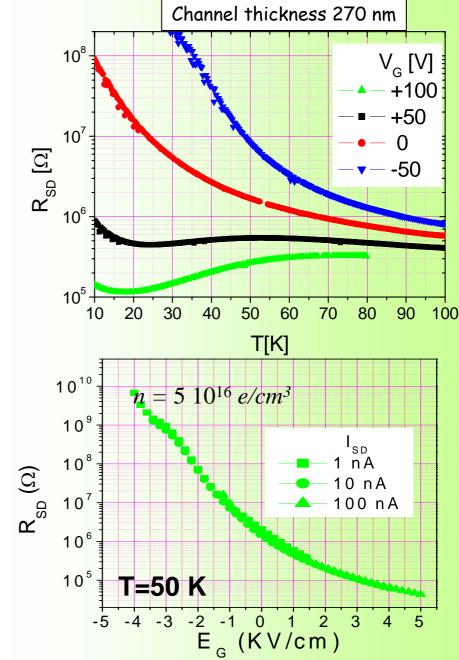


## Oxides Semiconductors

## Easy case : ZnO Back-gate devices

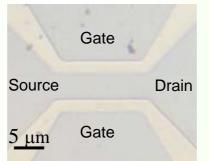
Double side polished SrTiO<sub>3</sub> 110 substrate



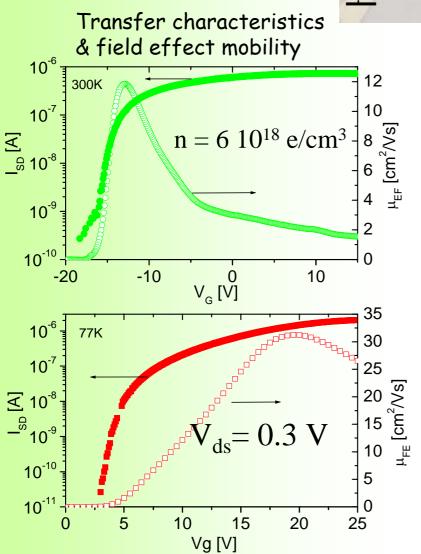


More then 5 order of magnitude R<sub>SD</sub> modulation at 50K

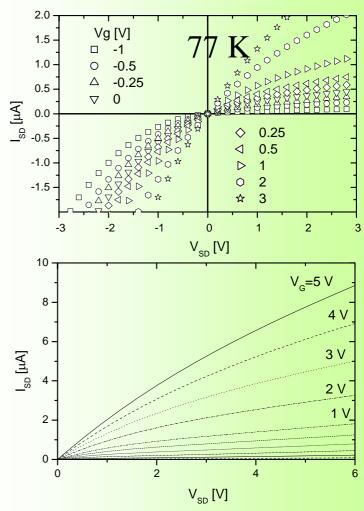
## ZnO Side gate devices



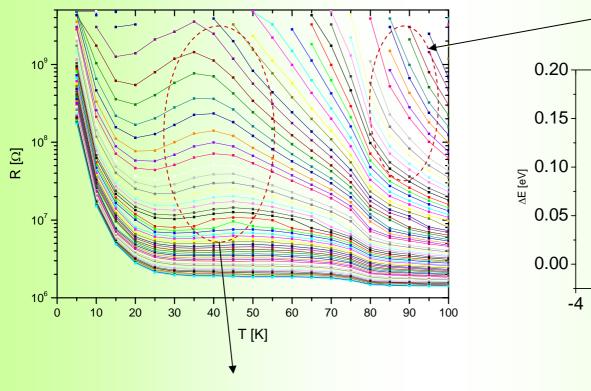
## 20 nm thick ZnO film by two-step method



