Flexoelectricity in oxide thin films

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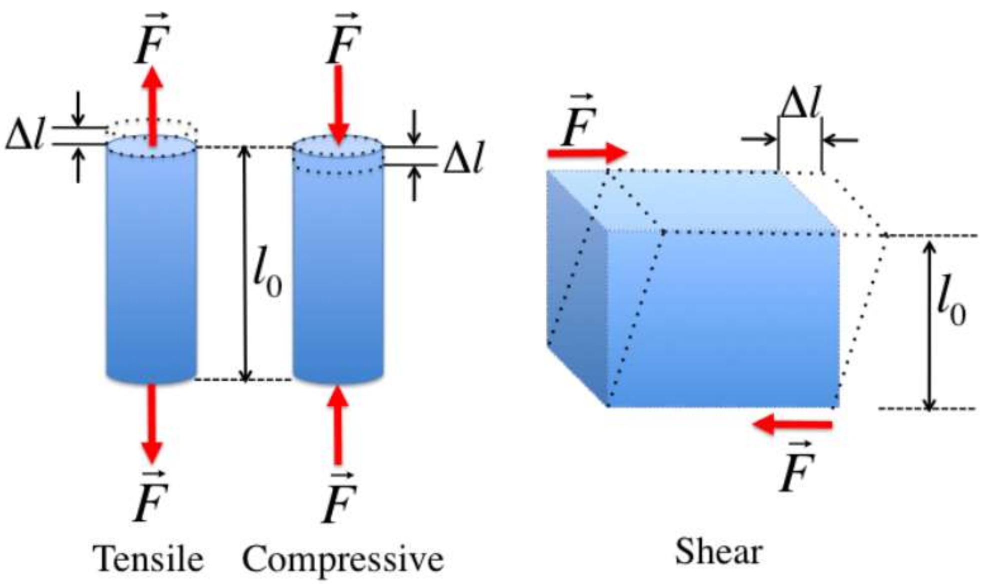
Outline

1. Introduction on flexoelectricity
2. Controlling physical properties of oxide thin films by flexoelectric effects
3. Functional manipulation of oxide thin films by applying pressure with an AFM tip
4. Potential applications
5. Summary

Deformation & Strain

Stretch

Squeeze



(linear) strain
= $\Delta L/L$

Strain engineering

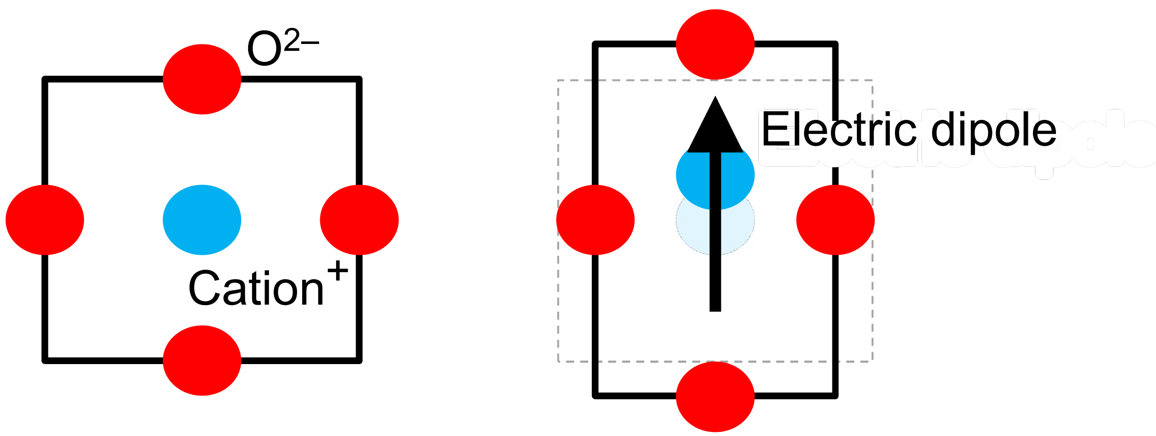
- Hydrostatic pressure
- Diamond anvil pressing
- Thin-film epitaxy
- …..

Emergent Phenomena

Bandwidth control
- Metal-insulator transition

Nonpolar-polar transition
- piezoelectricity
- ferroelectricity

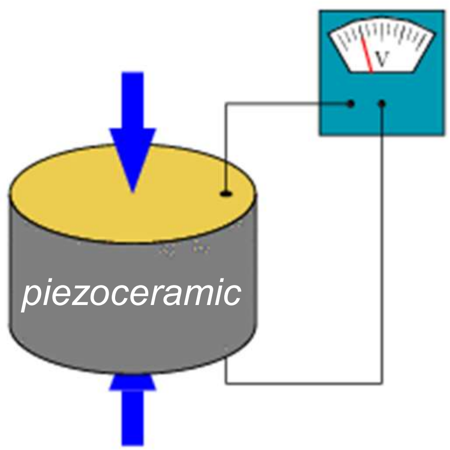
….



Piezoelectricity

Electromechanical coupling between charge and lattice (i.e. strain)

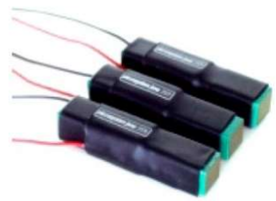
Piezoelectric effect



$$P_i = d_{ijk}\,\sigma_{jk}$$

d_{ijk} : piezoelectric coefficient
σ_{jk} : stress component

Under homogeneous strain



Actuator & motor

Sensor ….

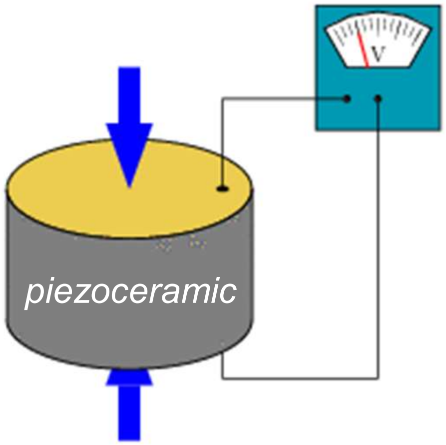
. Industrial equipment
. Security and defense
. Medical application
. Power generation
…..

Global market (2022): US$ 30.66 billion
(Source: "Global Piezoelectric Device Market")

Piezoelectric *vs* Flexoelectric effects

Electromechanical coupling between charge and lattice (i.e. **strain gradient**)

Piezoelectric effect

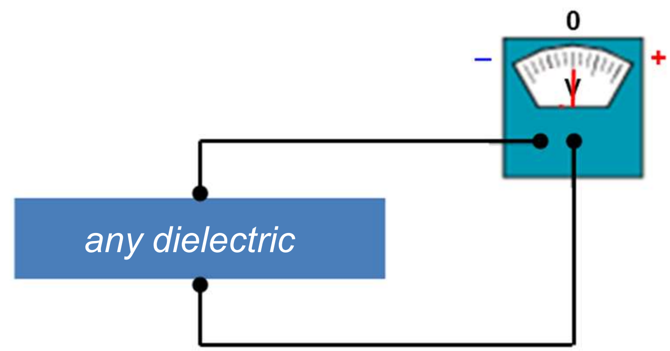


$$P_i = d_{ijk}\, \sigma_{jk}$$

d_{ijk} : piezoelectric coefficient
σ_{jk}: stress component

Under homogeneous strain

Flexoelectric effect



$$P_i = \mu_{ijkl} \frac{\partial u_{kl}}{\partial x_j}$$

μ_{ijkl} : flexoelectric coefficient
$\partial u_{jk}/\partial x_l$: strain-gradient term

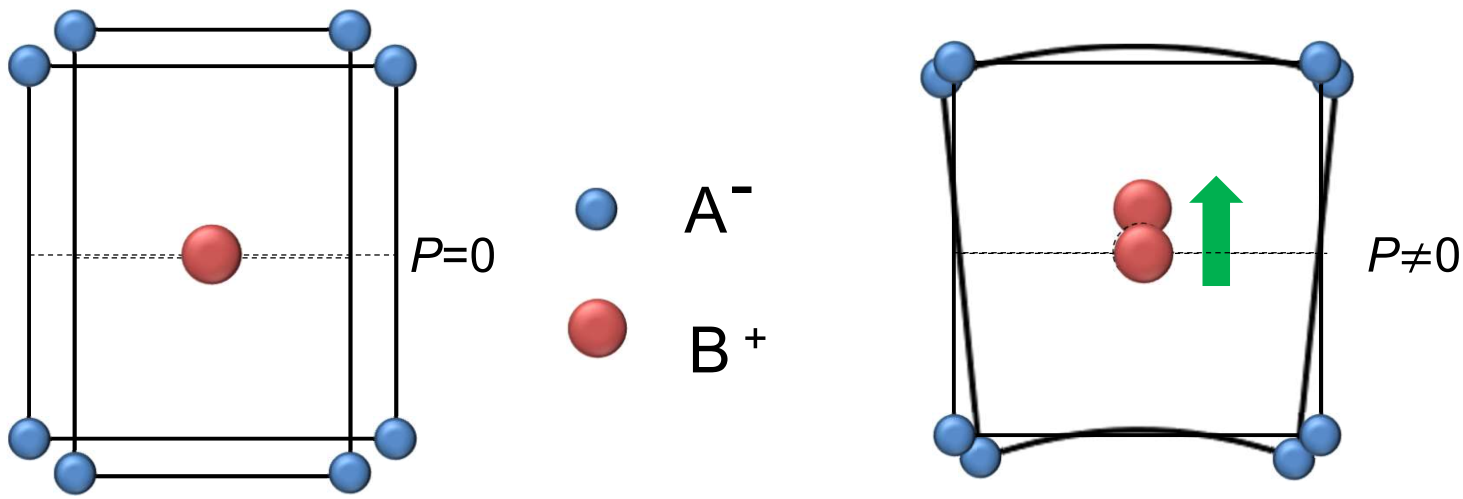
From inhomogeneous strain
(i.e. strain gradient)

S. M. Kogan, Sov. Phys. Solid State (1964)

Microscopic origins & required crystal symmetries

Homogeneous strain
:preserves the inversion symmetry

Strain gradient
:breaks the inversion symmetry



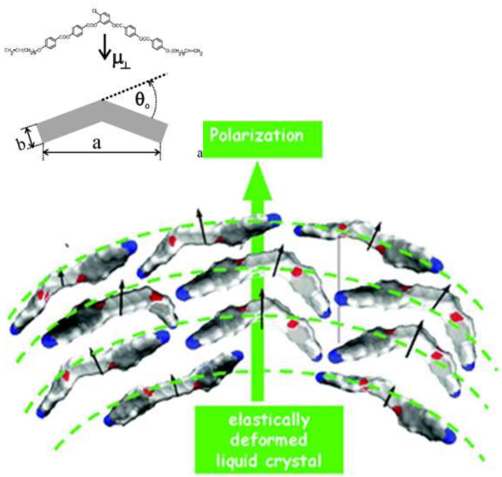
Piezoelectric effects can occur only in polar materials without inversion symmetry
(only 20 groups out of 32 crystalline point groups in nature)

However, flexoelectric effects can occur in any dielectric materials.

Flexoelectricity in nature

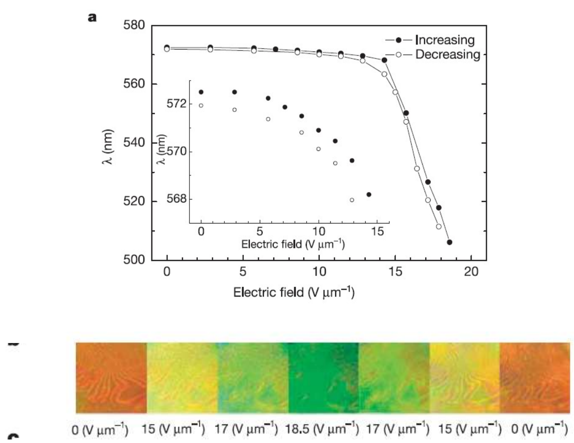
Flexoelectric effects have been reported in some *soft matters* (such as liquid crystals and polymers) *and biological systems.*

<E-field generation in liquid crystals>



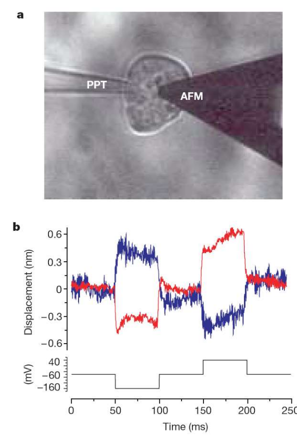
J. Harden *et al*., Phys. Rev. Lett. (2006)

<Phase stabilization in liquid crystals>



H. J. Coles *et al*., Nature (2005).

<E-field-induced membrane movement>

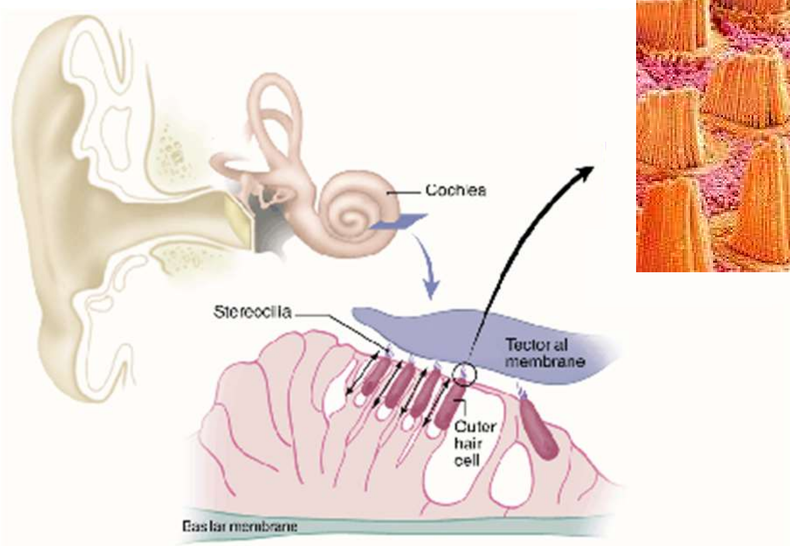


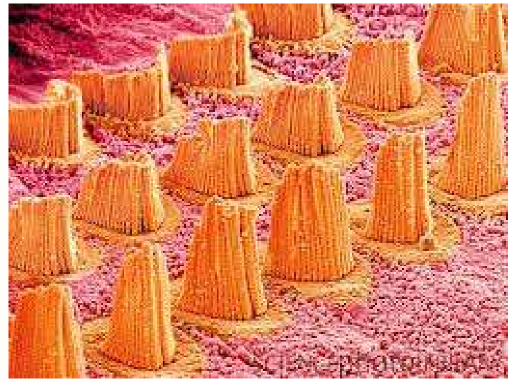
P. C. Zhang *et al*., Nature (2001)

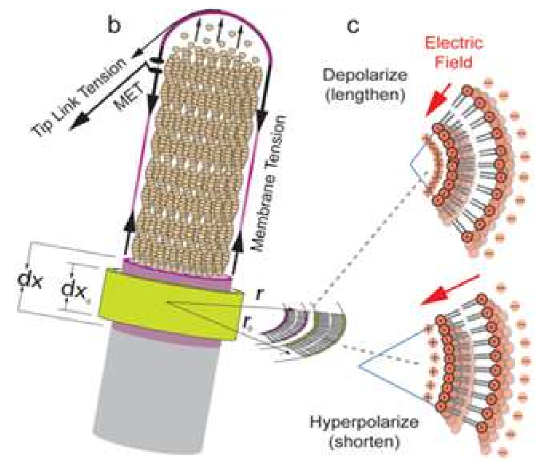
flexoelectric effect !

converse flexoelectric effects !

Flexoelectricity in our everyday Life







K. D. Breneman *et. al*., PLoS (2009)

Flexoelectricity is a contributing factor in mammalian hearing sensitivity !

