Oxides for Electronics, Memory, Magnetics, Photonics, Energy and Health

T. Venkatesan

NUS National University of Singapore | NUS Nanoscience & Nanotechnology Initiative

National University of Singapore

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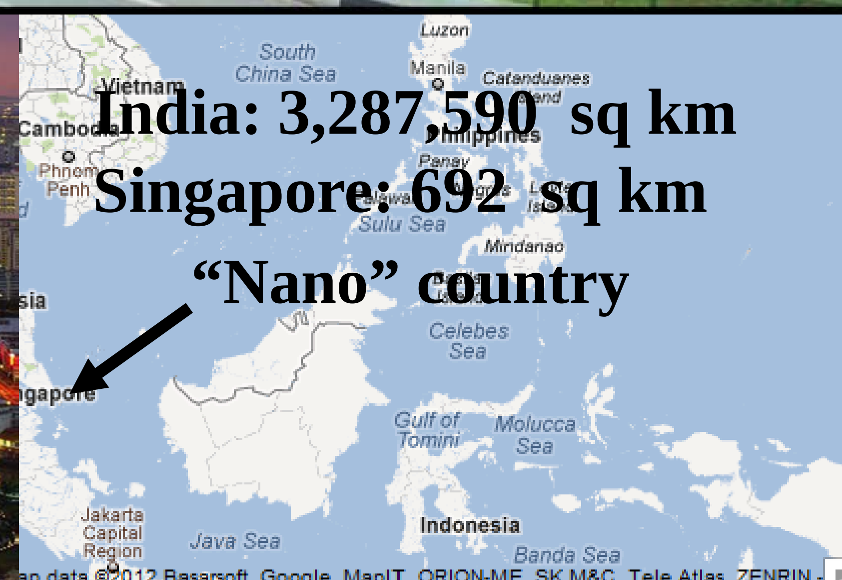
NanoCore

http://www.nusnni.nus.edu.sg/



India: 3,287,590 sq km

Singapore: 692 sq km

“Nano” country

Collaborators

Electronics/ Magnetics/ Memory	Energy	Health/ Bio- Inorganic Interfaces
Chen Jingsheng	Nripan Mathew (NTU)	Georgia Pastorin
Ramesh Ramamoorthy (ORNL)	Yang Shao Horn (MIT)	Markus R Wenk
Hans Hilgenkamp (Twente)	Antonio Castro- Neto	Paul Bigliardi (IMB)
Jeremy Levy (Pittsburgh)	Loh Kian Ping	Lim Chwee Teck
Michael Coey (Trinity)	Qing Wang	GV Shivashankar (MBI)
Eva Olsson (Chalmers)	Chua Soo-Jin	Linda Kenney (UIC)
S. Maekawa (JAST)	Lydia Helena Wong (NTU)	Wilson Wang
M. Bosman (IMRE)	Xu Rong (NTU)	Frank Watt
Seiji Yunoki (Riken)	Sum Tze Chian (NTU)	Walter Hunziker (IMCB)
Qinfang Zhang (Riken)	**Zhao Yongliang**	Lim Kah Leong
Bo Gu (JAST)	**Matthew Sinu**	Wang Haifeng
Mark Golden (Amsterdam)	**Wan Dongyang**	Kah James
Zhiqi Liu (ORNL)	**Zhao Lingchao**	Thomas Osipowicz
X Wang (MIT)	**Li Changjian**	**Kingshuk Poddar**
A Annadi (Pittsburgh)	**Meenakshi Annamalai**	**Stuti Kaivalya Desai**
Ding Jun	**Amar Srivastava**	**Shefali Talwar**
Mark Breese	**Tarapada Sarkar**	**Michal Dykas**
Ariando	**Brijesh kumar**	**Abhijeet Patra**
Andrivo Rusydi	**Syed Nizar**	**Federico Tesio Torta**
Hyunsoo Yang	**Kelsey Stoerzinger (MIT)**	**Amaury Gassiott**
Chen Jianqiang		**Mallikarjuna Rao Motapothula**
Chow Gan Moog		**Abhimanyu Singh Rana**
John Wong		**Viknish Krishnan Kutty**
Yuan Ping Feng		**Yoong Sia Lee**
Randall Law		**Sophia Lee**

A Bit of History

Materials like TiO_2 have been around for centuries

Oxide research in ferroelectrics, magnetics, superconductors, photonics etc…dates back to the 1950s or even earlier

Ferroelectrics like titanates (PZT), optical materials like $LiNbO_3$, Fe_3O_4, VO_2, manganites etc.. were well studied in the 1950-1970 time frame.

What rejuvenated this field in the 1980s?

It was a TSUNAMI!!

The High Tc TSUNAMI!!

1986 was a year of SUPERS

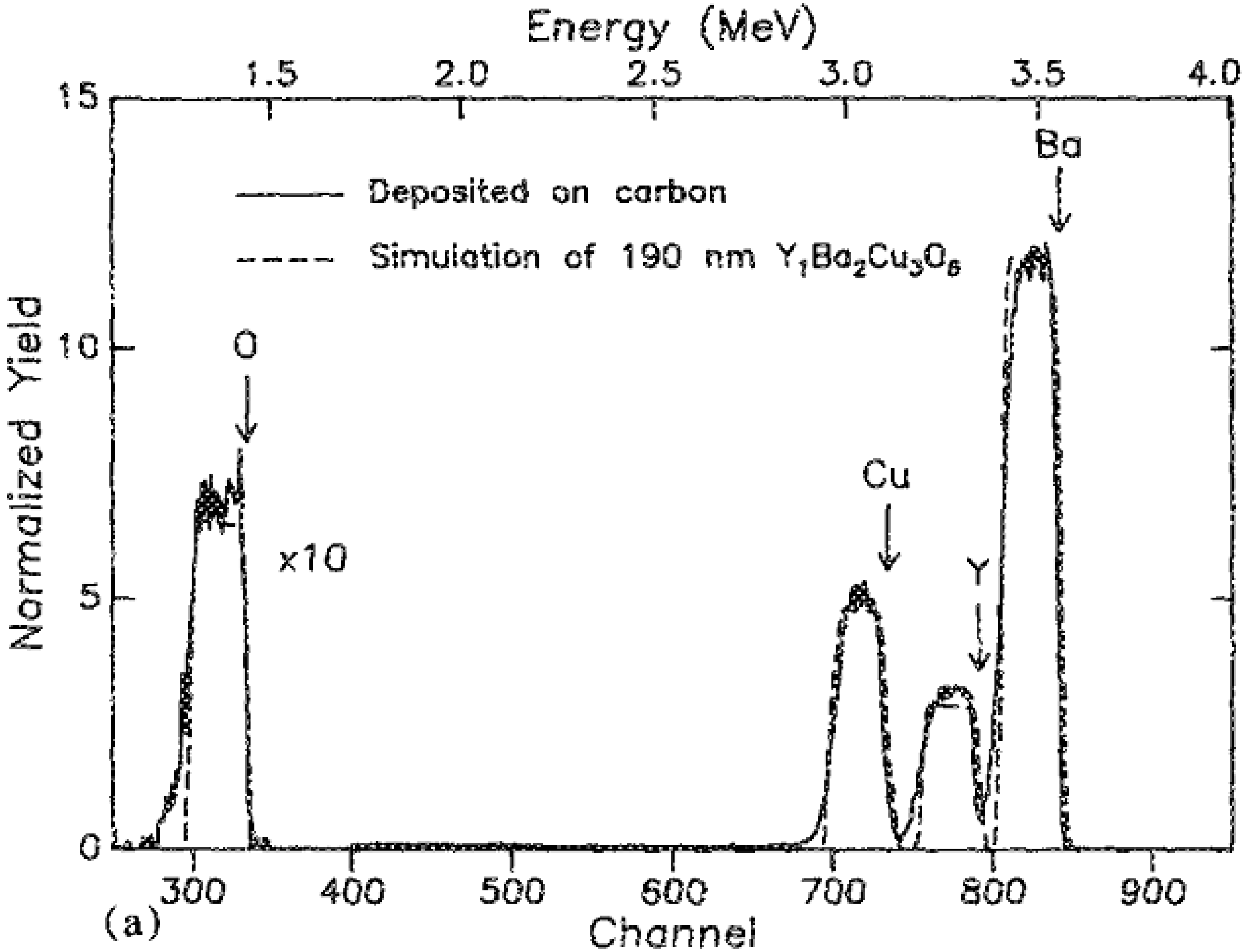
1. Super Super Nova Explosion
2. Super Wall Street Crash
3. Super Superconductors (High Tc)

Invention of the PLD Process

- The process reproduced the composition of a target of a multi-component material in the form of a smooth thin film
- The process enabled the rapid prototyping of virtually any oxide material one could synthesize in the Laboratory in the form of a pellet with size > 1 cm
- There has been a global adaptation of this technology with oxide researchers leading the pack

D. Dijkkamp, T. Venkatesan, APL, Applied Physics Letters 51 (8), 619(1987)
T. Venkatesan et al, APL, Applied physics letters 52 (14), 1193 (1988)

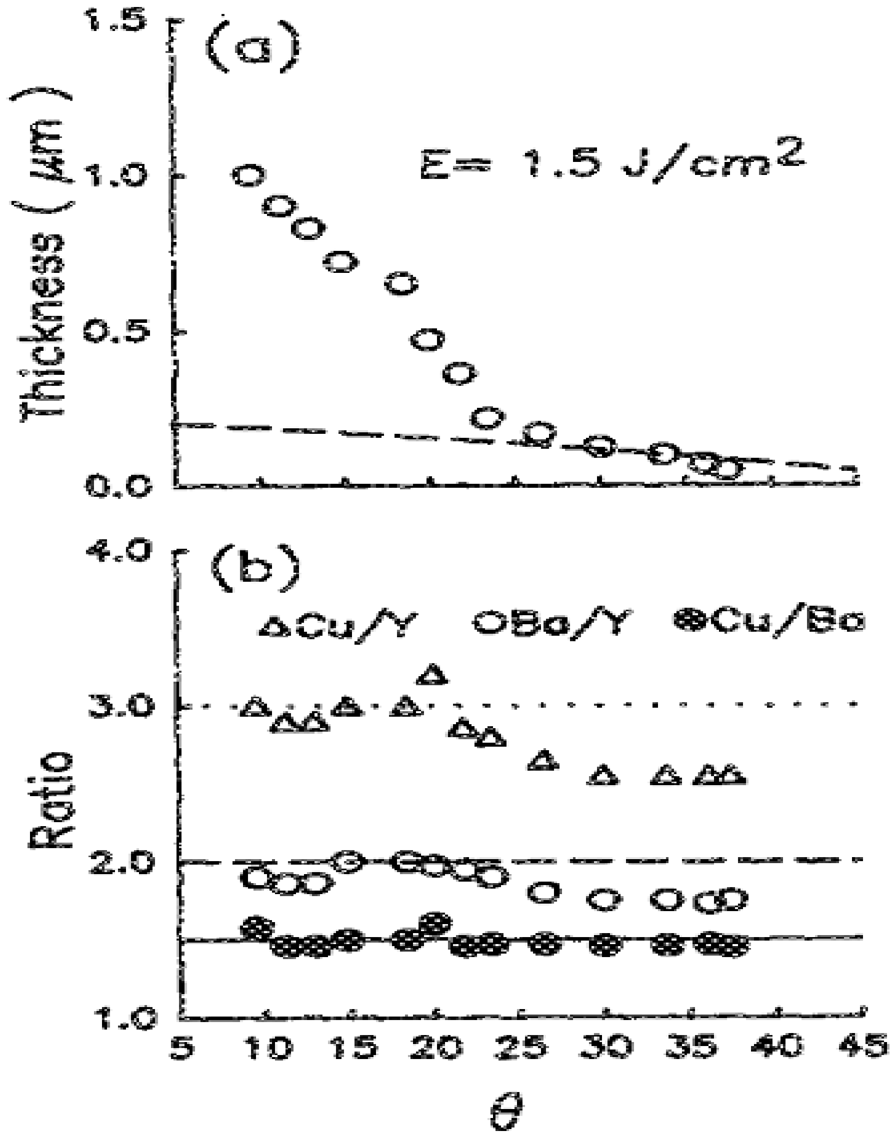
Composition Preservation of a Multicomponent Target in the deposited film



April 30, 1987 at Bellcore Labs at Rutgers University

D. Dijkkamp, T. Venkatesan, X. D. Wu, S. A. Shaheen, N. Jisrawi, Y. H. Min-Lee, W. L. McLean, M. Croft, Appl. Phys. Lett. 51, 619 (1987)

Angle and energy density Dependence of Composition



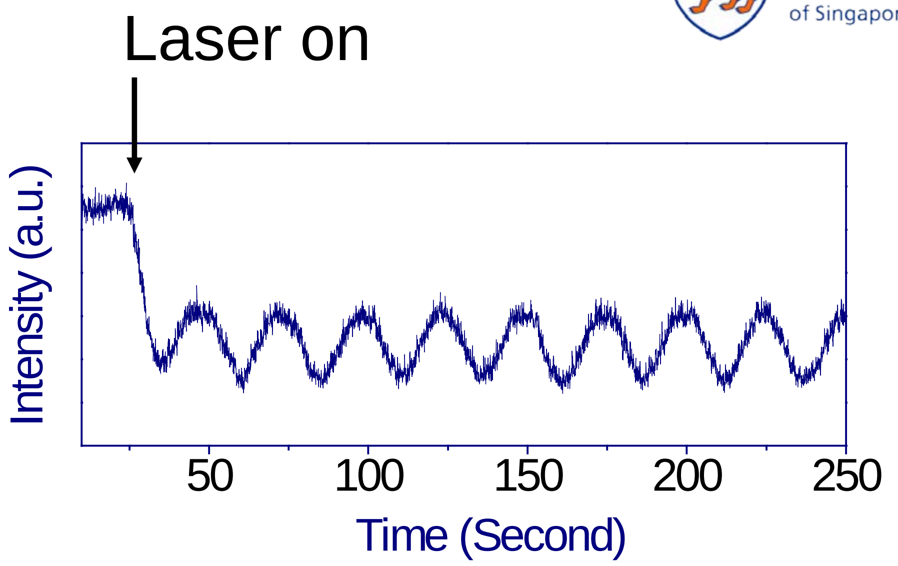
End of 1987: ***T. Venkatesan, X. D. Wu, A. Inam, J. B. Wachtman, Appl. Phys. Lett. 52, 1193 (1988)***

Key for progress

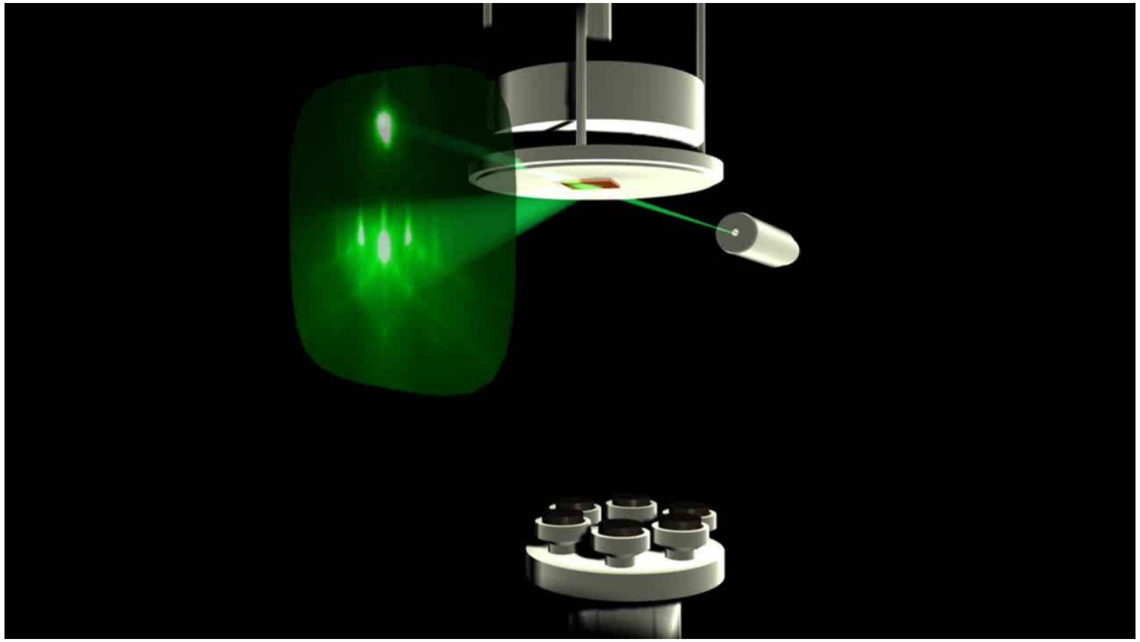
Atomic layer growth:

Pulsed laser deposition with in-situ RHEED

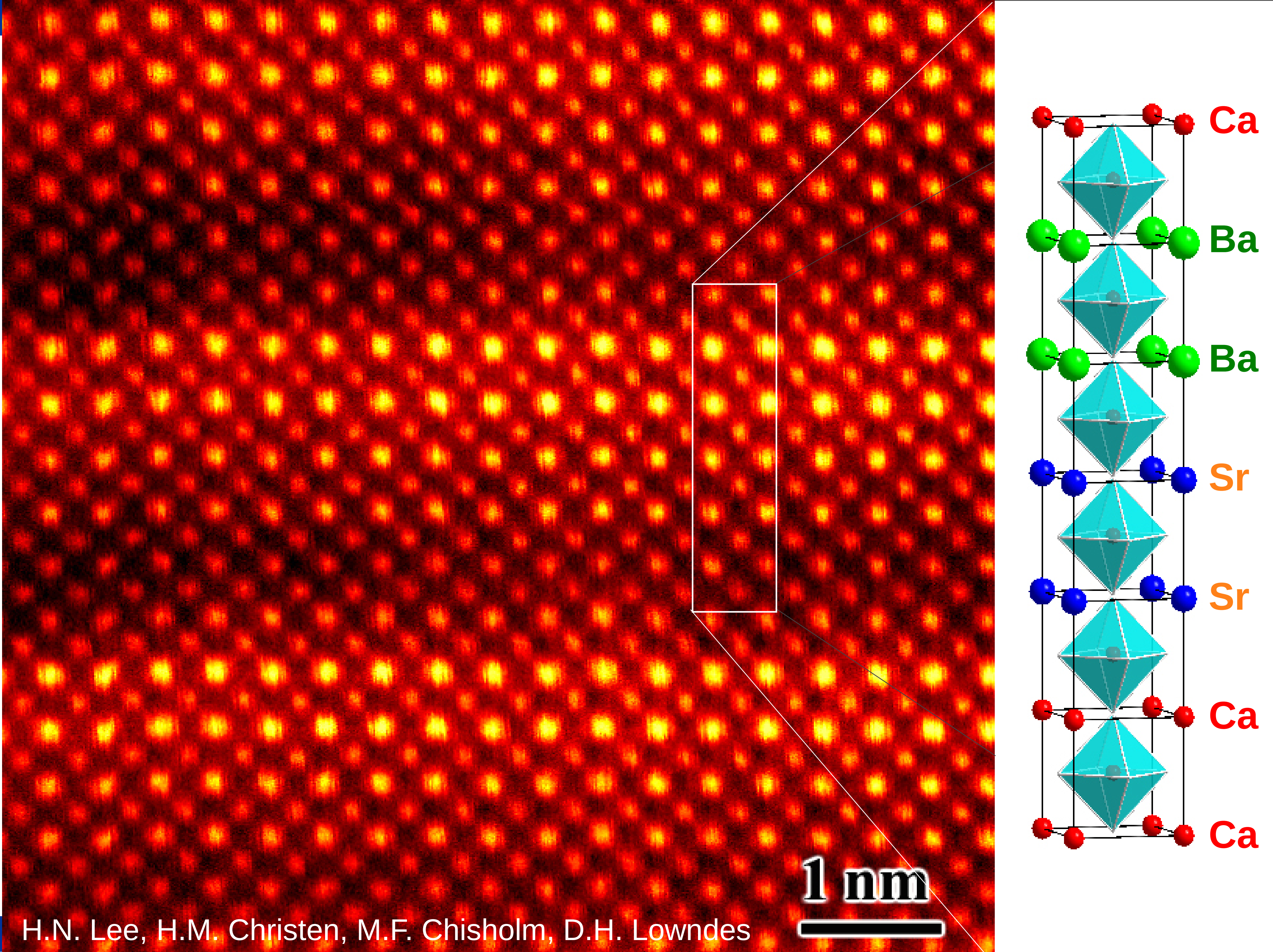
- **Stoichiometric transfer**
- **Easy to control oxygen**
- **Easy to vary composition**
- **High throughput**



- **Layer-by-layer growth**
- **Large O_2 pressure range (10^{-2}-10^{-6} Torr)**
- **T = 750 °C**



Freedom to design, stacking sequence not realized by nature in the bulk



Ca
Ba
Ba
Sr
Sr
Ca
Ca
1 nm
H.N. Lee, H.M. Christen, M.F. Chisholm, D.H. Lowndes

The Field That Gives!!

High Tc- 1986- 1994…….

Colossal Magneto Resistance Manganites- 1993- 1998

Diluted Magnetic Semiconducting Oxides- 1997- “UFOs”

Multiferroics- 1997-…………..

Polar/ Non-Polar Interfaces

Energy Materials

Novel Plasmonic Materials

Novel Bio- Interfaces???

1971-1986 (15 years):

Pion Deuteron scattering Cross section using Glauber's Eikonal Approximation
Heitler's paradox- Narrower than natural linewidth fluorescence from Mg atoms
Optical Bistability- Road to all optical computing
Laser Annealing of semiconductors
Ion beam interaction with organic surfaces
Bell's first Ga focused ion beam system and liquid metal ion sources
GeSe2 as high resolution ion beam resist
Si induced enhanced diffusion in GaAs/GaAlAs heterostructures
High power semiconductor lasers based on curved resonators

1987- Now (29 years):

Oxides
High Tc superconductors
CMR Manganites
DMSO
Oxide Interfaces
Bio- Inorganic Interfaces

Major Research Programs

- In-situ study of quantum interfaces (Electronics)
- Low Energy Field Effect Memories (Memory)
- Oxide Electronics On Silicon-Beyond Moore (Electronics, Magnetics, photonics)
- SINBERISE-Solar Photons to fuel (Energy)
- Science of Bio-Inorganic Interfaces (Biology and health)

Science of Bio-Inorganic Interfaces

Preparation of material library

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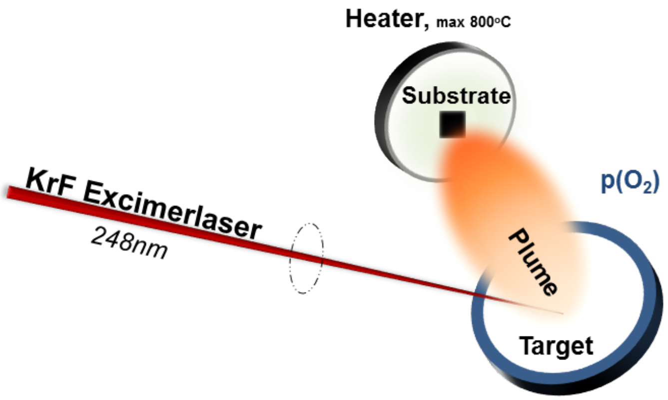
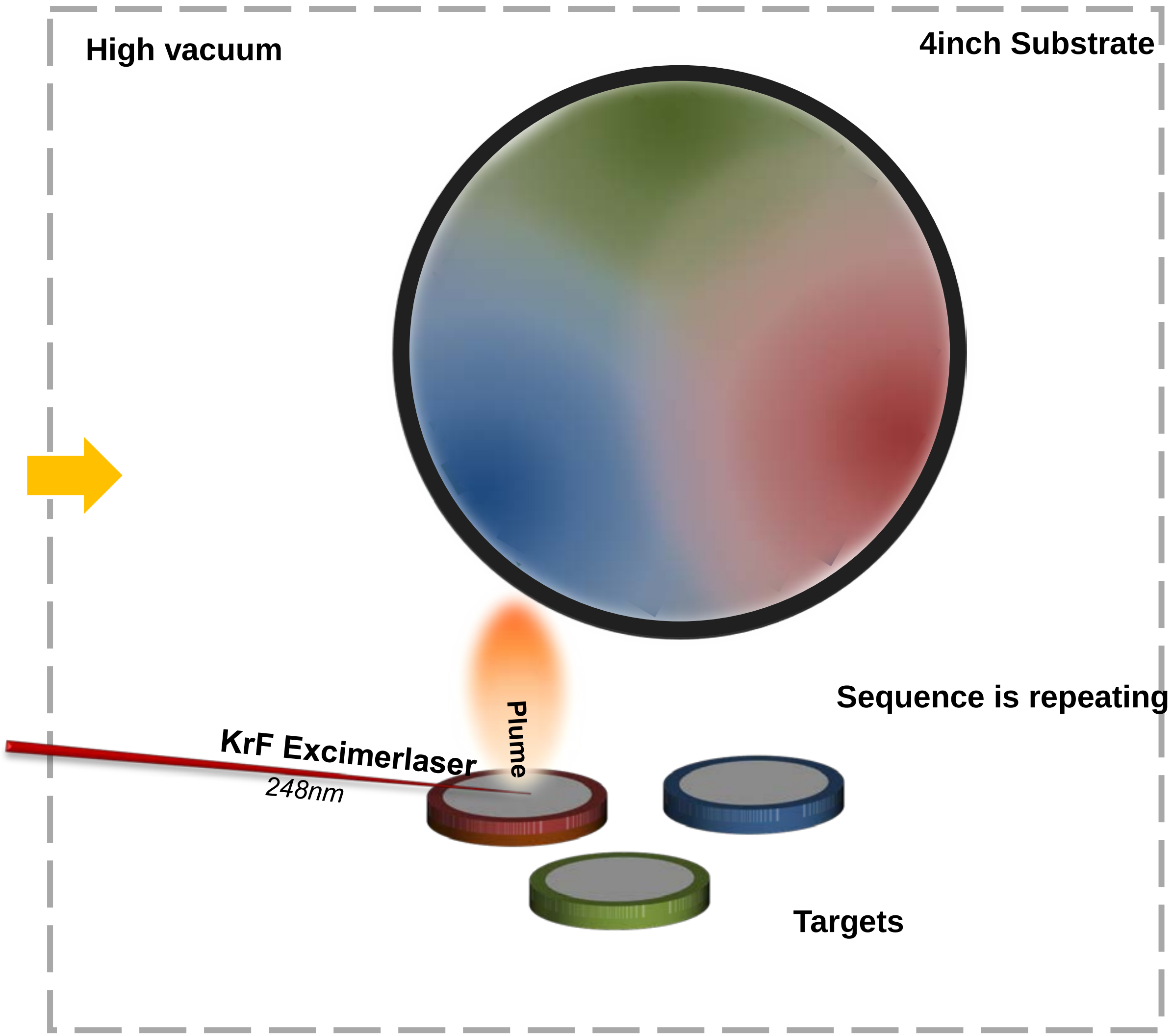
Conventional PLD