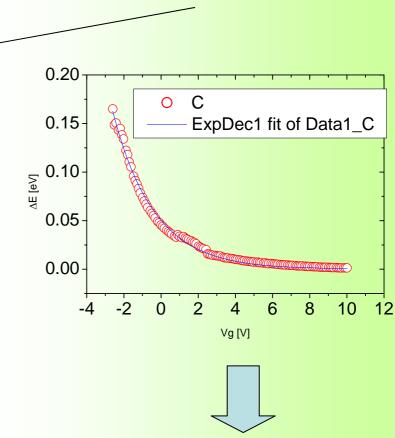
#### Characteristic curves



#### Thermal activated behaviour



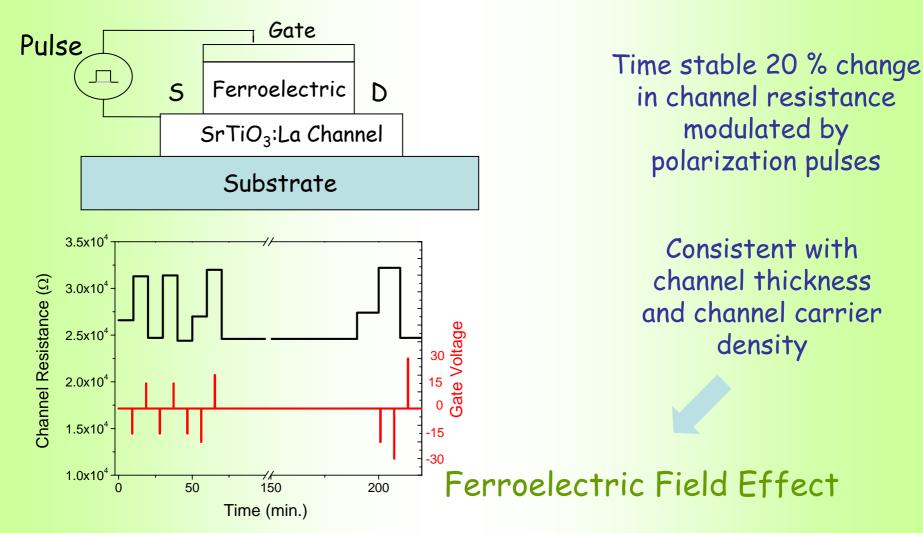
MIT driven  $\varepsilon_r$  of the STO substrate Competition between T and V dependences of  $\varepsilon_r$ 



Estimation of the density of state (impurity band)

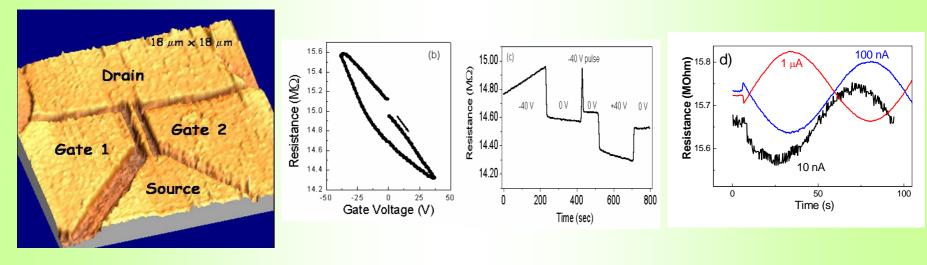
 $D(E) \propto 1/(E-Ec)$ 

## Ferroelectric field effect on SrTiO<sub>3</sub> channel



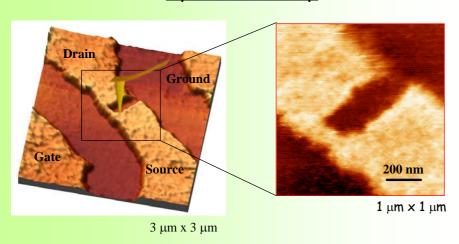
Strontium titanate resistance modulation by ferroelectric field effect
D. Marré, A. Tumino, E. Bellingeri, I. Pallecchi, L. Pellegrino, A.S. Siri,
J. Phys. D: Appl. Phys. 36 No 7 896-900 (2003)

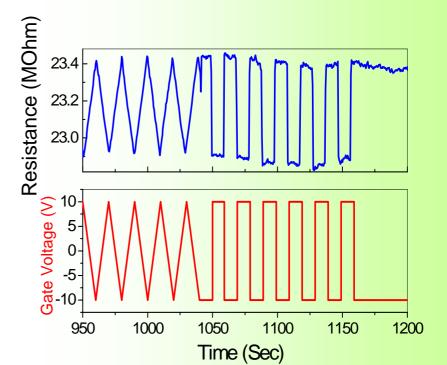
### First example of side gate devices (SrTiO<sub>3</sub> on LaAlO<sub>3</sub>)