First experiment in hard solids, including oxides

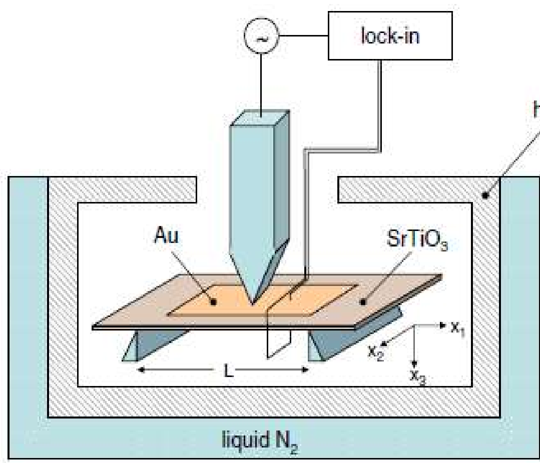
PRL 99, 167601 (2007) PHYSICAL REVIEW LETTERS week ending 19 OCTOBER 2007

Strain-Gradient-Induced Polarization in $SrTiO_3$ Single Crystals

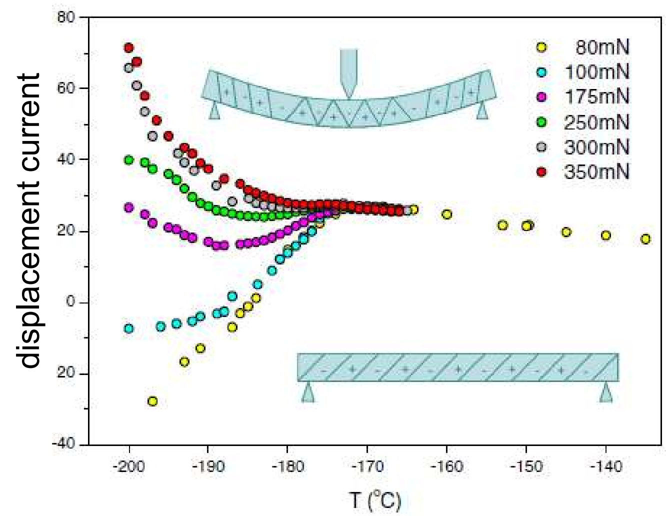
P. Zubko,* G. Catalan,† A. Buckley, P. R. L. Welche, and J. F. Scott

Centre for Ferroics, Department of Earth Sciences, University of Cambridge, Cambridge CB2 3EQ, United Kingdom

(Received 26 July 2007; published 19 October 2007)



dynamic mechanical analyzer

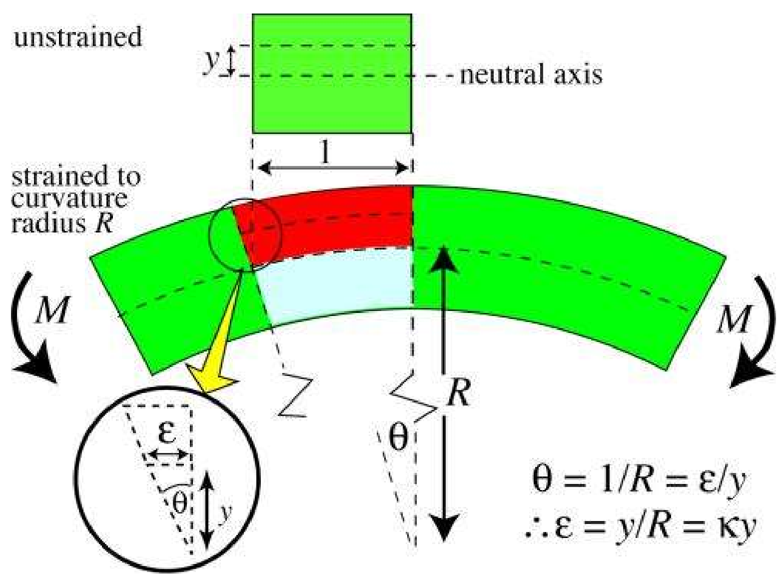


Flexoelectricity in solids at macroscopic scale !

Little studies on flexoelectric effects in oxides?

In *oxides*, there have been little investigations on flexoelectricity: ***Why ?***

1. most strain gradient : *highly inhomogeneous*
   → difficult to measure and control the strain gradient distribution
2. Macroscopically tiny effect due to its very small flexoelectric coefficients and restricted elastic deformation at macroscopic scale.



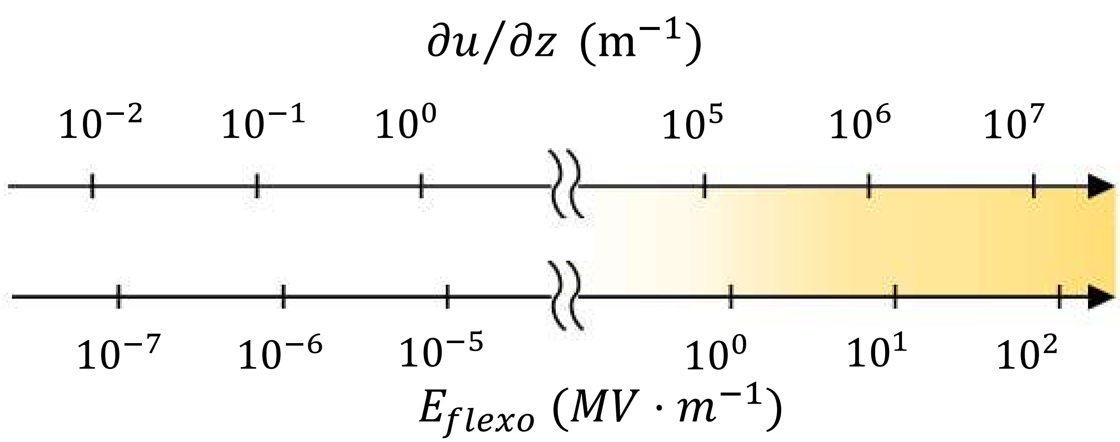
Strain gradient ~ $1/R$
(R: Radius of curvature)

If R = 10 cm, E_{flexo} ~ 10 V/m
→ negligibly small in bulk!

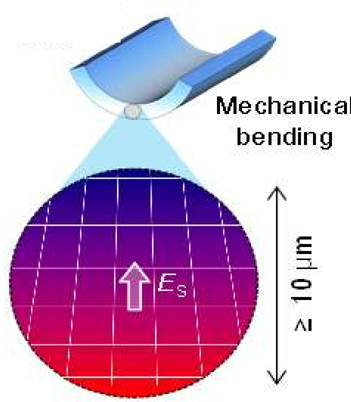
Source: : https://eng.libretexts.org/

Flexoelectricity: macroscale vs. nanoscale

- ✓ Piezoelectric effect : *P* nearly independent of sample size
- ✓ ***Flexoelectric effect*** : *P* inversely proportional to size

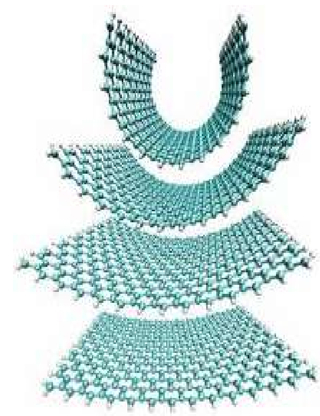


macroscopic scale



Flexoelectric field negligibly small

nanoscale



10^6 – 10^8 Enhancement!

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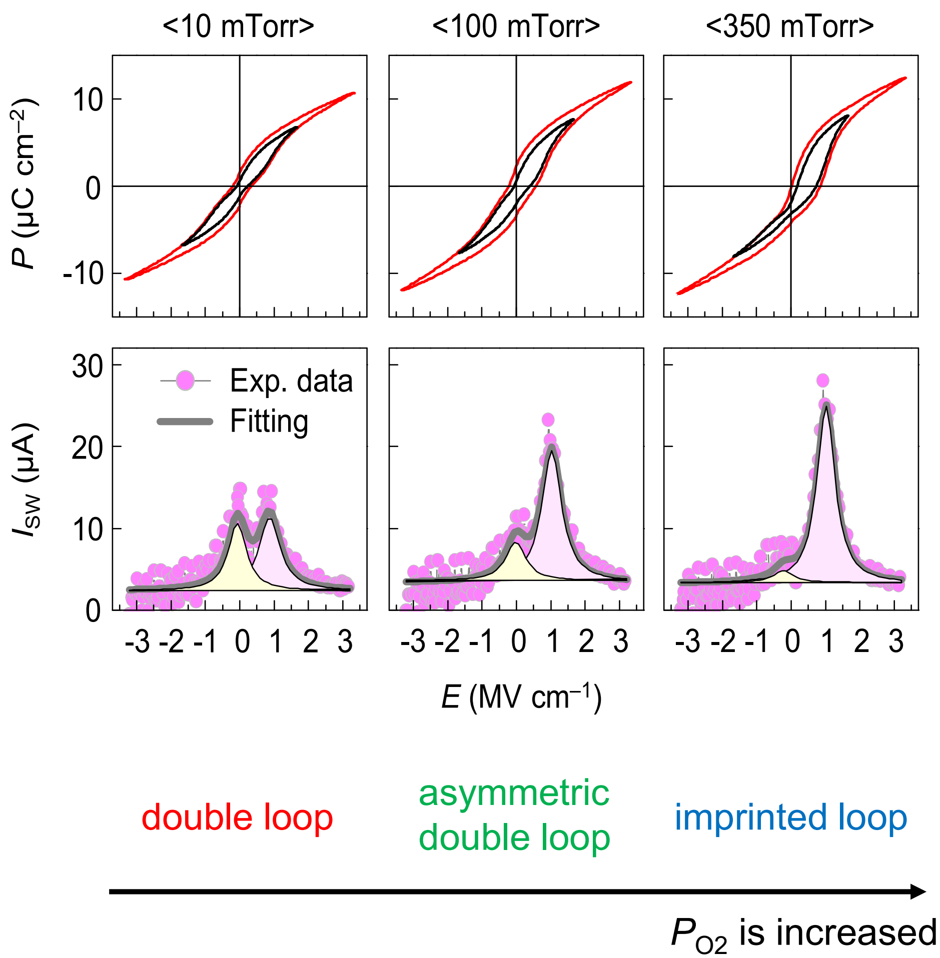
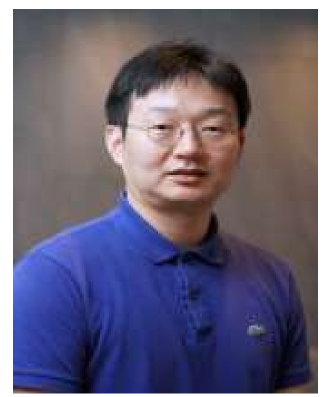
4. Potential applications
5. Summary

Dependence of P_{O_2} for ferroelectric $HoMnO_3$ films



Similar phenomena had been observed in many FE films with small P values. → origins?

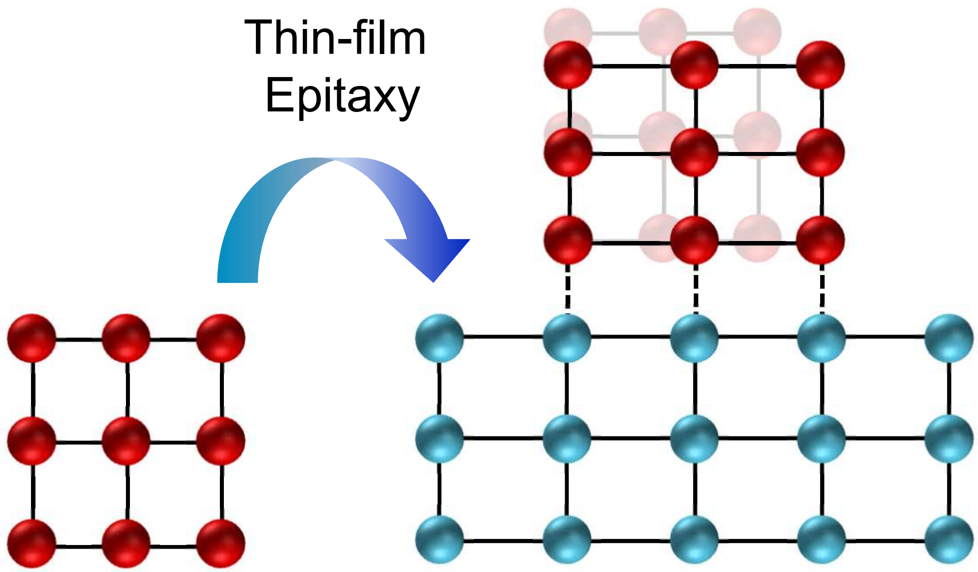


Prof. Dae Su Lee (POSTECH)

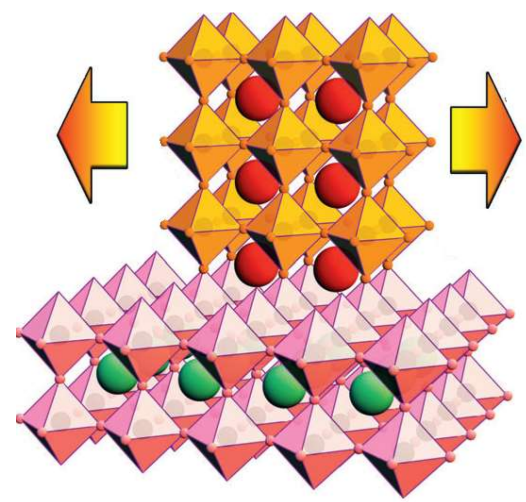
D. Lee *et al.*, Phys. Rev. Lett. (2011)

Epitaxial growth of an oxide thin film

Growth of fully strained oxide thin film

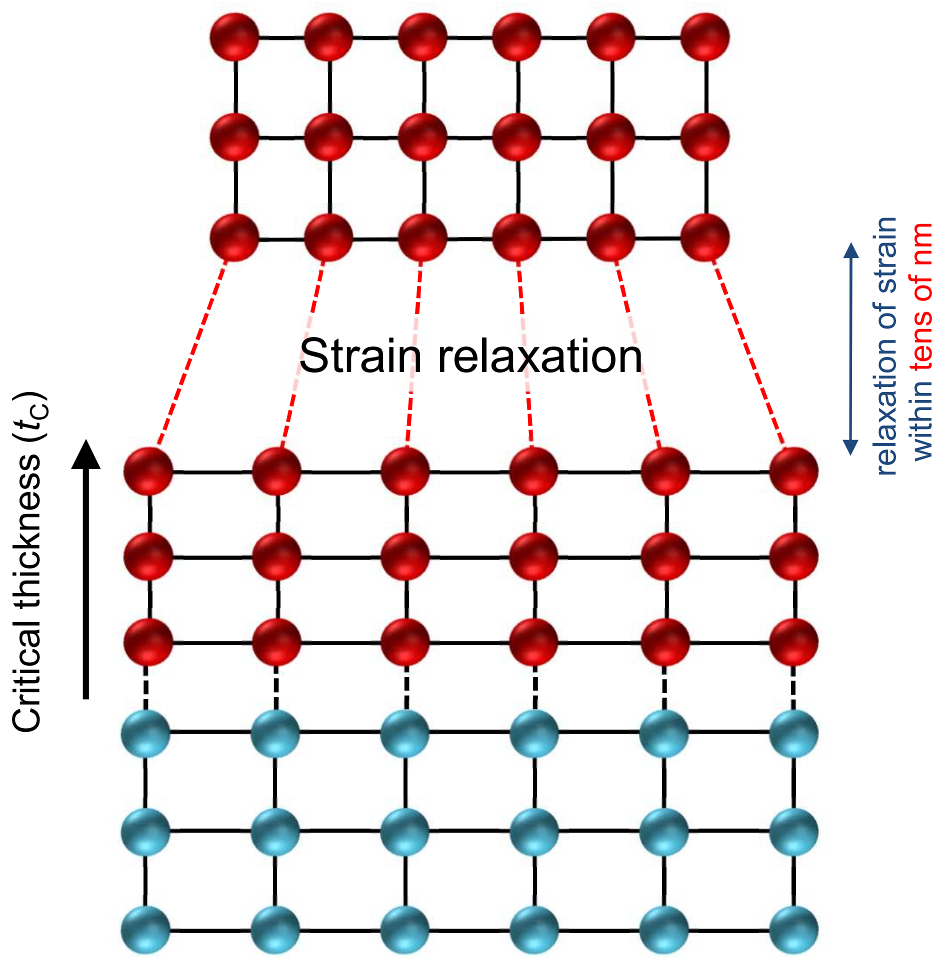


epi-stabilization



Strain gradient engineering using epitaxial Films

Growth of relaxed oxide thin film



- Above t_C, the strain usually become relaxed by elastic deformation, dislocation, etc.

$$\left\langle \frac{\partial u}{\partial z} \right\rangle \approx \frac{1\,\%}{10^1 \sim 10^2 \text{ nm}} \approx 10^5 \sim 10^6 \text{ m}^{-1}$$

"Strain Gradient Engineering" ?!