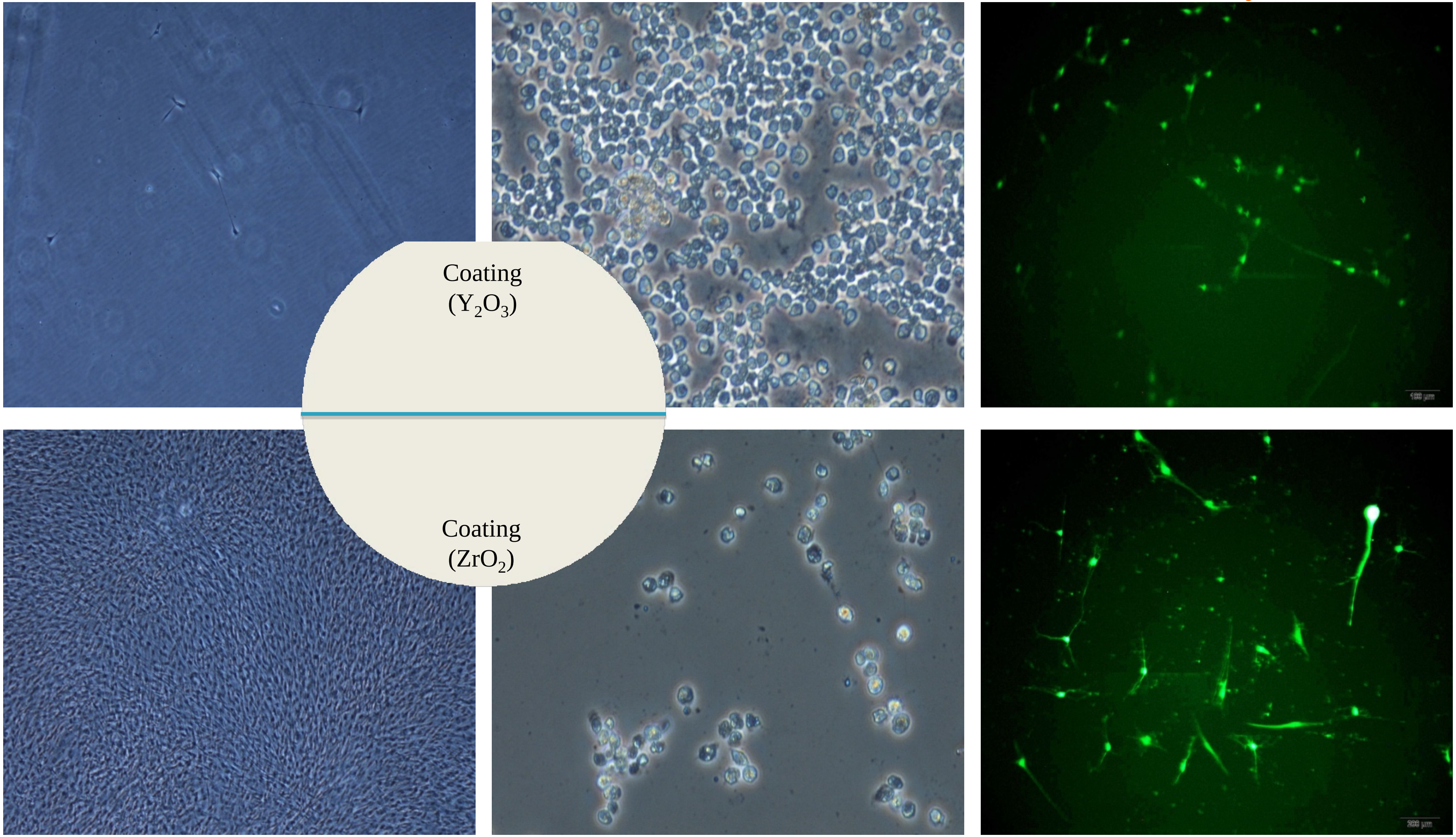


Continuous composition spread PLD

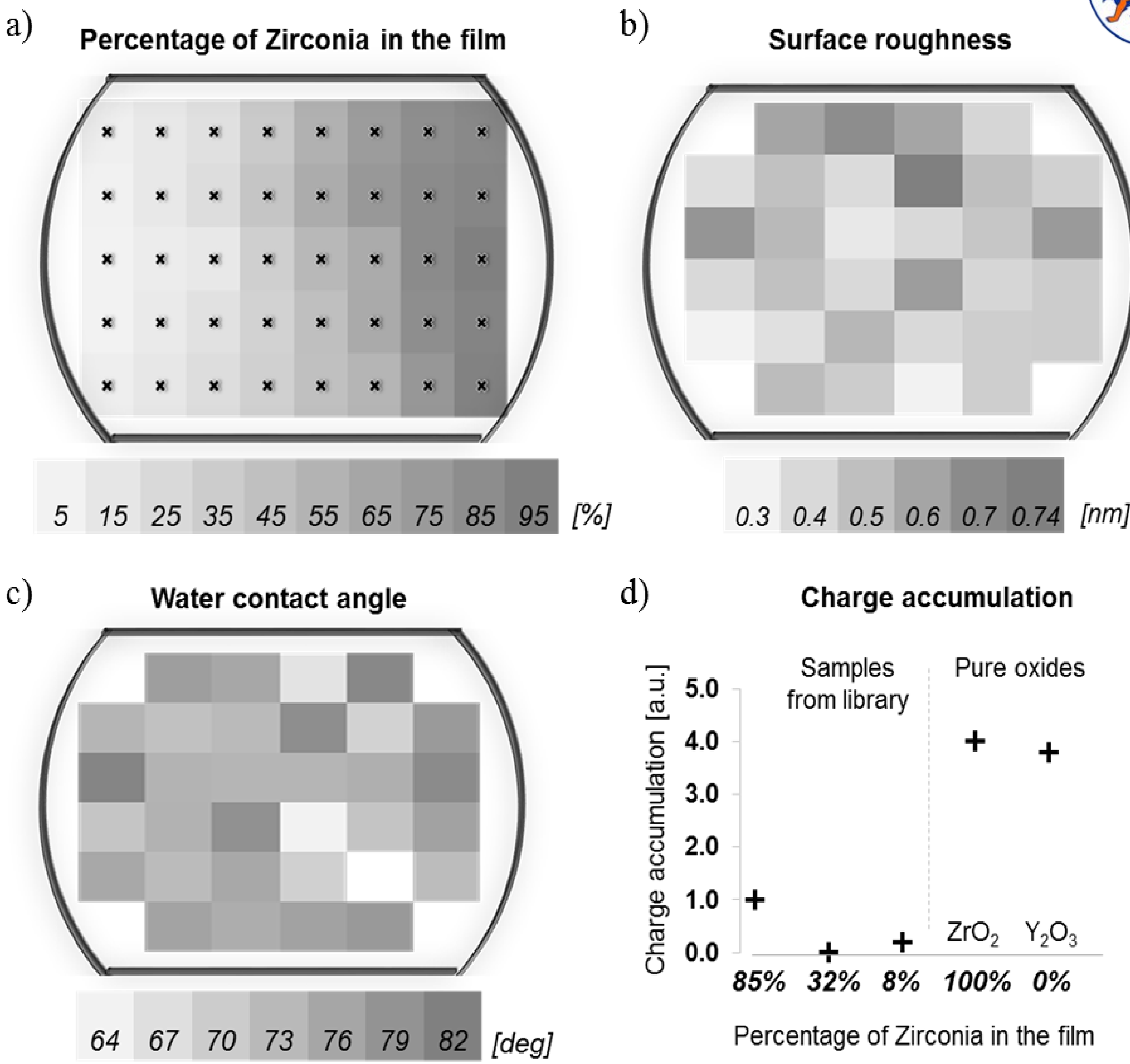


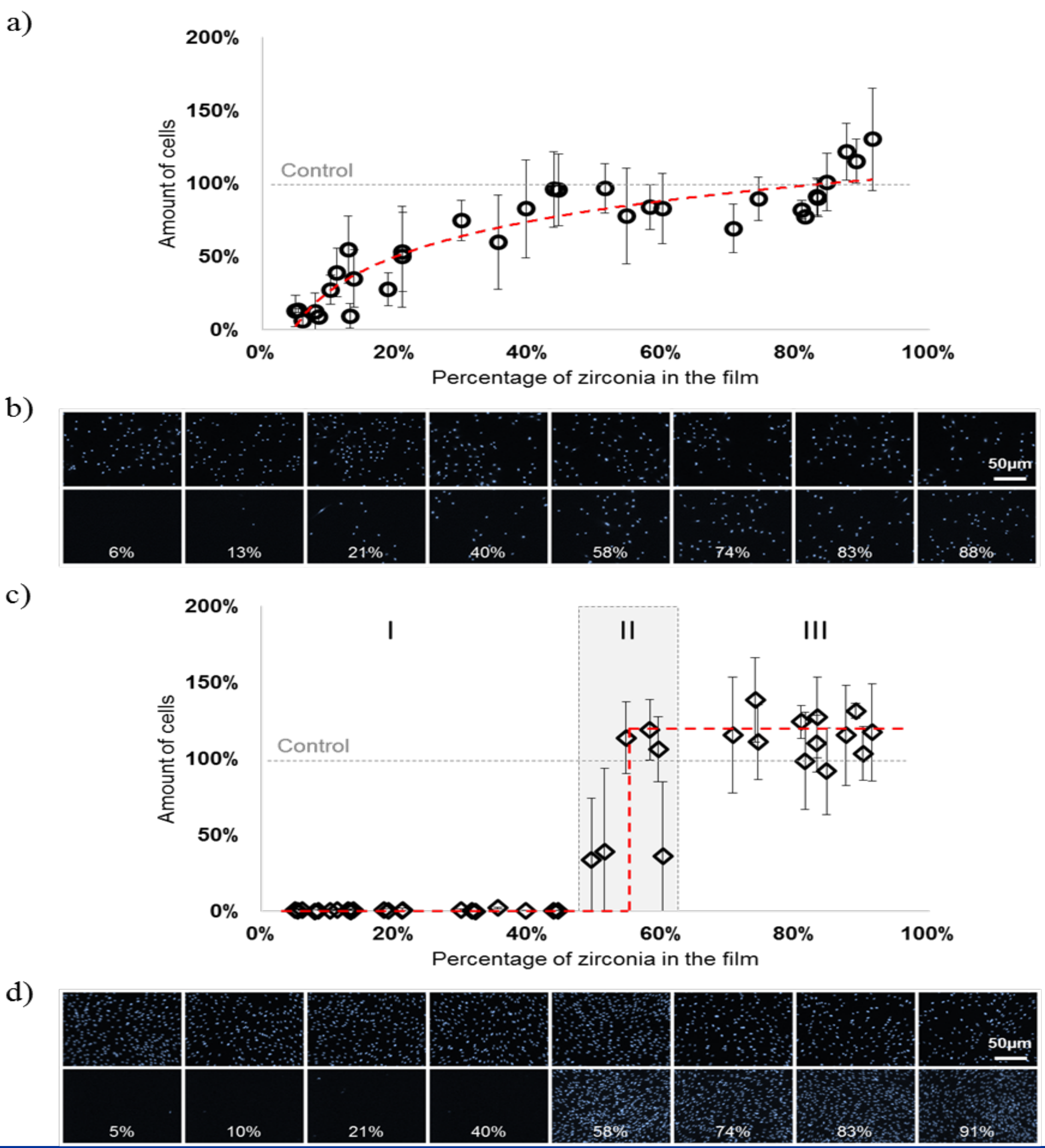
Differential culture of human cells on inorganic oxide surface

Growth of Human Primary Fibroblasts | **Growth of Human Primary Melanoma** | **Differentiation of Human Neural Crest Stem Cells into Sensory Neurons**



Surface roughness, wetting angle, charge





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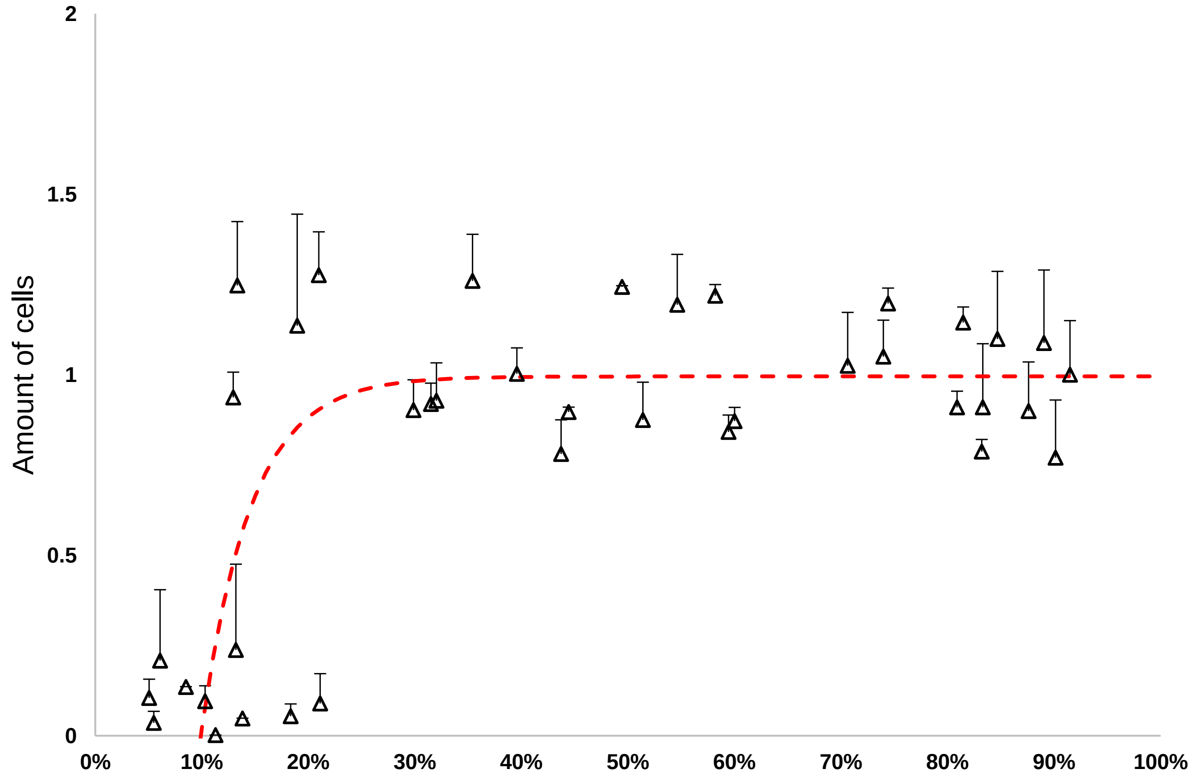
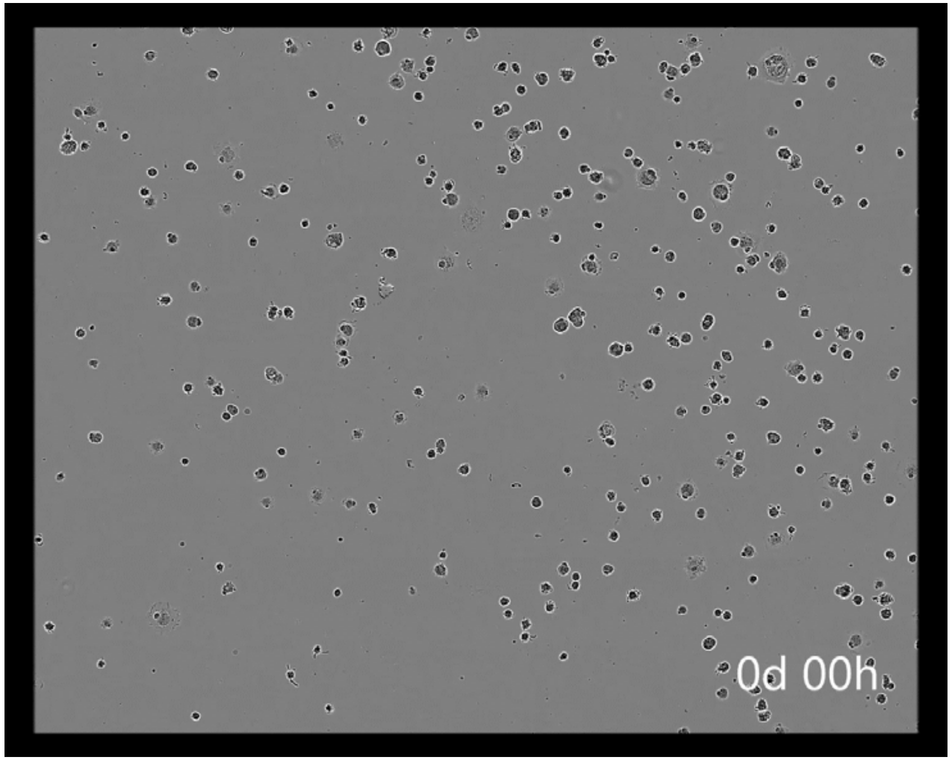
Neuronal Stem Cells on Material Library



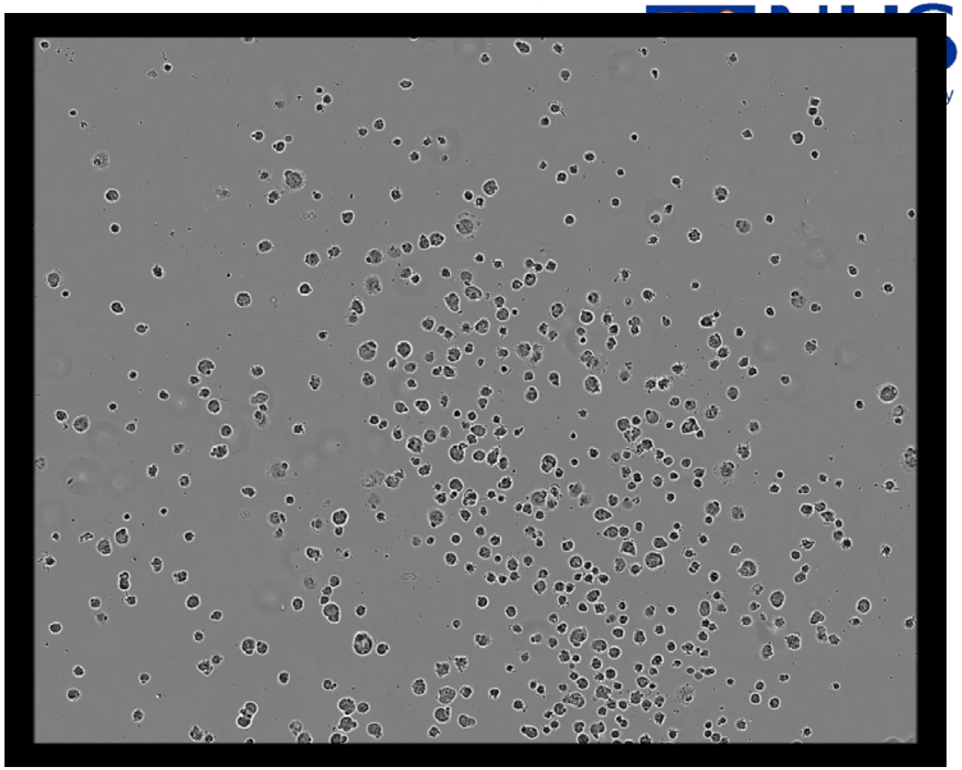
Critical Concentration and Adhesion parameters

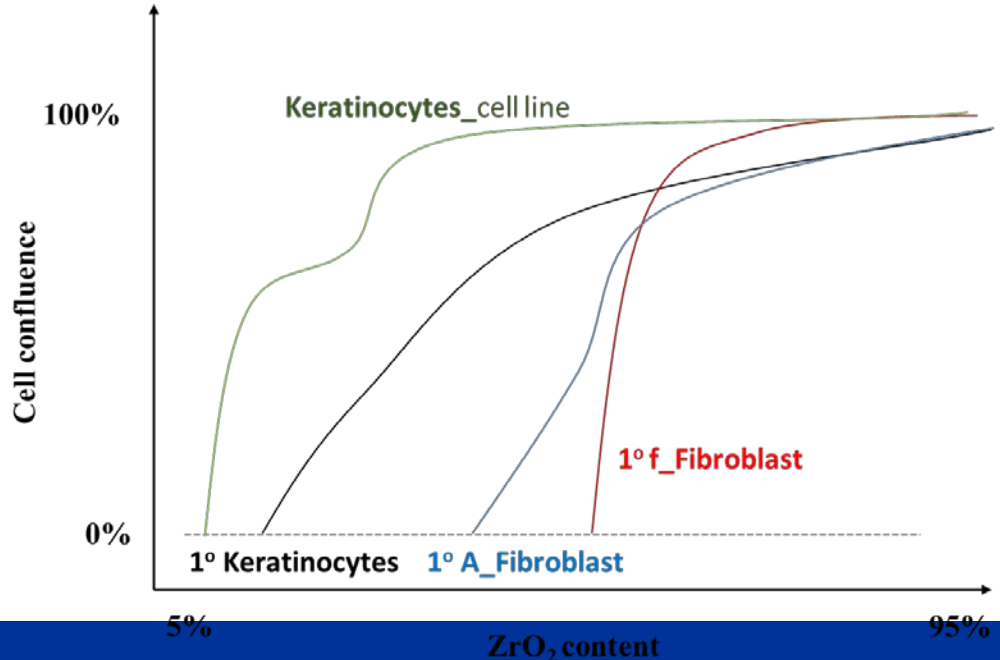
Cell Type	x_c	x_o	$\alpha_{Zirconia}$ α_{max}	α_{Yttria} α_{min}
NSCs	*0.099*	*0.046*	*0.995*	*-0.109*
FBs	*0.486*	*0.035*	*1.163*	*-1.099*
KCs	*0.008*	*0.281*	*1.043*	*-0.008*

Yttria region



Zirconia region

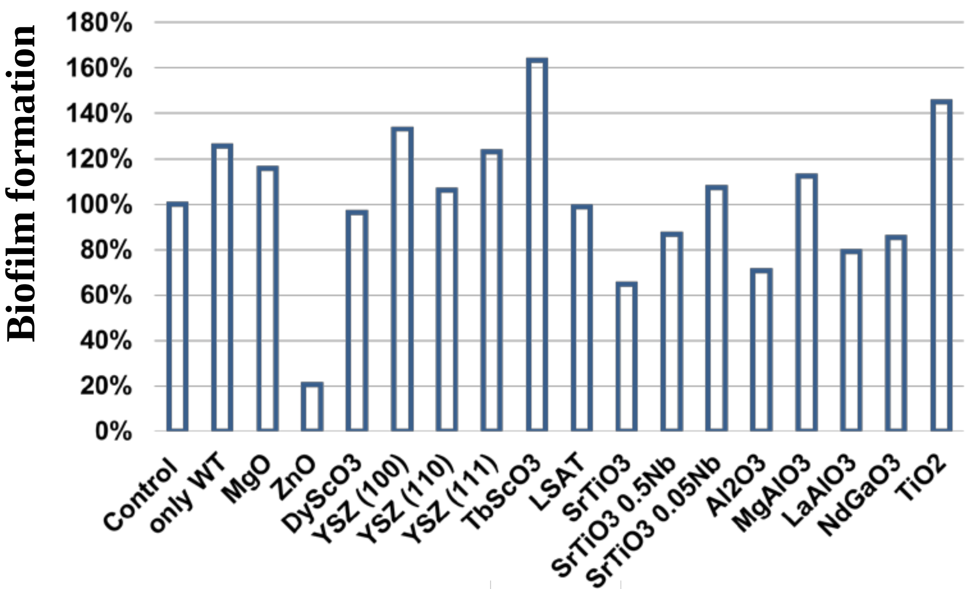


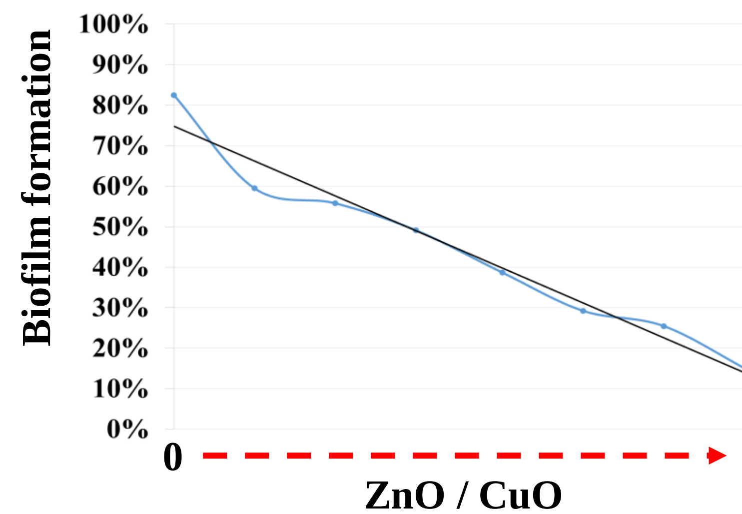


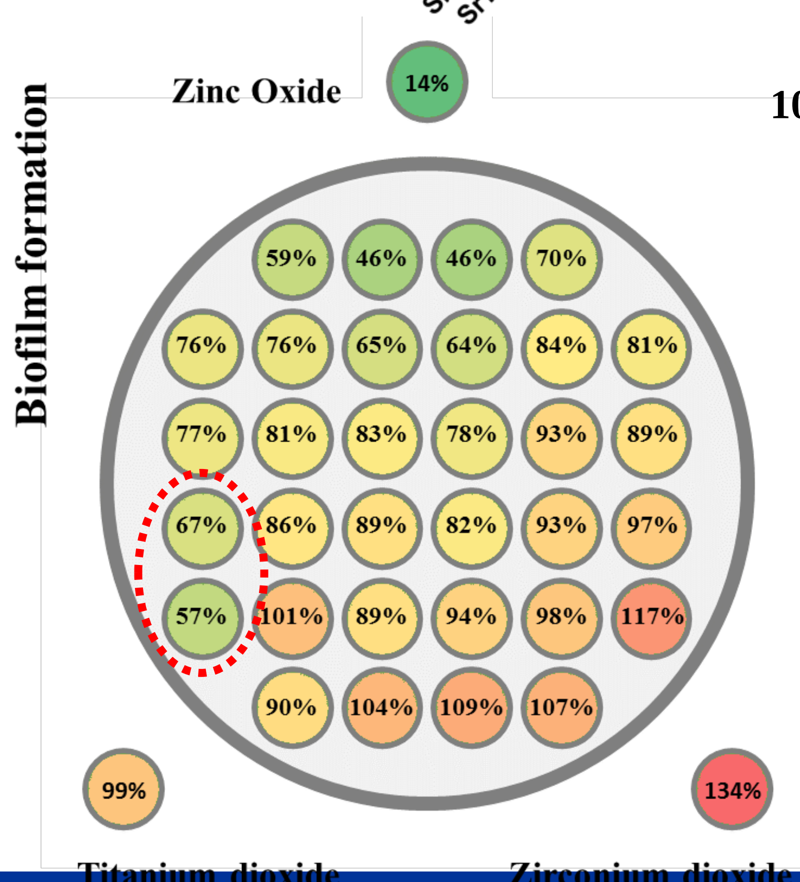
Cell types:

Keratinocytes_cell line - NTERT cell line
1° Keratinocytes - from human subject
1° A_Fibroblasts - from human adult skin
1° f_Fibroblasts - from human foreskin

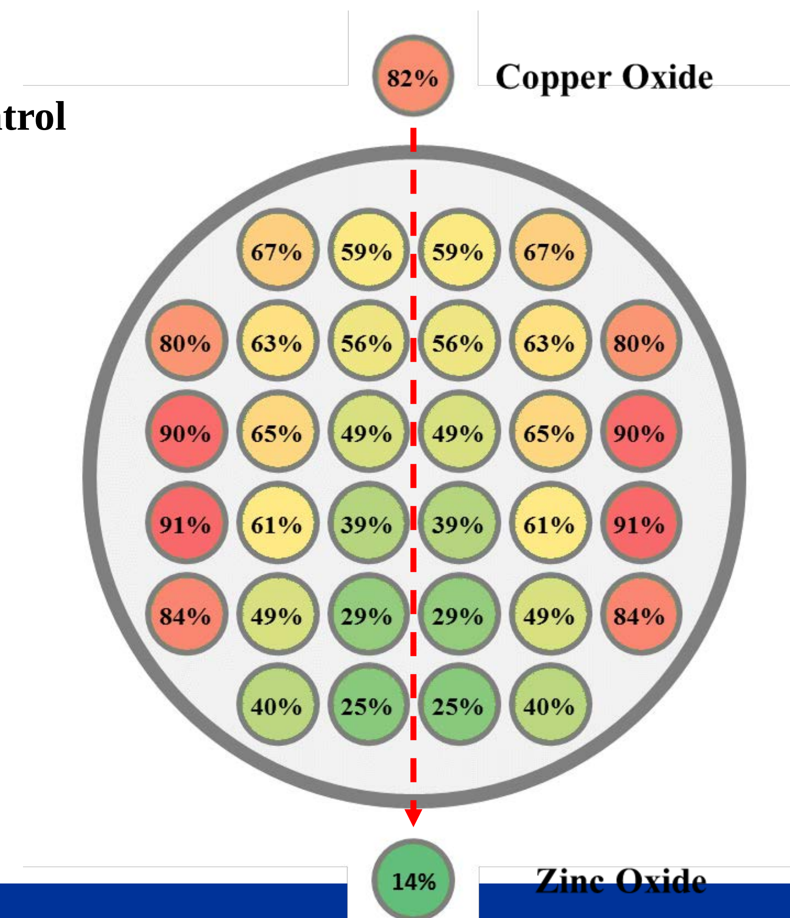
Biofilm formation of *Salmonella*



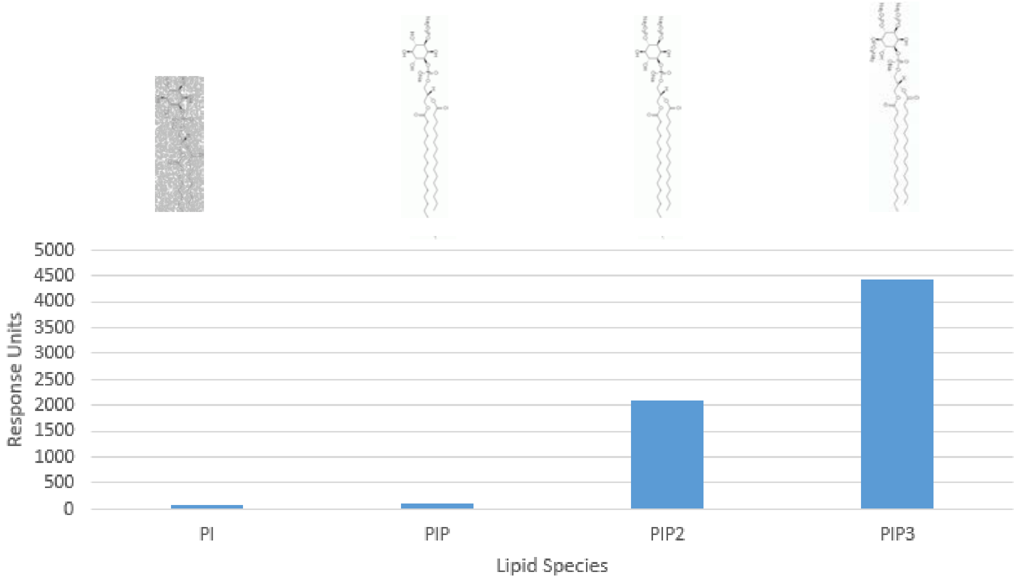


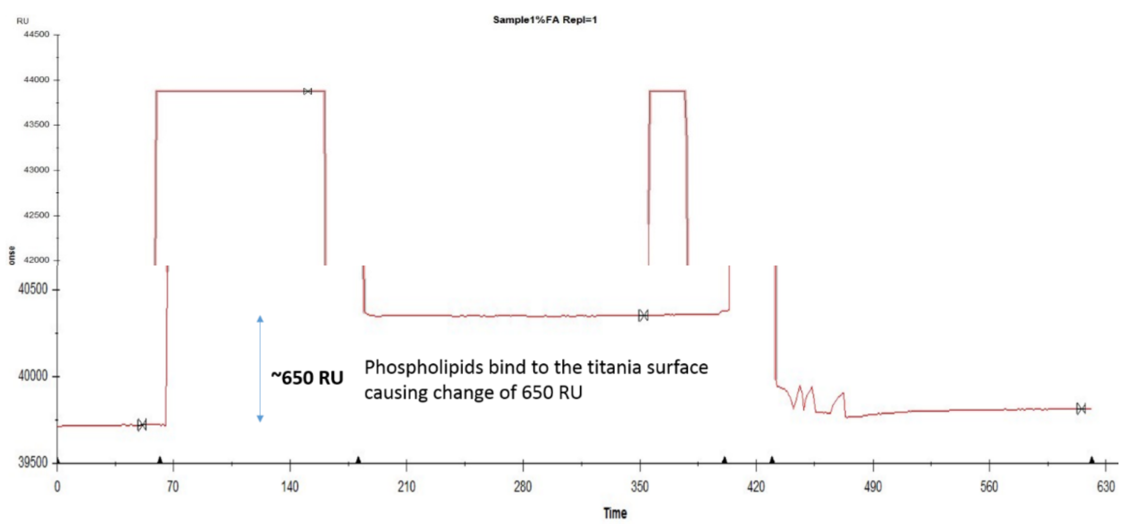


100% - control



SPR chips with a stable layer of transition metal oxide for studying interaction of biomolecules to surfaces





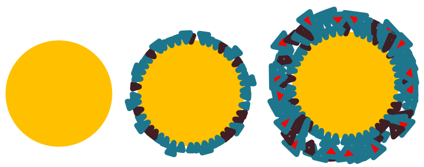
1. Very sensitive and accurate measurement of adhesion of biomolecules to surfaces of interest
2. Minimum errors/artefacts – will enable better decision in choice of material for solid phase extraction and engineered cell fate applications