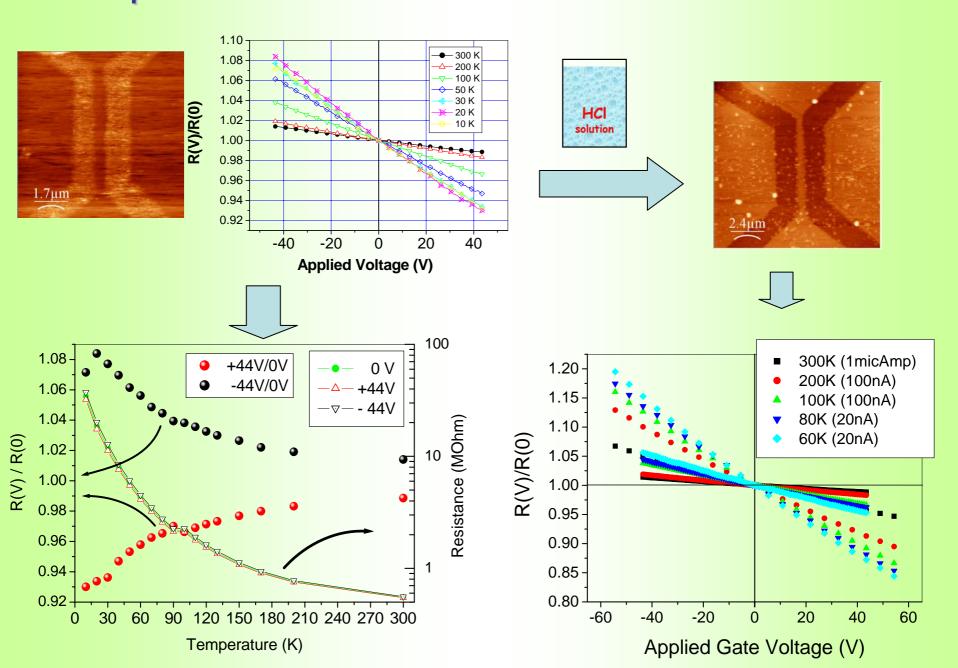
Fabrication of submicron-scale SrTiO<sub>3</sub> devices by an atomic force microscope L. Pellegrino, at al Appl. Phys. Lett. 81, 3849 (2002)

## Successive Modification of the channel by the AFM tip

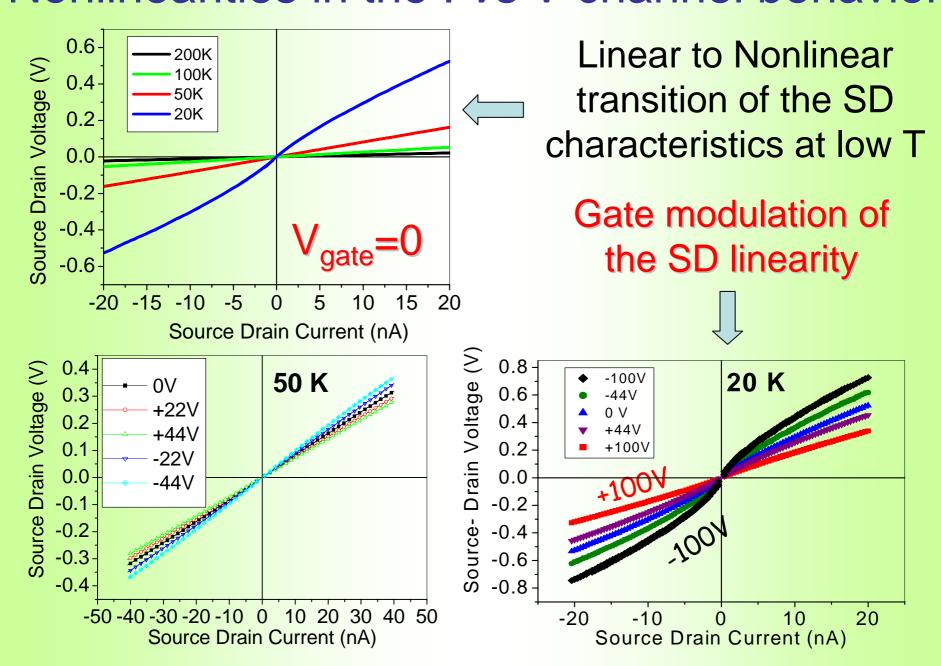




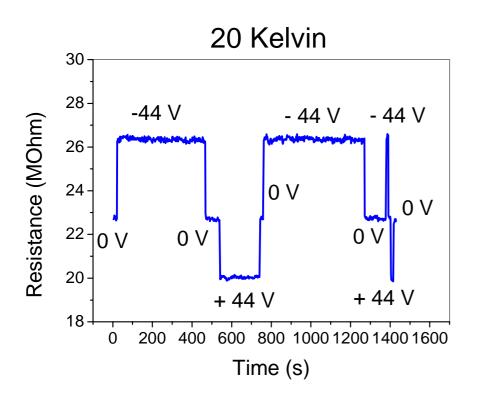
## Temperature characterization of the side FET