Flexoelectric effects →
Control of FE domain, MPB, self-polarization, defects, etc

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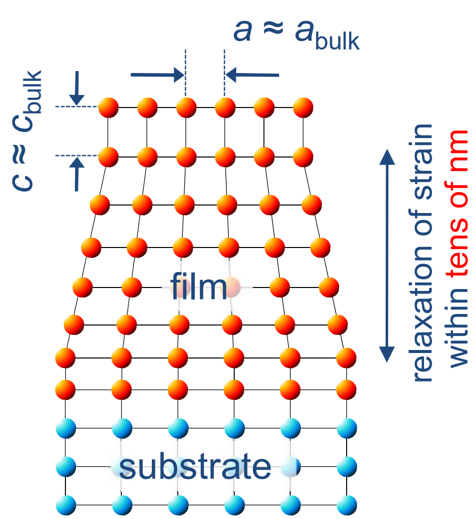
Controlling strain gradient inside FE film with P_{O2}

In most oxide thin films,

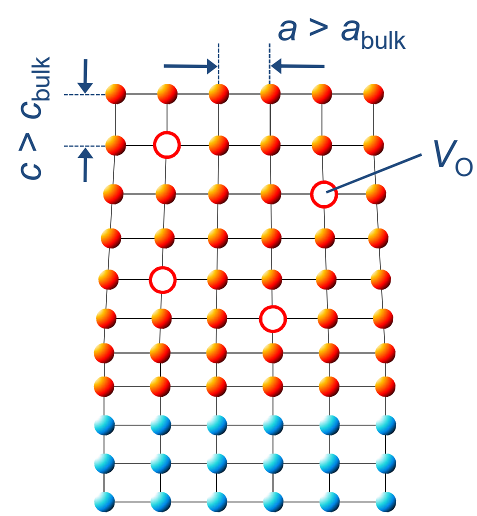
- Strain gradient can be controlled by introducing oxygen vacancies (V_O).

High oxygen pressure



<large strain gradient
by relaxation of strain>

Low oxygen pressure

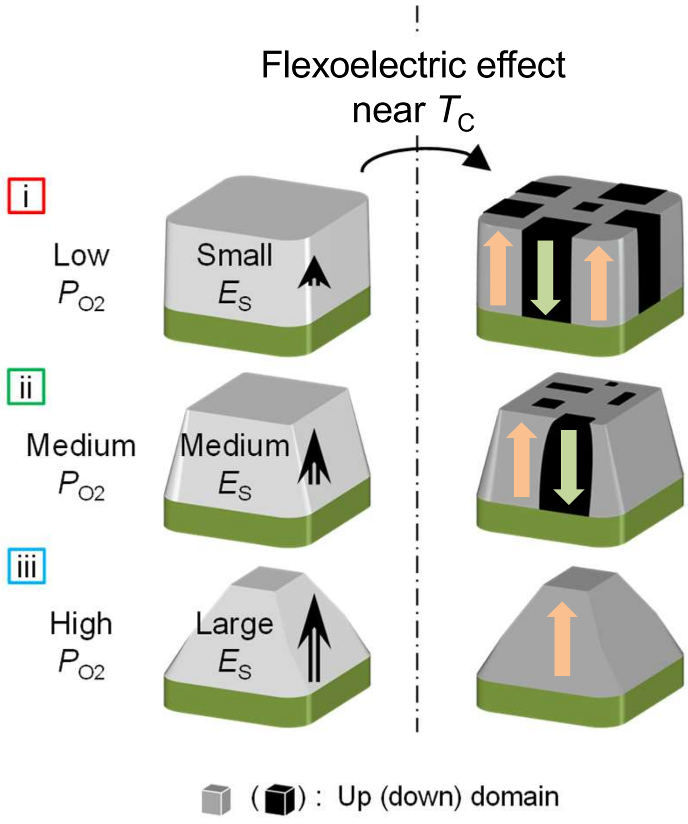


<suppressed strain gradient
by crystal volume expansion>

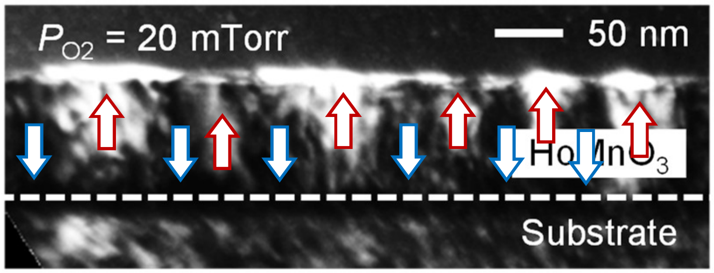
Confirmed by gazing incidence XRD

D. Lee *et al*., Phys. Rev. Lett. (2011)

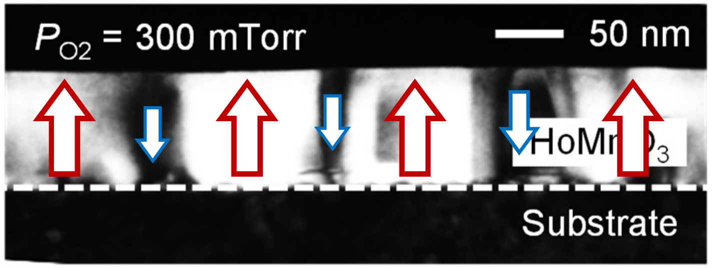
Flexoelectricity in domain formation of $HoMnO_3$ film



Modulation of FE domain pattern
by flexoelectric effect



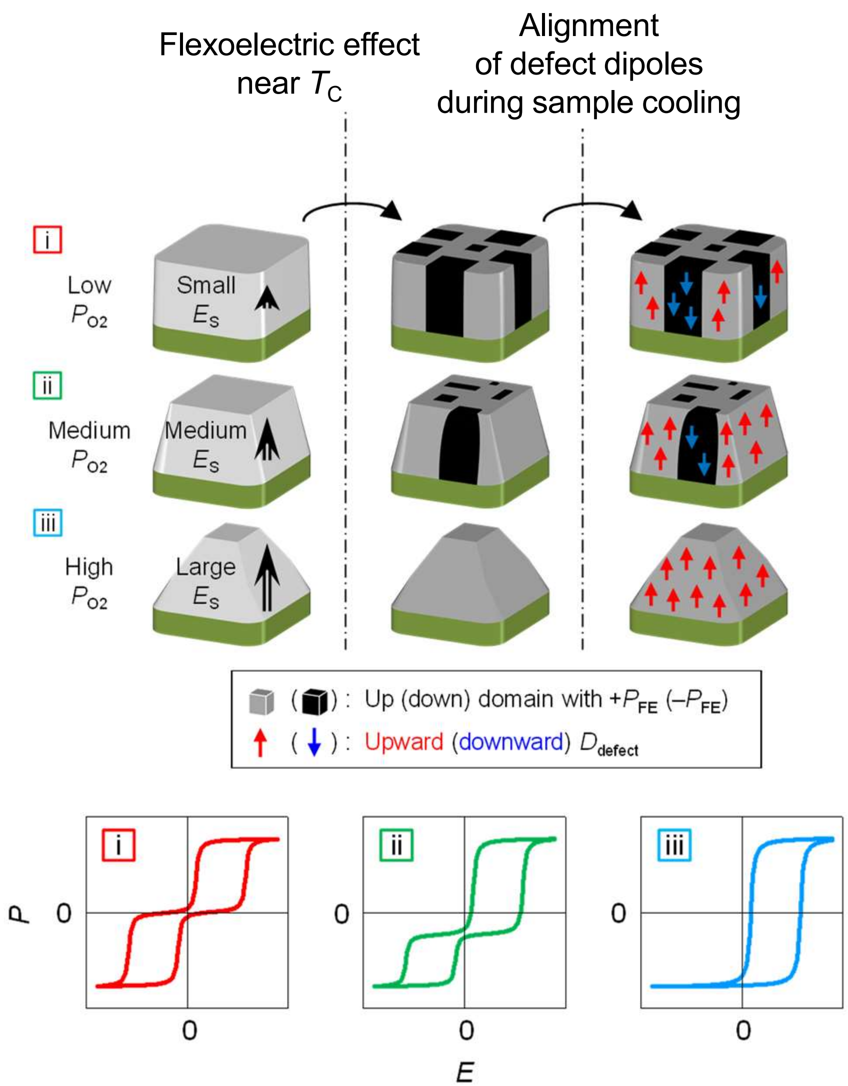
Low P_{O2} → small polydomain preferred

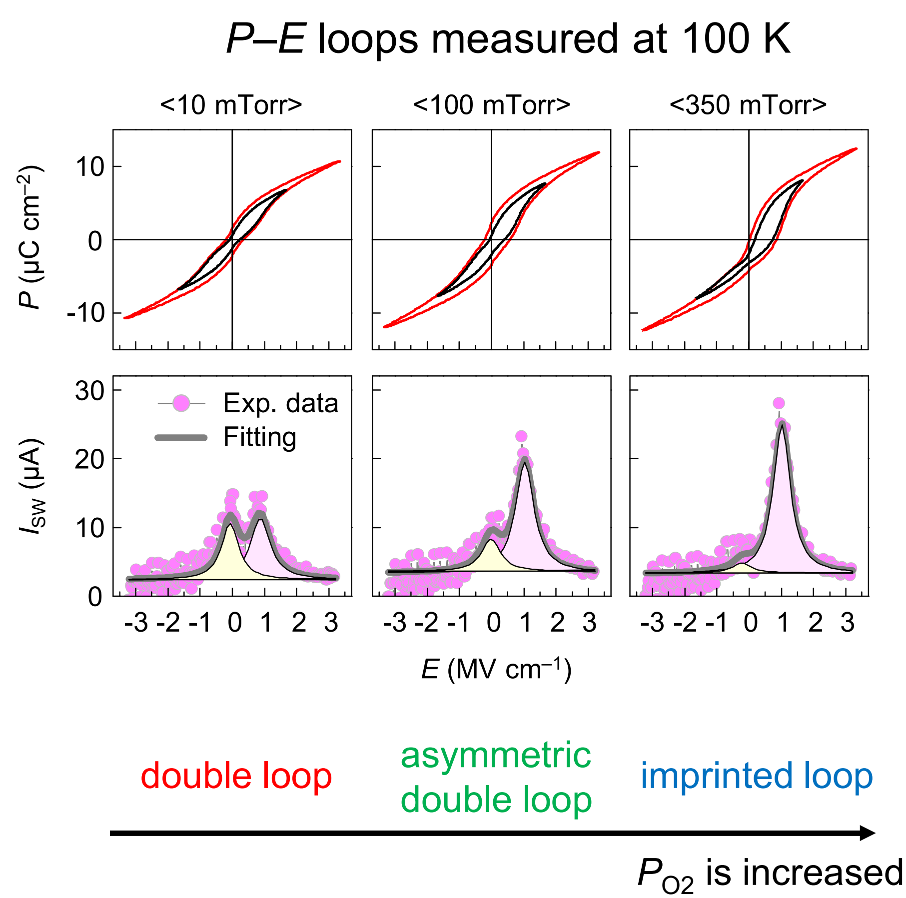


High P_{O2} → up-polarization domain preferred

D. Lee *et al*., Phys. Rev. Lett. (2011)

Flexoelectric control of *P–E* loops of $HoMnO_3$





D. Lee *et al.*, Phys. Rev. Lett. (2011)

Nanoscale flexoelectricity in solids

PRL **107,** 057602 (2011) PHYSICAL REVIEW LETTERS week ending 29 JULY 2011

Giant Flexoelectric Effect in Ferroelectric Epitaxial Thin Films

D. Lee,[1] A. Yoon,[2] S. Y. Jang,[1] J.-G. Yoon,[3] J.-S. Chung,[4] M. Kim,[2] J. F. Scott,[5] and T. W. Noh[1,*]
[1]*ReCFI, Department of Physics and Astronomy, Seoul National University, Seoul 151-747, Korea*
[2]*Department of Materials Science and Engineering, Seoul National University, Seoul 151-747, Korea*
[3]*Department of Physics, University of Suwon, Suwon, Gyunggi-do 445-743, Korea*
[4]*Department of Physics and CAMDRC, Soongsil University, Seoul 156-743, Korea*
[5]*Department of Physics, University of Cambridge, Cambridge CB3 0HE, United Kingdom*
(Received 4 April 2011; published 29 July 2011)

We report on nanoscale strain gradients in ferroelectric $HoMnO_3$ epitaxial thin films, resulting in a giant flexoelectric effect. Using grazing-incidence in-plane x-ray diffraction, we measured strain gradients in the films, which were 6 or 7 orders of magnitude larger than typical values reported for bulk oxides. The combination of transmission electron microscopy, electrical measurements, and electrostatic calculations showed that flexoelectricity provides a means of tuning the physical properties of ferroelectric epitaxial thin films, such as domain configurations and hysteresis curves.

First demonstration of giant & controllable flexoelectric effect at the nanoscale

Nanoscale strain relaxation in epitaxial ferroelectric $HoMnO_3$ thin films
→ Critical influence on ferroelectric domain structures

D. Lee *et al*., Phys. Rev. Lett. (2011)

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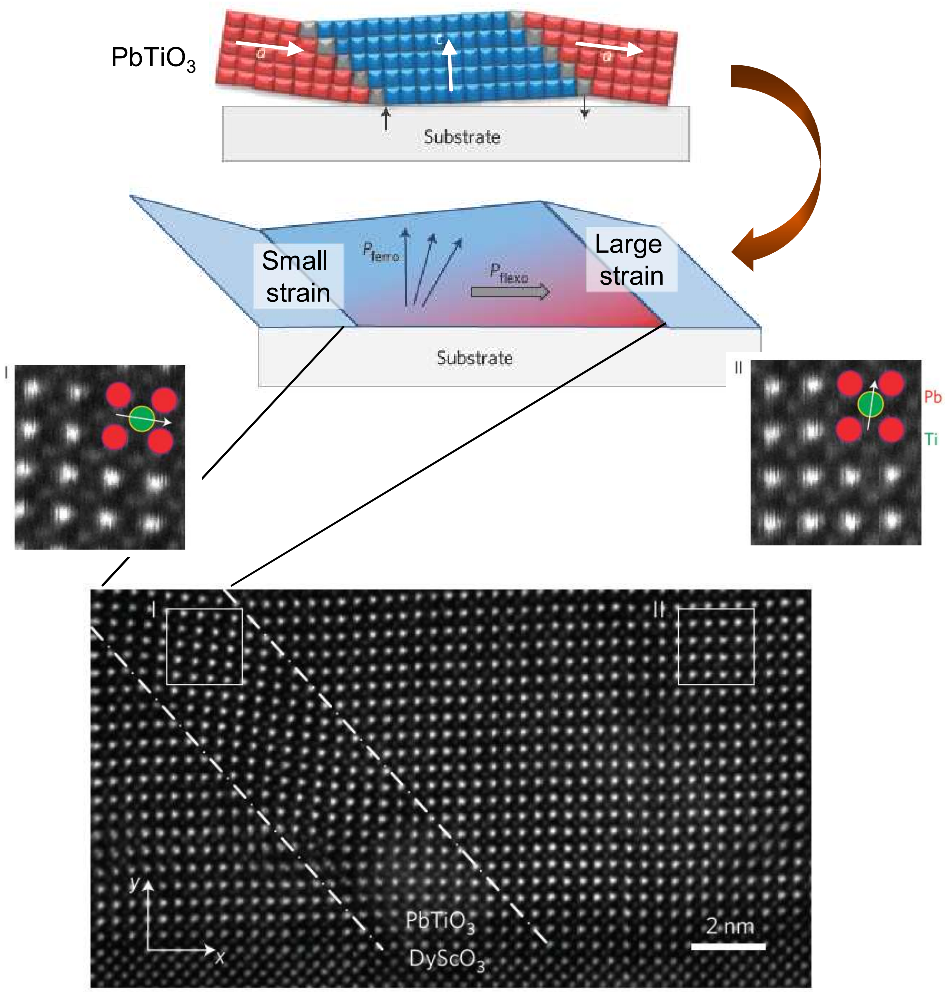
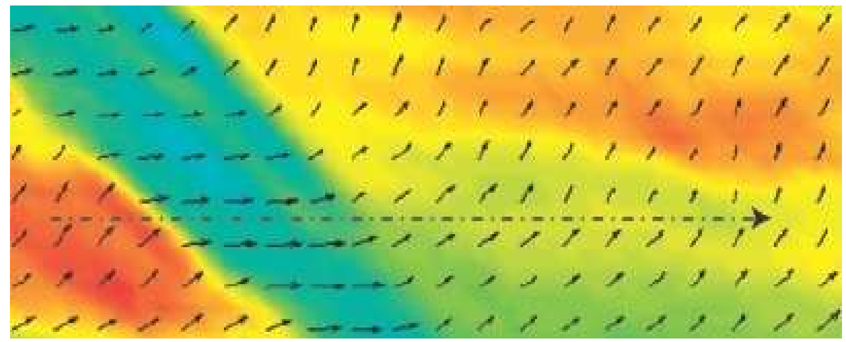
Domain Wall: Polarization Rotation due to FlexoE



Polarization vector map



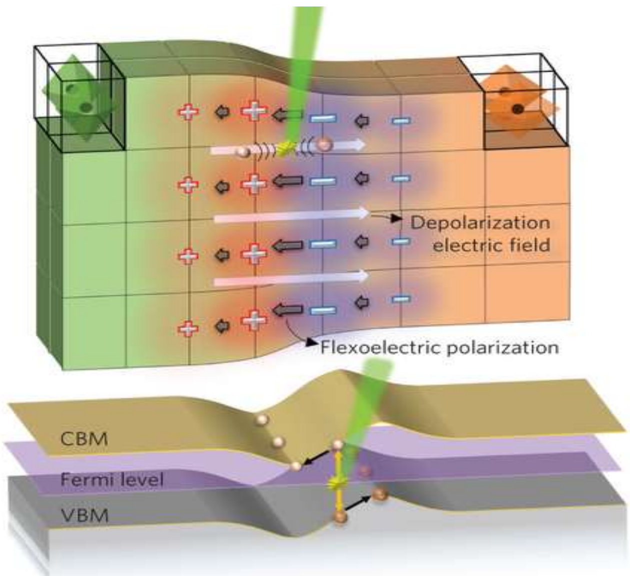
Large (in-plane) strain gradient in ferroelastic (*c*/*a*/*c*/*a*) domains

- Flexoelectric effect
- Rotation of polarization from vertical to horizontal direction.