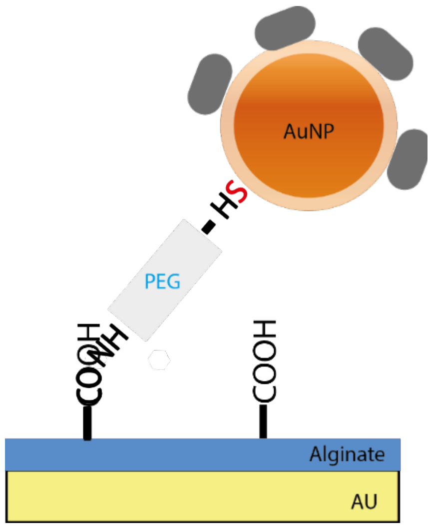
SPR for studying protein-NP interactions

Protein	k_a	k_d	K_D
HSA	2.26E+02	7.23E-05	**3.19E-07**
Fibrinogen	2.36E+03	1.84E-05	**7.80E-09**
IgG	9.09E+01	8.24E-06	**9.06E-08**

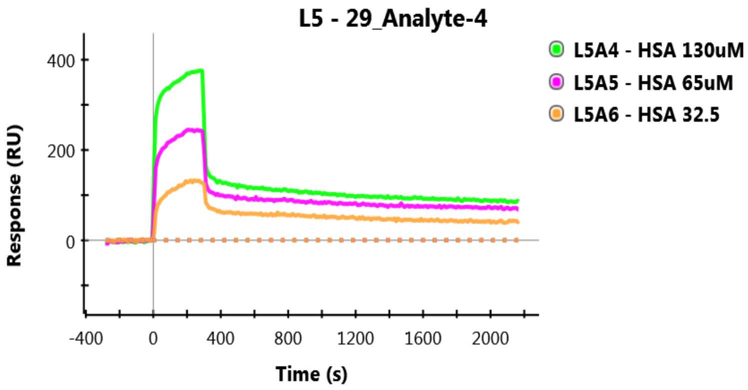
Kd is very accurate in this chart

Ka requires model fitting and theory support for the model used

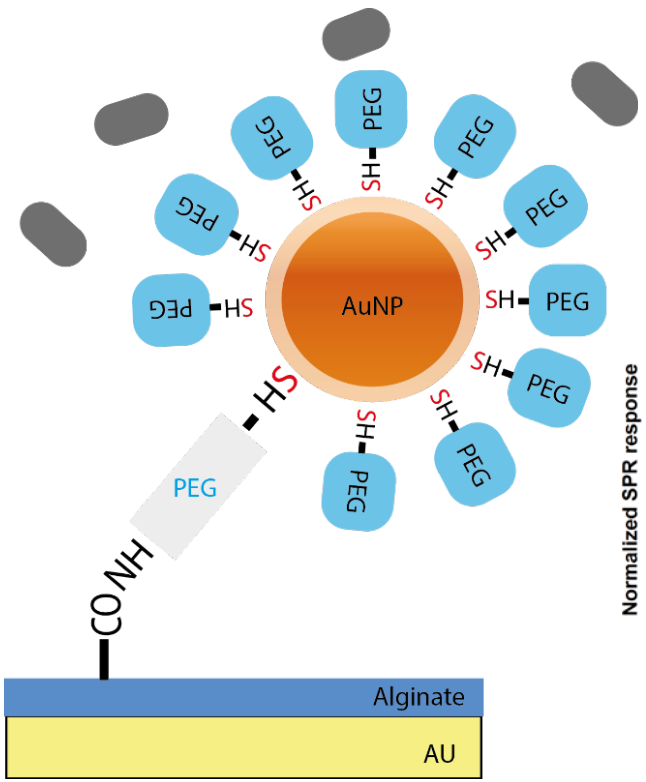


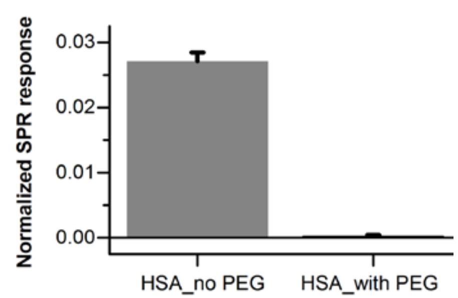


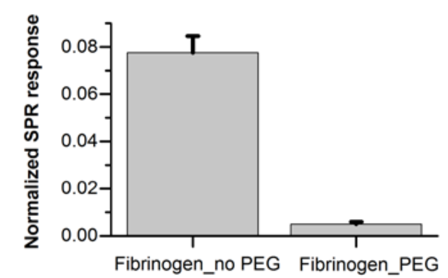
EDC/NHS coupling



Probing effect of PEGylation

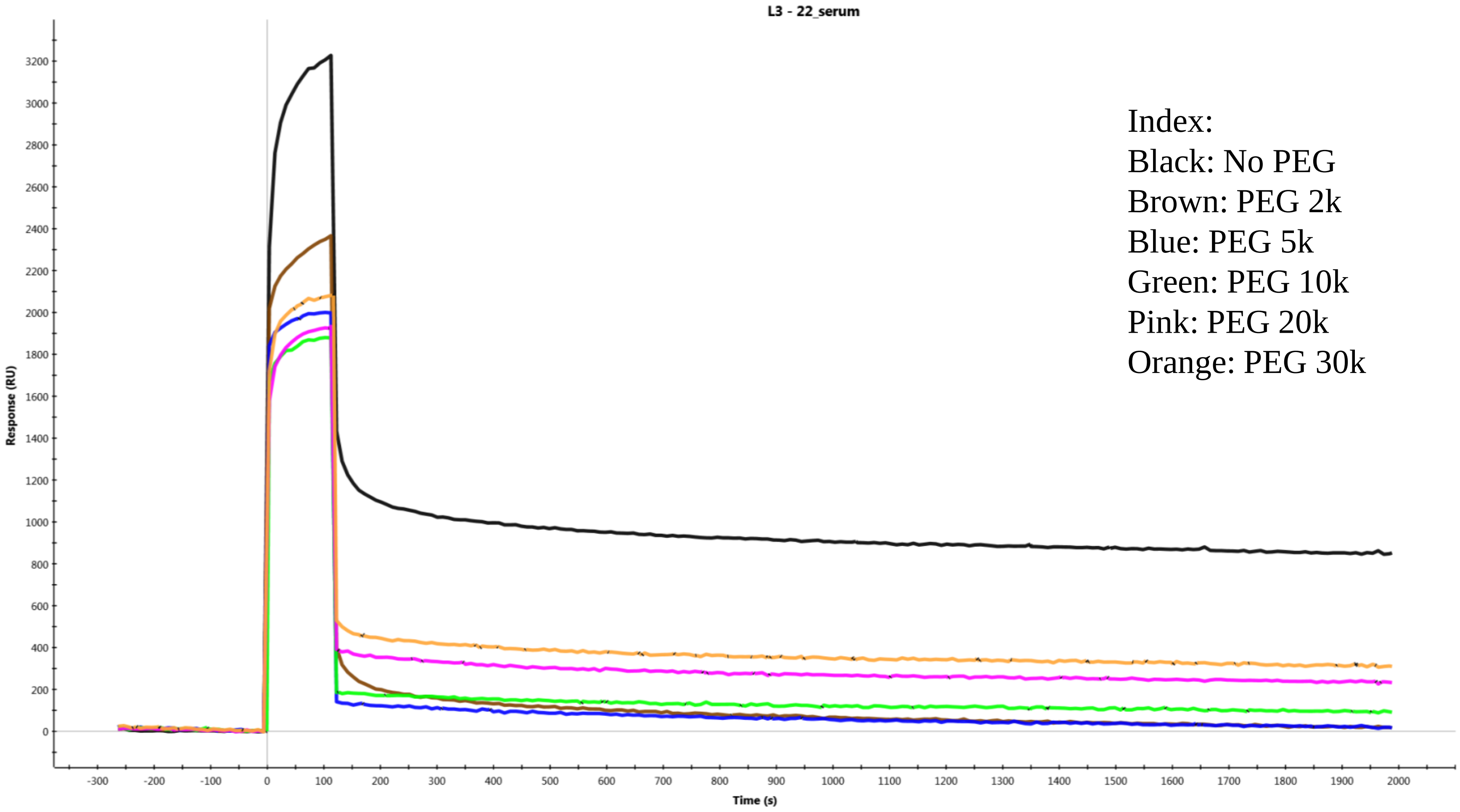






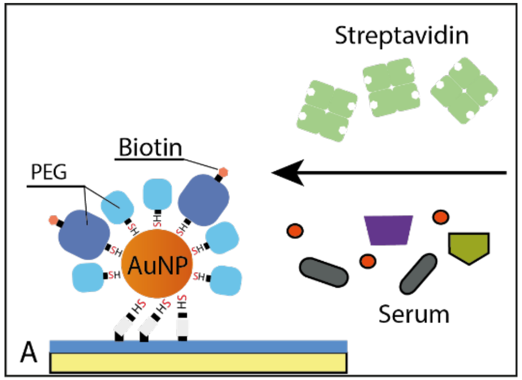
Advantage: We get to know the finer detail – PEGylation is not equally effective against all proteins.

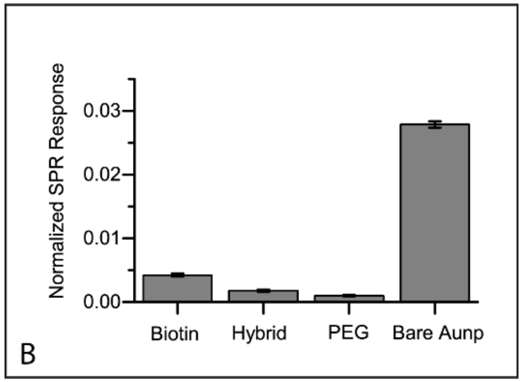
Effect of PEG chain length
(20% human serum)

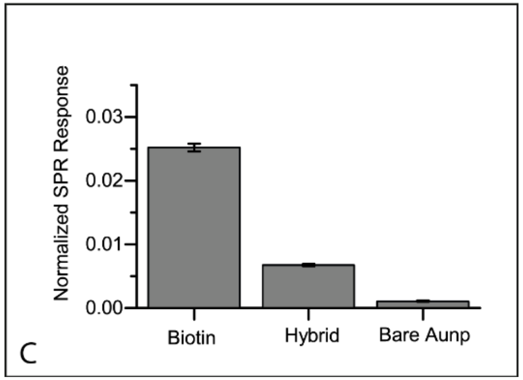


Index:
Black: No PEG
Brown: PEG 2k
Blue: PEG 5k
Green: PEG 10k
Pink: PEG 20k
Orange: PEG 30k

Screening NP based drug formulations







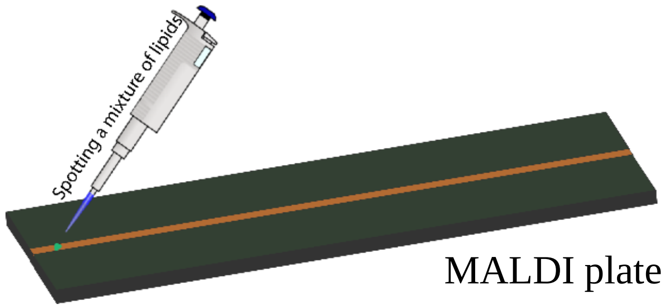
Formation of corona

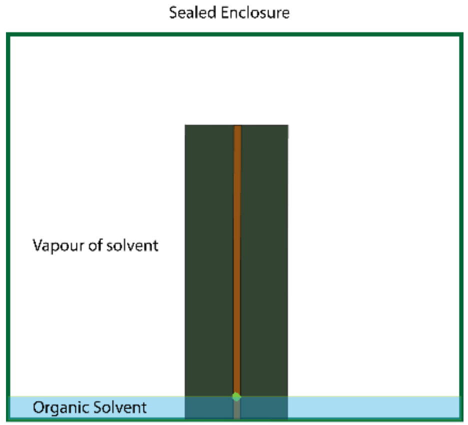
Ability to bind specific target

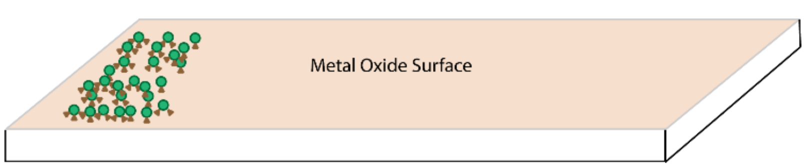
In the future, biologists can screen NP based formulations quickly for key parameters and choose to proceed to cell studies with candidates that clear the screening process. Huge cut in downtime.

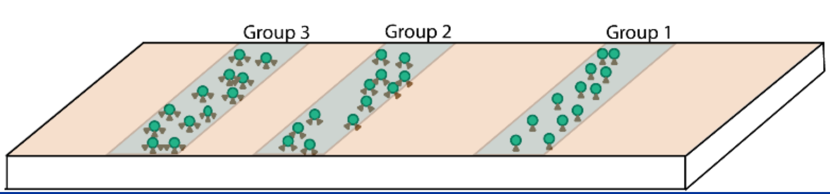
Planar columns of transition metal oxide for on target enrichment of lipids

Schematic Diagram

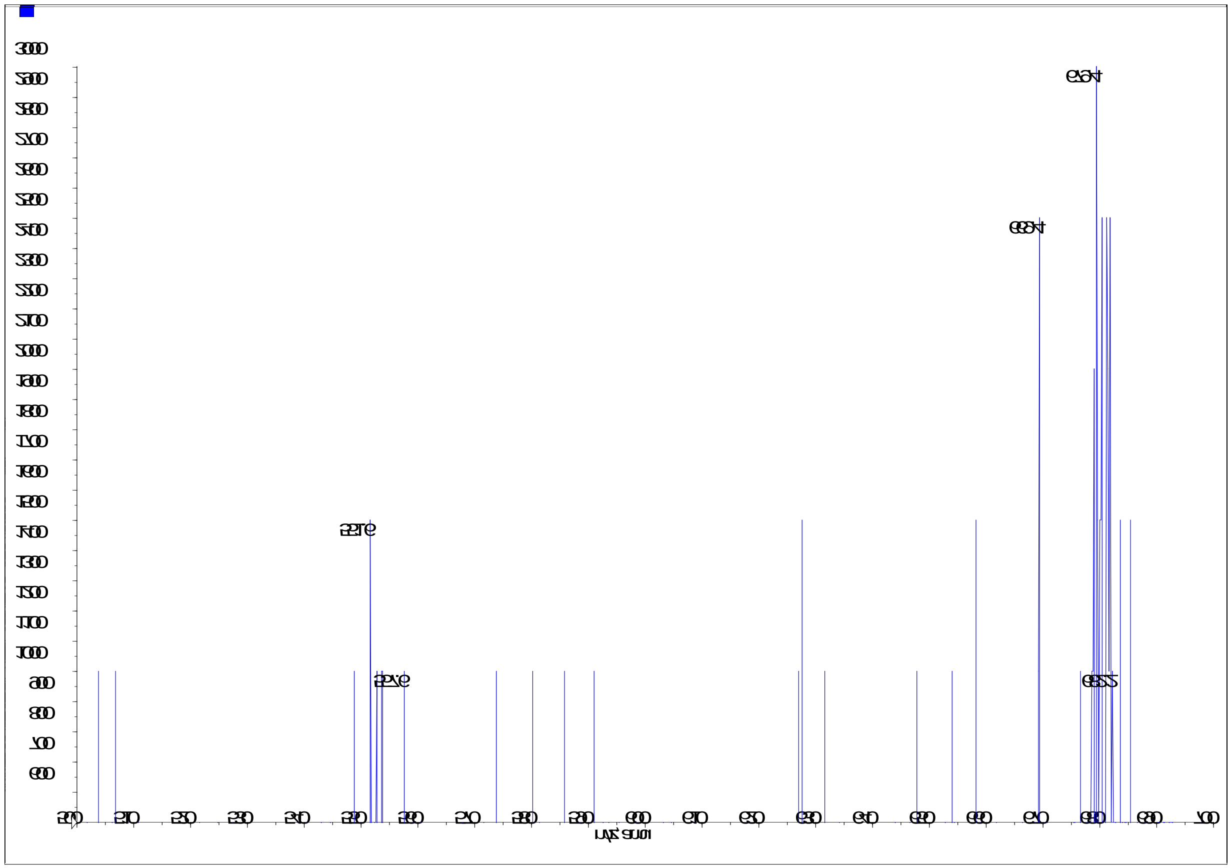








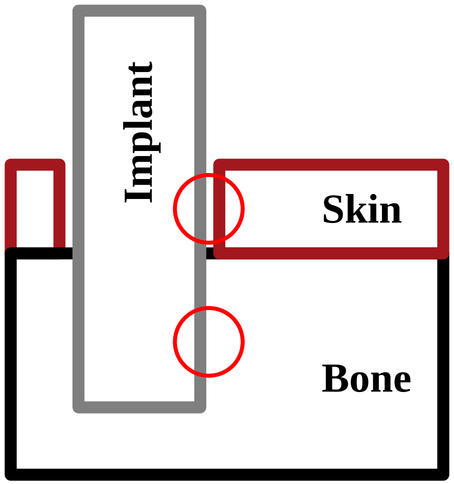
Results



1. Migration of 3 cm observed
2. Different metal oxides can be used to enhance separation distance

Planar columns of transition metal oxide for on target enrichment of multi and mono phosphorylated lipid species prior to analysis with Matrix-Assisted Laser Desorption-Ionization Mass Spectrometry

YSZ for Percutaneous Implant



Implant – skin interface

Implant – bone interface

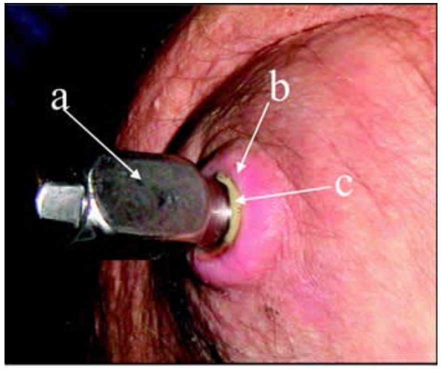
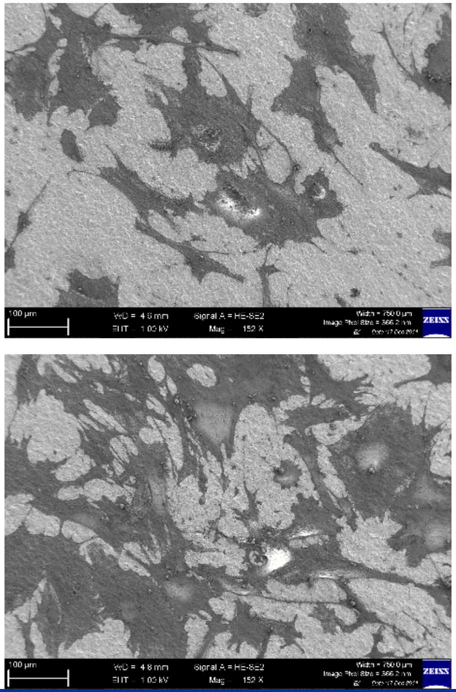


Figure 1.
(a) Solid titanium abutment penetrating the residuum skin. **(b)** Surrounding skin. **(c)** Layer of pus between skin and abutment.

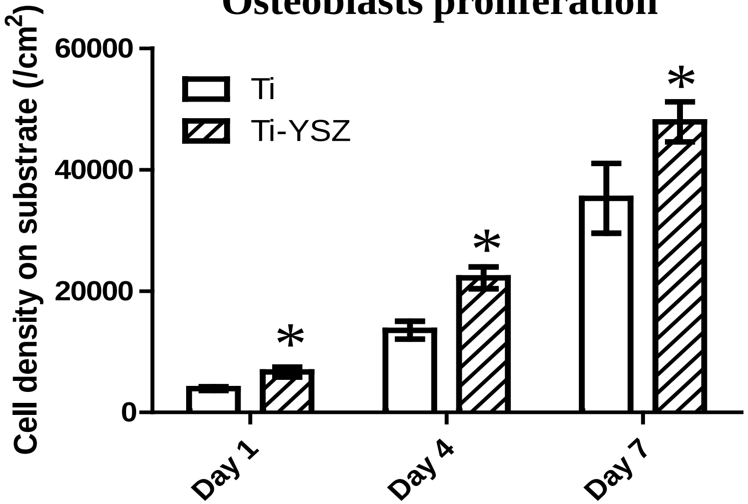
Day 1

Ti

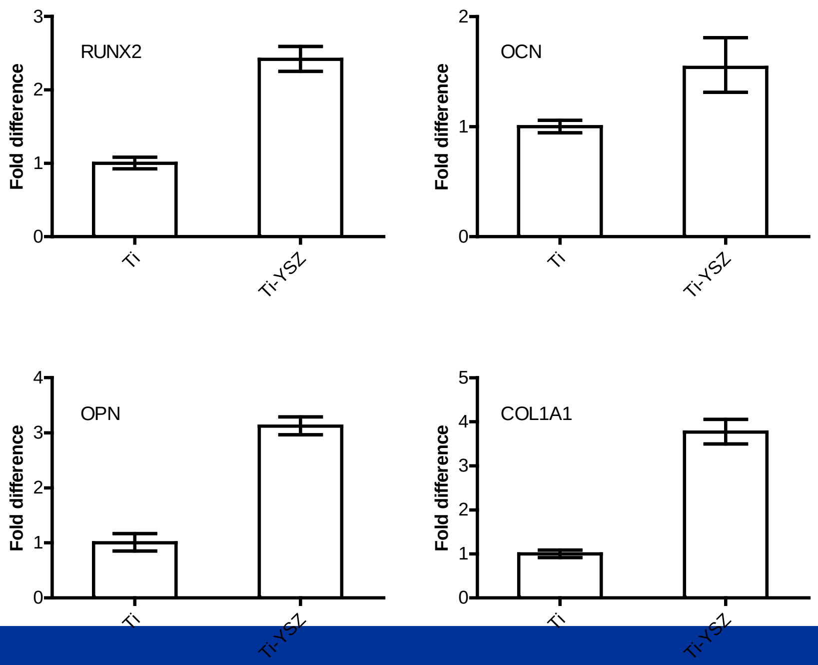
Ti-YSZ



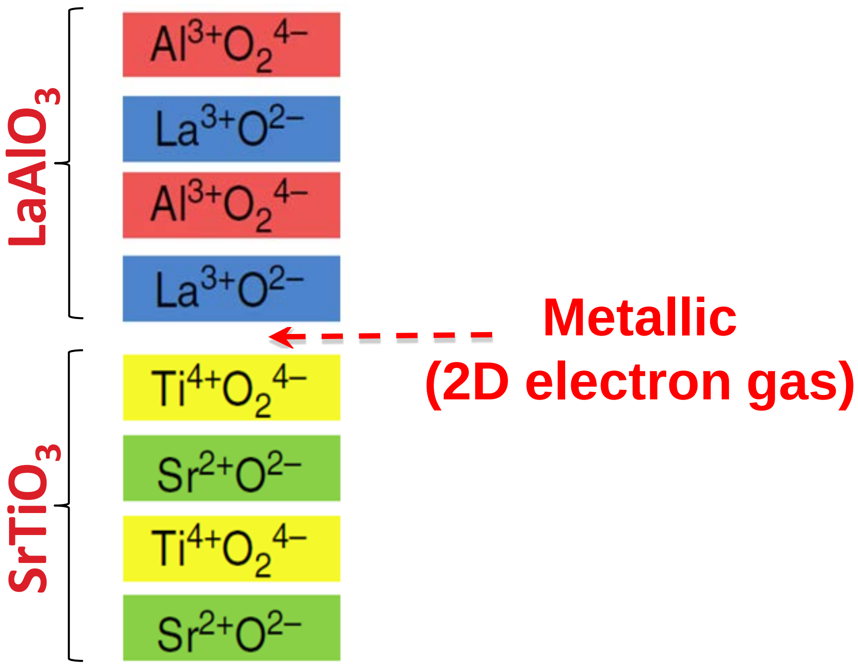
Osteoblasts proliferation



Osteoblasts gene expression



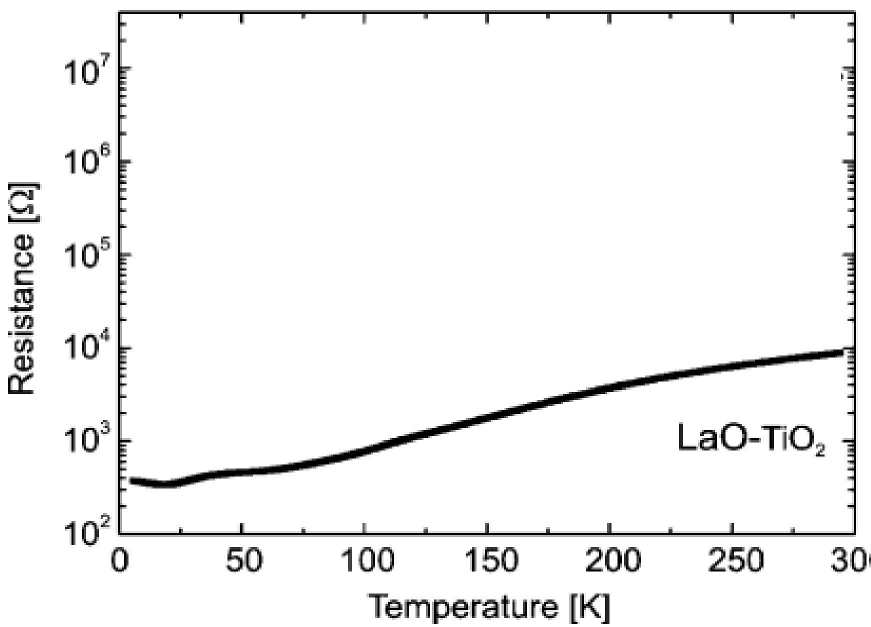
The LAO/STO interface



A high-mobility electron gas at the $LaAlO_3/SrTiO_3$ heterointerface

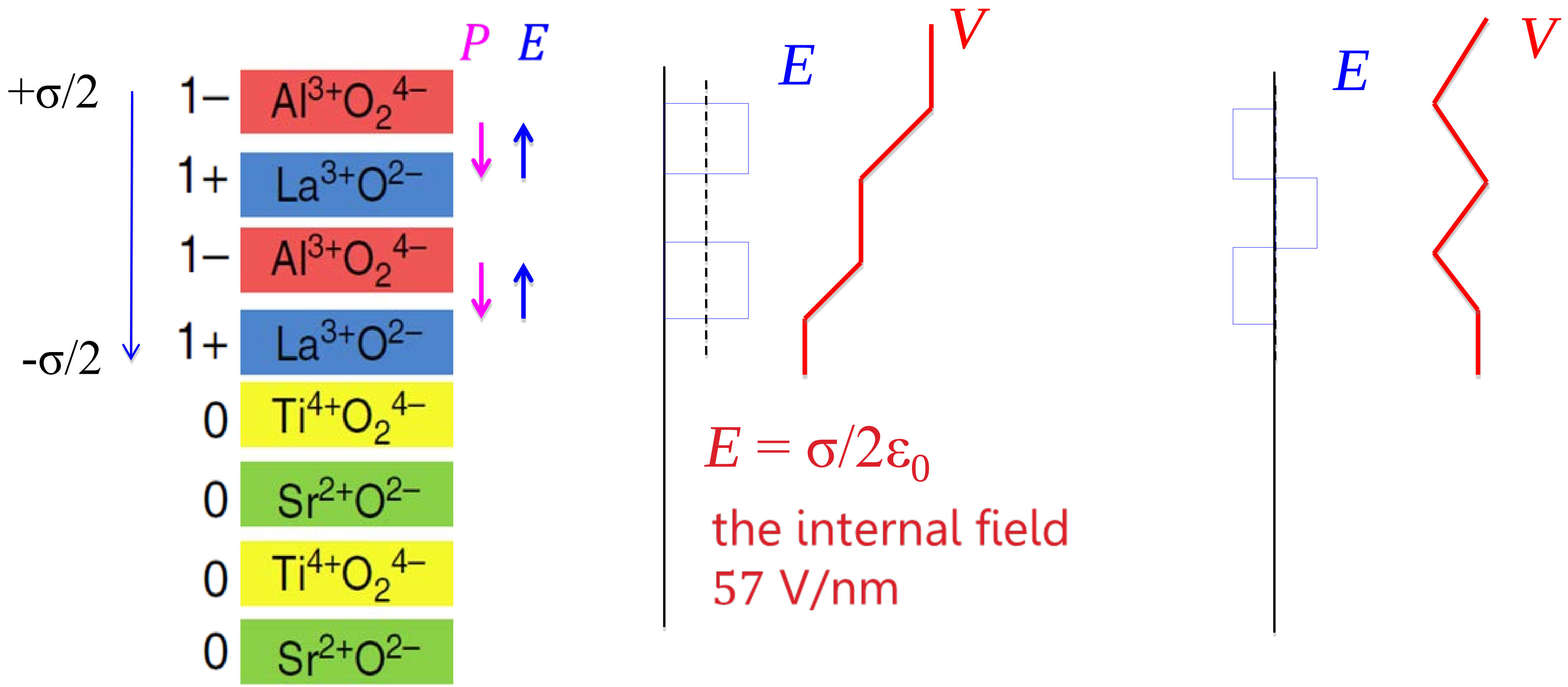
A. Ohtomo[1,2,3] & H. Y. Hwang[1,3,4]

[1] *Bell Laboratories, Lucent Technologies, Murray Hill, New Jersey 07974, USA*
[2] *Institute for Materials Research, Tohoku University, Sendai, 980-8577, Japan*
[3] *Japan Science and Technology Agency, Kawaguchi, 332-0012, Japan*
[4] *Department of Advanced Materials Science, University of Tokyo, Kashiwa, Chiba, 277-8651, Japan*



Polarization catastrophe

Electronic reconstruction



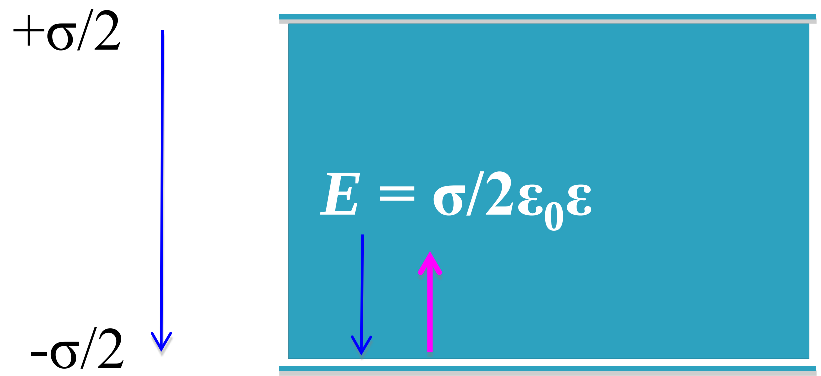
Ohtomo & Hwang, *Nature* **427**, 423 (2004)