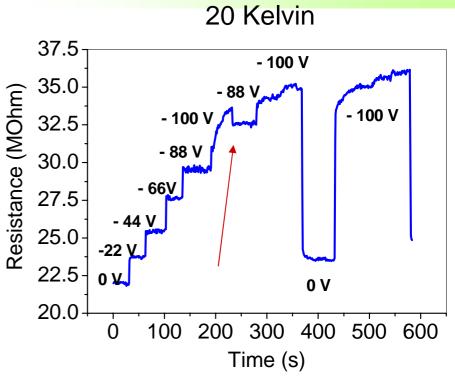


## Nonlinearities in the I vs V channel behavior



## **Observation of E-Field Induced Drift**





Low Gate field



No hysteresis

High Gate field



Memory effects

### ... Exploiting the Dielectric Constant of the Substrate

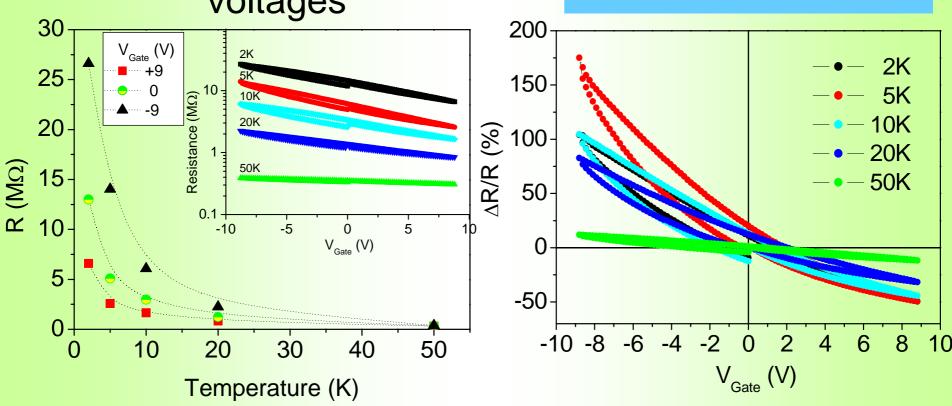
## Maximum resistance modulation observed on homoepitaxial STO devices

STO:La on STO

**STO** substrate

Homoepitaxial thin films have better conductivity

 Higher effect at lower gate voltages

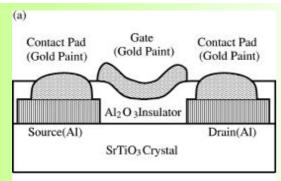


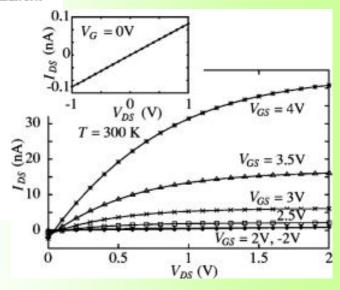
#### Field-effect transistor on SrTiO<sub>3</sub> with sputtered Al<sub>2</sub>O<sub>3</sub> gate insulator

K. Ueno, <sup>a)</sup> I. H. Inoue, H. Akoh, M. Kawasaki, <sup>b)</sup> Y. Tokura, <sup>c)</sup> and H. Takagi<sup>d)</sup>
Correlated Electron Research Center (CERC), National Institute of Advanced Industrial Science and Technology (AIST), Tsukuba 305-8562, Japan

(Received 14 March 2003; accepted 1 July 2003)

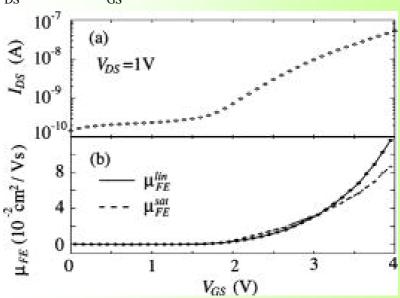
A field-effect transistor has been constructed that employs a perovskite-type  $SrTiO_3$  single crystal as the semiconducting channel. This device functions as an n-type accumulation-mode device. The device was fabricated at room temperature by sputter-deposition of amorphous  $Al_2O_3$  films as a gate insulator on the  $SrTiO_3$  substrate. The field-effect (FE) mobility is  $0.1~cm^2/V$  s and on-off ratio exceeds 100 at room temperature. The temperature dependence of the FE mobility down to 2~K shows a thermal-activation-type behavior with an activation energy of 0.6~eV. © 2003~American~Institute~of~Physics. [DOI: 10.1063/1.1605806]





Drain-source current  $I_{\rm DS}$  plotted against the drain-source bias  $V_{\rm DS}$  of the  ${\rm Al_2O_3/SrTiO_3}$  FET at 300 K. A channel length and a width of the FET device were 25 and 300  $\mu$ m, respectively. The inset shows the blow-up of the  $I_{\rm DS}-V_{\rm DS}$  curve for  $V_{\rm GS}=0$  V

. (a) The gate-source bias  $V_{\rm GS}$  dependence of the drain-source current  $I_{\rm DS}$  for a fixed drain-source bias  $V_{\rm DS} = +1$  V of the same device used for Fig. 2. The on-off ratio between  $V_{\rm GS}$  of 0 and 4 V for  $V_{\rm DS}$  of 1 V exceeds 100. (b)  $V_{\rm GS}$  dependence of the field effect mobility  $\mu_{\rm FE}$ .  $\mu$  and  $\mu$  were deduced from Fig. 3(a) by using Eqs. (1) and (2), respectively. Both increase monotonically with  $V_{\rm GS}$  and no saturation was observed even for large gate bias