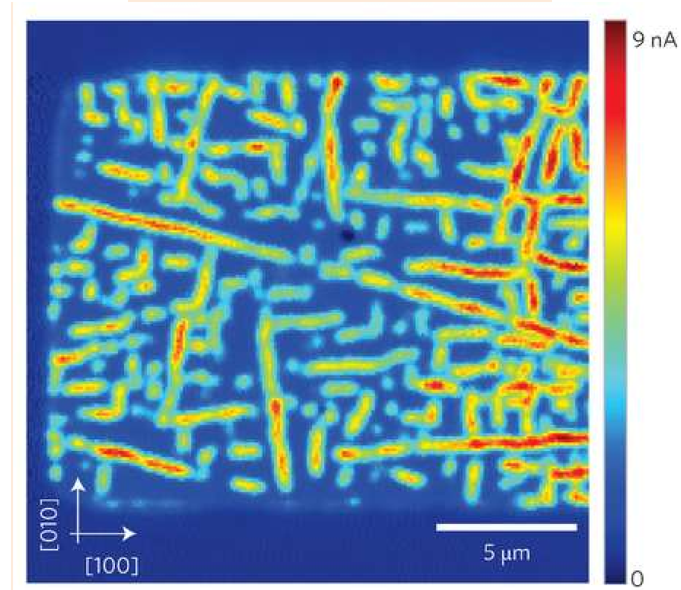
G. Catalan *et al.*, Nature Mater (2011)

Structural phase boundary: photocurrent enhancement

Tetragonal (T) $BiFeO_3$　　*Rhombohedral (R) $BiFeO_3$*



Photocurrent map



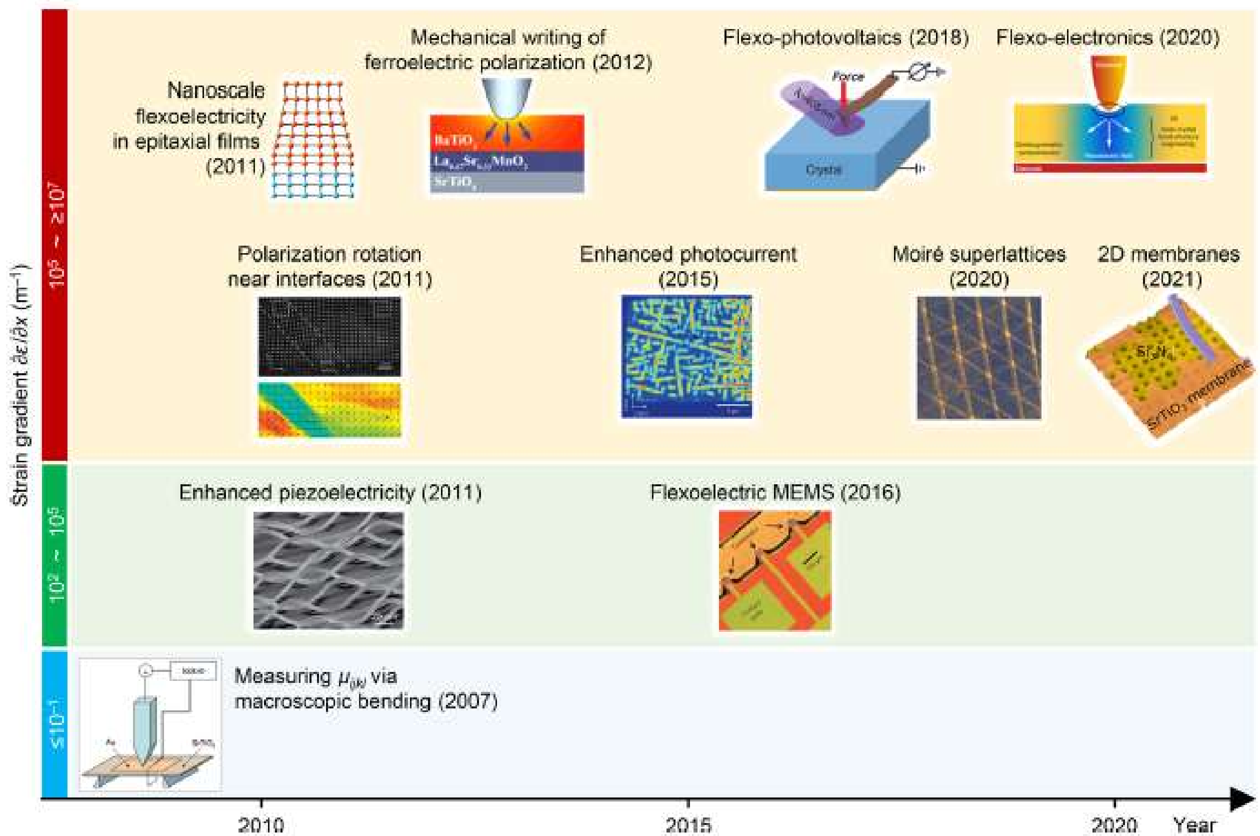
Strain gradient near structural phase boundary

- Flexoelectric polarization
- Splits the photo-induced electron-hole pairs

- T-BFO: low photo-current
- T+ R BFO : large photo-current

K .Chu, C.H. Yang *et al*., Nature Nanotech (2015)

Key milestones in the flexoelectric research



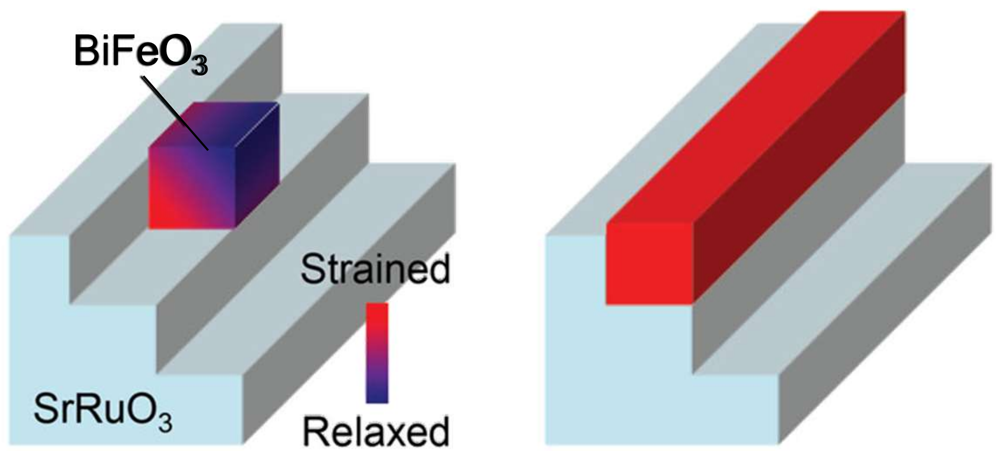
Review articles

D. Lee & T. W. Noh, Philos. Trans. R. Soc., A **370**, 4944 (2012); P. Zubko *et al.*, Annu. Rev. Mater. Res. **43**, 387 (2013); P. V. Yudin & A. K. Tagantsev, Nanotechnology **24**, 432001 (2013); T. D. Nguyen *et al.*, Adv. Mater. **25**, 946 (2013); L. Shu *et al.*, J. Adv. Ceram. **8**, 153 (2019); B. Wang *et al.*, Prog. Mater. Sci. **106**, 100570 (2019); …..
D. Lee, APL Mater. **8**, 090901 (2020); S. M. Park *et al*, Appl. Phys. Rev. **8**, 041327 (2021)

Controlling strain gradient with growth temperature

In $BiFeO_3$ thin film

- Adatom mobility, determining film growth, can differ by growth temperature
- Strain gradient can be controlled by deposition temperature T_D



Low T_D

Low adatom mobility

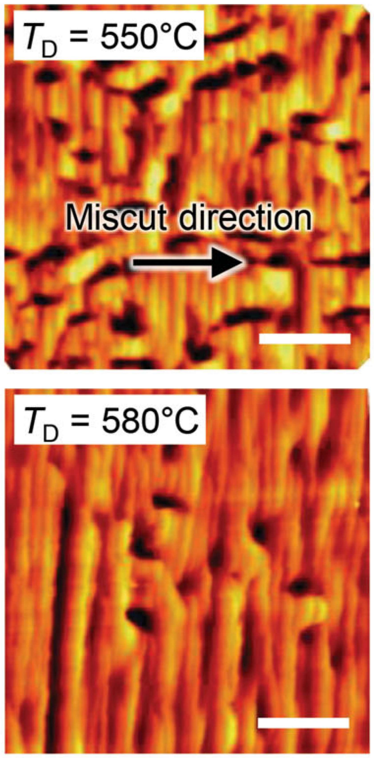
→ easily relaxed

High T_D

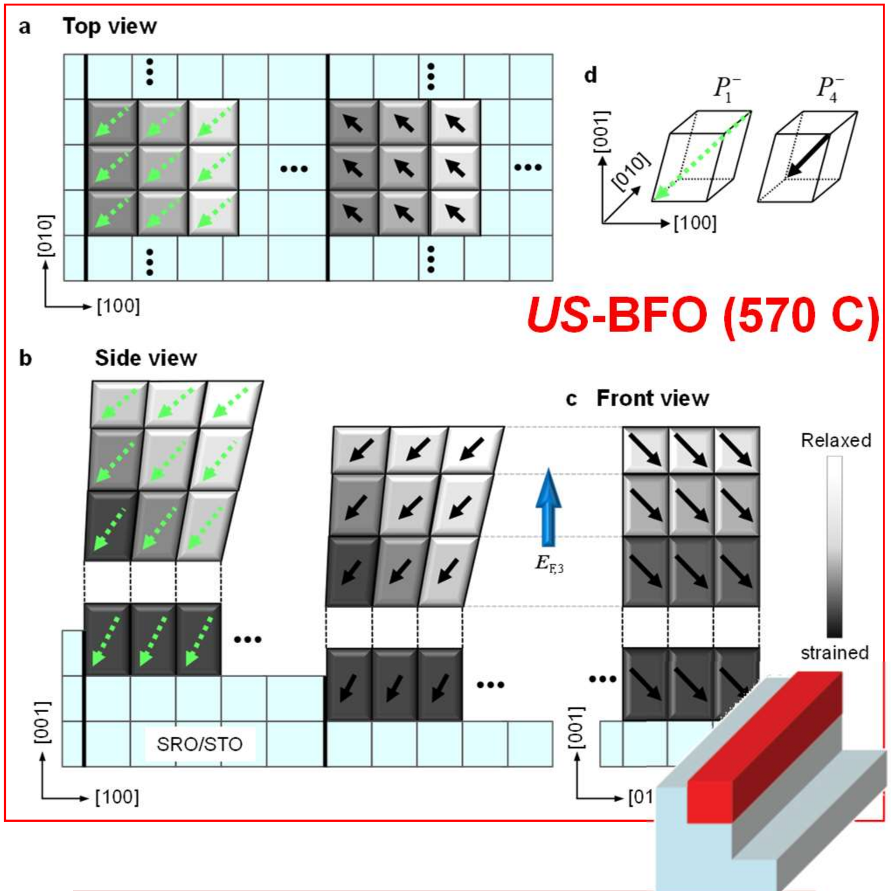
High adatom mobility

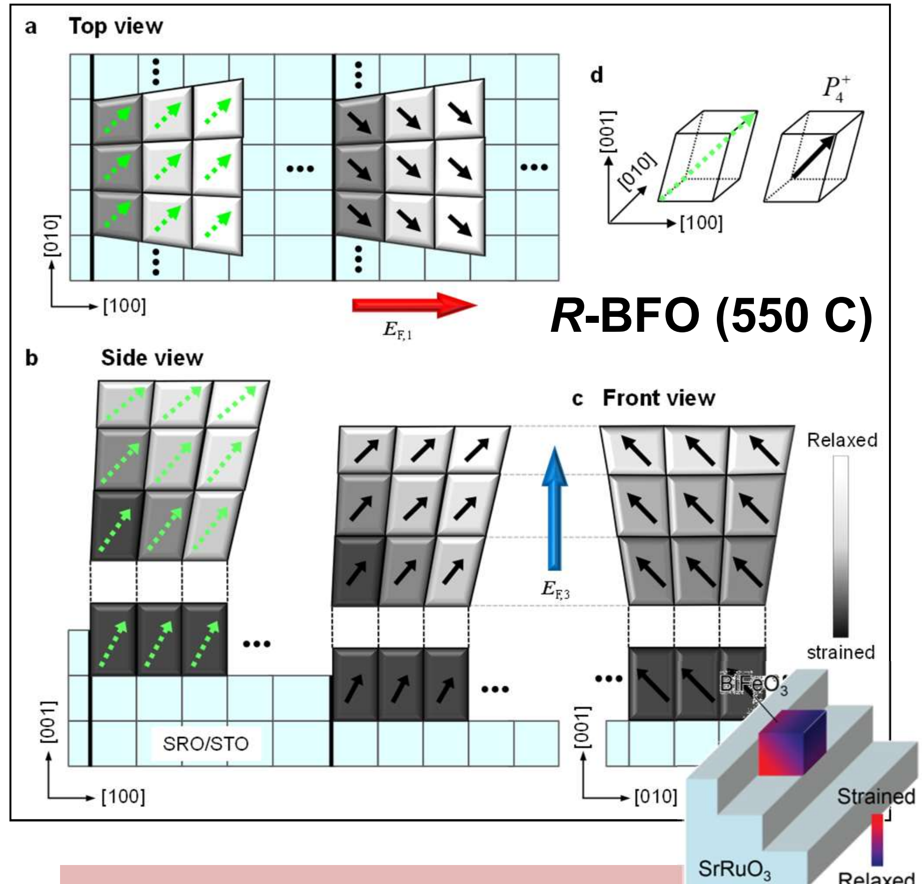
→ little relaxation along step edge

AFM image



Controlling FlexE field : T_D dependence



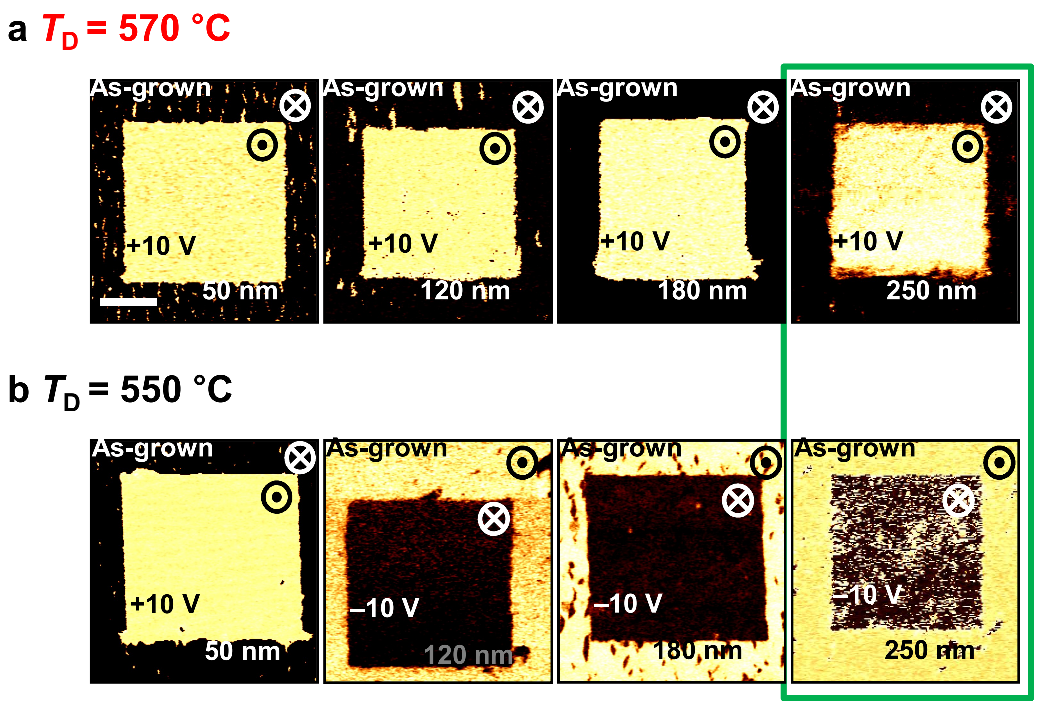


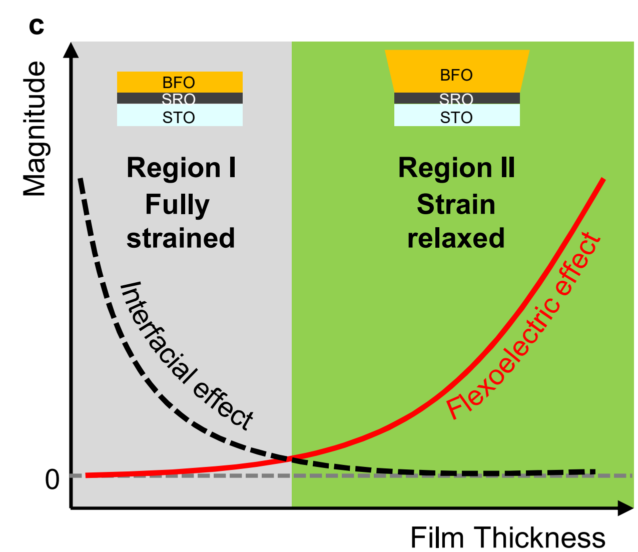
Uniaxially strained lattice
- **Out-of-plane** $E_{flexo,3} \approx 2.3$ MV m^{-1}

All lattice constants are relaxed
- **Out-of-plane** $E_{flexo,3} \approx 3.2$ MV m^{-1}
- **In-plane** $E_{flexo,1} \approx 8.7$ MV m^{-1}

B. C. Jeon et al., Adv. Mat. (2013)

Reversal of self-polarization





Direction of the self-polarization is determined by competition of interfacial and flexoelectric effects in $BiFeO_3$ films.