Nakagawa, Hwang and Muller, *Nature Materials* **5**, 204 (2006)

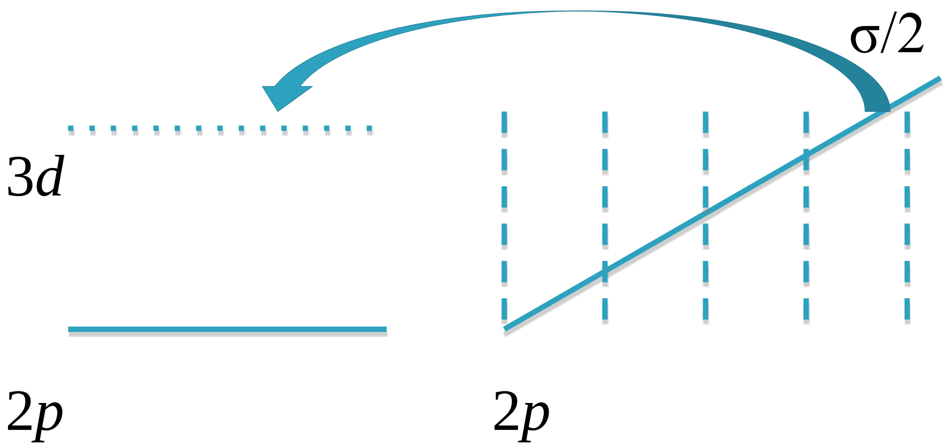
“Irresponsible Physicist Deposits Excess Polar layers on top of Non-polar substrates”

Where does the -σ/2 charge come from ?

$$E_{ext} = \sigma/2\varepsilon_0$$



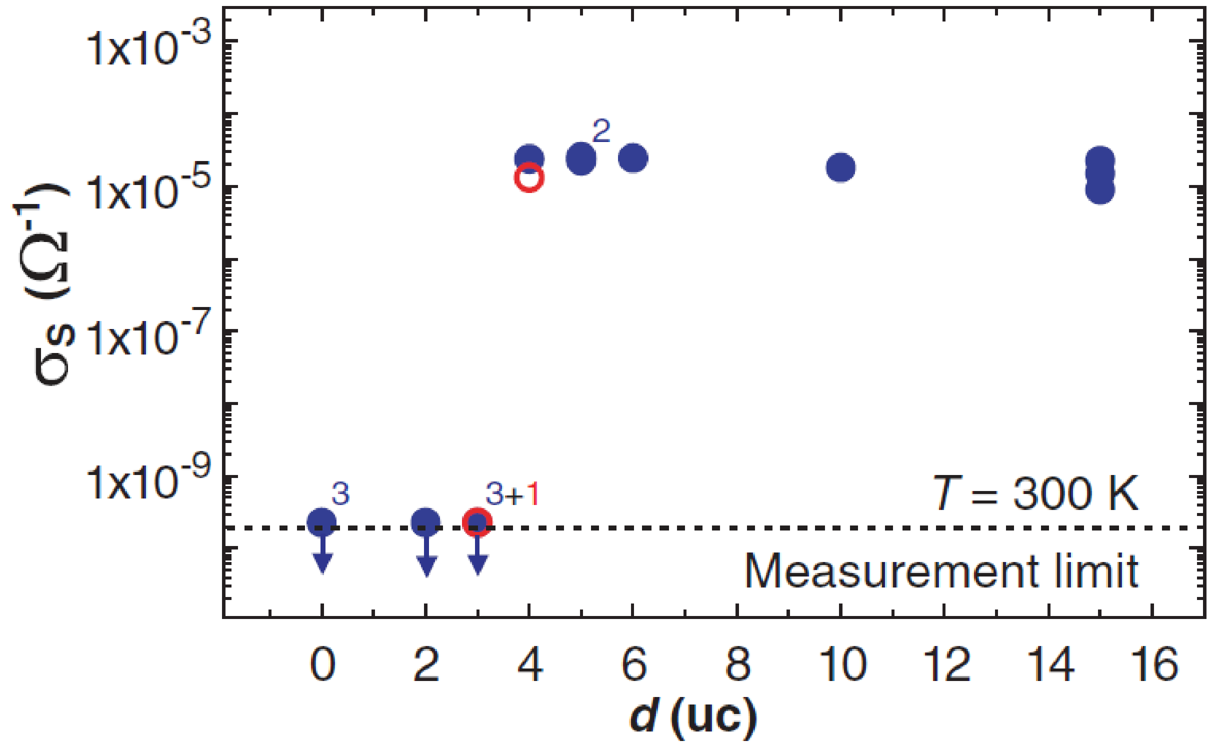
The internal field is reduced by a factor ε from 57×10^9 V m^{-1} to 2.4 V nm^{-1} or **0.9 V per unit cell**.

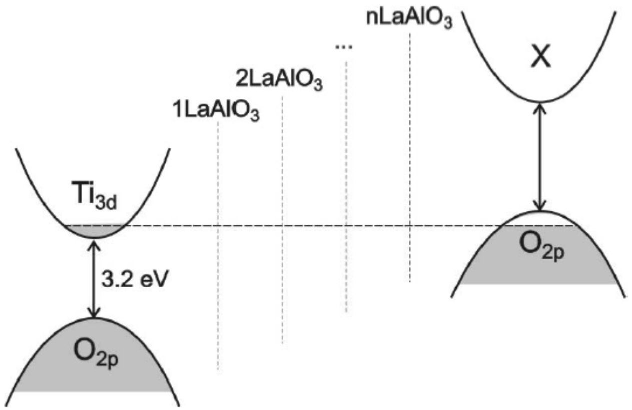


Charge transfer at the interface needed to avert the polar catastrophe is **0.5 e/uc** or **3.3×10^{14} electrons cm^{-2}**

Critical thickness for the conductivity

Thiel *et al.*, *Science* **313**, 1942 (2006)





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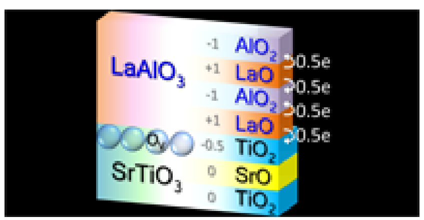
Phys. Rev. X 3, 021010 (2013) [9 pages]

Origin of the Two-Dimensional Electron Gas at $LaAlO_3/SrTiO_3$ Interfaces: The Role of Oxygen Vacancies and Electronic Reconstruction



Popular Summary: Insulating polar oxides, consisting of charged layers [e.g., (100) $LaAlO_3$ (LAO) as layers of LaO^{+1} and AlO_2^{-1}], have generated a great deal of excitement in the last decade. At the interface of the polar LAO with a nonpolar insulating oxide $SrTiO_3$ (STO), a two-dimensional electron g… Read Full Popular Summary

Abstract	References	No Citing Articles	Supplemental Material

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Z. Q. Liu[1,2], C. J. Li[1,3], W. M. Lü[1,*], X. H. Huang[4], Z. Huang[1], S. W. Zeng[1,2], X. P. Qiu[4], L. S. Huang[5], A. Annadi[1,2], J. S. Chen[5], J. M. D. Coey[1,6], T. Venkatesan[1,2,3,4], and Ariando[1,2,†]
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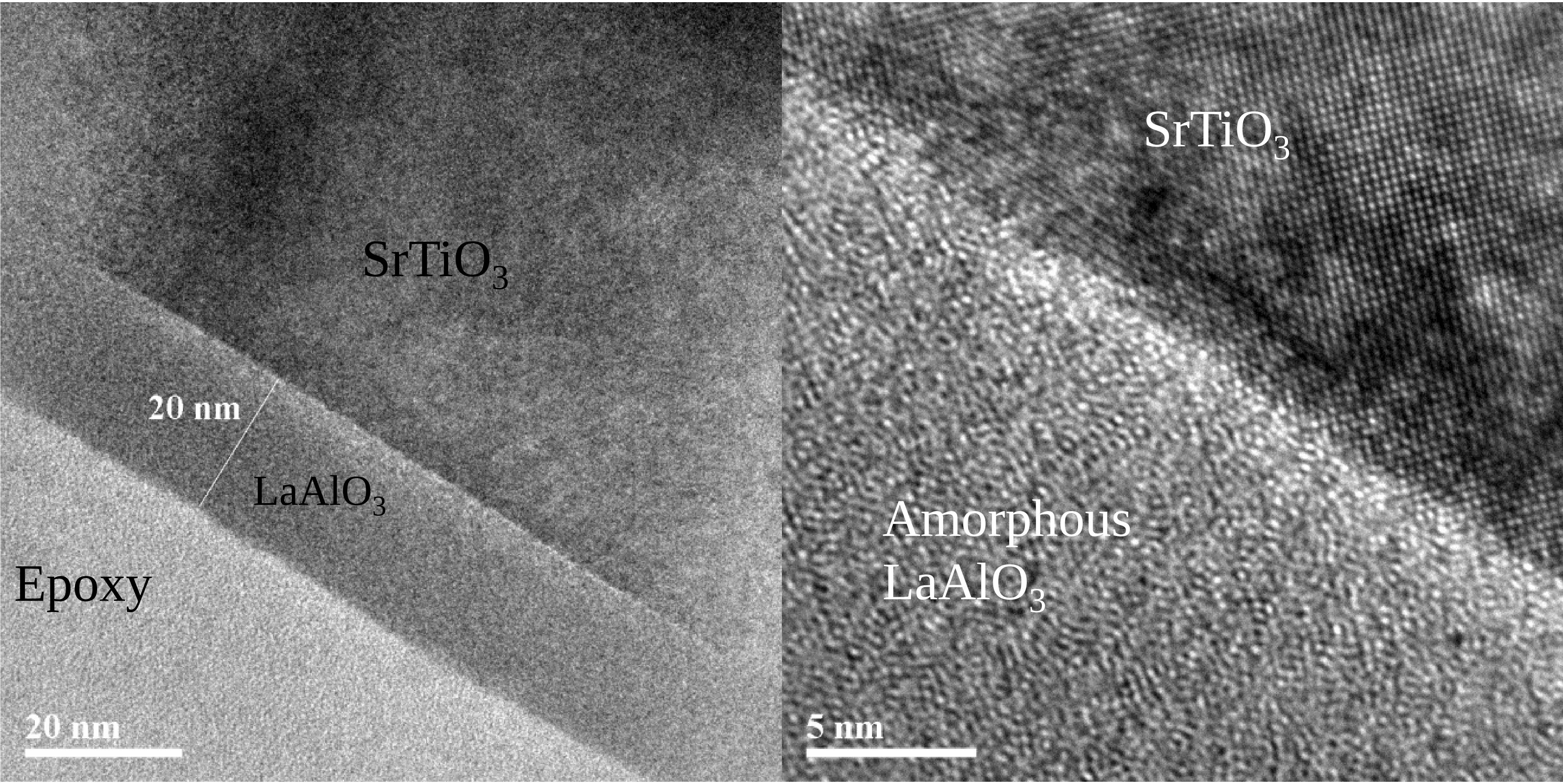
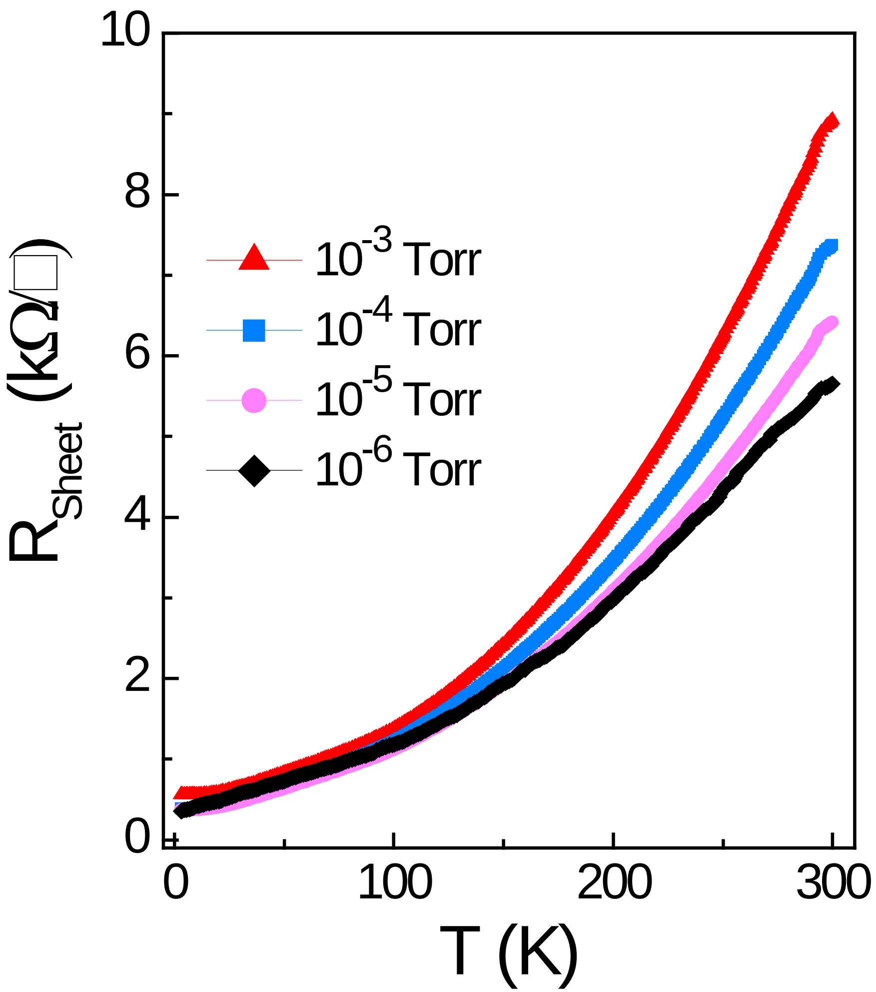
[5]*Department of Material Science and Engineering, National University of Singapore, 117576 Singapore*
[6]*Department of Pure and Applied Physics, Trinity College, Dublin 2, Ireland*

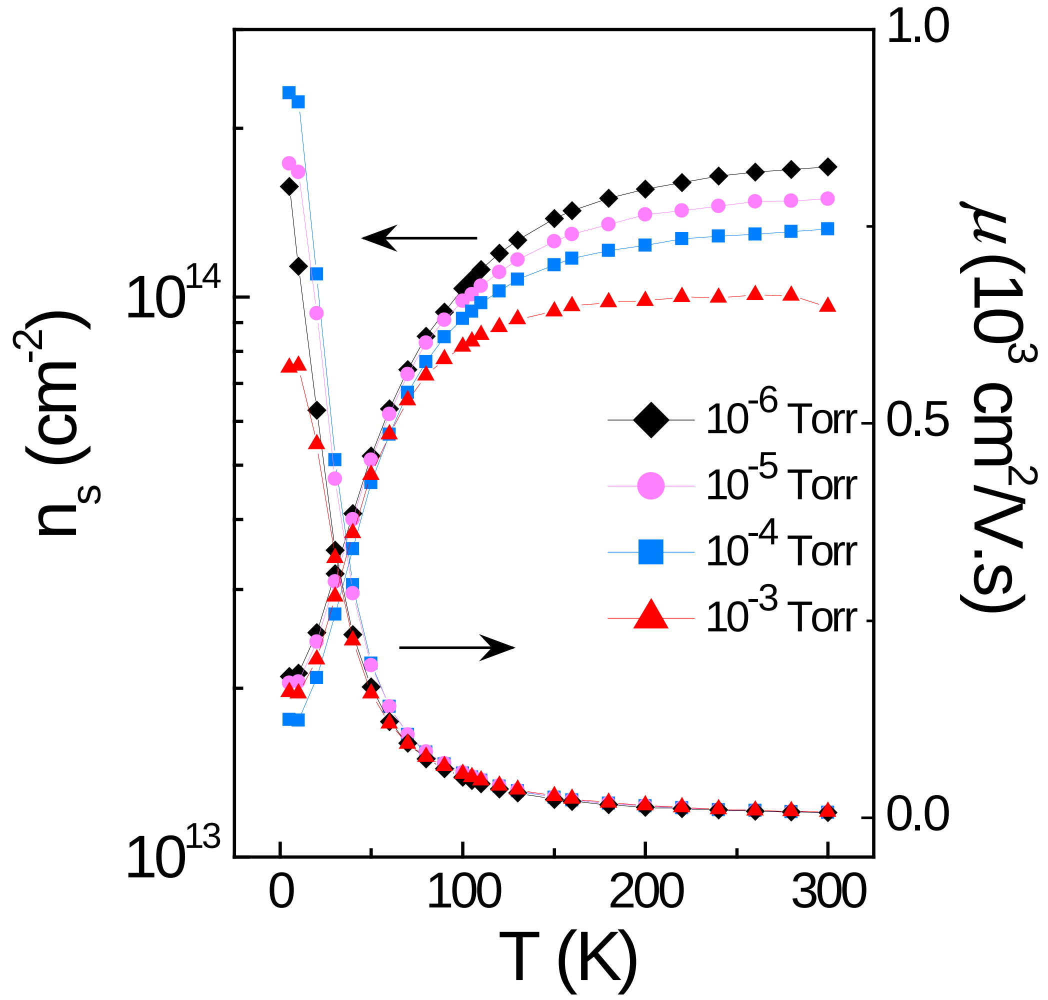
Amorphous LAO/STO Interface



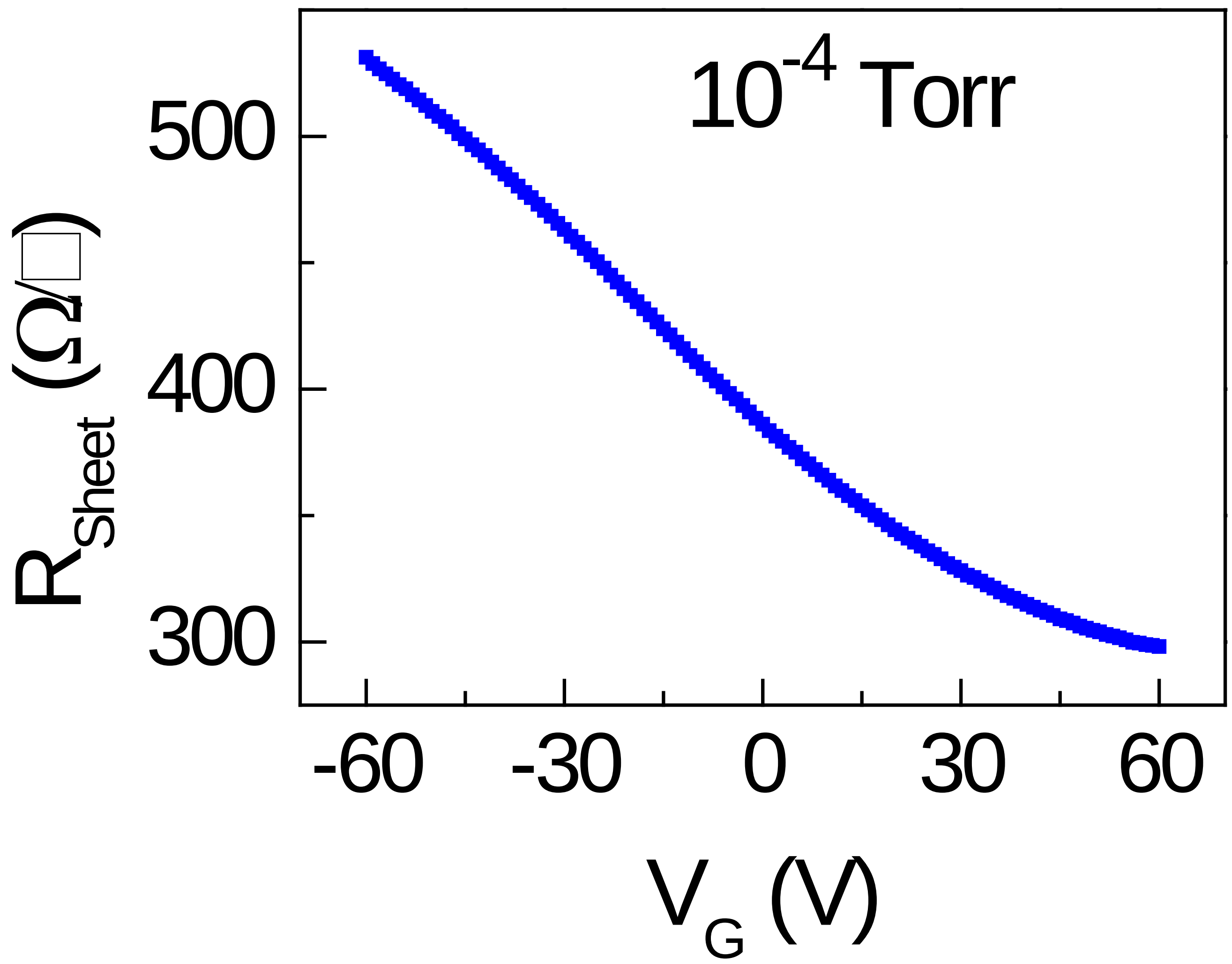
Room temperature deposition at 10^{-6} Torr

Electrical transport of amorphous LAO/STO



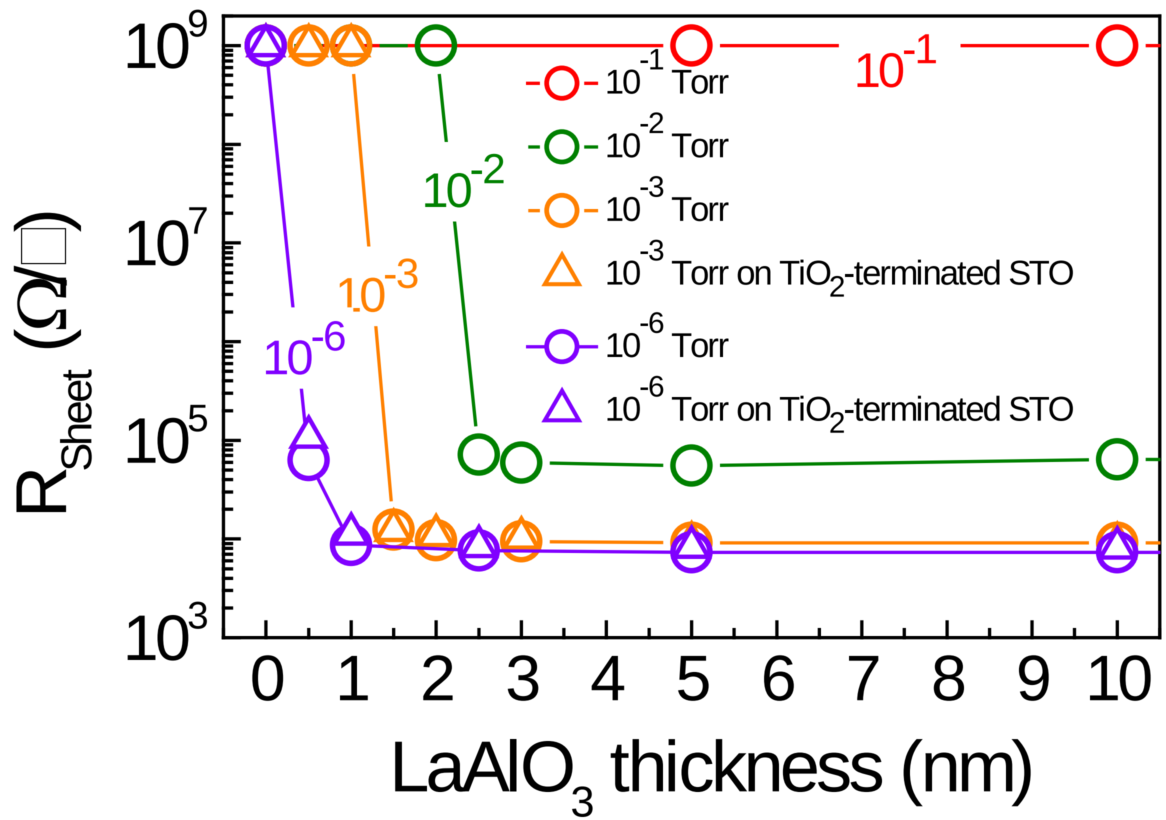


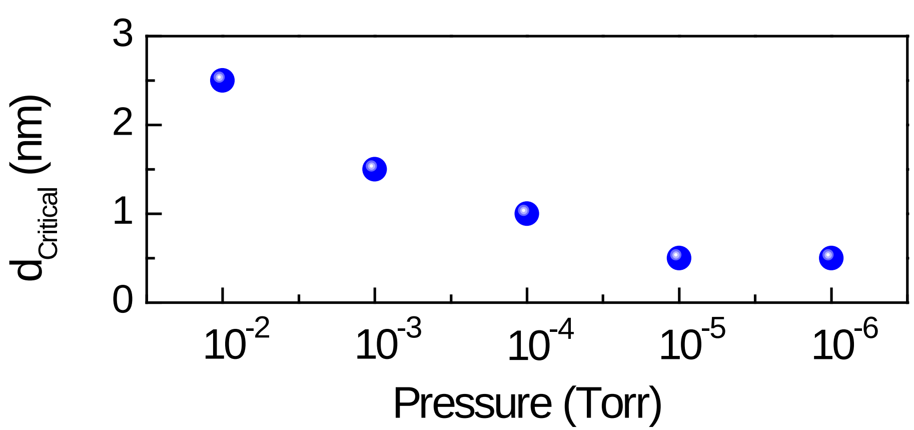
Electric field effect



Back gate voltage dependence

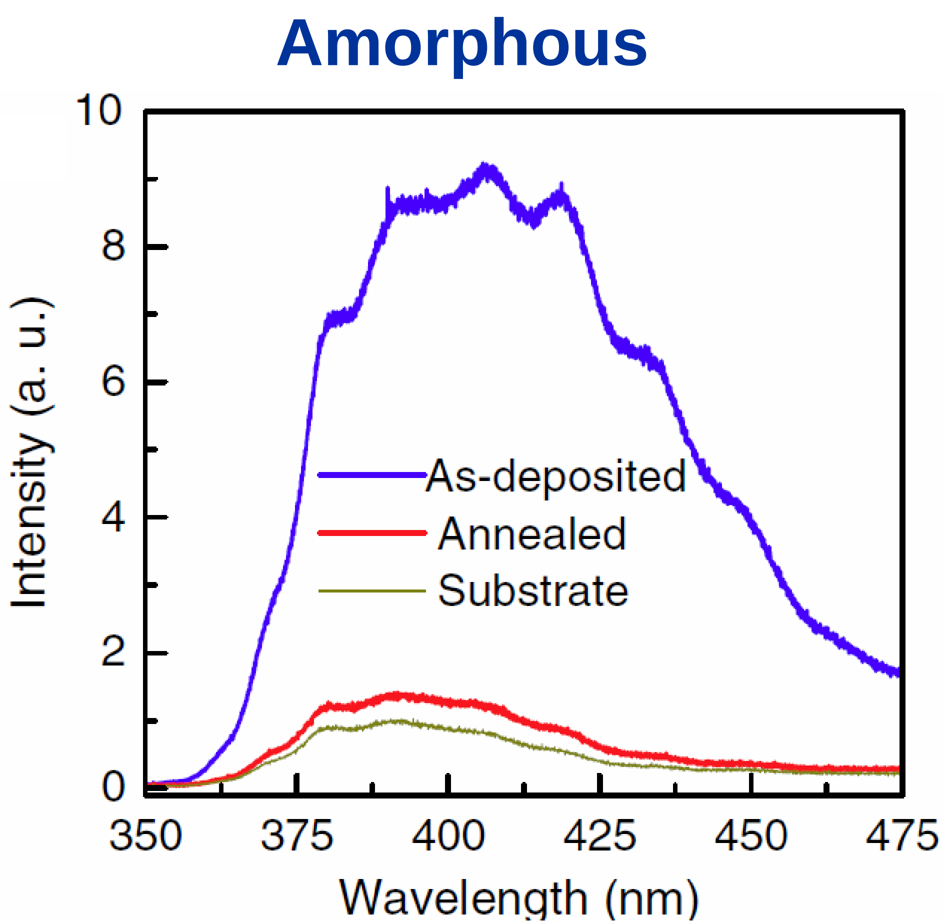
Critical thickness

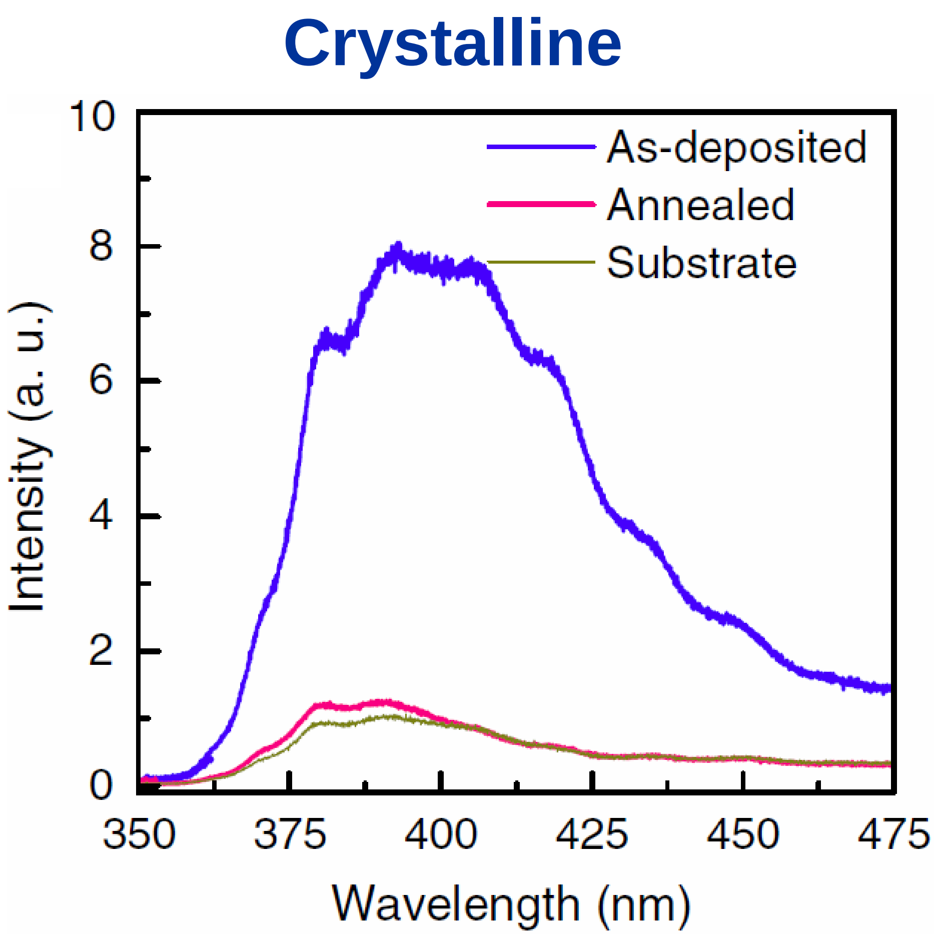




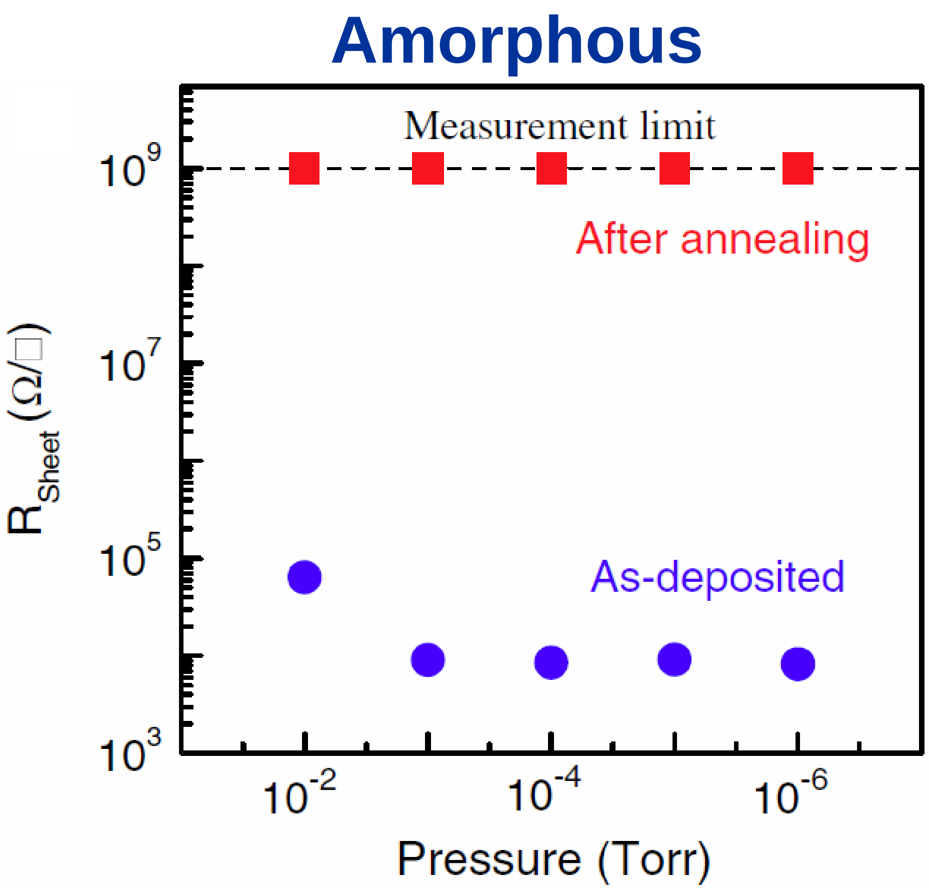
- Oxygen partial pressure
- Laser fluence
- Substrate – target distance
- …