APPLIED PHYSICS LETTERS VOLUME 84, NUMBER 19 10 MAY 2004

#### Field-effect transistor based on KTaO<sub>3</sub> perovskite

K. Ueno, a) I. H. Inoue, T. Yamada, H. Akoh, Y. Tokura, b) and H. Takagi<sup>c)</sup>
Correlated Electron Research Center (CERC), National Institute of Advanced Industrial Science and Technology (AIST), Tsukuba 305-8562, Japan

(Received 16 December 2003; accepted 23 February 2004; published online 29 April 2004)

An *n*-channel accumulation-type field-effect transistor (FET) has been fabricated utilizing a KTaO<sub>3</sub> single crystal as an active element and a sputtered amorphous Al<sub>2</sub>O<sub>3</sub> film as a gate insulator. The device demonstrated an ON/OFF ratio of 10<sup>4</sup> and a field-effect mobility of 0.4 cm<sup>2</sup>/V s at room temperature, both of which are much better than those of the SrTiO<sub>3</sub> FETs reported previously. The field-effect mobility was almost temperature independent down to 200 K. Our results indicate that the Al<sub>2</sub>O<sub>3</sub>/KTaO<sub>3</sub> interface is worthy of further investigations as an alternative system of future oxide electronics. © 2004 American Institute of Physics. [DOI: 10.1063/1.1703841]

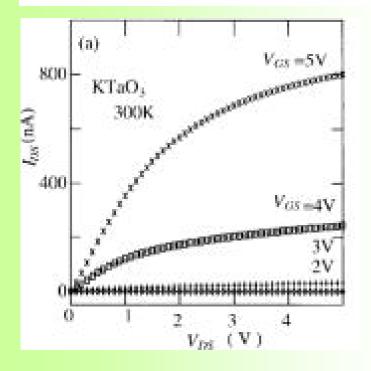


Fig. 1. (a) Drain-source current  $I_{\rm DS}$  plotted against the drain-source bias  $V_{\rm DS}$  of the  ${\rm Al_2O_3/KTaO_3}$  FET for various gate voltages  $V_{\rm GS}$  at 300 K. The KTaO<sub>3</sub> single crystal was annealed at 700 °C prior to the device fabrication.

## Manganites

## Ferroelectric-field-induced tuning of magnetism in the colossal magnetoresistive oxide $La_{1-x}Sr_xMnO_3$

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A ferroelectric field effect approach is presented for modulating magnetism in the colossal magnetoresistive oxide  $La_{1-x}Sr_xMnO_3$  (LSMO). The ferromagnetic Curie temperature of ultrathin LSMO films was shifted by 35 K reversibly using the polarization field of the ferroelectric oxide  $Pb(Zr_xTi_{1-x})O_3$  in a field effect structure. This shift was also observed in magnetoresistance measurements, with the maximum magnetoresistance ratio at 6 T increasing from 64% to 77%. This model system approach does not introduce substitutional disorder or structural distortion, demonstrating that regulating the carrier concentration alone changes the magnetic phase transition temperature and leads to colossal effects.

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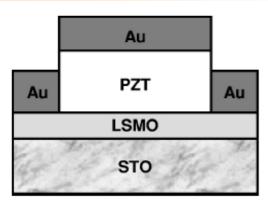
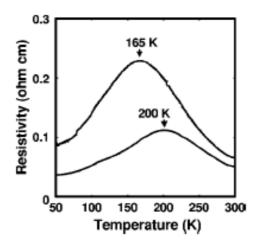


FIG. 2. Schematic view of a PZT/LSMO heterostructure deposited on a SrTiO<sub>3</sub> (STO) substrate. Gold electrodes are deposited for electrical transport measurements.



PACS number(s): 73.50.-h, 75.47.Gk

FIG. 6. Resistivity as a function of temperature for the two polarization states of the PZT layer. The upper curve corresponds to depletion of holes and is termed the depletion state; the lower curve corresponds to accumulation of holes and is termed the accumulation state. The resistivity peak temperatures are 165 K and 200 K for the depletion and accumulation states, respectively.