B. C. Jeon et al., Adv. Mat. (2013)

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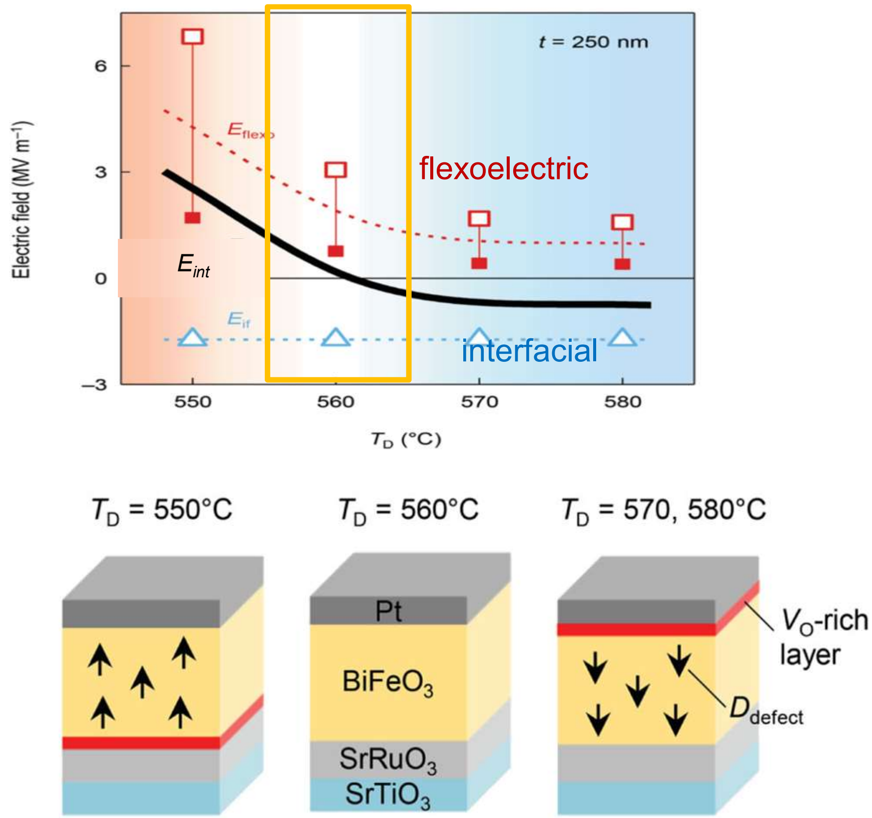
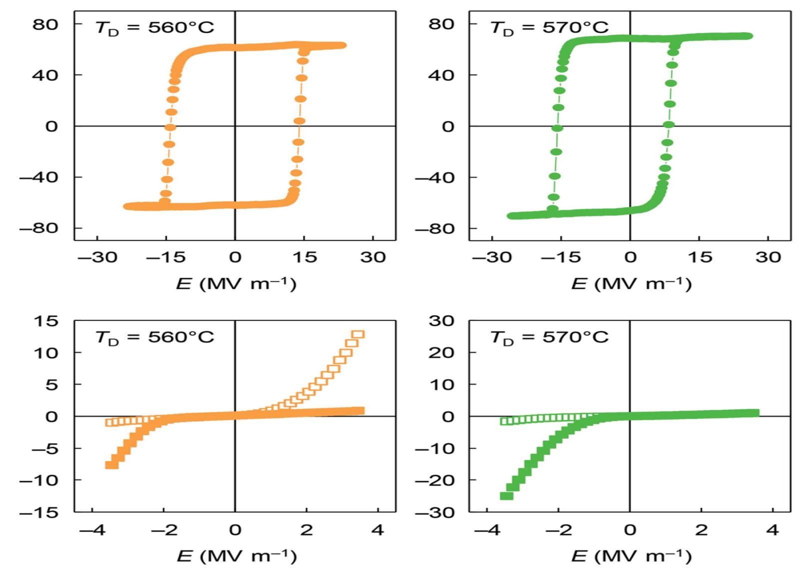
More examples of flexoelectric effects

***Defect Formation**



D. Lee *et al.*, Adv. Mat. (2014)

***Transport Properties**



Nano. Lett. (2012); Adv. Mat. (2014)

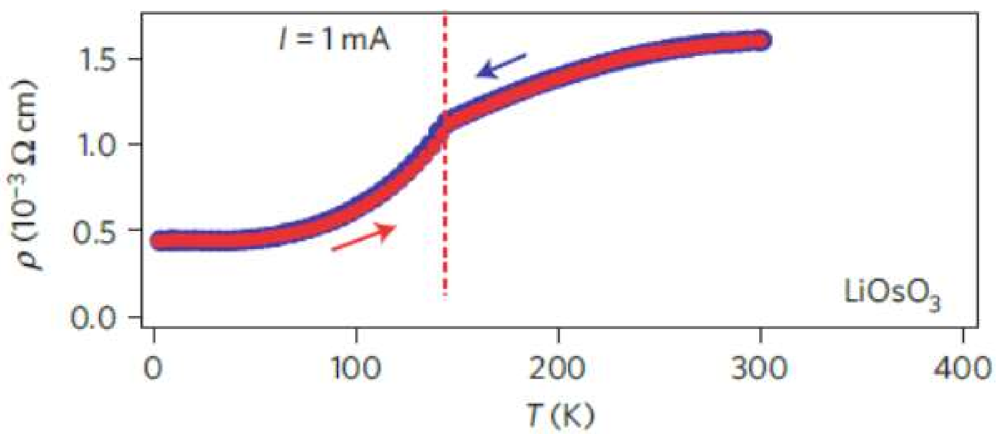
polar metal?

- **A metal that contains an electric dipole moment.** And, its components have an ordered electric dipole.
- With common sense, such metals should be unexpected, because the free electrons will neutralize the polarized charge.
- However, P.W. Anderson and E. I. Blunt theoretically predicted the existence of polar metals from the symmetry consideration on Martensitic transformation. [Phys. Rev. Lett. (1965)]
- Up to this point, they have been reported in $LiOsO_3$, $LaNiO_3$, and $(Sr,Ca)TiO_3$ 2DEGs. They usually have small amounts of free carriers.

Y. Shi *et al.*, Nature Mater. (2013)

T. H. Kim *et al.*, Nature (2016)

J. Brehin, *et al.*, Nature Physics (2023)



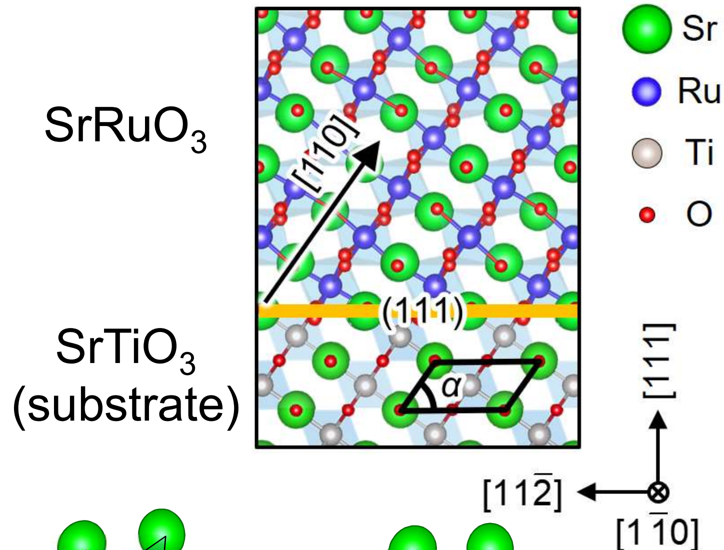
Flexo-polar metal

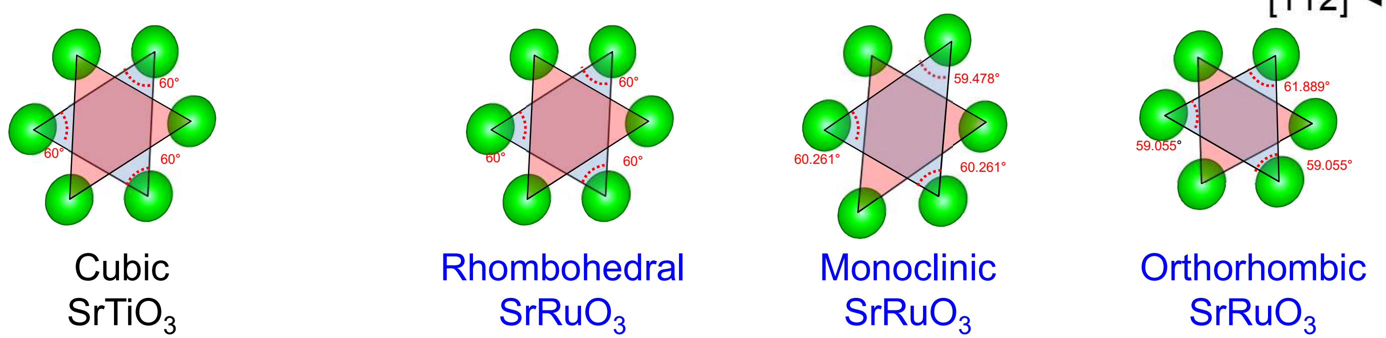
- How to design a large polarization inside a metallic film?

$SrRuO_3$ (bulk)

- highly metallic
- ferromagnetism (T_C ~ 160 K)
- orthorhombic structure

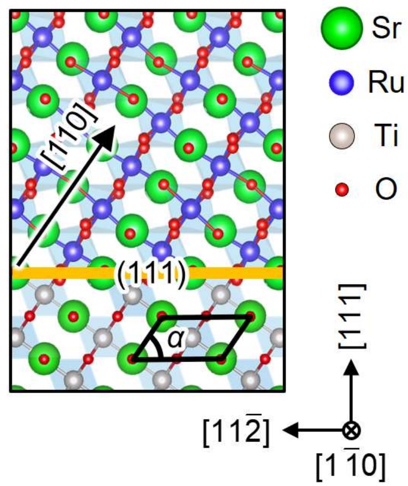
$SrRuO_3/SrTiO_3(111)$ film

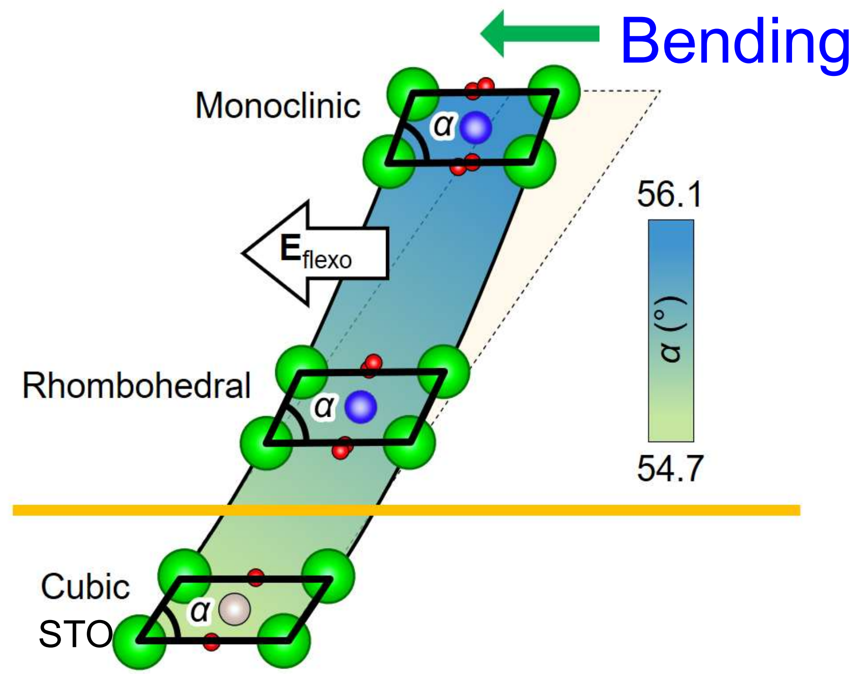




Flexo-polar metal

- How to design a large polarization inside a metallic film?

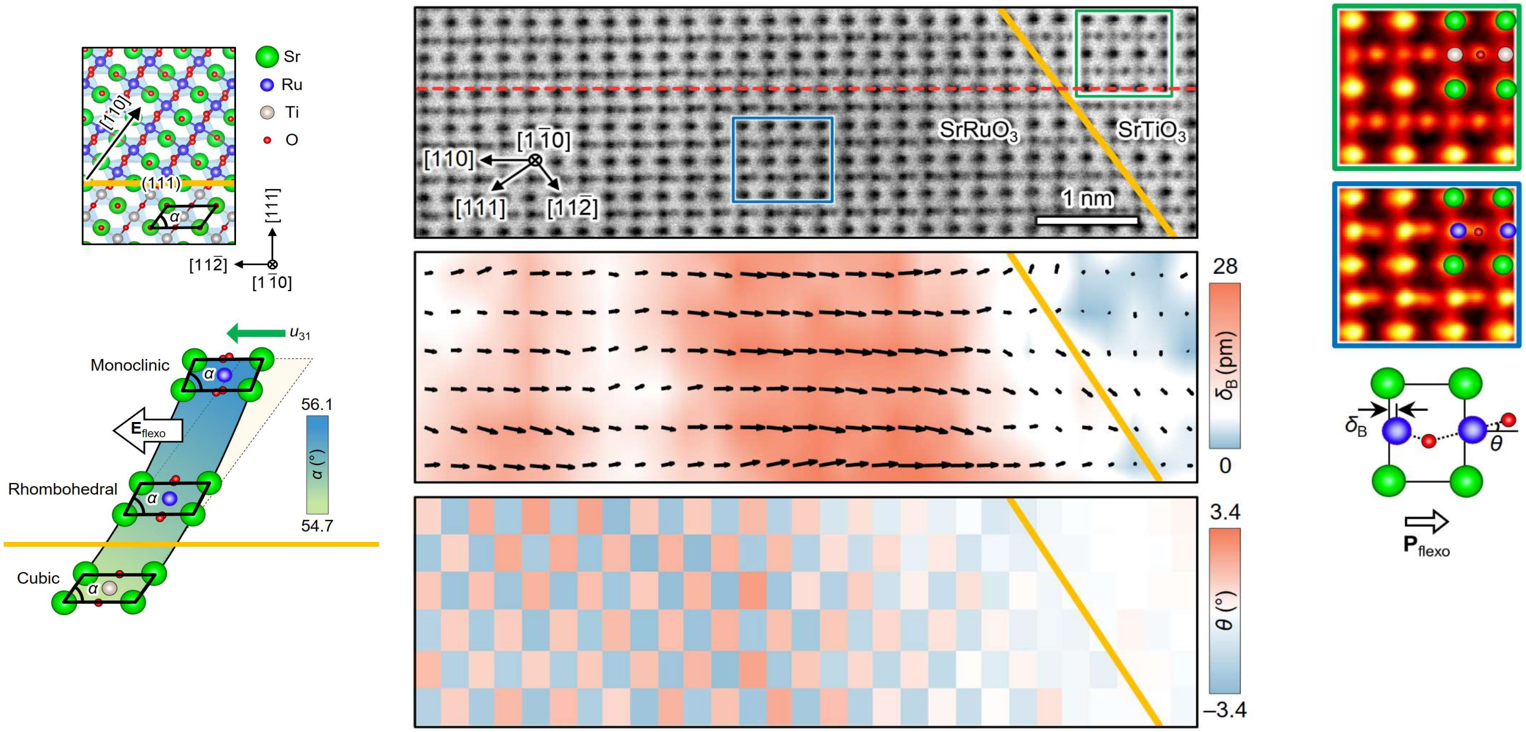




Inherent symmetry-lowering structural evolution
→ Strain gradient (> 10^6 m^{-1}) in $SrRuO_3$ (111) films