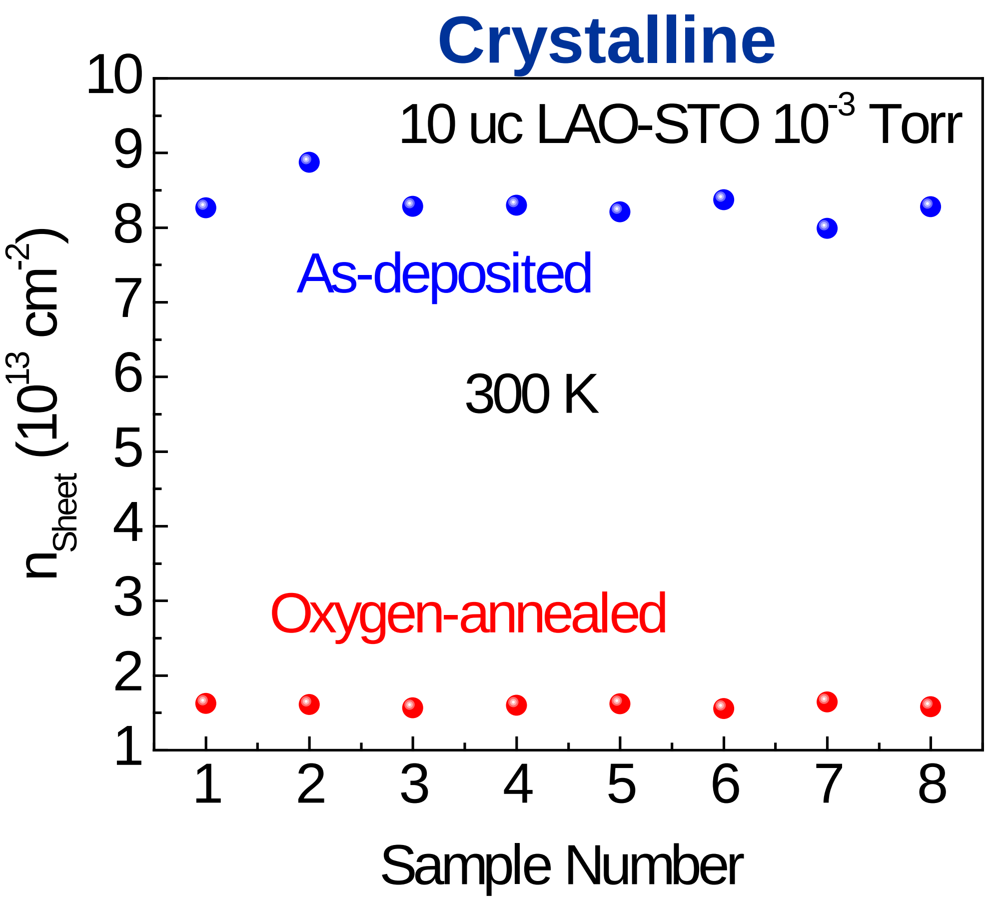
Photoluminescence





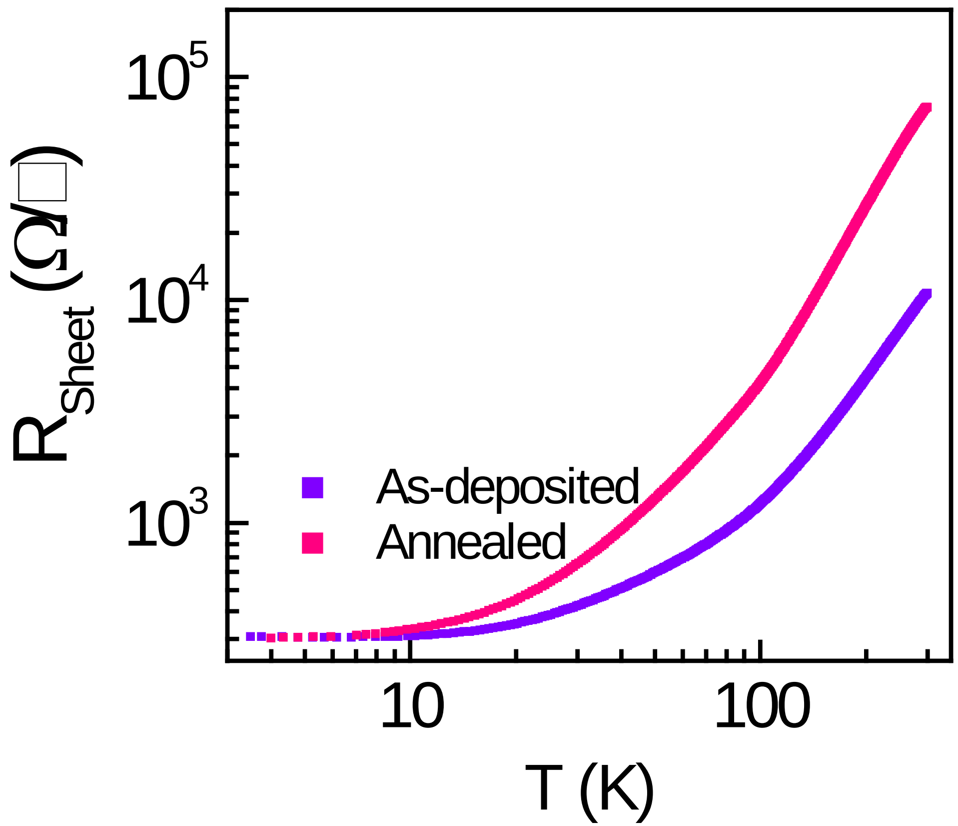
After annealing - resistance

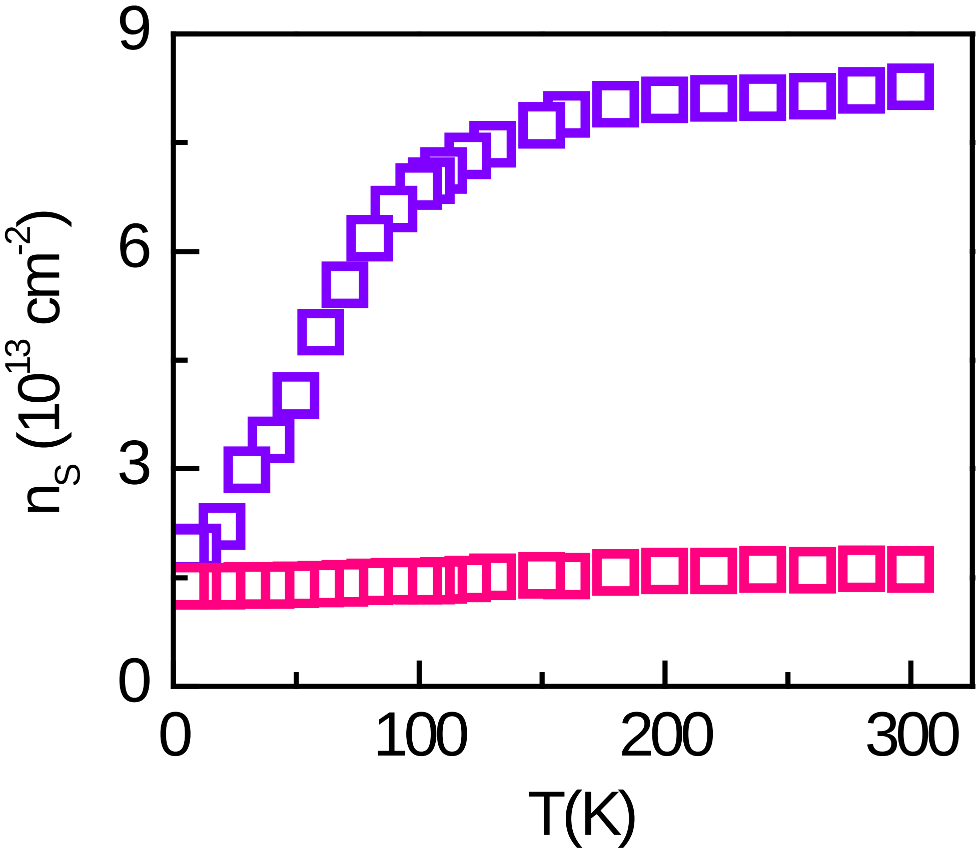




Crystalline samples - after annealing

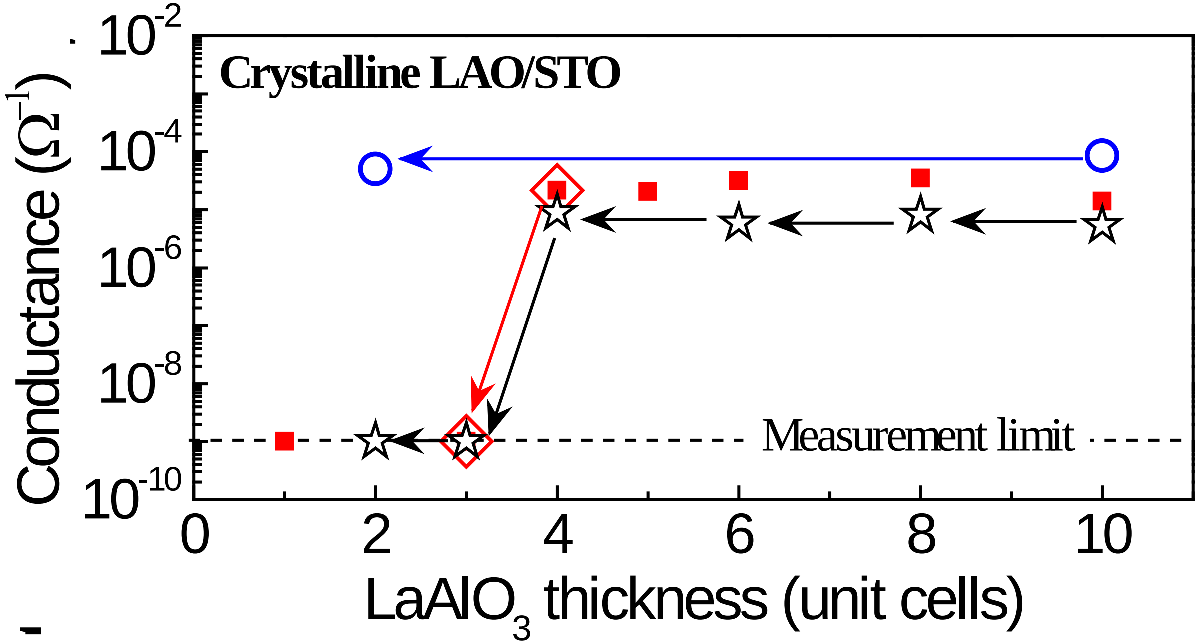
Activation energy E = 4.2 meV

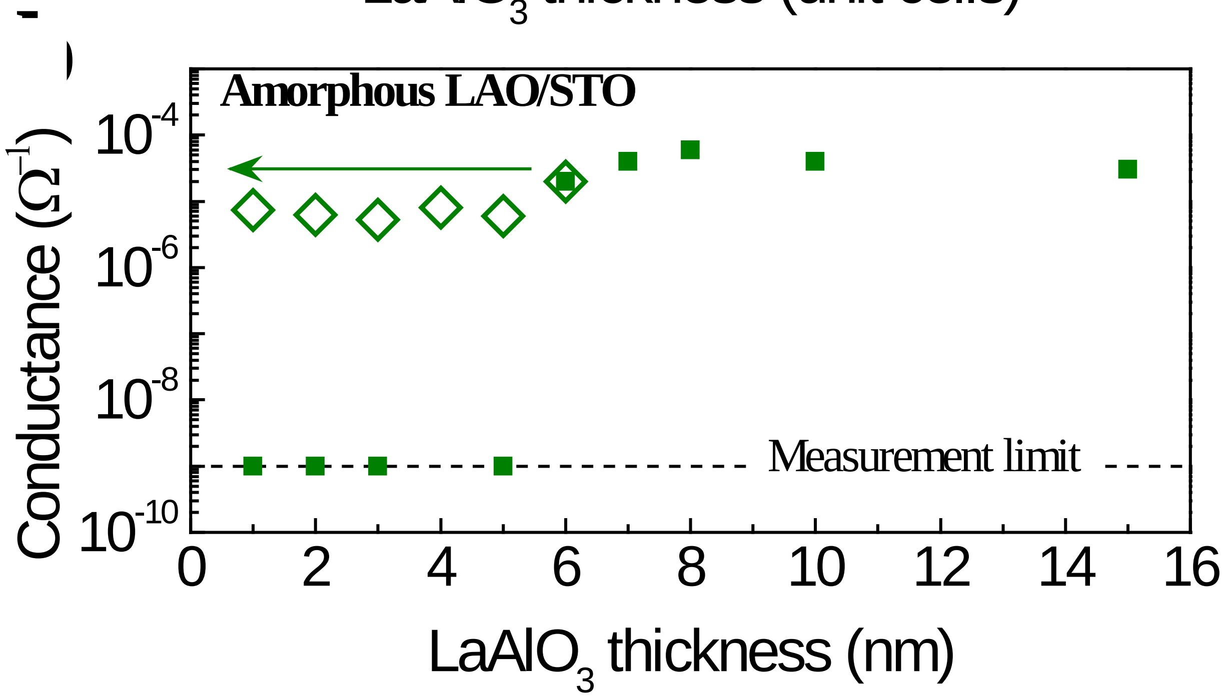




Activation energy E = 0.5 meV

Argon milling experiment





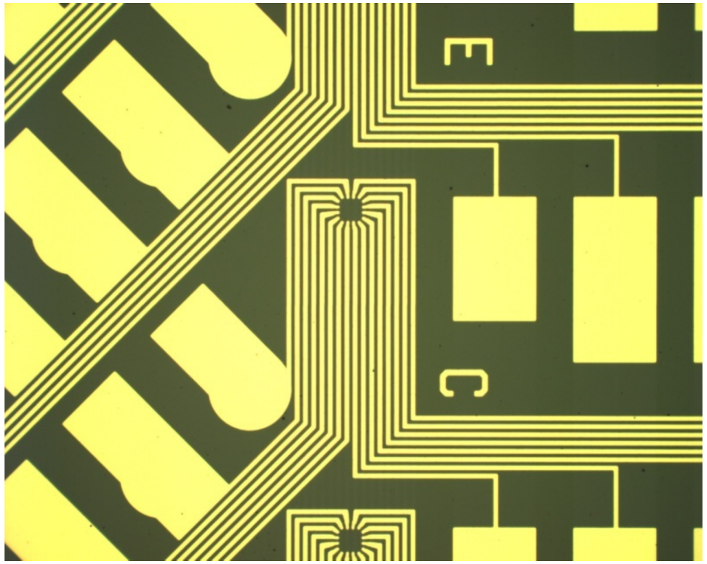
unannealed

vs.

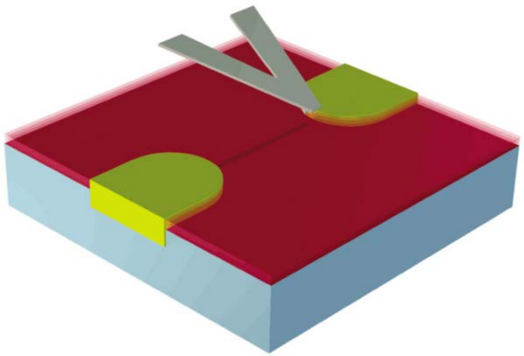
annealed

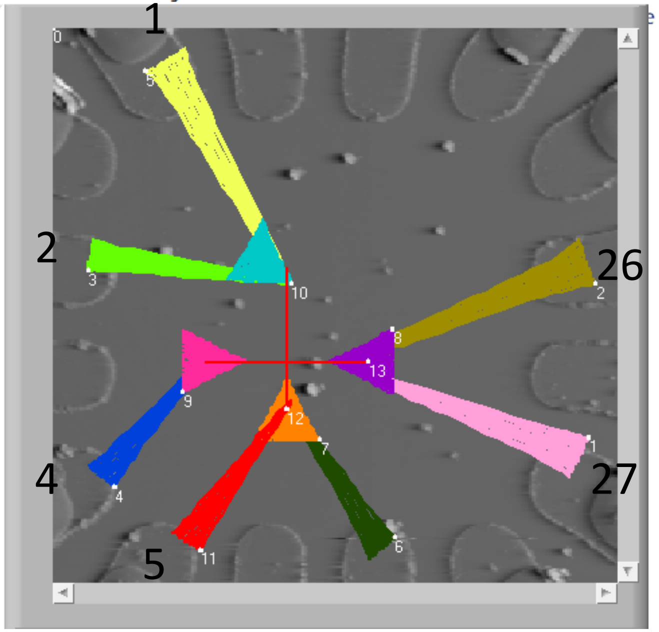
Percolation carrier density of ~10^{13} cm^{-2} (r_0 of a few nm, $\epsilon_r = 300$, $m^* = 5m_e$)

Anisotropic 1D superconductivity?

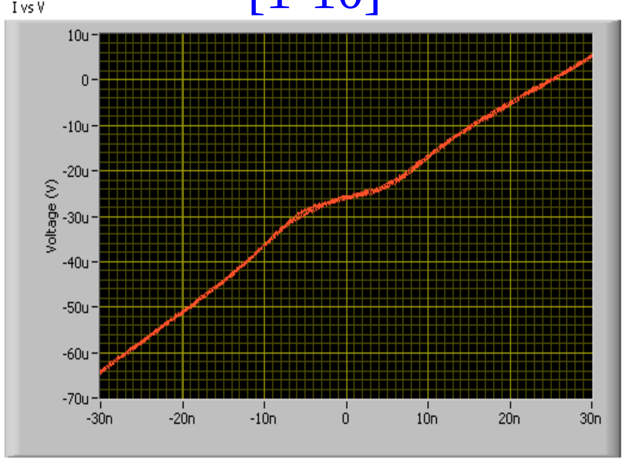


AFM written 7 nm wide conducting line on the 3.50uc LAO/STO(110) samples



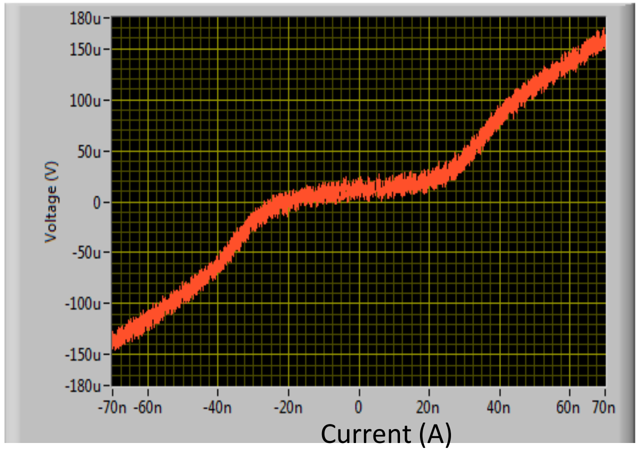


[1-10]



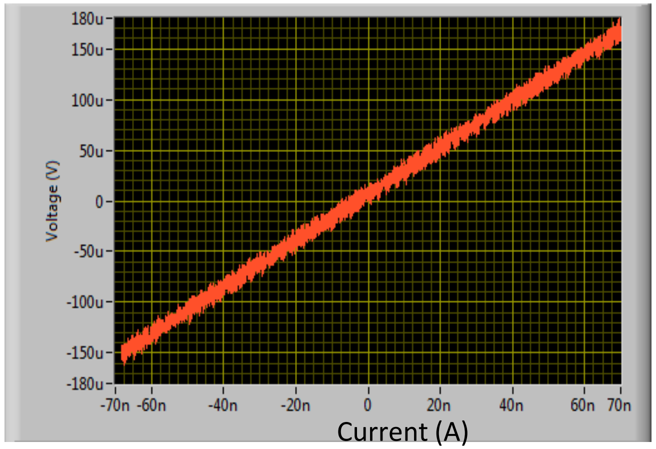
50 mK, 0 Oe

[001]



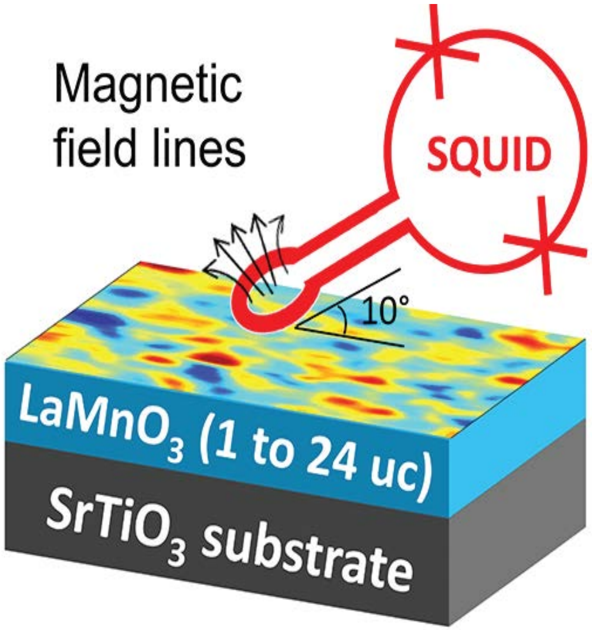
50 mK, 0 Oe

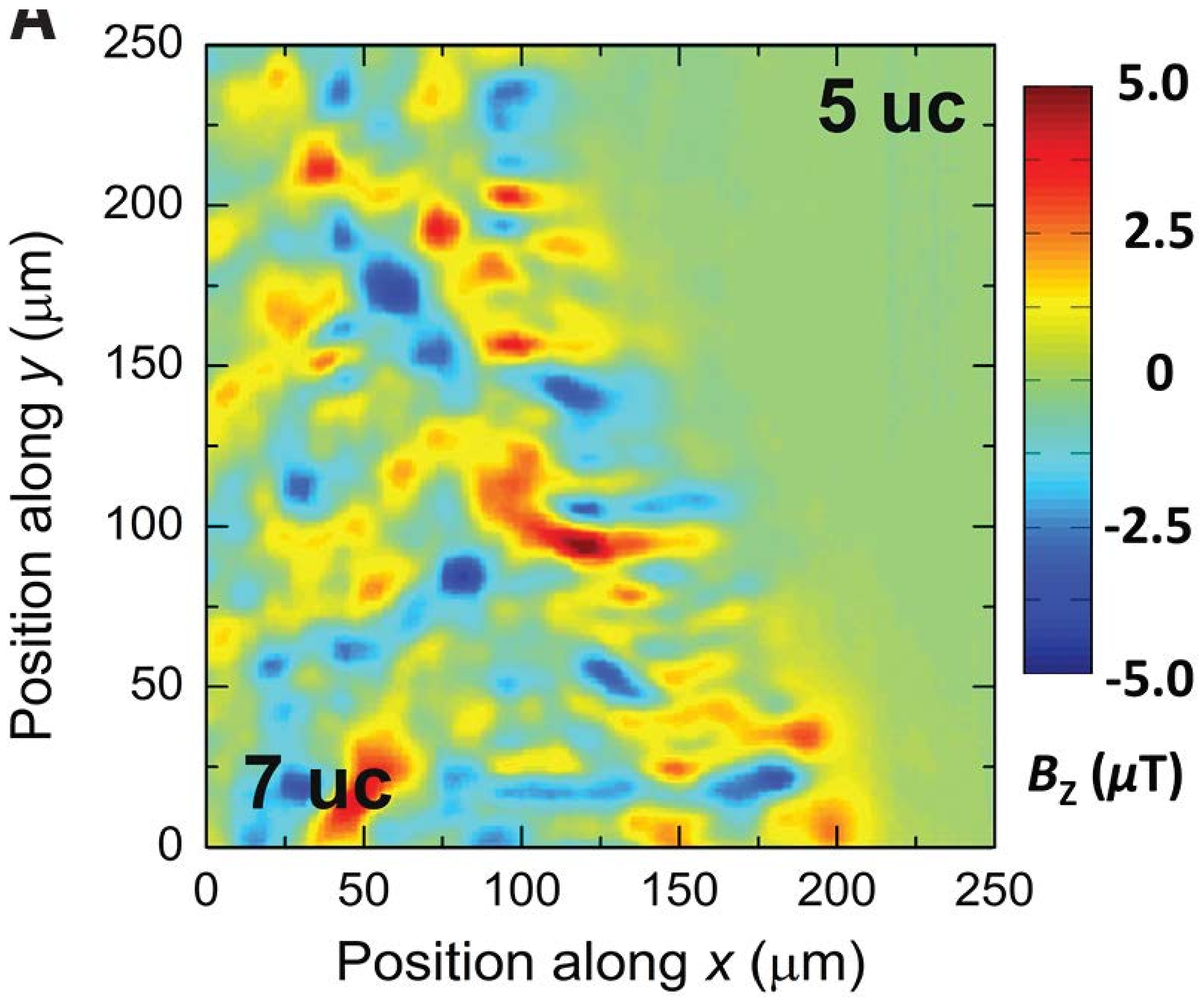
[001]



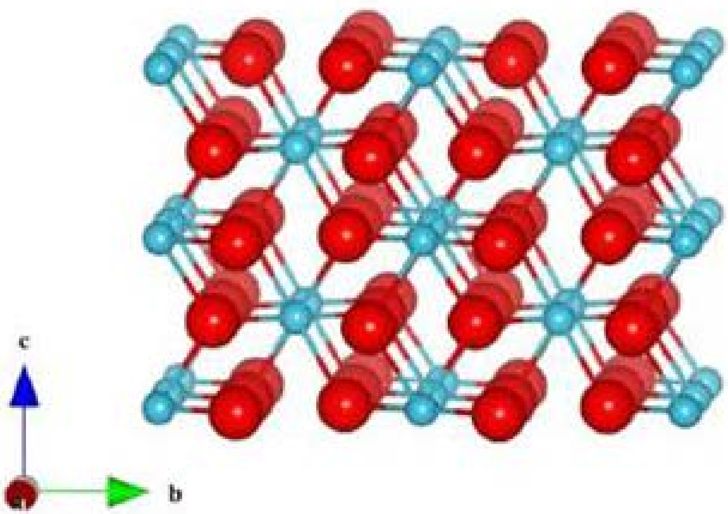
50 mK, 3000 Oe

Magnetic Field Imaging of LMO Layers

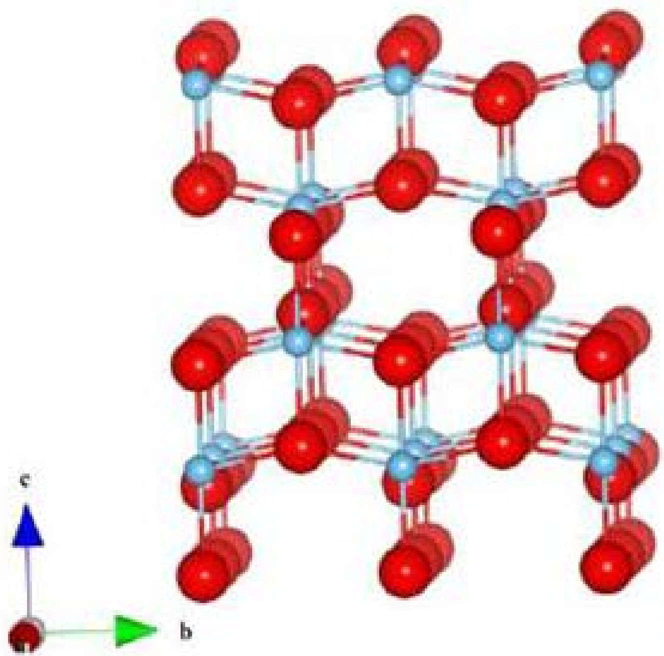




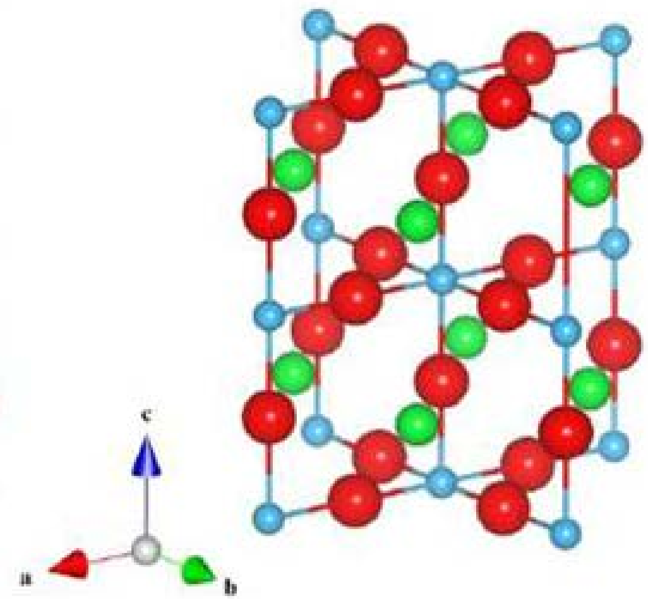
Crystal structure of rutile TiO_2, anatase and $SrTiO_3$