#### Side-gate devices in a La<sub>0.67</sub>Ba<sub>0.33</sub>MnO<sub>3</sub> exhibiting metallic behavior

Reversible shift of the transition temperature of manganites in planar field-effect devices

patterned by atomic force microscope I. Pallecchi et al., Appl. Phys. Lett. 83, 4435 (2003)

Field effect on planar devices made of epitaxial manganite perovskites I. Pallecchi et al., J. Appl. Phys. 95, 8079 (2004)

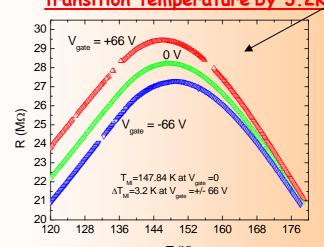
Channel width 2.3 mm

Gate barrier width 1.4 mm

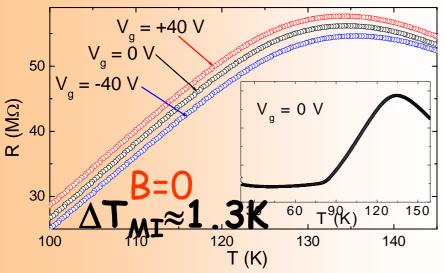
Film thickness 22 nm  $\Rightarrow C \approx 7.6 \cdot \varepsilon_n pF/m$ 

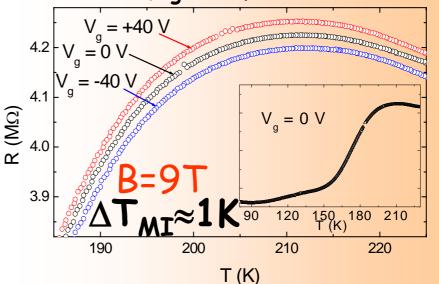
For  $\varepsilon_r \approx 1000$ , the equivalent charge per unit volume accumulated/depleted by a gate voltage of  $\pm 66 \text{V}$  in the channel is  $6.2 \cdot 10^{19} \text{ cm}^{-3}$ 

Reversible shift of the metal-semiconductor transition temperature by 3.2K

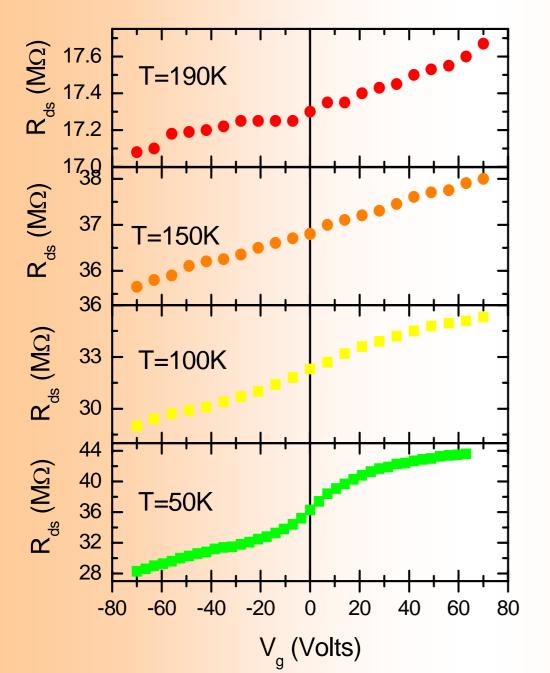


Comparison of the effects of electric and magnetic fields





#### Is this true field effect?

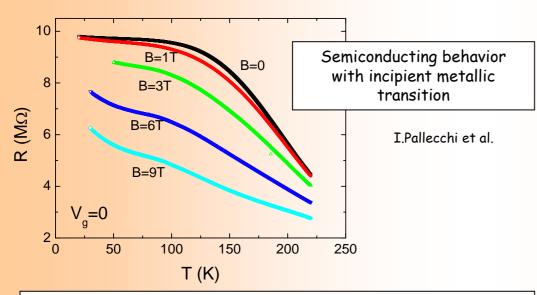


The relative change in channel resistance behavior as a function of the applied electric field is odd and linear at T>100K; at lower temperature the observed non-linearities may be due to non-linear dielectric permittivity of the SrTiO<sub>3</sub> substrate.

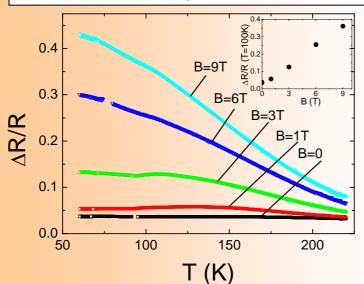
## Sheet charge or volume charge?

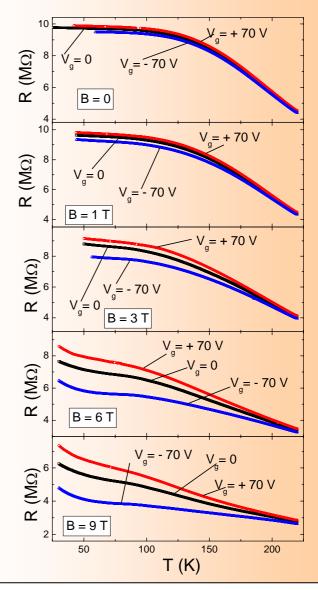
How much in depth does the electric field penetrate in a film with more than  $10^{20}$  carriers/cm<sup>3</sup>? Shall we invoke a phase separation scenario?