Flexo-polar metal

- Atomic-scale imaging : shear strain gradient and flexo-polar phase

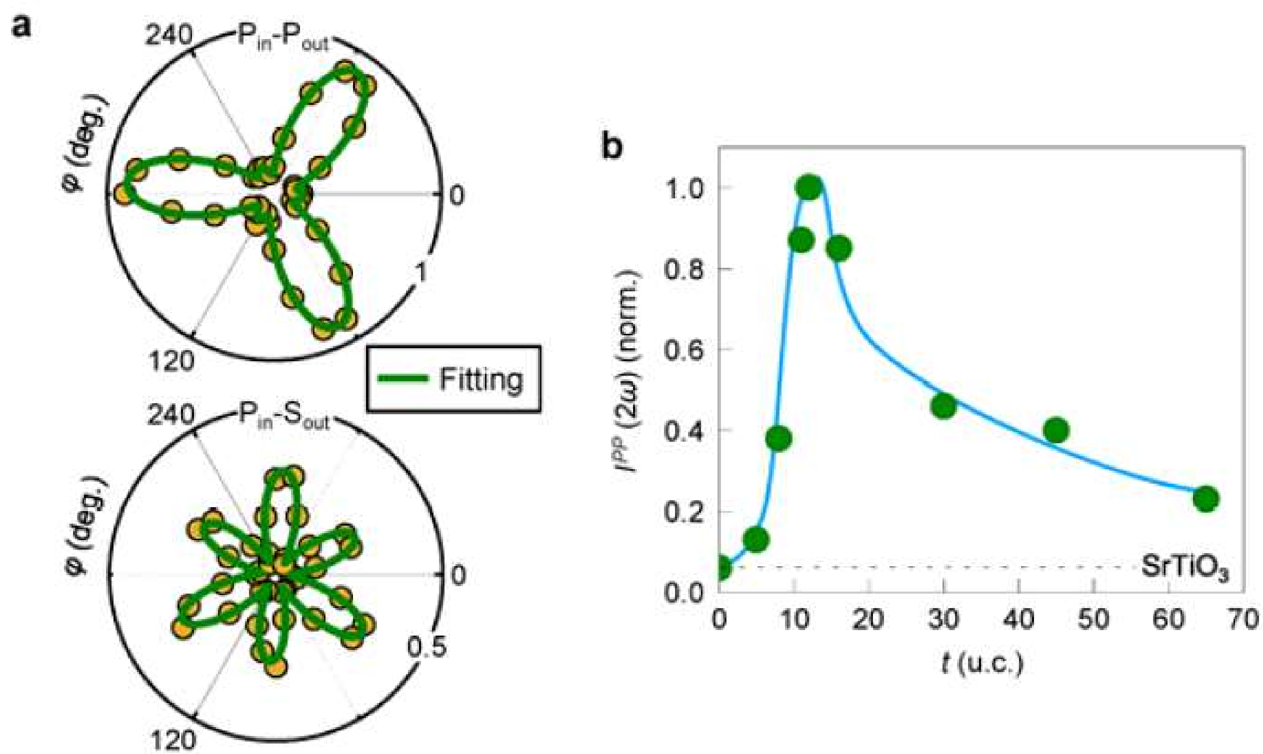


- Shear strain gradient (a few 10^6 m^{-1})
- Strong bulk polarity via Ru off-centering
- Gradual structural evolution

W Peng et al., (submitted)

Flexo-polar metal

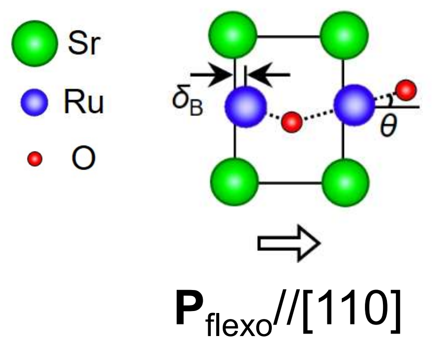
- Second Harmonic Generation (SHG) to probe broken inversion-symmetry

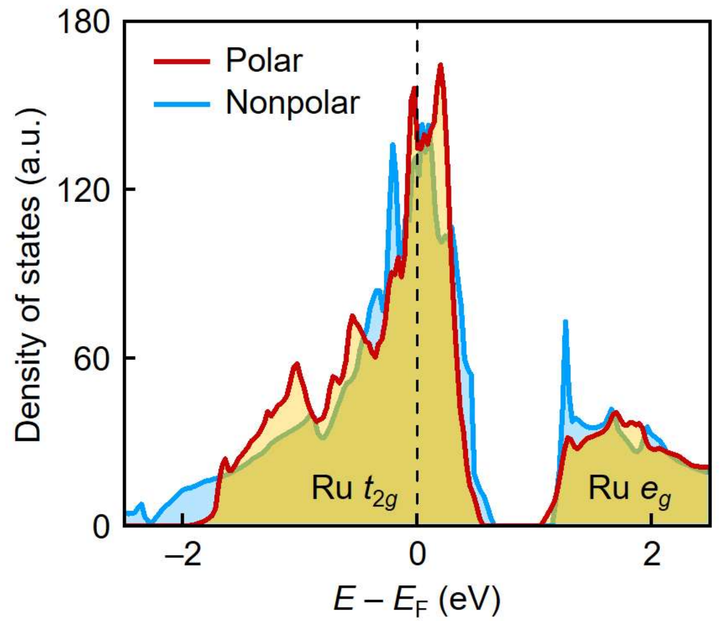


First demonstration of flexo-polar metal?
A new way to make polar metals?

W Peng et al., (submitted)

Flexo-control of electronic properties





Ru-driven bulk polarity → Ru *d*-orbital band reconstruction

Notable band narrowing → Increased electron correlation

Enhanced magnetic ordering by Stoner's theory:
Increase of M (or T_C) by 25% (or 10 K)

Outline

1. Introduction on flexoelectricity

2. Controlling physical properties of oxide thin films by flexoelectric effects
   : ferroelectric domain formation & switching, polarization rotation, defect pinning and related transport, photocurrent, ….

3. Functional manipulation of oxide thin films by applying pressure with an AFM tip

4. Potential applications

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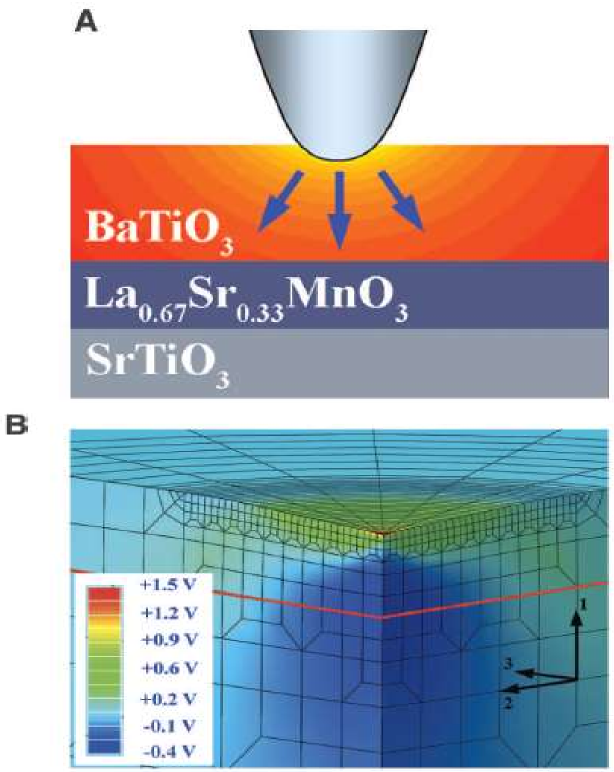
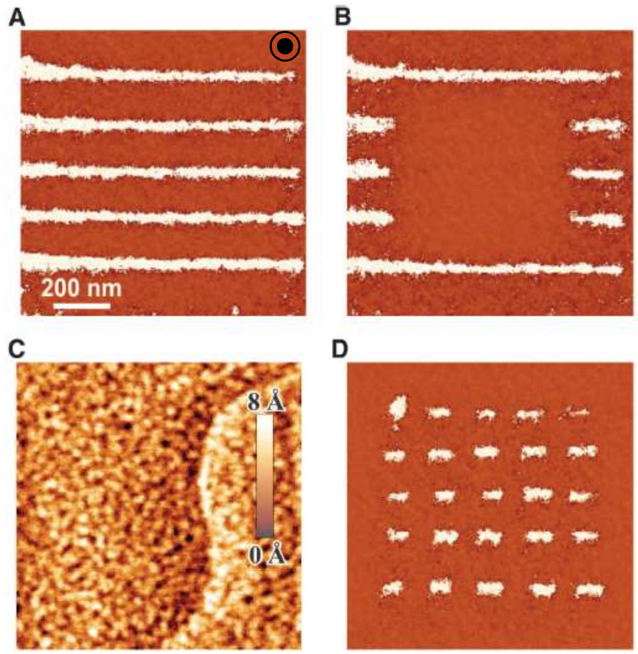
Mechanical switching of polarization in FE $BaTiO_3$

Domain switching with force

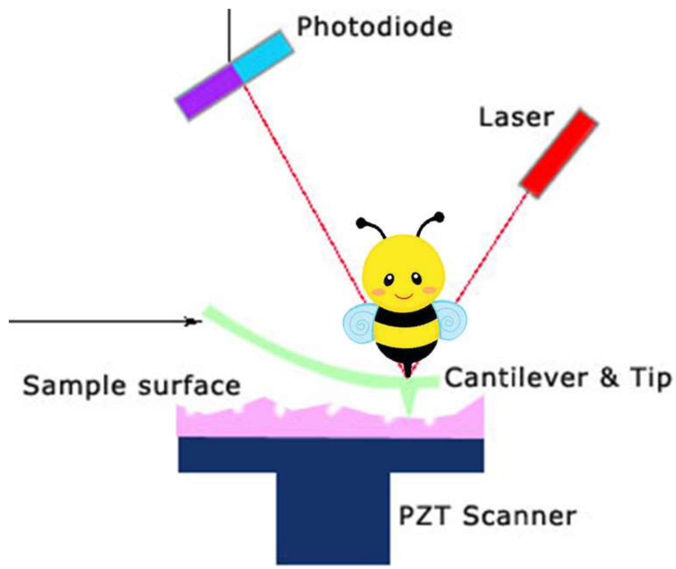


Mechanically switched domain



H. Lu *et al.*, Science (2012)

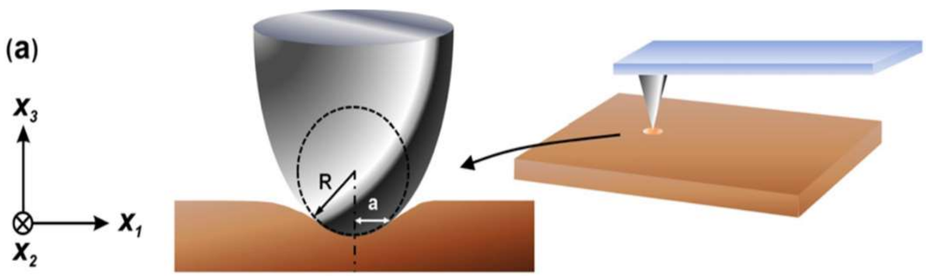
Functional manipulation with an AFM tip



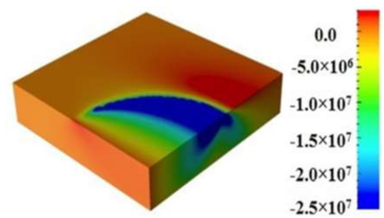
Up to ~ 1 GPa

~ 1 GPa

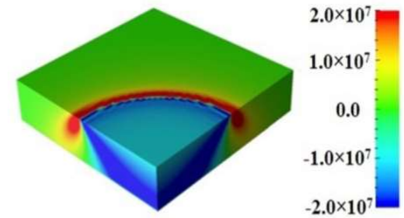
Mechanical switching : Theoretical explanation



Flexoelectric field (V/m) $$E_k^{flexo} = f_{ijkl}\frac{\partial \varepsilon_{ij}}{\partial x_l}$$

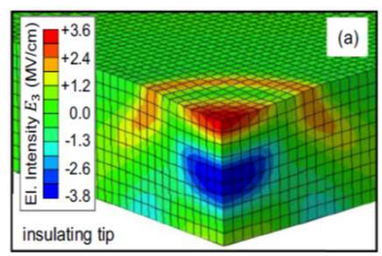


In-plane flexofield (V/m)

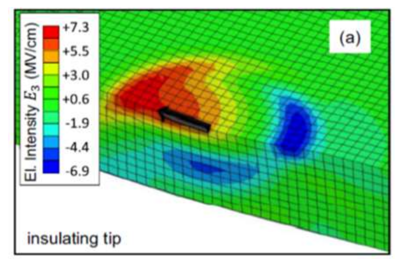


Out-of-plane flexofield (V/m)

Gu et al., Appl. Phys. Lett. 2015



Static tip



Sliding tip

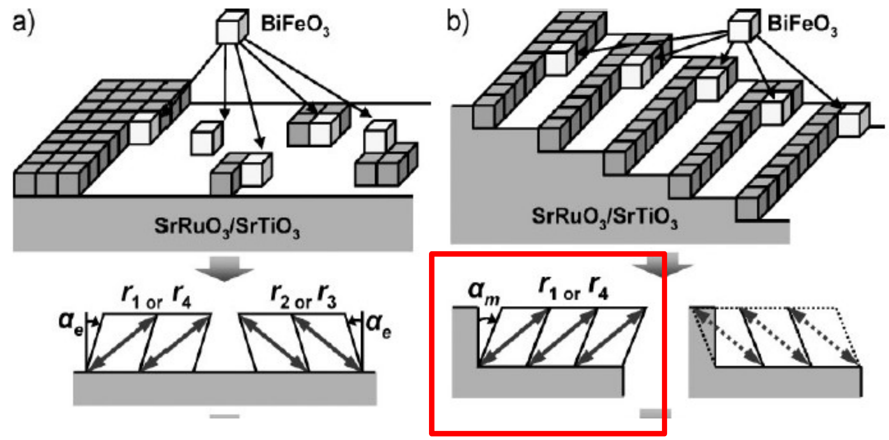
Ocenesak et al., Phys. Rev. B 2015

- The in-plane flexofield is comparable with out-of-plane.
- The scanning probe motion changes the shape of flexofield.

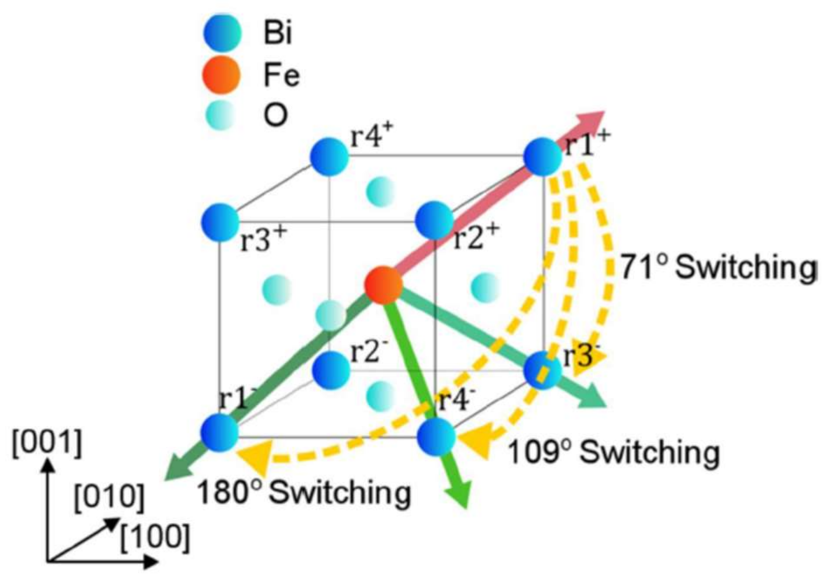
Ferroelectric $BiFeO_3$ Thin Film

- $BiFeO_3$: A well-known multiferroic
  - – Rhombohedral structure (bulk)
  - – FE & AFM
  - –T_C~1103 K, T_N~843 K
  - – P_{FE} ~ 100 μC cm^{-2} along [111]
  - – 8 possible polarizations

on $SrTiO_3$(100) substrates with step edges



H. W. Jang *et al*., adv. Mater. 21, 817 (2009)



Possible pathways: 71° & 180°

There are three different ferroelectric switching pathways in $BiFeO_3$:
i.e. 71°, 109°, 180° switchings.