Rutile TiO_2

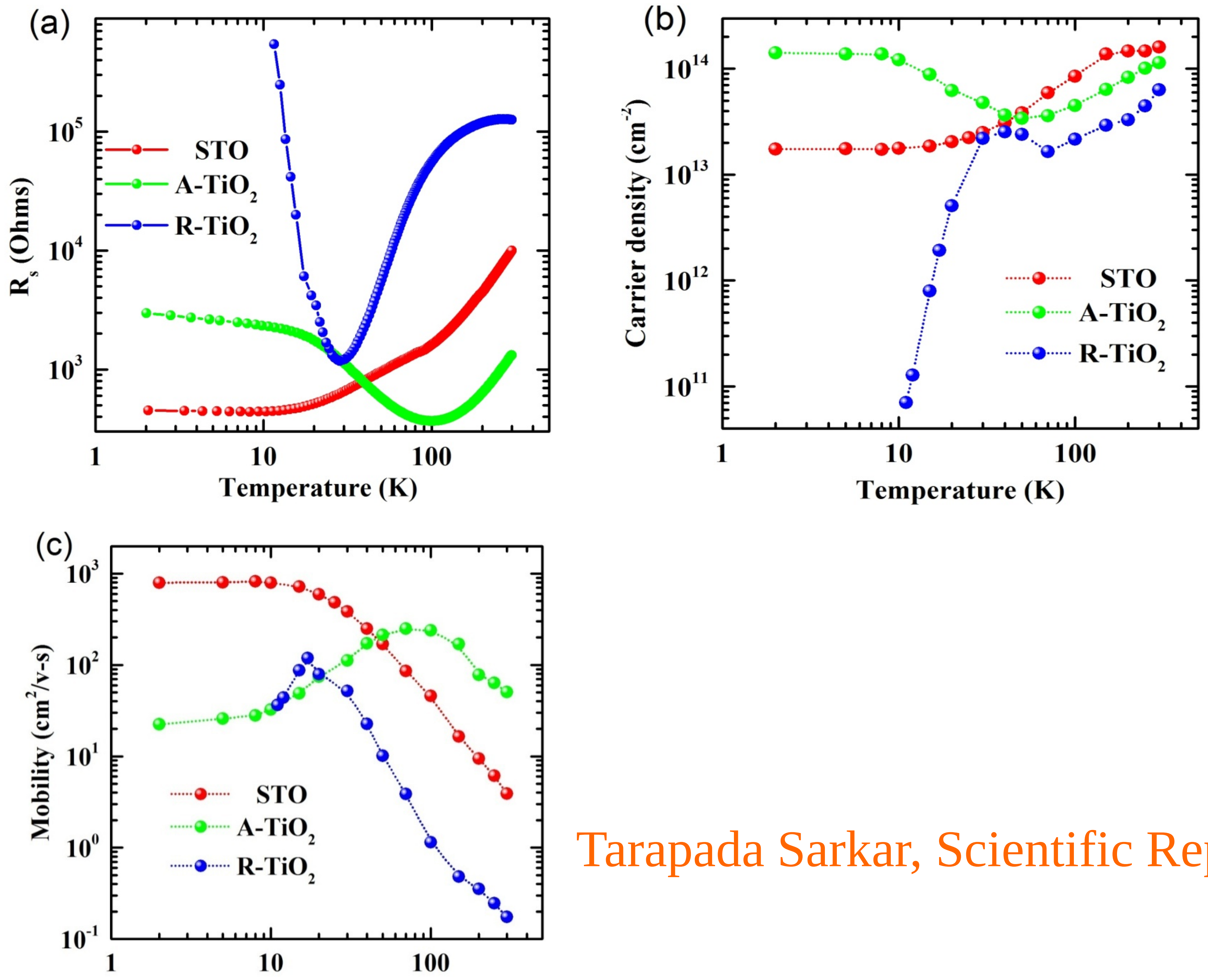


Anatase TiO_2



Perovskite $SrTiO_3$

Electrical transport



Tarapada Sarkar, Scientific Reports

Before I Forget the Memory Application

2 unit cells of BTO appear to be ferroelectric

Theoretically below 3 uc BTO should not be ferroelectric

Ferroelectric tunnel Junctions can be prepared on silicon

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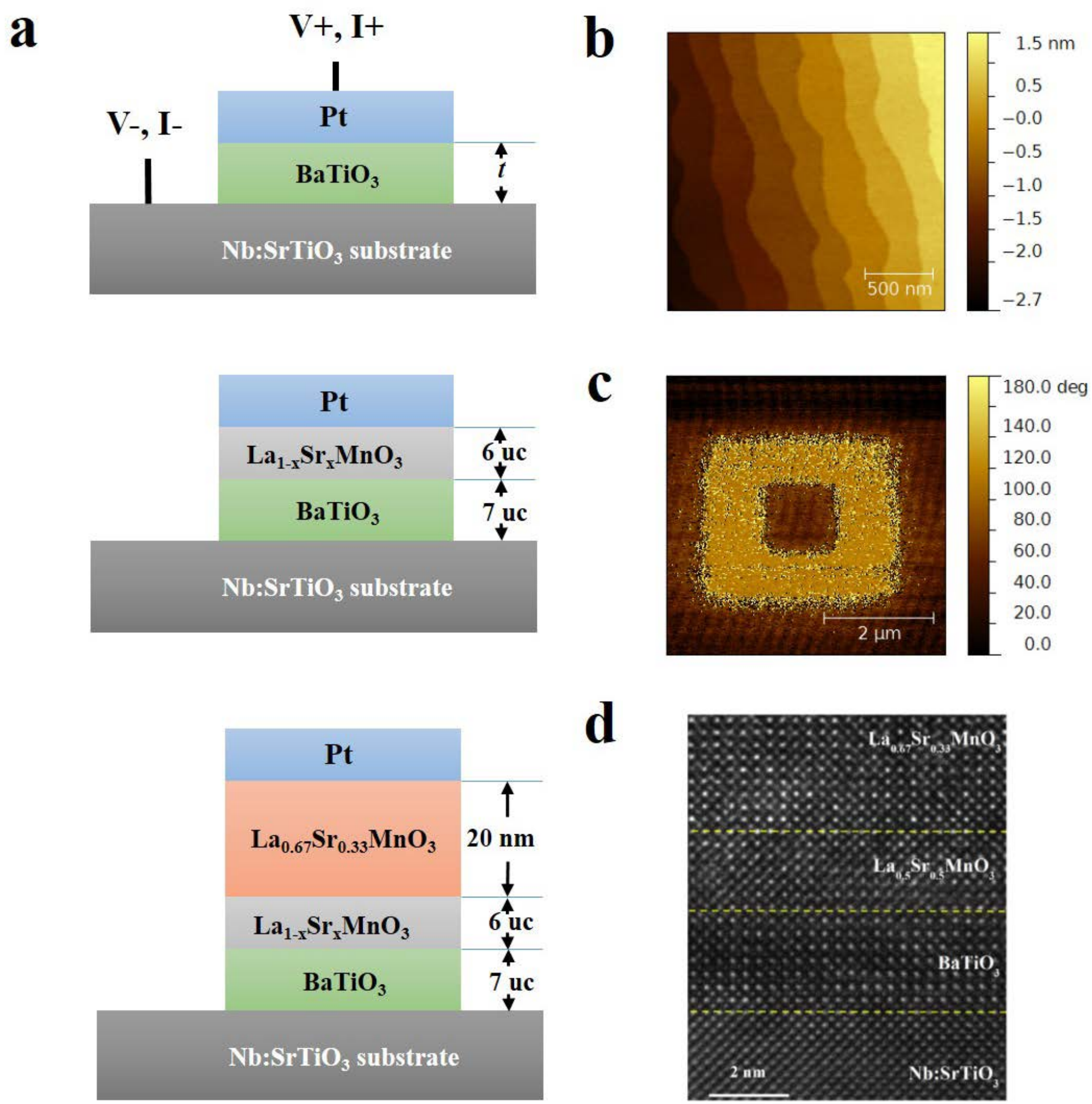
Device Structure



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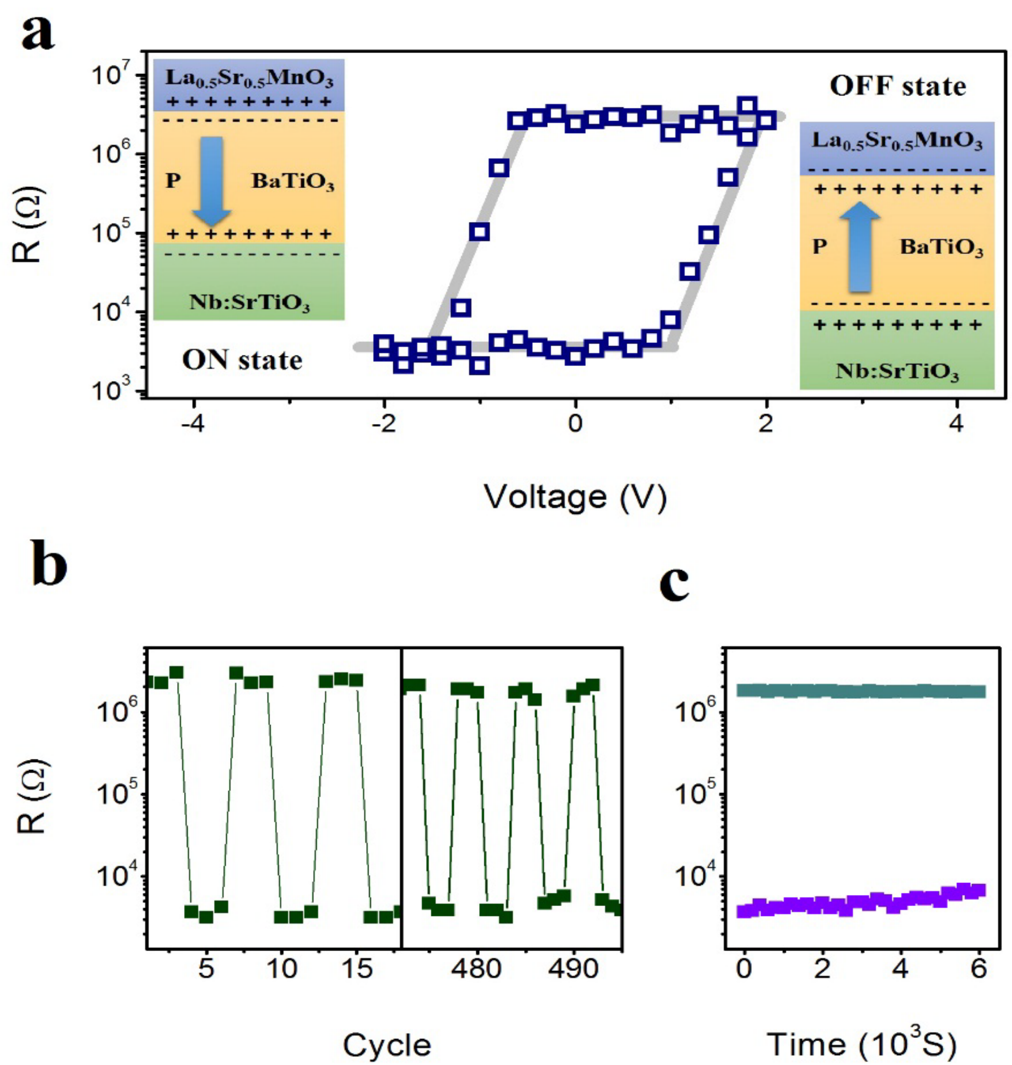
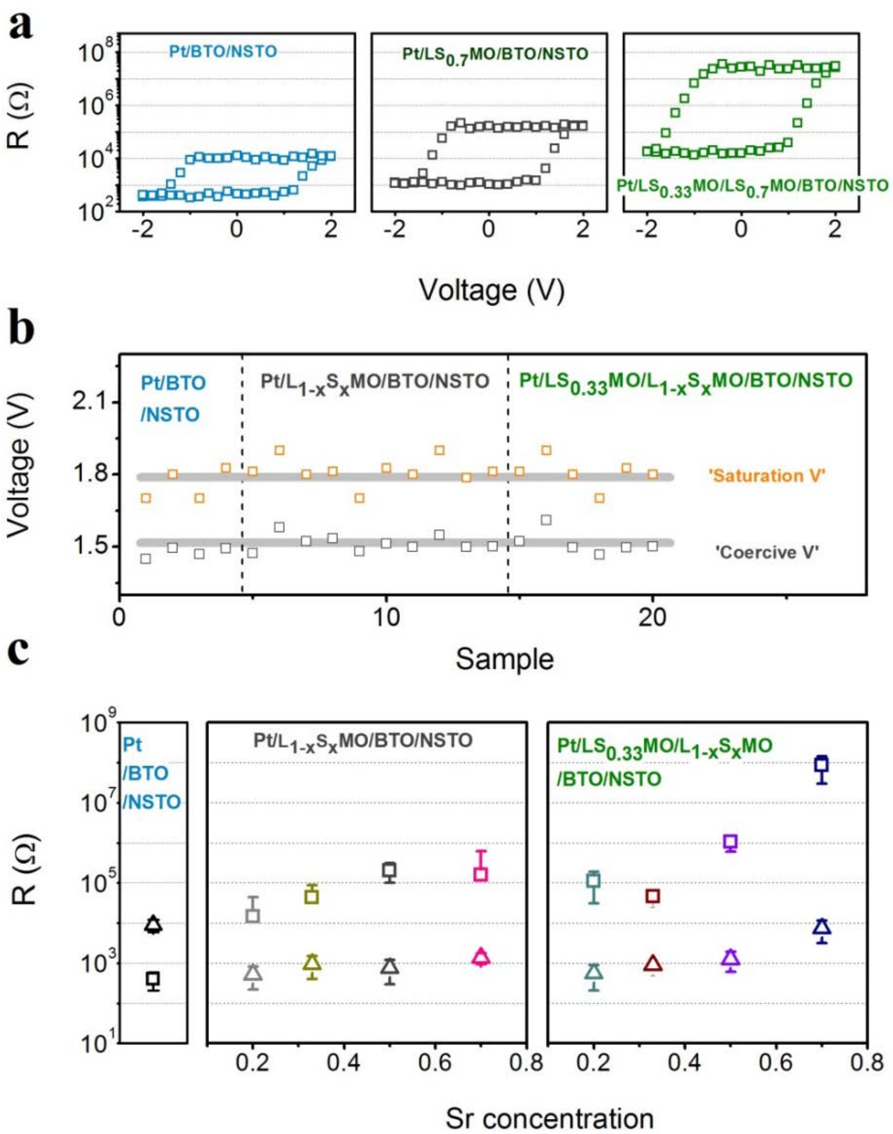
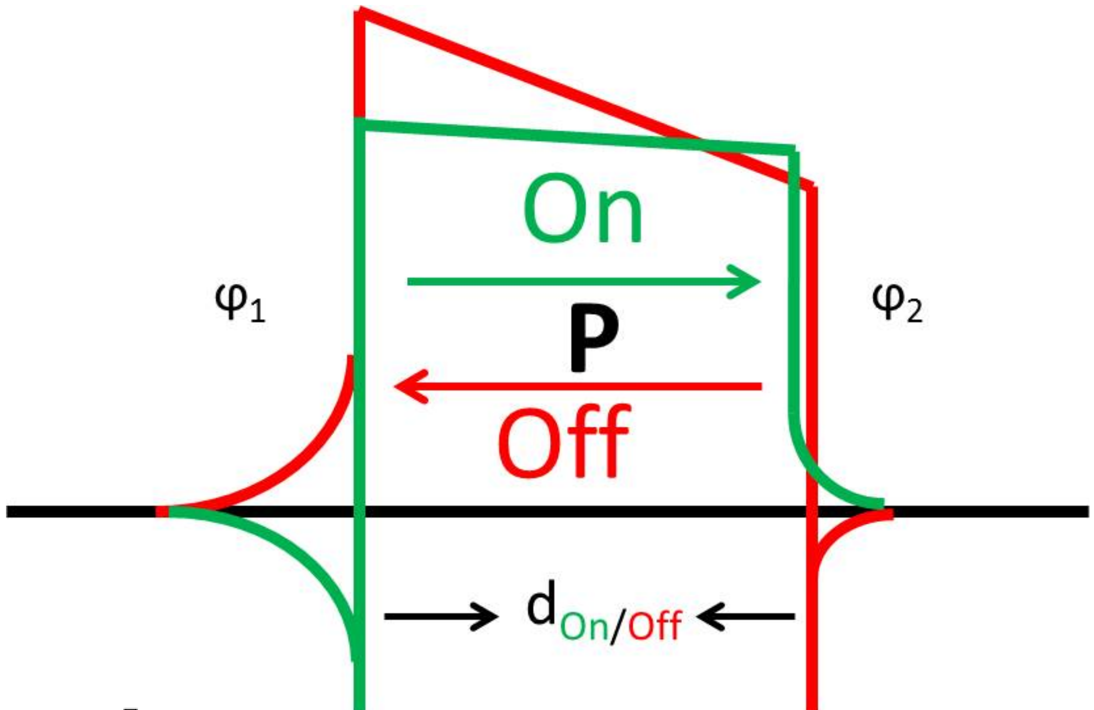
FTJ performance



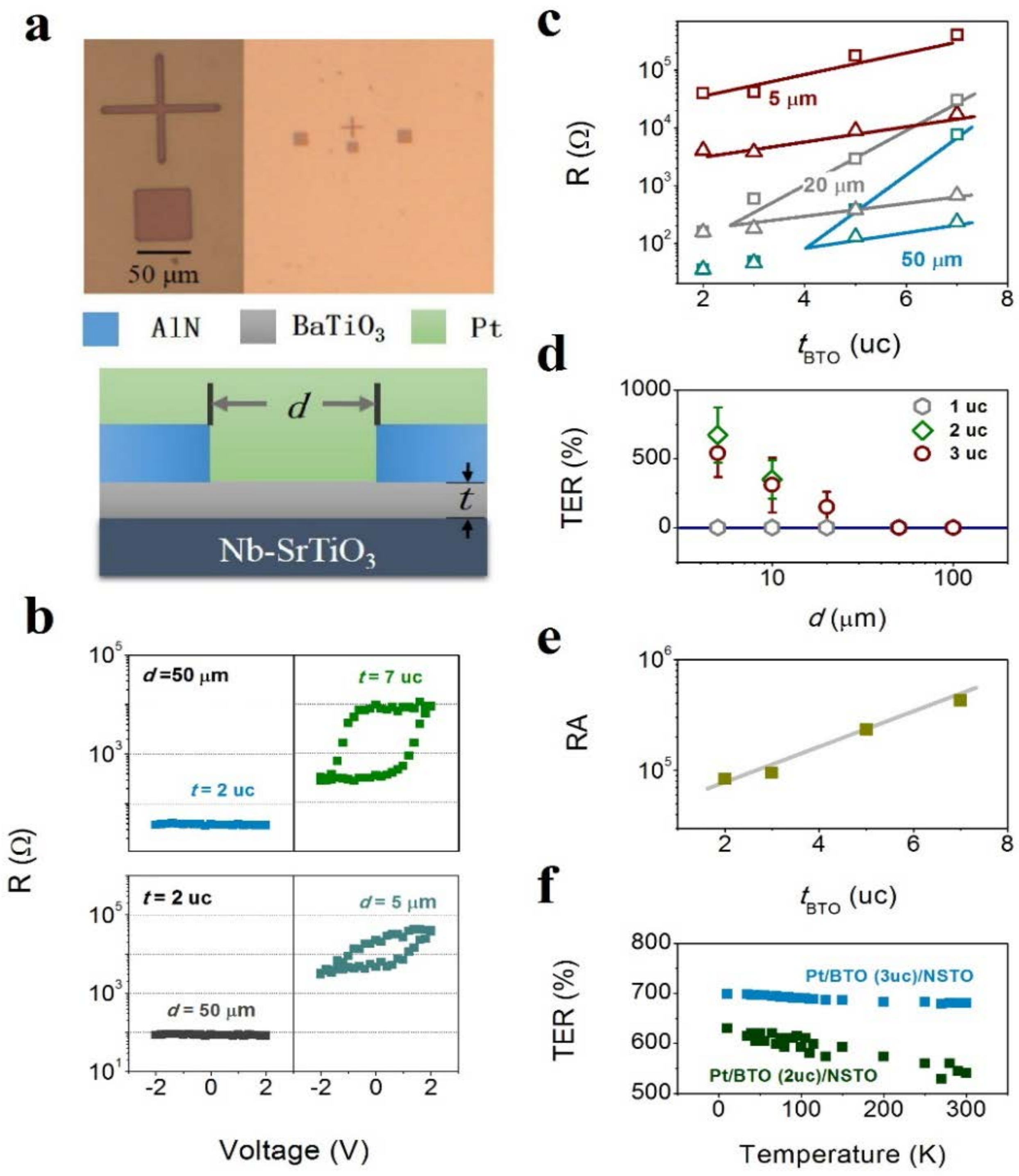
Different Structured FTJs



Analysis



$$J \cong -\frac{4em}{9\pi^2\hbar^3}\frac{exp\left\{\alpha(V)\left[\left(\varphi_2-\frac{eV}{2}\right)^{3/2}-\left(\varphi_1+\frac{eV}{2}\right)^{3\cdots}\right.\right.}{\alpha^2(V)\left[\left(\varphi_2-\frac{eV}{2}\right)^{1/2}-\left(\varphi_1+\frac{eV}{2}\right)^{1/2}\right.}\cdots$$



BTO thickness and Device size

Changjian Li, Venkatesan, Nano Lett. 15 (4), 2568 (2015)

Magnetism

Electronic Phase Separation at LAO/STO interfaces

Cationic Defect mediated Magnetism

Novel Magnetic Coupling via Polar Layers

Atomically Controlled Oxide Heterostructures

nature COMMUNICATIONS

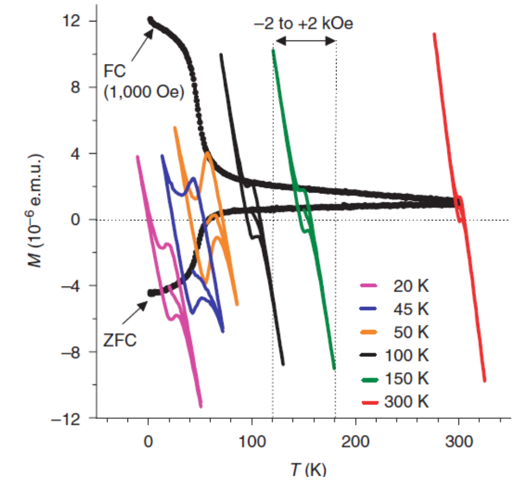
ARTICLE

Received 3 Sep 2010 | Accepted 12 Jan 2011 | Published 8 Feb 2011

DOI: 10.1038/ncomms1192

Electronic phase separation at the $LaAlO_3/SrTiO_3$ interface

Ariando[1,2,*], X. Wang[1,2,*], G. Baskaran[3], Z. Q. Liu[1,2], J. Huijben[4], J. B. Yi[5], A. Annadi[1,2], A. Roy Barman[1,2], A. Rusydi[1,2,6], S. Dhar[1,7], Y. P. Feng[1,2], J. Ding[5], H. Hilgenkamp[1,2,4,7,8] & T. Venkatesan[1,2,7]



news & views

OXIDE INTERFACES

Moment of magnetism

Electrons at an interface between two insulating oxides are now shown to exhibit electronic state not seen in the bulk of either individual oxide.

Andrew J. Millis

NATURE PHYSICS | ADVANCE ONLINE PUBLICATION |

roughly one unit cell of the interface. Earlier this year, Ariando *et al.*[12] showed by direct magnetization measurements that magnetism occurs in interfaces grown at high oxygen partial pressure, with evidence of ordering persisting up to room temperature. As the oxygen partial pressure during growth is reduced, the size of the moment is found to decrease, and becomes very small at the growth pressures that other workers have found optimized the superconducting properties.

Ariando, Rusydi, Venkatesan, Hilgenkamp

FM via Cationic Vacancies

VOLUME 89, NUMBER 21 PHYSICAL REVIEW LETTERS 18 NOVEMBER 2002

Possible Path to a New Class of Ferromagnetic and Half-Metallic Ferromagnetic Materials

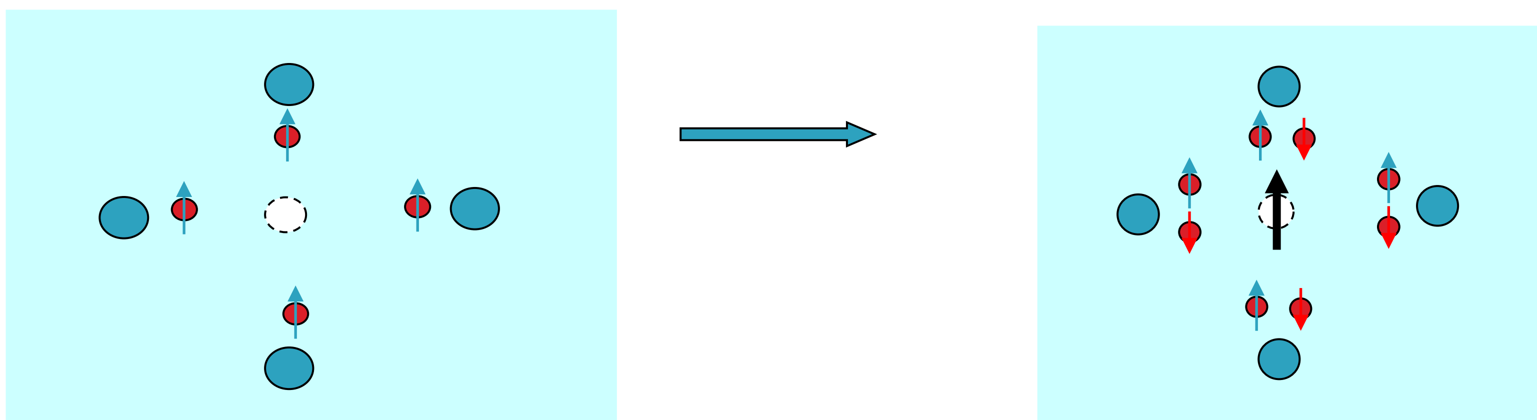
I. S. Elfimov,[1] S. Yunoki,[1] and G. A. Sawatzky[1,2]
[1]*Solid State Physics Laboratory, Materials Science Center, University of Groningen, Nijenborgh 4, 9747 AG Groningen, The Netherlands*
[2]*Department of Physics and Astronomy, University of British Columbia, 6224 Agricultural Road, Vancouver, British Columbia, Canada V6T 1Z1*
(Received 24 May 2002; published 4 November 2002)

We introduce a path to a possibly new class of magnetic materials whose properties are determined entirely by the presence of a low concentration of specific point defects. Using model Hamiltonian and *ab initio* band structure methods we demonstrate that even large band gap nonmagnetic materials as simple as CaO with a small concentration of Ca vacancies can exhibit extraordinary properties. We show that such defects will initially bind the introduced charge carriers at neighboring sites and depending on the internal symmetry of the clusters so formed, will exhibit "local" magnetic moments which for concentrations as low as 3% transform this nonmagnetic insulator into a half-metallic ferromagnet.