#### Side-gate devices in a La<sub>0.7</sub>Sr<sub>0.3</sub>MnO<sub>3</sub> below the percolation threshold



In a **phase-separation** scenario, metallic ferromagnetic regions are embedded in a semiconducting paramagnetic matrix and their volume fraction is below the percolation threshold

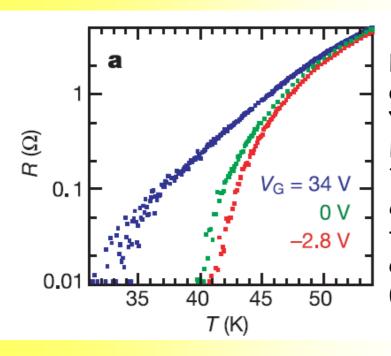




The electric field enlarges or shrinks the metallic ferromagnetic domains, while the magnetic field enlarges and also polarizes them.

## High Temperature Superconductors

#### Mannhart, J. High-Tc transistors. Supercond. Sci. Technol. 9, 49-67 (1996).

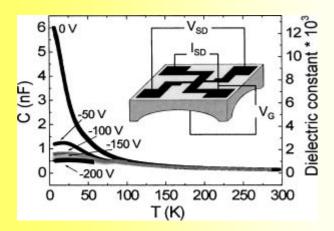


Field effects in superconducting films. Change of the DS resistance of an ,8-nm-thick YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7-d</sub> channel with a ,300-nm-thick Ba<sub>0.15</sub>Sr<sub>0.85</sub>TiO<sub>3</sub> gate insulator. The blue curve corresponds to depletion of the carrier density, and the red curve corresponds to enhancement of the carrier density in the DS (drain-source) channel.

APPLIED PHYSICS LETTERS VOLUME 83, NUMBER 18 3 NOVEMBER 2003

#### Field-effect experiments in NdBa<sub>2</sub>Cu<sub>3</sub>O<sub>7- $\delta$ </sub> ultrathin films using a SrTiO<sub>3</sub> single-crystal gate insulator

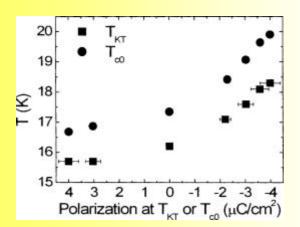
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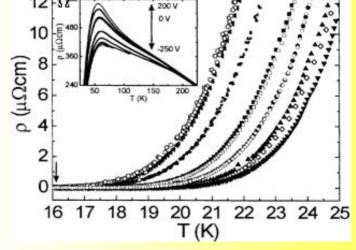


We report on the electrostatic modulation of superconductivity in very thin films of cuprate superconductors using a field-effect device based on a SrTiO<sub>3</sub> single-crystal gate insulator. A  $T_c$  modulation of 3.5 K and a 37% change of the normal state resistance have been observed in an epitaxial bilayer composed of an insulating PrBa<sub>2</sub>Cu<sub>3</sub>O<sub>7- $\delta$ </sub> layer deposited on top of a superconducting NdBa<sub>2</sub>Cu<sub>3</sub>O<sub>7- $\delta$ </sub> film, two unit cells thick. To achieve large electric fields, the thickness of the commercial dielectric single-crystal SrTiO<sub>3</sub> substrate (also used as the gate insulator) was reduced to 110  $\mu$ m. The dielectric properties of the gate insulator were characterized as a function of temperature and electric field and the magnitude of the field effect was quantified. A  $T_c$  enhancement of 2.8 K was obtained for an applied field of  $-1.8 \times 10^6$  V/m, corresponding to a polarization of  $-4 \mu$ C/cm<sup>2</sup>. © 2003 American Institute of Physics. [DOI: 10.1063/1.1624635]

Temperature and electric field dependence of the capacitance and dielectric constant of the STO single-crystal gate insulator. Inset: schematic of the device

Resistivity as a function of temperature of the PBCO/NBCO heterostructure close to the foot of the transition and over the whole temperature range (inset) for different applied fields across the gate dielectric. The resistivity was calculated using the thickness of the NBCO layer. The arrow indicates the value of *T*KT for zero applied field.