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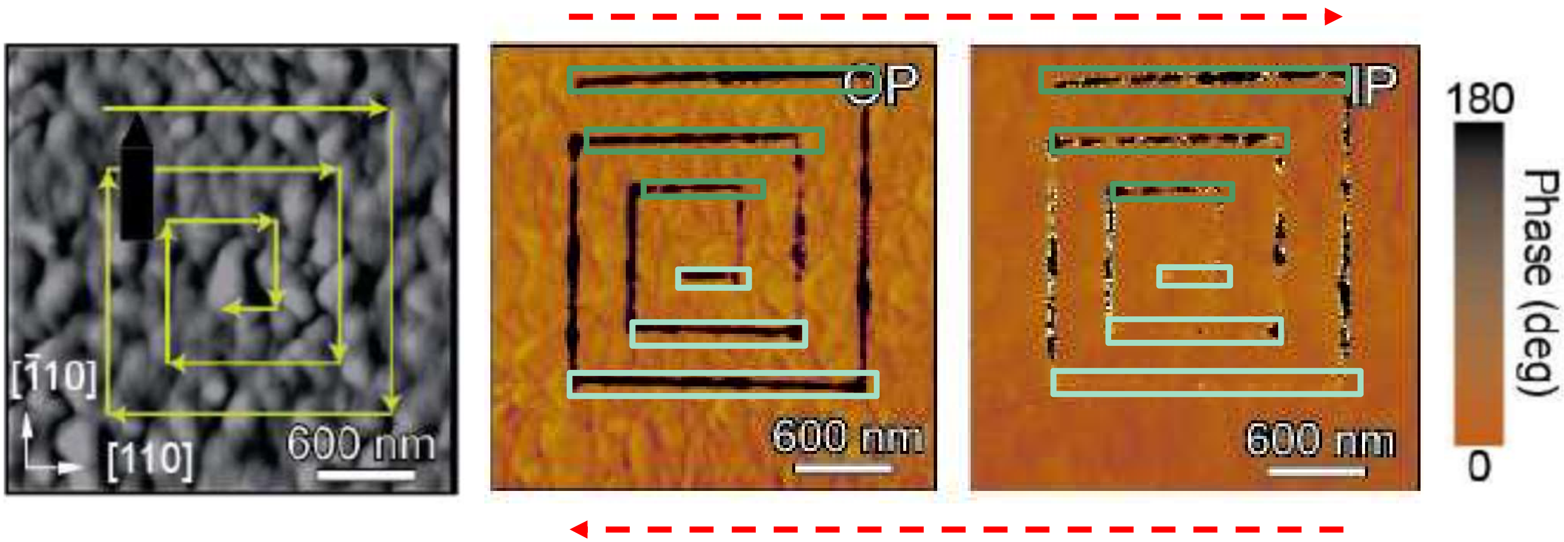
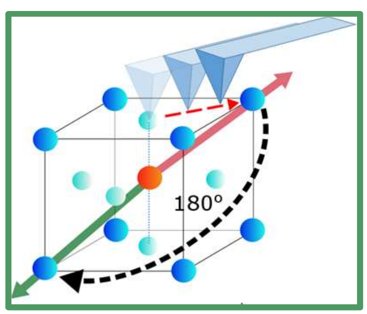
Selective ferroelectric switching by controlling tip motion

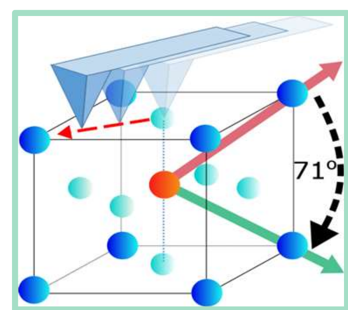
Sample: $BiFeO_3$ thin film (*P* along body-diagonals)

180° switching for tip motion along [110]



71° switching for tip motion along [-1-10]





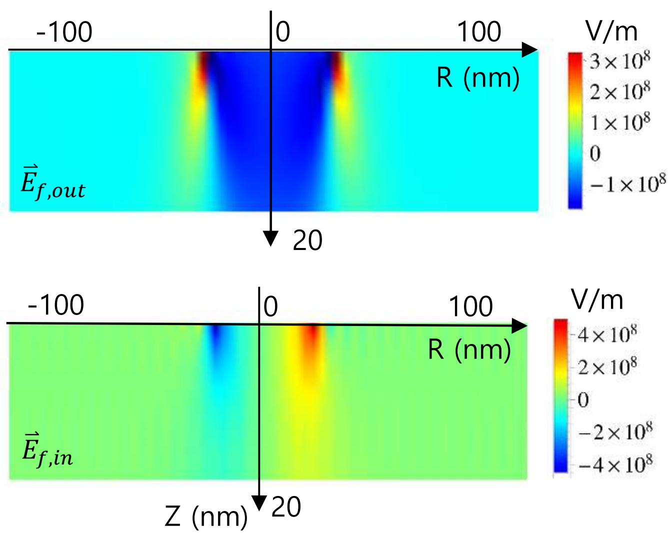
S. M. Park *et al*., Nature Nanotech. (2018)

Selective ferroelectric switching by controlling tip motion

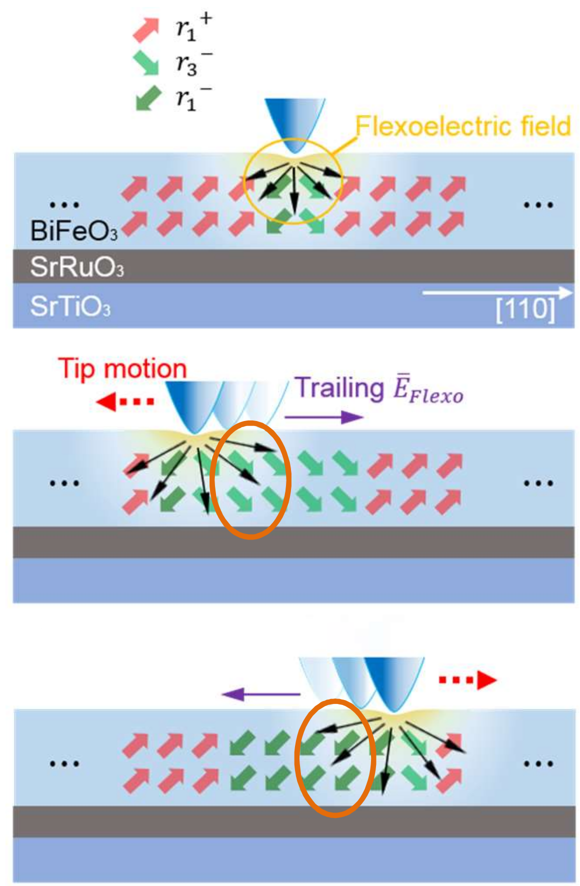
Phase field simulation: trailing flexoelectric field

Prof. L. Q. Chen (PSU)

Flexoelectric field distribution (static case)



Effect of moving tip

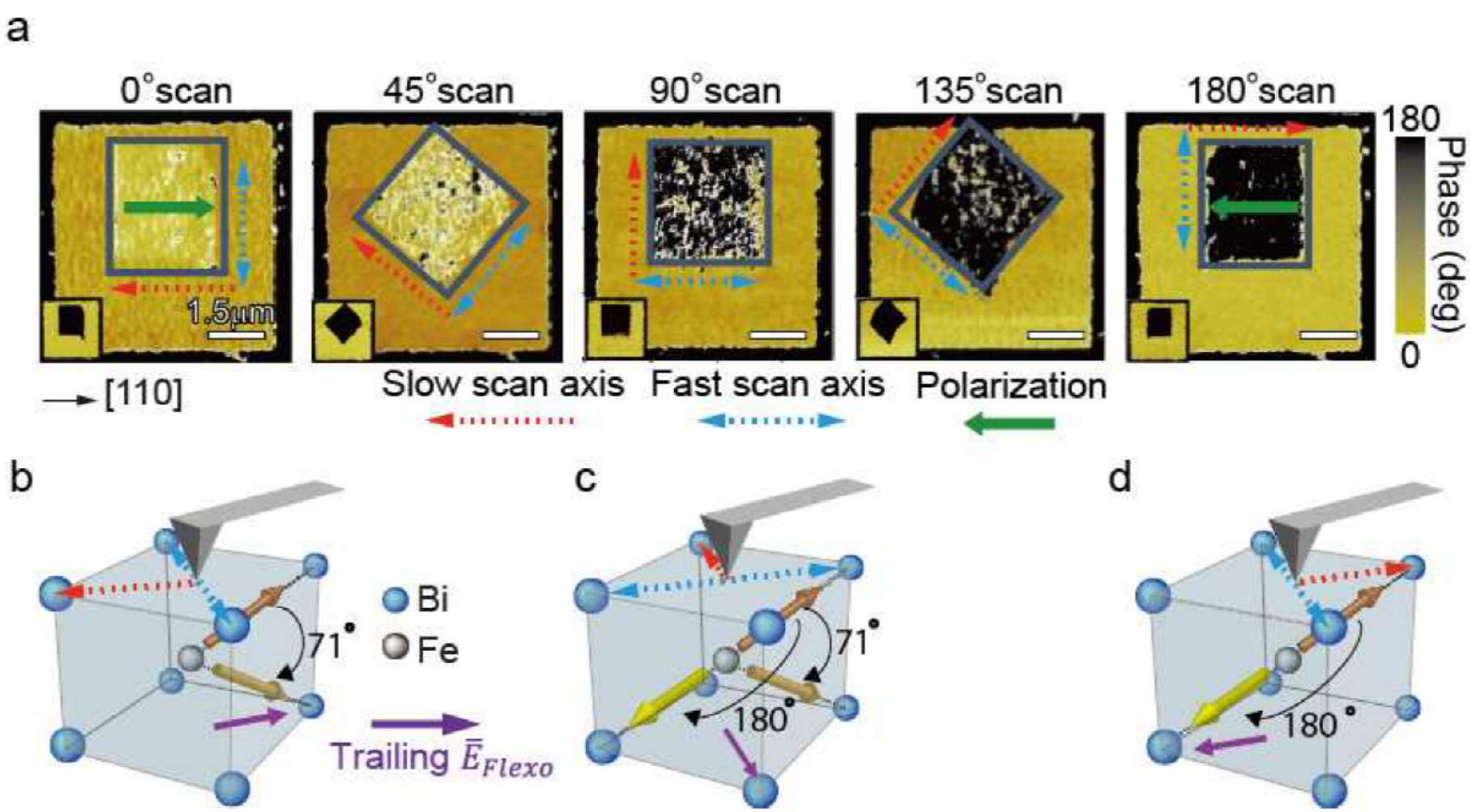


Motion of the tip effectively breaks the symmetry of the FlexE field.

Generates an effective field at the wake of the tip–trailing FlexE field.

S. M. Park *et al*., Nature Nanotech. (2018)

FE polarization switching by mechanical 2D scan



- The direction of trailing flexoelectric field is opposite to the slow scan axis in 2D scan case.

S. M. Park *et al*., Nature Nanotech. (2018)

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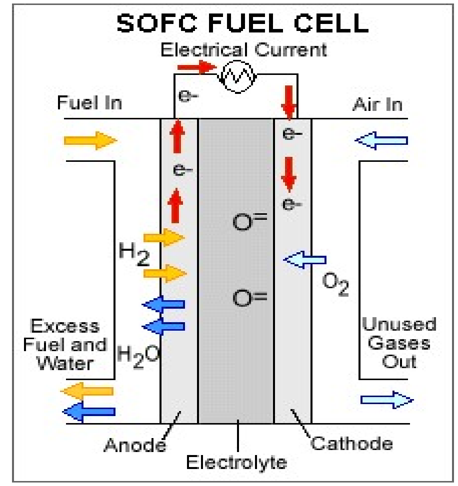
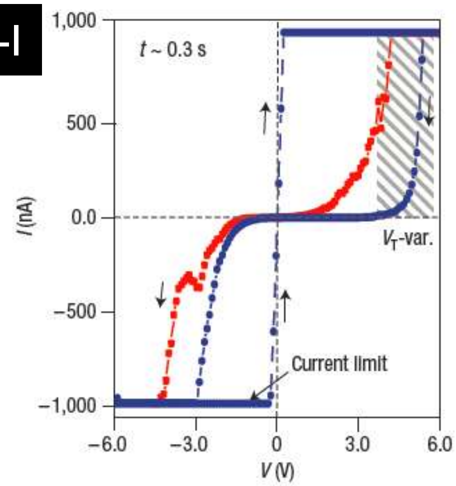
Importance of oxygen vacancy control in oxides

Oxygen vacancies are important, since they can determine the physical properties of oxides. So their control is very important for oxide electronics. Can we use AFM for moving V_O?

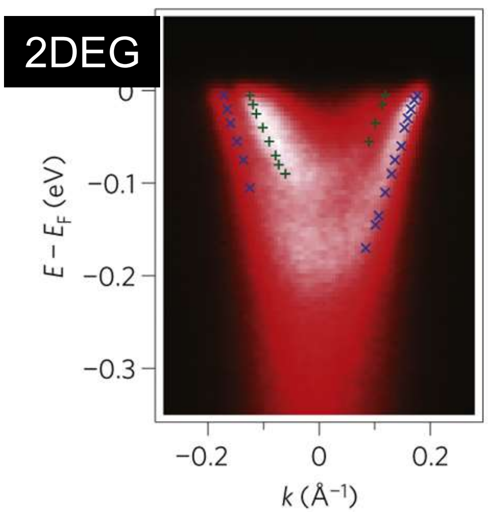


M-I



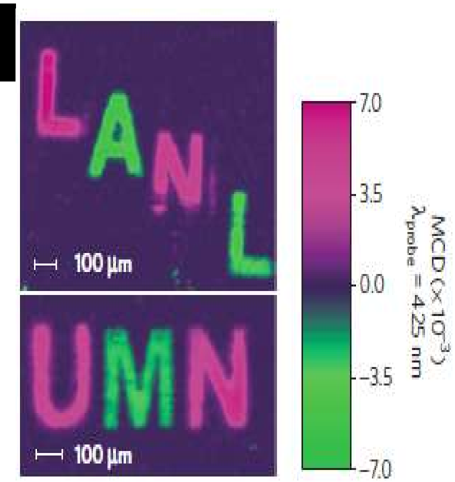
K. Szot *et al*., Nat. Mater. (2006)

2DEG



W. Meevasana *et al*., Nat. Mater. (2011)

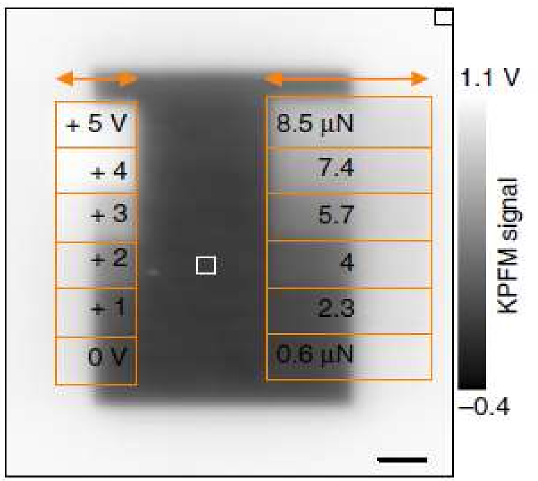
Magnetism

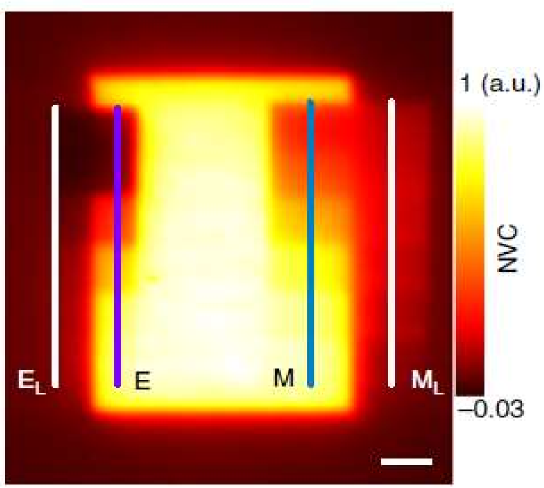


W.D. Rice *et al*., Nat. Mater. (2014)