Elfimov et al. Phys. Rev. Lett. 98, 137202 (2007);
Zunger et al., Phys. Rev. Lett. 96, 107203 (2006)

Anion

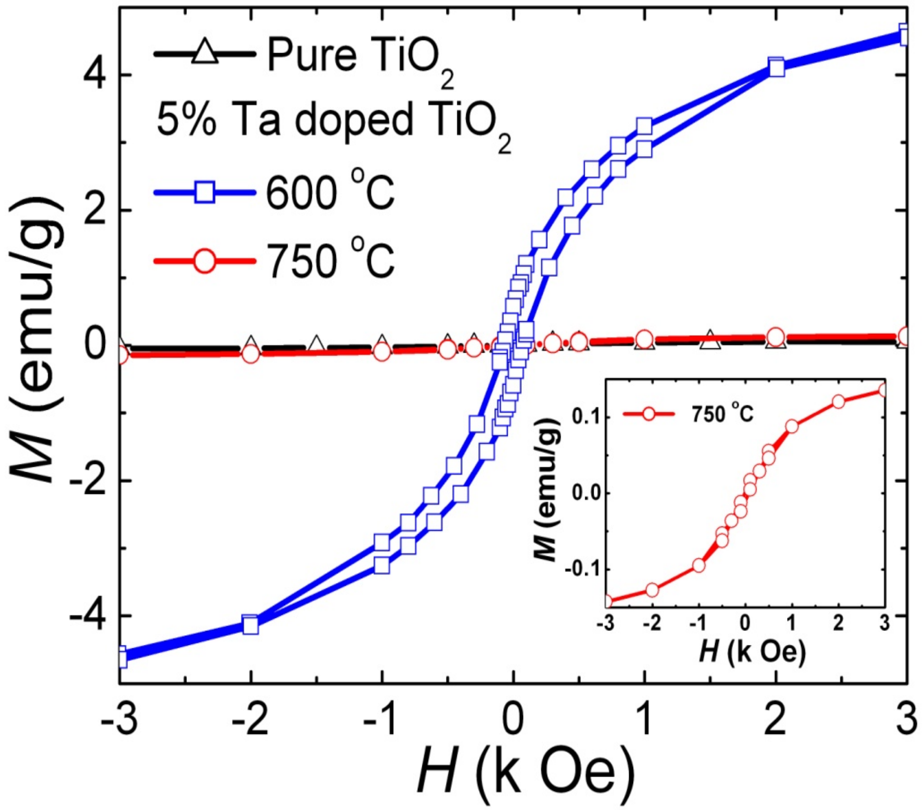
Cation

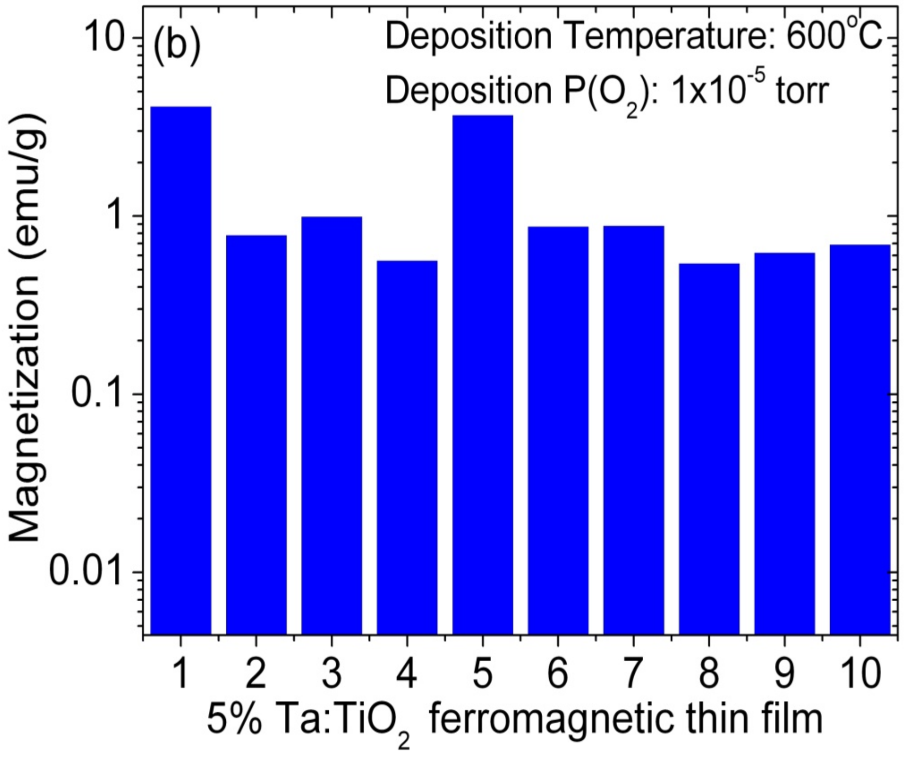


Cation vacancy

Local moments coming from holes

FM in Anatase 5% Ta-TiO_2



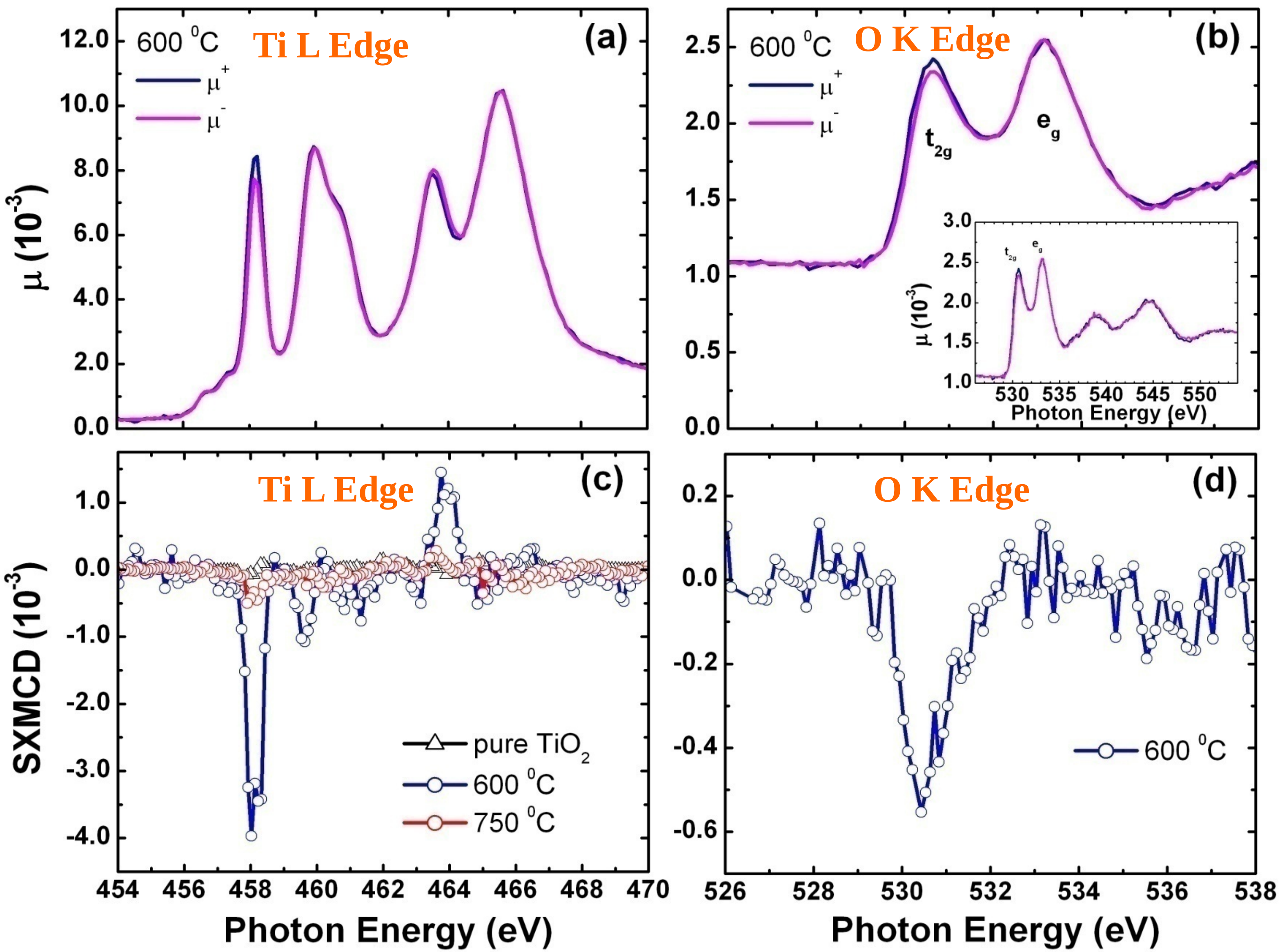


Maximum Saturation Magnetization : 1.1 μ_B/Ta ion

Maximum Remnant Magnetization : 0.14 μ_B/Ta ion

A. Rusydi, **Philosophical Transaction - Royal Soc. A 370, 4927 (2012)**

SXMCD: FM in 5% Ta-TiO_2

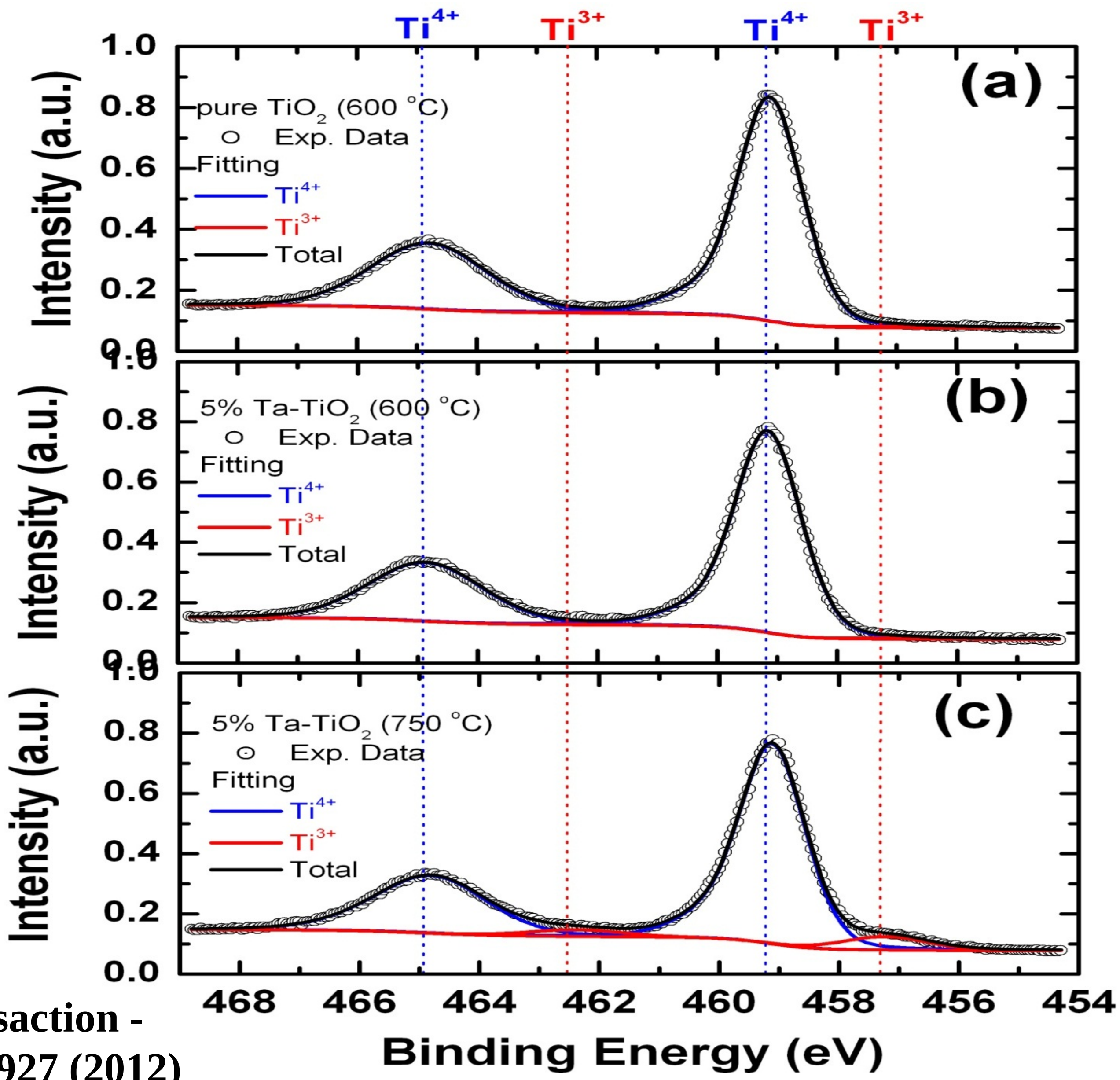


t_{2g} states at both Ti L and O K edge play the dominant role in FM & indicates p-d hybridizations

1.42 μ_B/ Ti defect

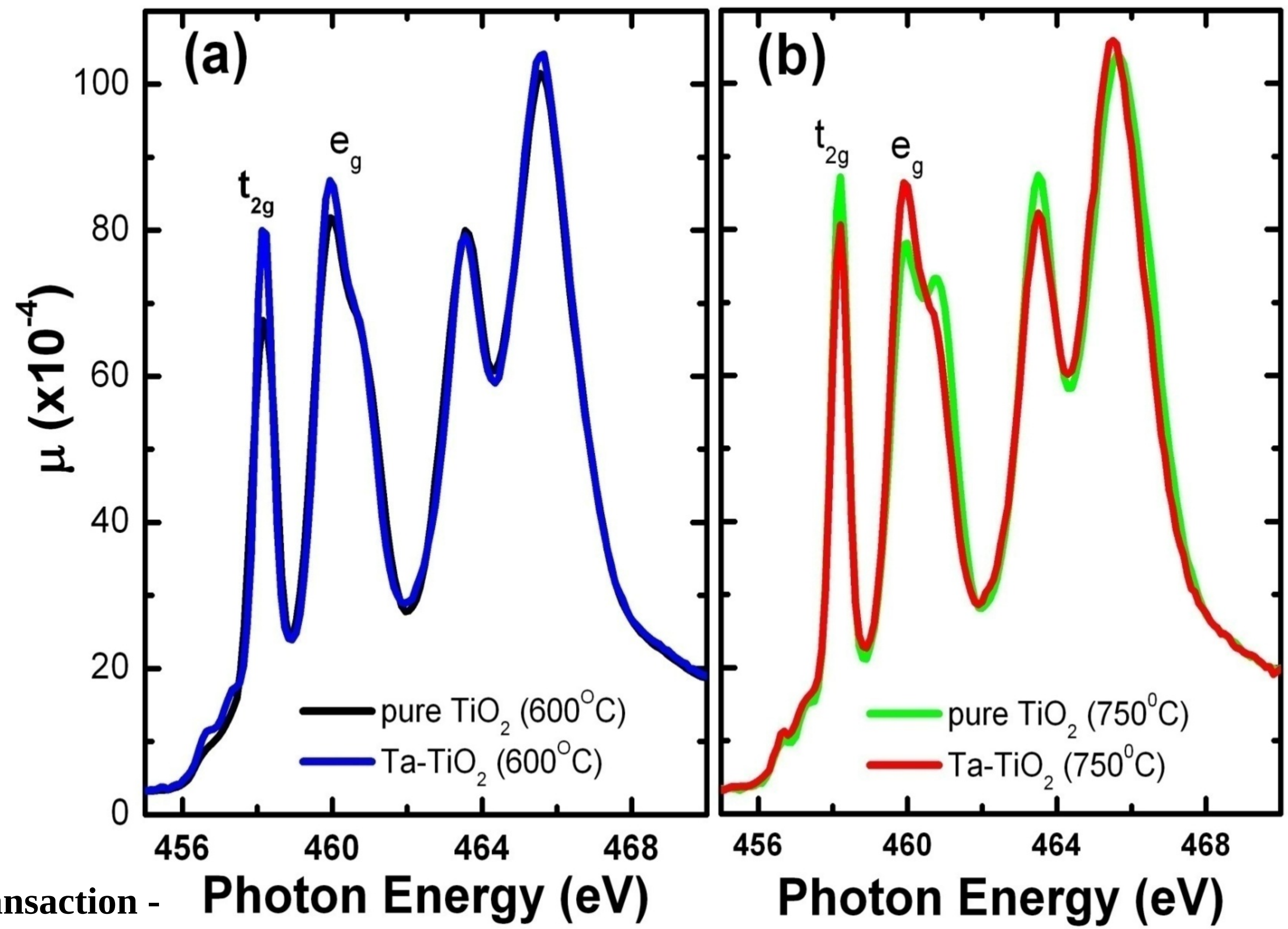
A. Rusydi, **Philosophical Transaction - Royal Soc. A 370, 4927 (2012)**

Ti $^{3+}$ in 600C and 750C Sample



A. Rusydi,
Philosophical Transaction - Royal Soc. A 370, 4927 (2012)

Cationic Vacancies in 600 and 750C samples



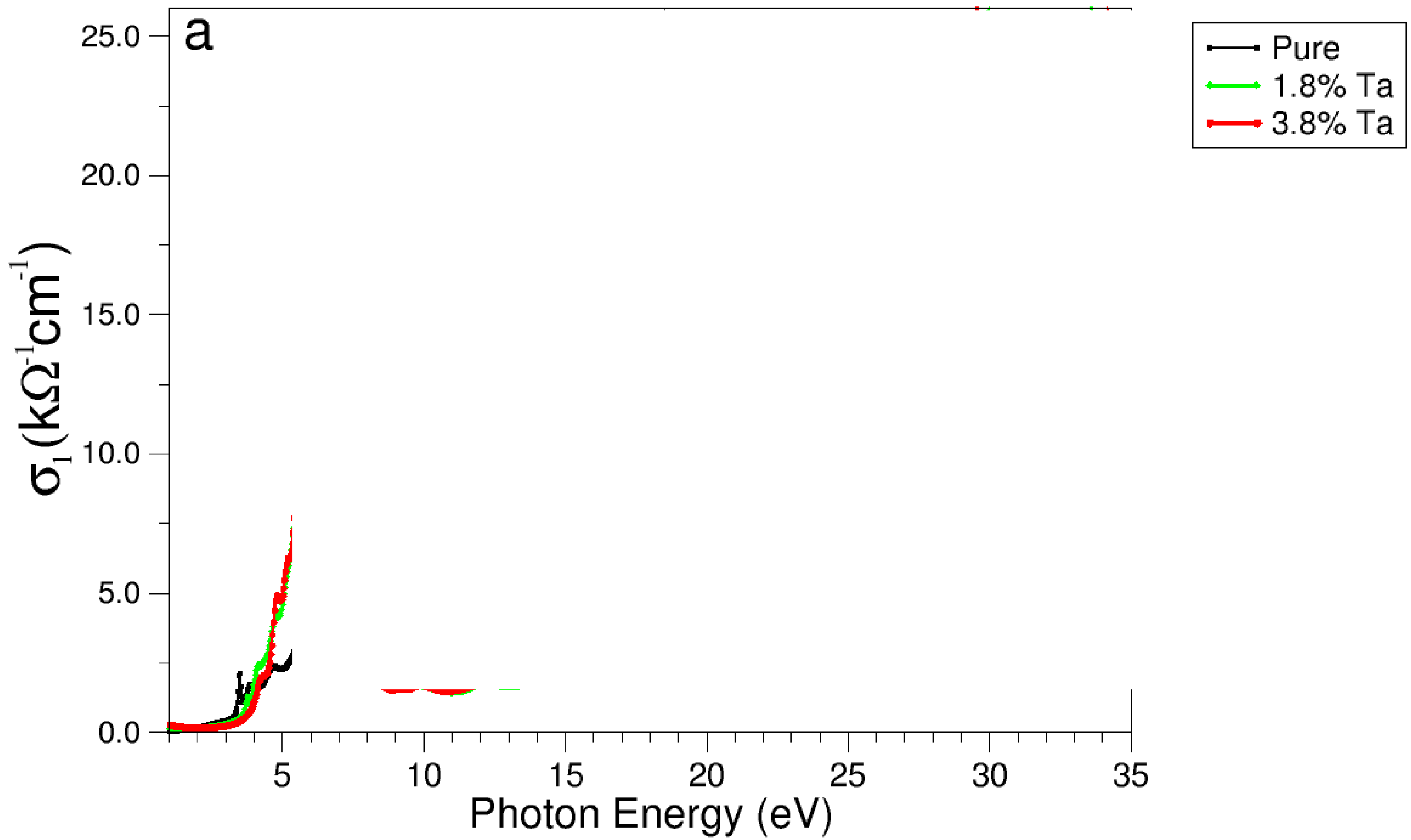
A. Rusydi,
Philosophical Transaction - Royal Soc. A 370, 4927 (2012)

Novel Optical Properties with potential Energy Applications

High energy exciton in TiO_2- Evidence of strong correlation

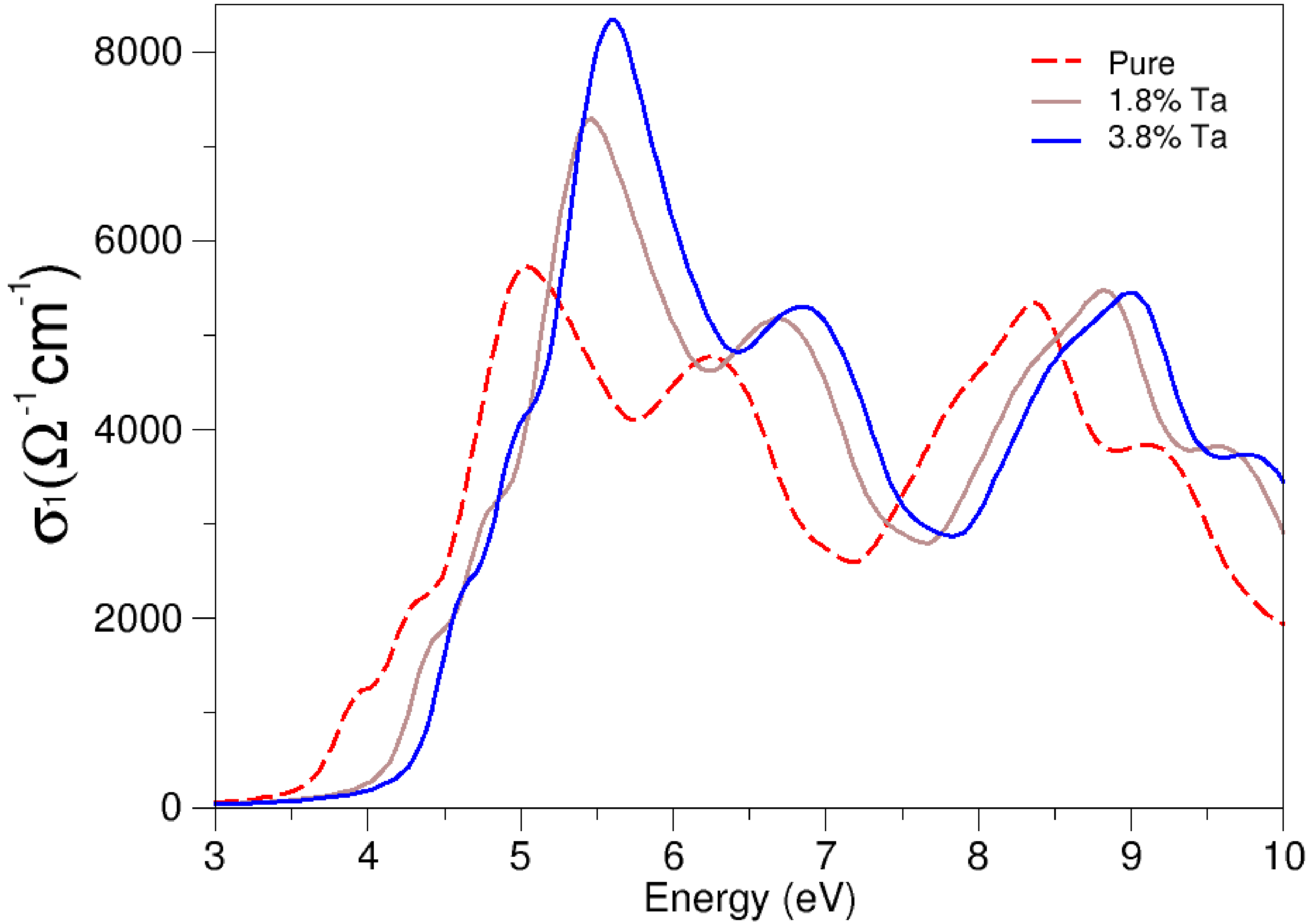
$SrNbO_3$- Correlated Plasmon Excitation

What do we know about TiO_2 and $Ta_xTi_{1-x}O_2$ films from conventional spectroscopic ellipsometry?



Yong Zhihua, Paolo E. Trevisanutto, Iman Santoso, Arkajit R. Barman, Teguh Citra Asmara, Sankar Dhar, L. Chiodo, A. Terentjevs, F. Della Sala, V. Olevano, Michael Rübhausen, T. Venkatesan, Andrivo Rusydi, submitted (2015)

Monte Carlo Espresso Computation including electronic Correlation



Yong Zhihua, Paolo E. Trevisanutto, Iman Santoso, Arkajit R. Barman, Teguh Citra Asmara, Sankar Dhar, L. Chiodo, A. Terentjevs, F. Della Sala, V. Olevano, Michael Rübhausen, T. Venkatesan, Andrivo Rusydi, submitted (2015)

Water splitting with $SrNbO_{3+\delta}$

NUS National University of Singapore

Semiconductors can be used in photocatalytic water splitting, how about metal or metallic oxide, nitrite, etc.?

First response: No, missing of internal field, low diffusion length…

Reality: Yes

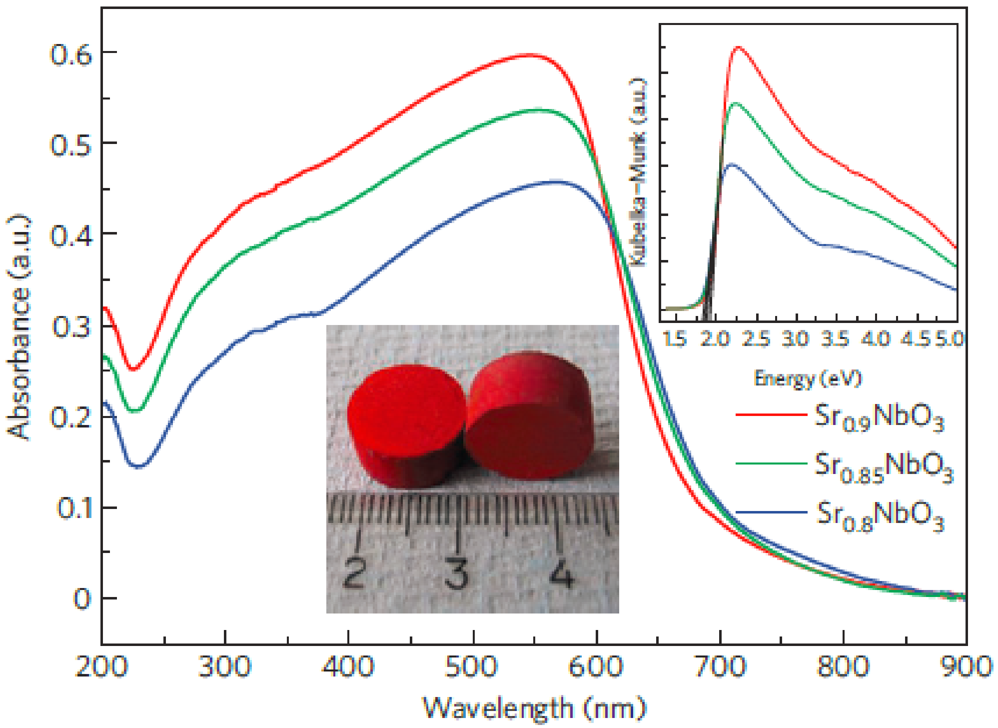
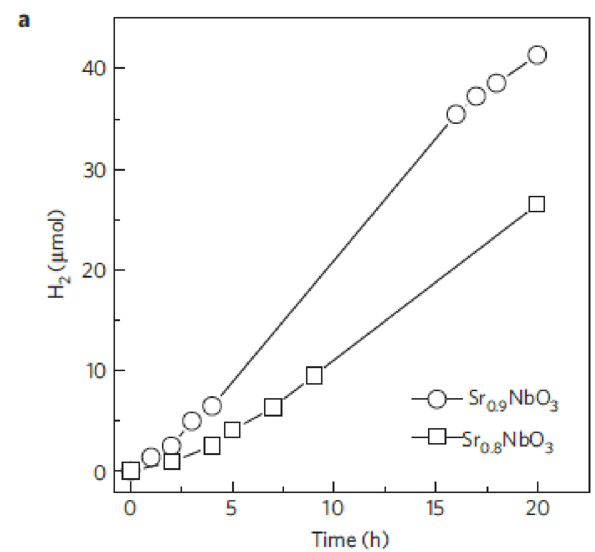
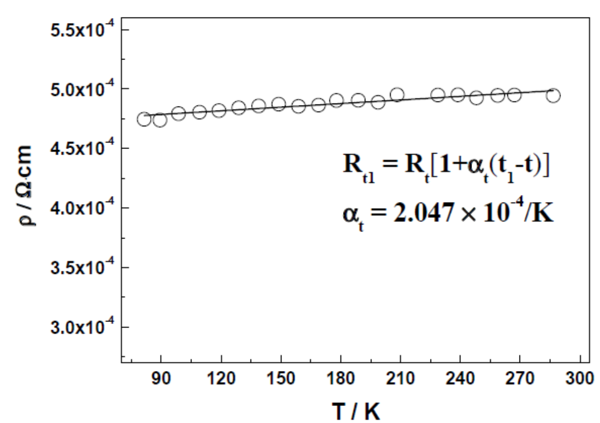


Figure 1 | Ultraviolet-visible absorbance spectra (converted from diffuse reflectance spectra) for $Sr_{1-x}NbO_3$ ($x = 0.1$, 0.15 and 0.2). Kubelka-Munk transformation of the absorption curves is shown in the top inset. Typical sintered pellets ($Sr_{0.9}NbO_3$) are shown in the bottom inset.





- Xu, X.X., et al., *A red metallic oxide photocatalyst.* Nature Materials, 2012. **11**(7): p. 595-598.

Why is SNO special?

High mobility induced high diffusion length?

$$\mathbf{L} = \sqrt{D\tau},\ \mathbf{D} = \left(\frac{kT}{e} \cdot \mu\right)$$

Where is the visible light absorption originating from?

No !

SNO needs to be carefully studied.