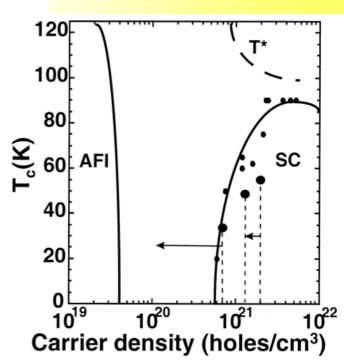


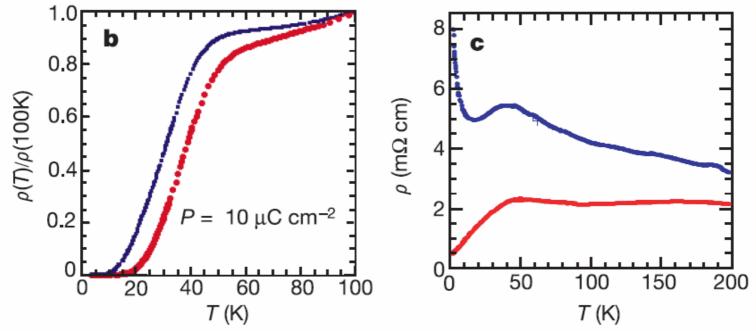
Critical temperature  $T_{\rm KT}$  (the Kosterlitz–Thouless temperature), and  $T_{c0}$ , the temperature at which R=0.1 of the NBCO layer as a function of the measured polarization at  $T_{\rm KT}$  or  $T_{c0}$ 

# Electrostatic Modulation of Superconductivity in Ultrathin GdBa<sub>2</sub>Cu<sub>3</sub>O<sub>7-x</sub> Films

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The polarization field of the ferroelectric oxide lead zirconate titanate  $[Pb(Zr_xTi_{1-x})O_3]$  was used to tune the critical temperature of the high-temperature superconducting cuprate gadolinium barium copper oxide  $(GdBa_2Cu_3O_{7-x})$  in a reversible, nonvolatile fashion. For slightly underdoped samples, a uniform shift of several Kelvin in the critical temperature was observed, whereas for more underdoped samples, an insulating state was induced. This transition from superconducting to insulating behavior does not involve chemical or crystalline modification of the material.





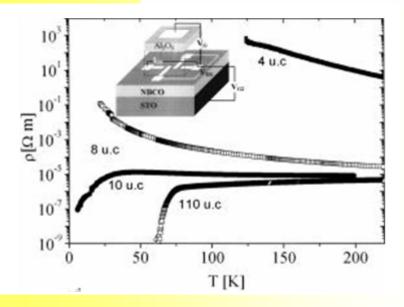
APPLIED PHYSICS LETTERS VOLUME 84, NUMBER 19 10 MAY 2004

#### Field-effect tuning of carrier density in Nd<sub>1.2</sub>Ba<sub>1.8</sub>Cu<sub>3</sub>O<sub>y</sub> thin films

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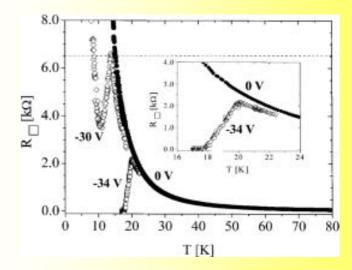
(Received 5 January 2004; accepted 14 March 2004; published online 29 April 2004)

Using a field effect device we modified the number of holes in the surface layers of 4 to 10 unit cell Nd<sub>1.2</sub>Ba<sub>1.8</sub>Cu<sub>3</sub>O<sub>y</sub> (NBCO) epitaxial films grown on (100) SrTiO<sub>3</sub> substrates. The results obtained on a set of 12 devices demonstrate that it is possible to induce reversible changes of the hole density of NBCO films by field effect. It is found that the field effect becomes less pronounced increasing the film thickness. Insulating—superconducting transition was observed in one 8 unit cell NBCO field effect device. © 2004 American Institute of Physics. [DOI: 10.1063/1.1745103]



Temperature dependence of the resistivity of Nd<sub>1.2</sub>Ba<sub>1.8</sub>Cu<sub>3</sub>O<sub>y</sub> films having different thicknesses: 4 u.c. (closed squares), 8 u.c. (open squares), 10 u.c. (closed circles), and 110 cells (open triangles). In the inset a sketch of the field effect device is shown.

Sheet resistance measured as a function of temperature on an 8 u.c. FET for  $V_g = 0$  (closed circles),  $V_g = -30$  V (open diamonds), and  $V_g = -34$  V (open circles). The dashed line indicates the value of the quantum resistance  $R_Q = 6.45$  k . In the inset the insulating—superconducting transition is shown



## Conclusions

- Field effect in transition metal oxides is possible adopting new geometries and high- $\kappa$  materials.
- Back and side gate geometry allow direct access to the channel under FE
- In ZnO the application is close
- FE proved to be a powerful tools for the study of strongly correlated electron system:
  - Magnetic transition and phase separation in manganites
  - Superconducting properties in HTCS