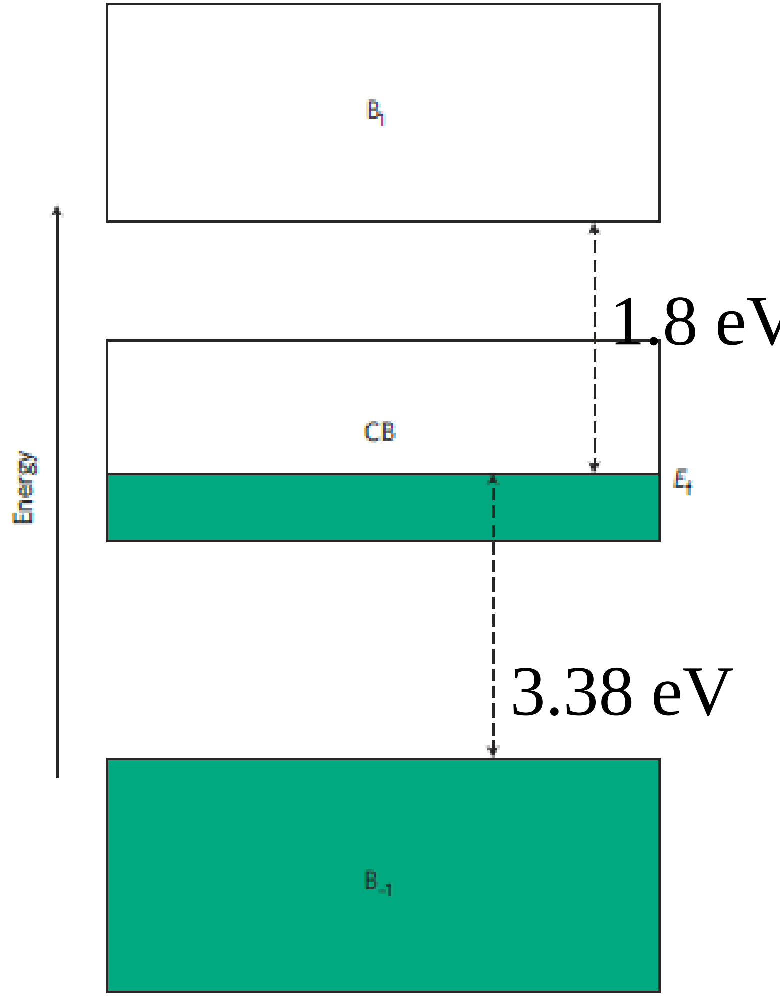
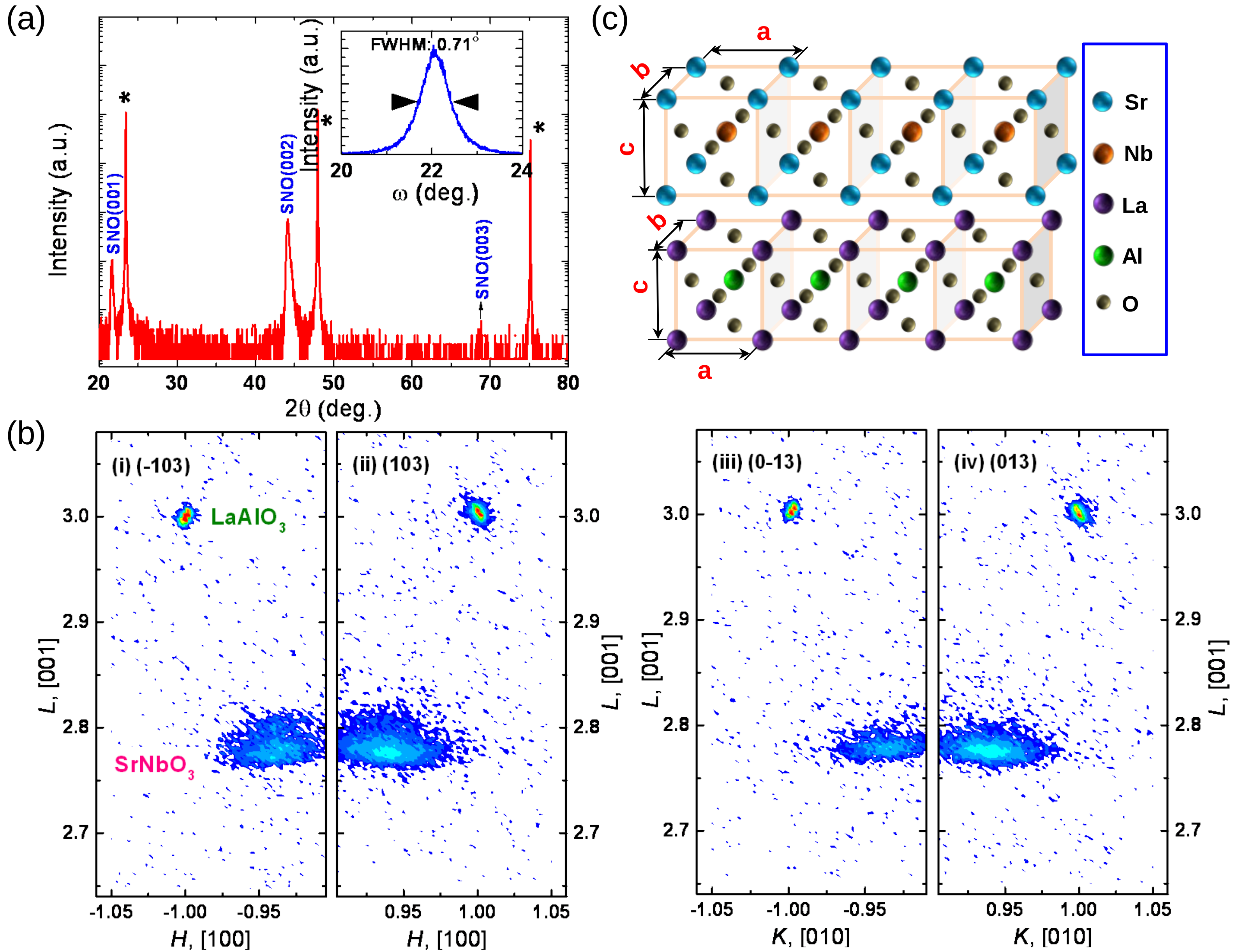


Figure 2 | Schematic of band structure for a metallic conductor. Bands below and above the conduction band (CB) are labelled as B_{-1} and B_1. Possible transitions associated with photon absorption are marked by arrows.

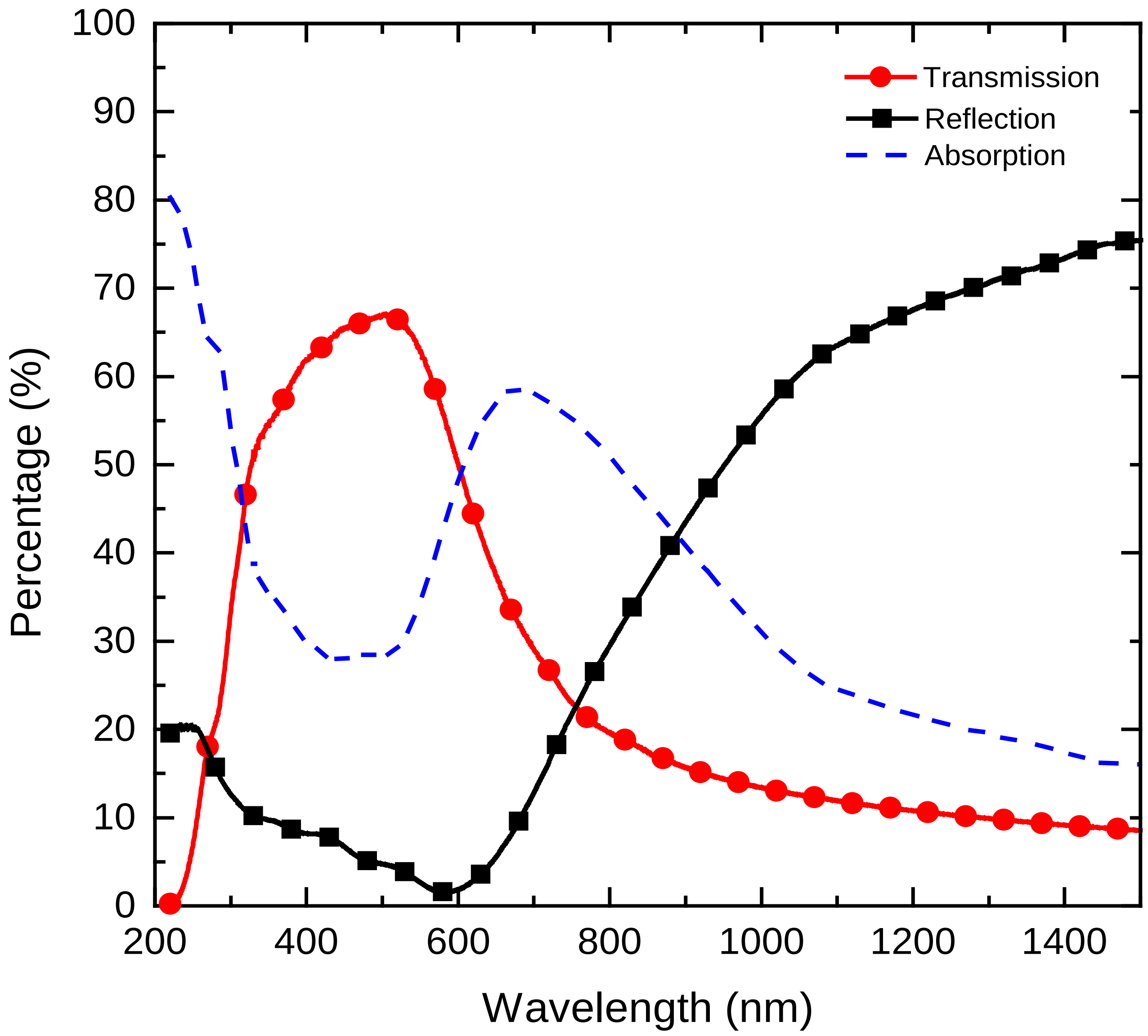
X. Xu, et al, Nature Mater. **11**, 595-598 (2012)

Epitaxial thin film preparation



LAO was selected as the substrate because we need to study the film's transport, optical and transient absorption properties.

Optical absorption

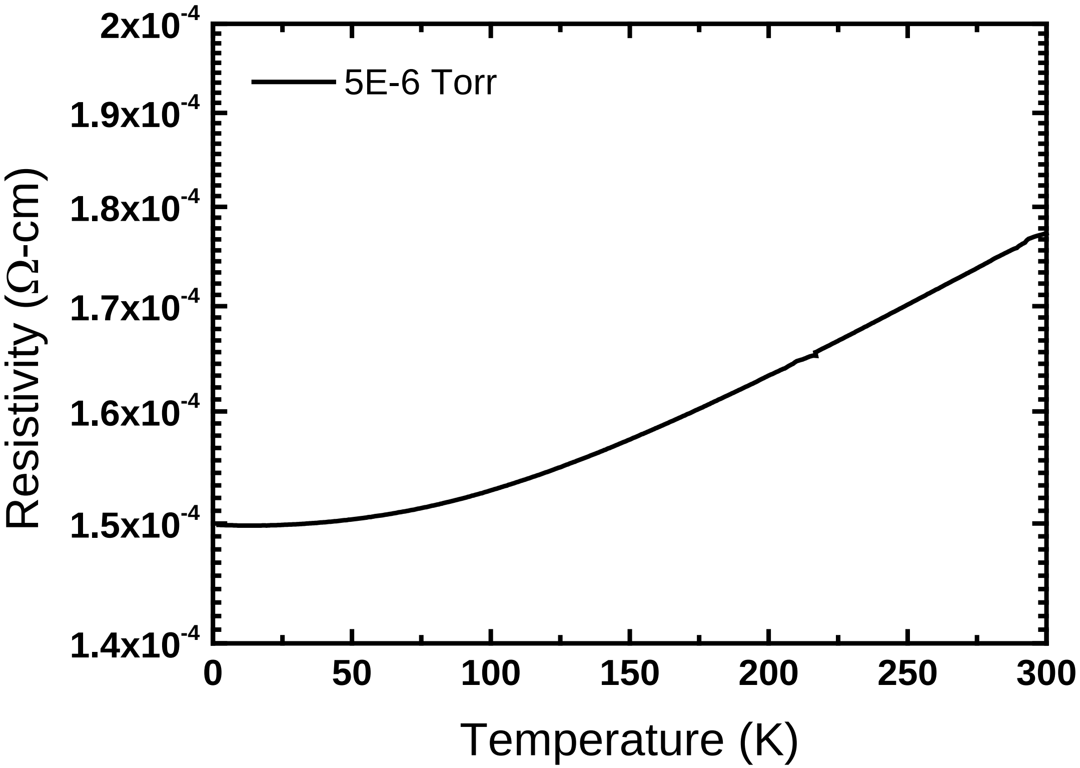


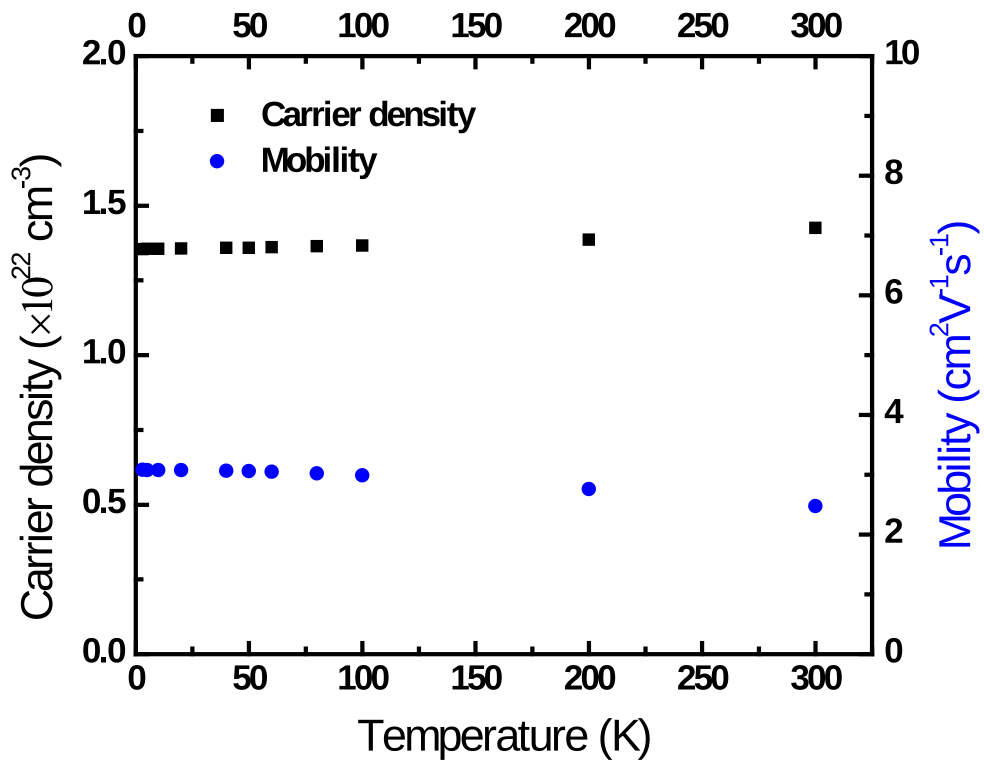
The optical band gap is measured as around 4.1 eV.

The absorption peak located at 700 nm may correspond to the visible light absorption.

Free electron absorption can not be rule out

High mobility?



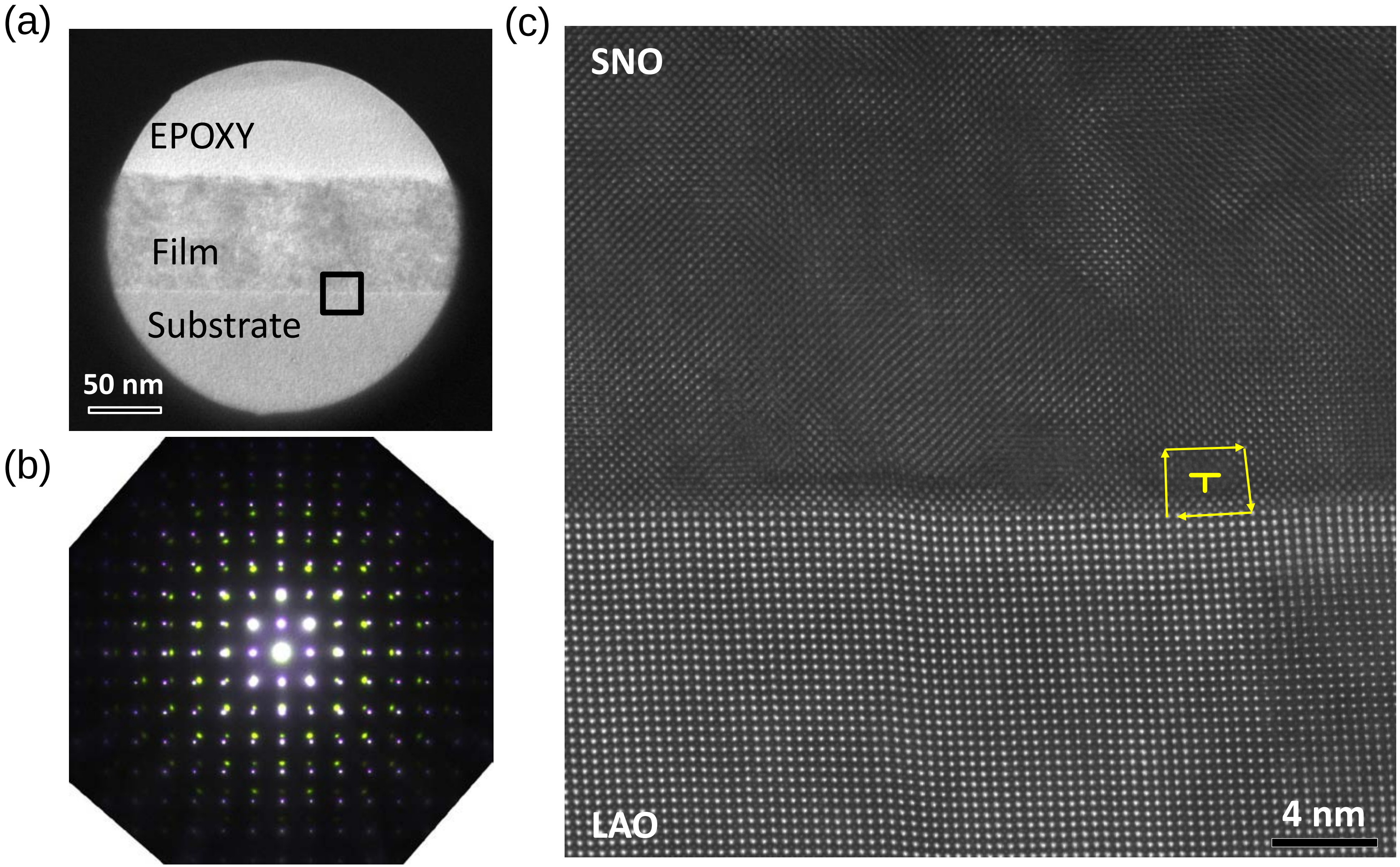


$L = \sqrt{D\tau}$, $D = (\frac{kT}{e} \cdot \mu)$

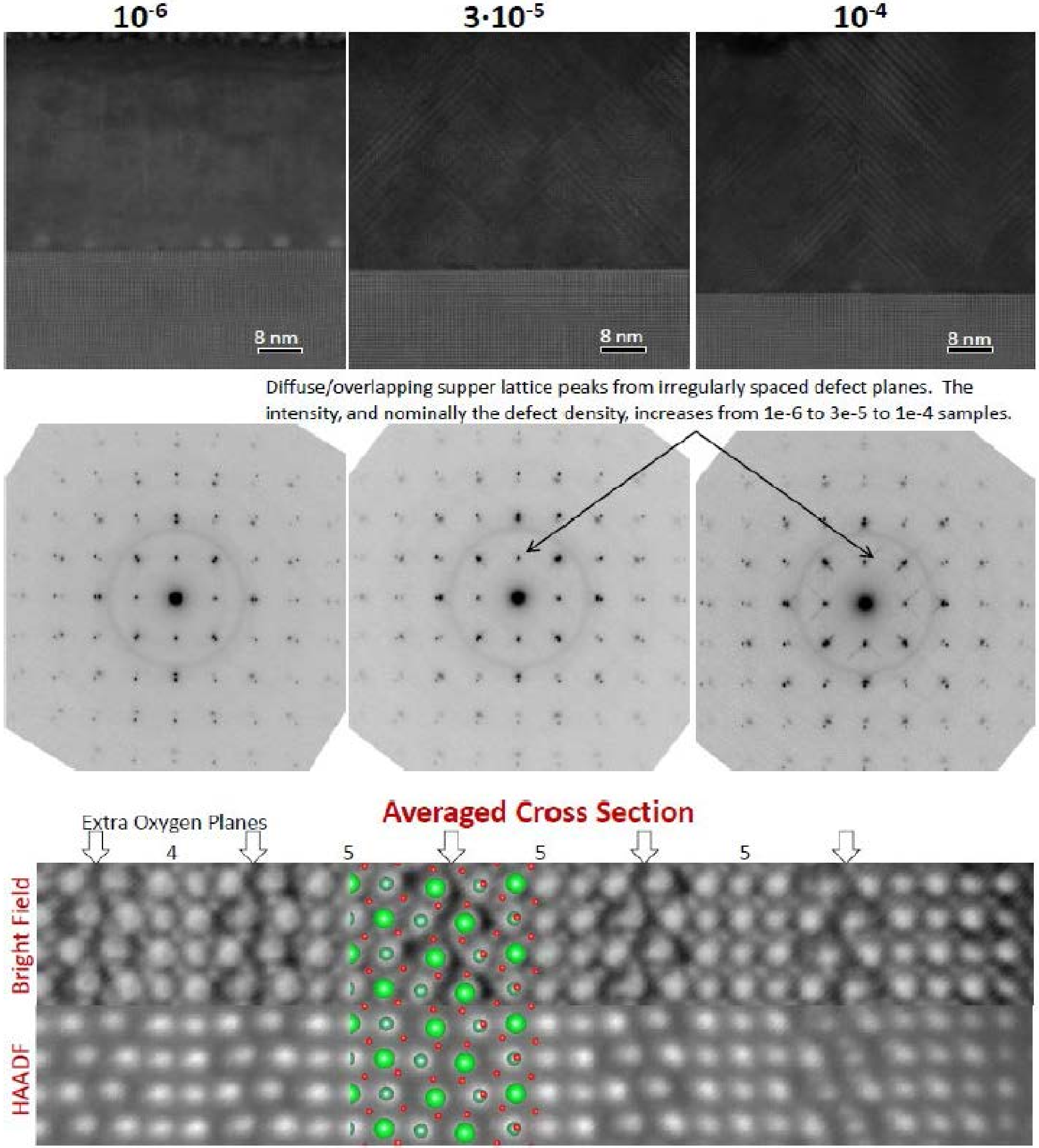
$\tau \sim 200$ ps, $\mu \sim 2$ cm^{2}V^{-1}s^{-1}, ➔ L ~ 32 nm

Explained ➔ Particle size dependence of the water splitting efficiency of the powder

Epitaxial thin film preparation

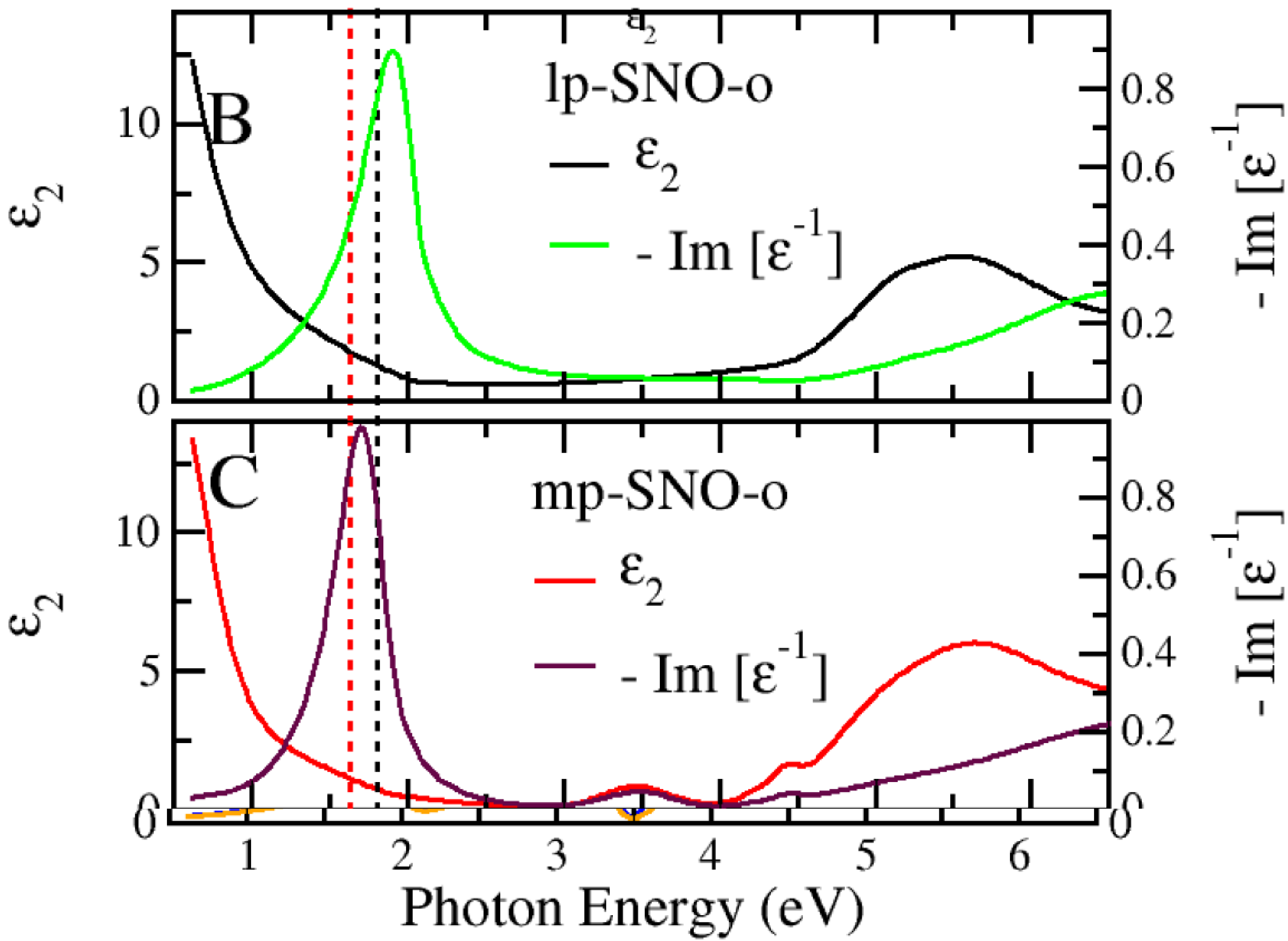


Epitaxial SNO film (with tetragonal perovskite structure) can be prepared under very high vacuum level (below 5E-6 Torr). Composition Sr:Nb:O= 1:1:3

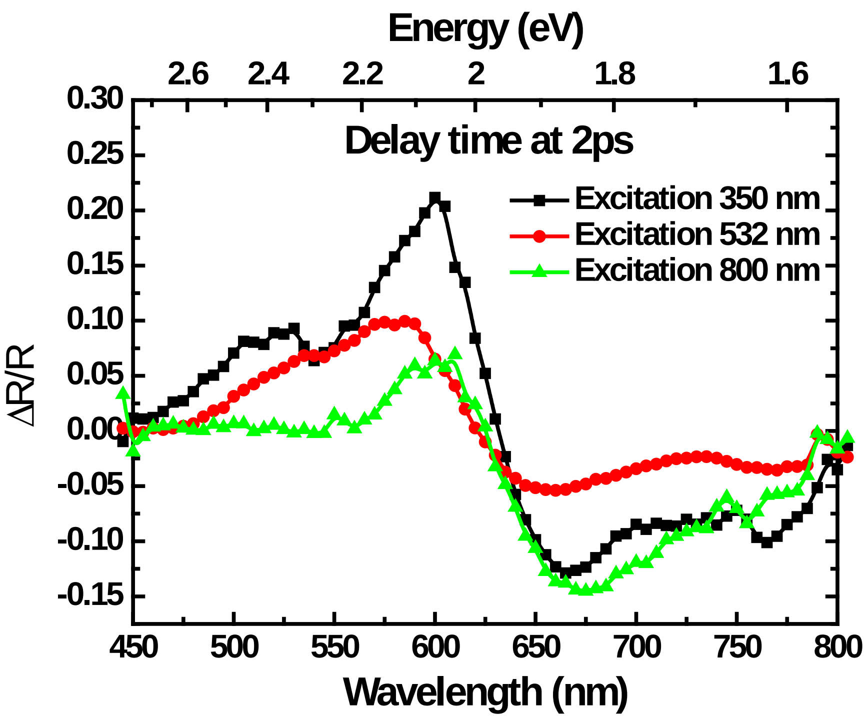


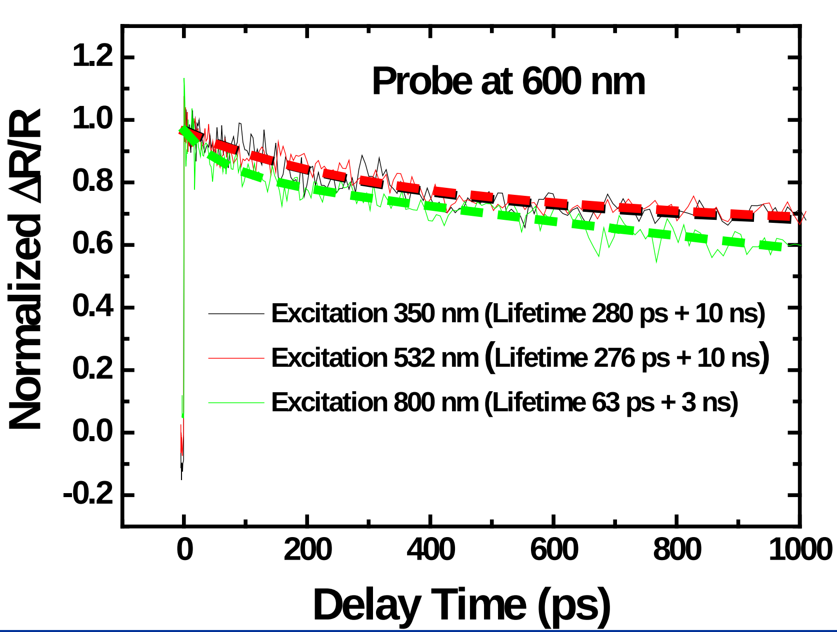
TEM Cross section of films as a function of oxygen excess. Over 3 the oxygen gets incorporated as insulating planes

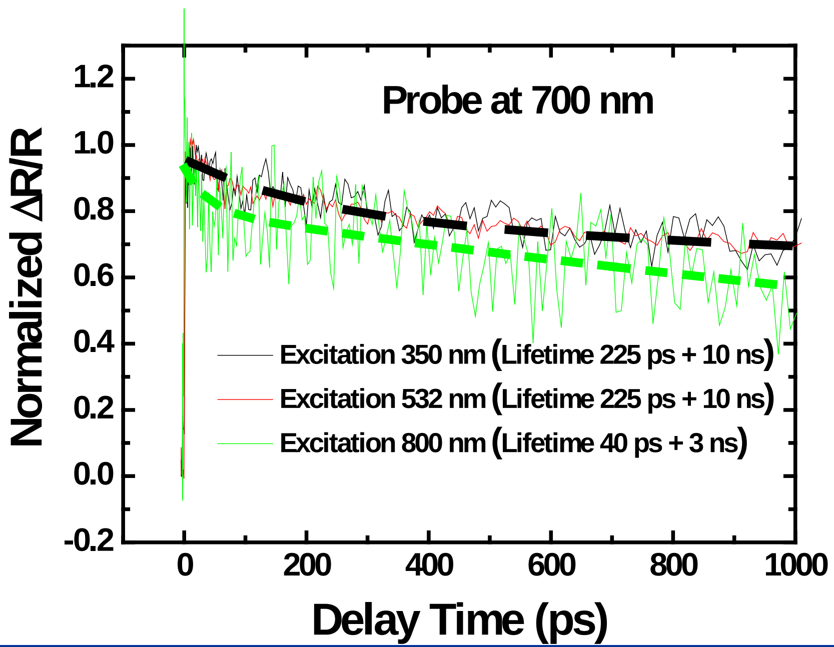
Resonant and Correlated Plasmon



Transient absorption







Time for a Break

Mechanism of VO2- Dimer driven or Crystal Phase driven?