Studying V_O and their diffusion by AFM tip

Sample: $SrTiO_3$/Nb:$SrTiO_3$

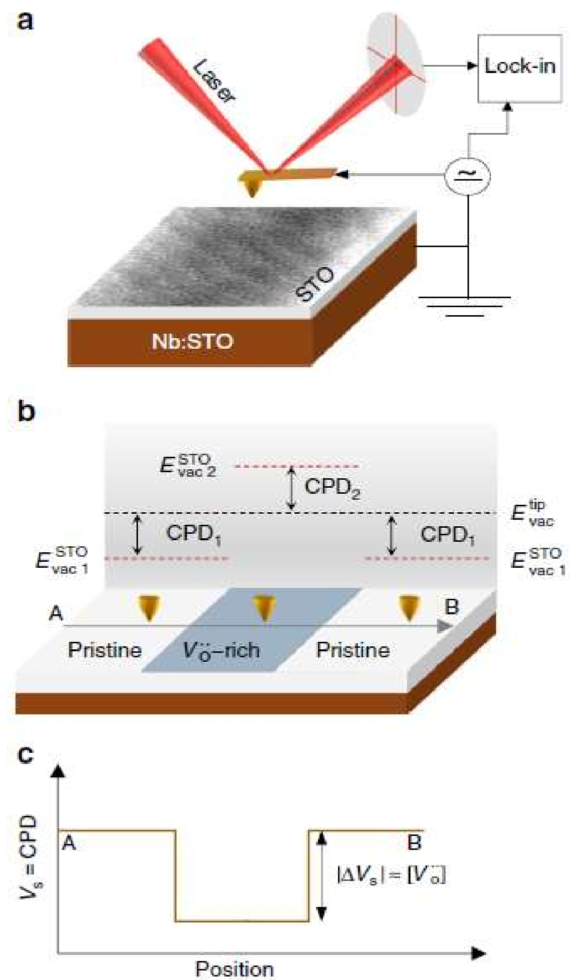




Kelvin probe force microscope (KPFM)

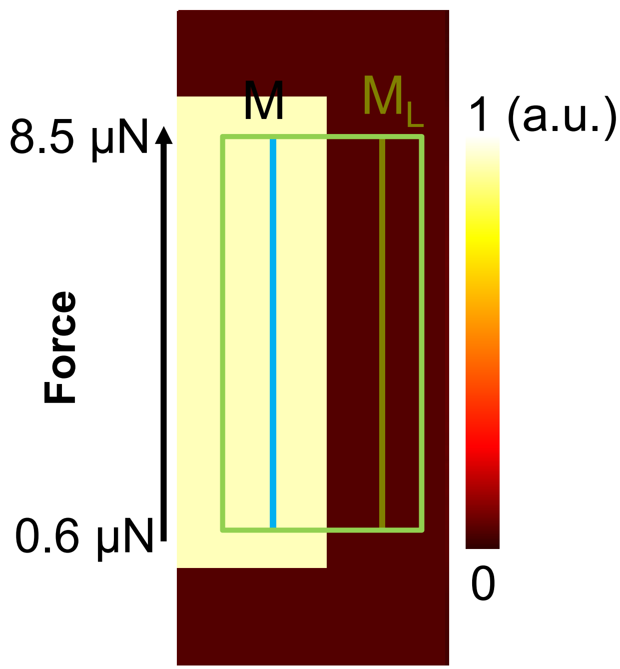
Measure contact potential difference (CPD)

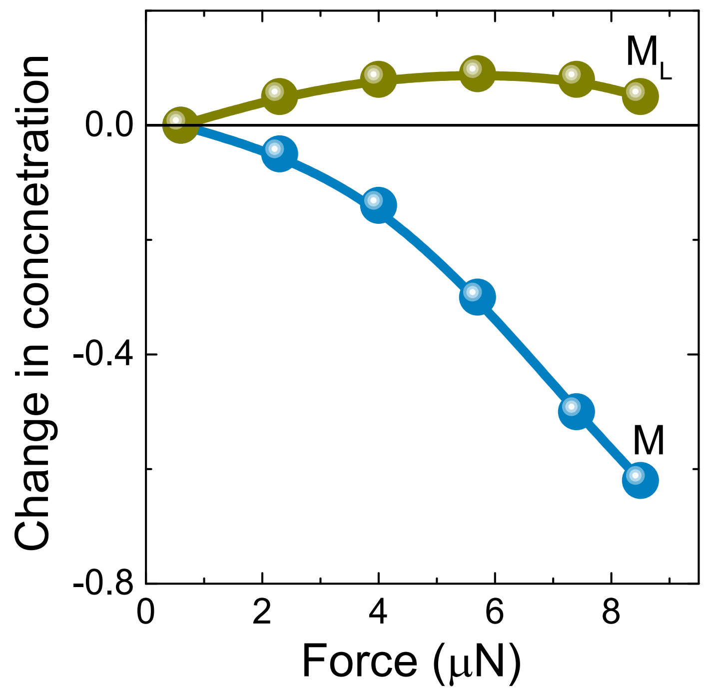
→ Change in V_O concentration



Manipulation of oxygen vacancies with FlexE

Normalized vacancy conce ntration map by **KPFM**





AFM-tip moves V_O along $SrTiO_3$ surface!

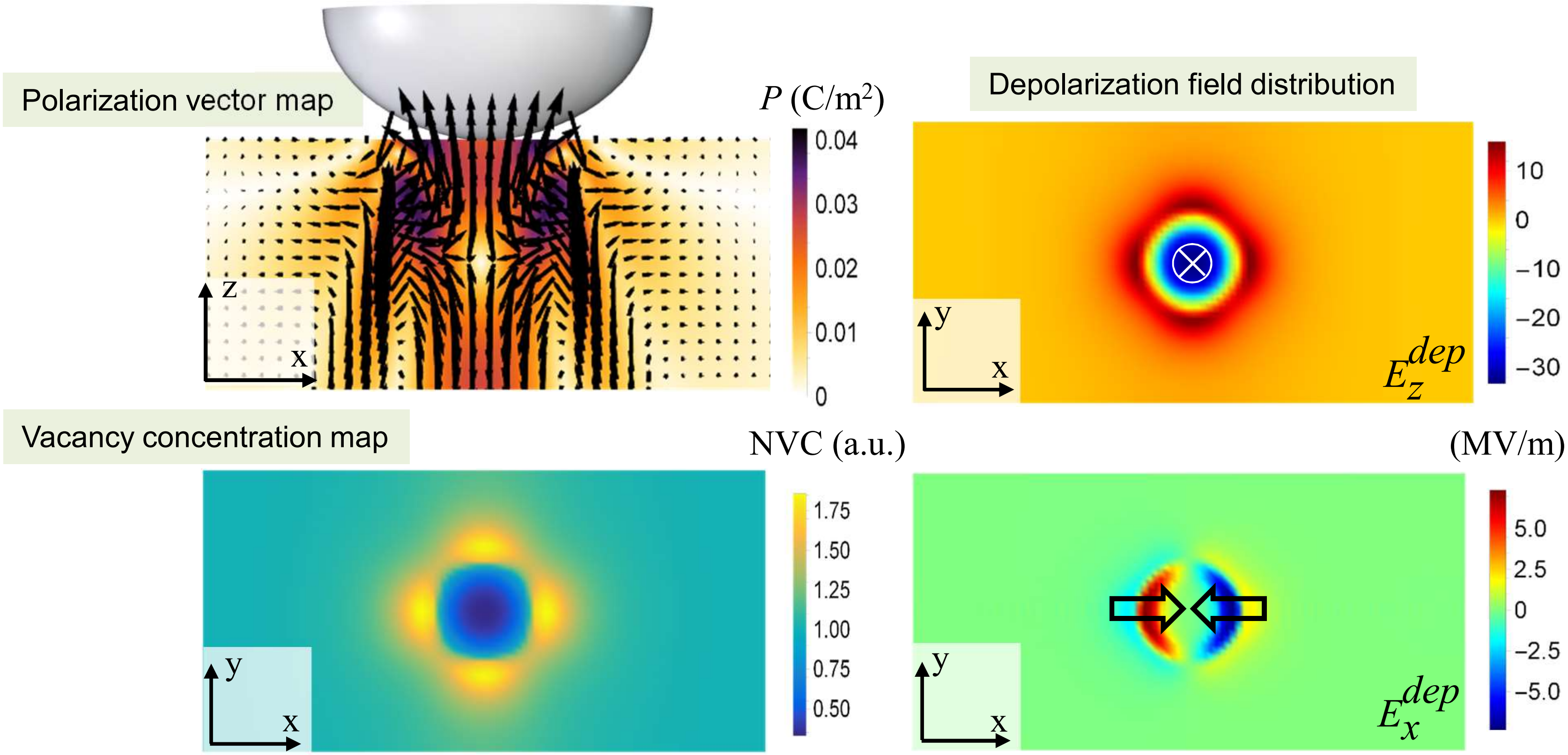
- Concentration decreased along M
- Concentration enhanced along M_L

S. Das *et al*., Nature Communications (2017)

Underlying mechanism

Collaboration with Prof. L. Q. Chen (Penn State U)

Phase field simulation

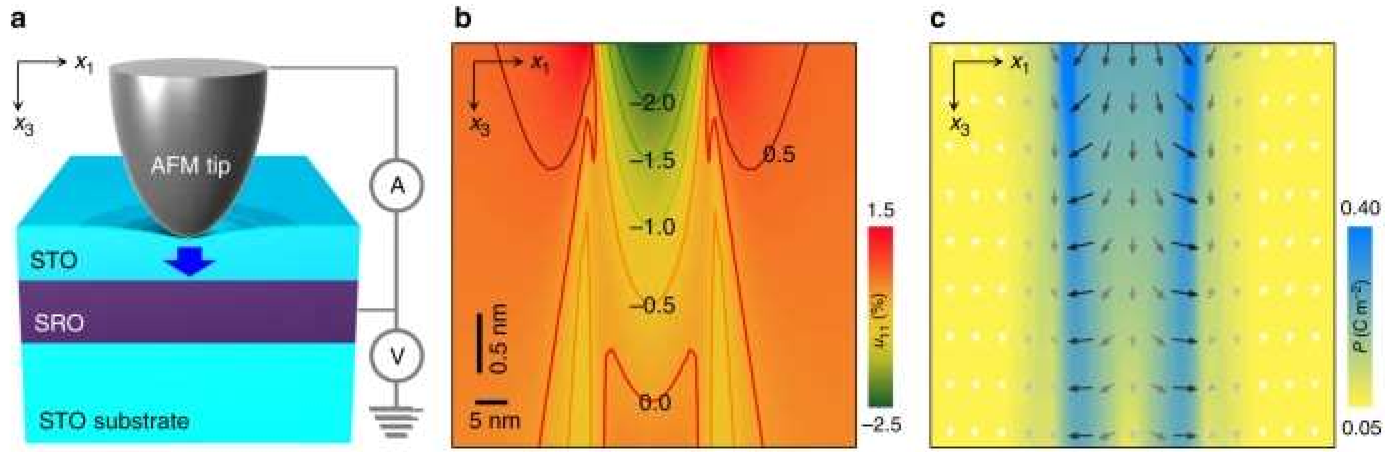


- The pressed tip can trap oxygen vacancies
- and move them with its motion. → "**flexoelectric V_O tweezer**"

S. Das *et al*., Nature Communications (2017)

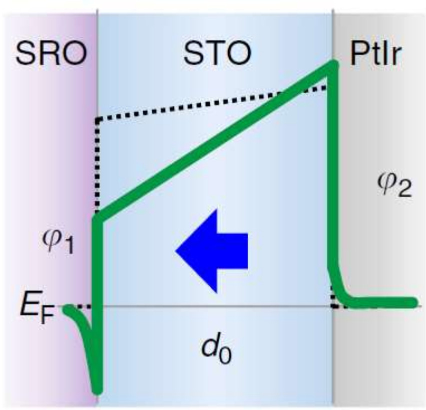
Mechanically tunable quantum tunneling

Flexoelectrically polarized ultrathin barrier using 3.5 nm-thick STO with 5μN



transverse strain

polarization profile
with phase-field simulation



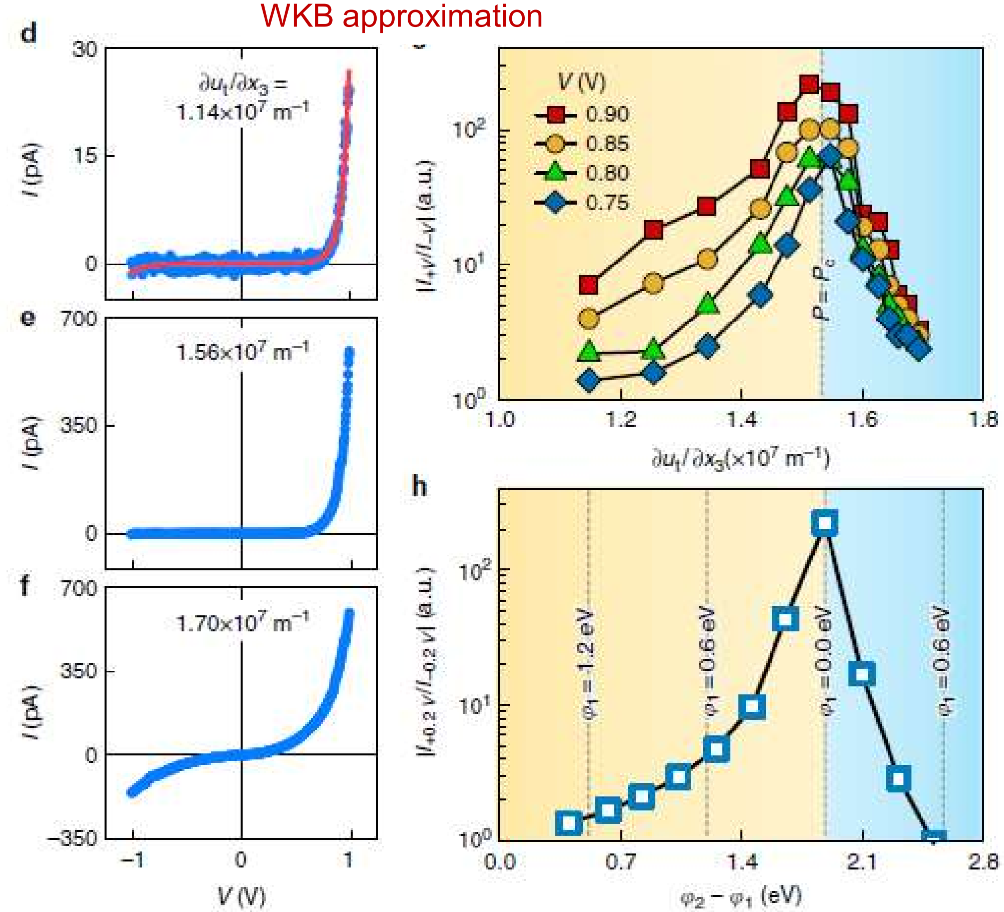
Neglecting flexoelectricity, pressue does not produce any polarization in STO.

S. Das *et al*., Nature Communications (2019)

Mechanically tunable quantum tunneling

- Flexoelectric control of electric tunneling

WKB approximation

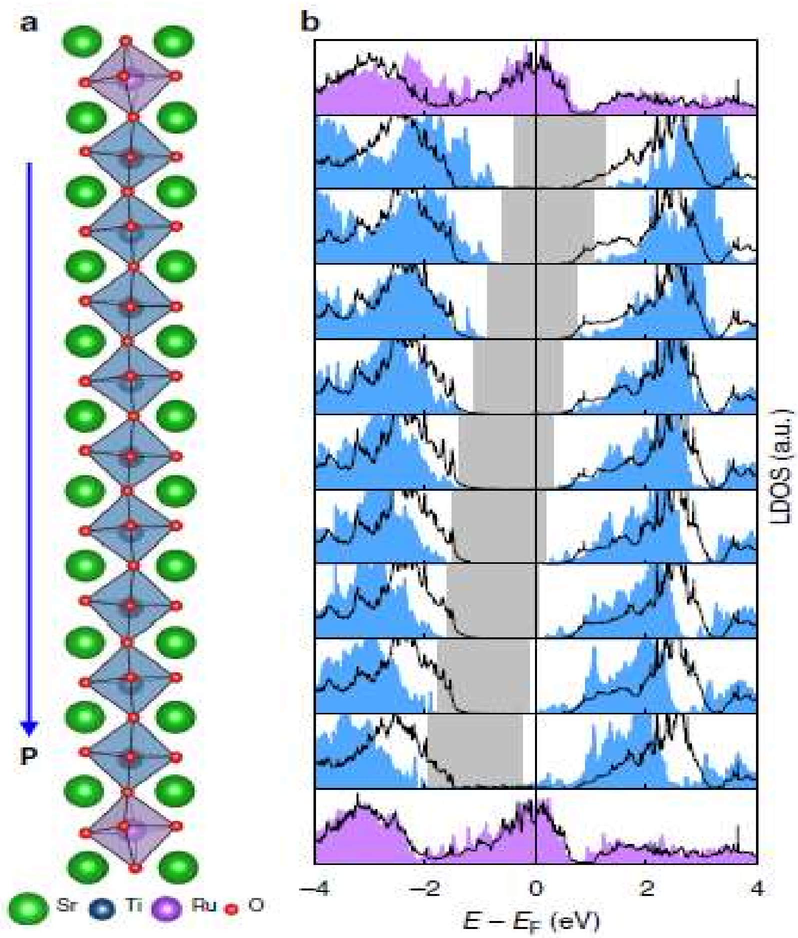


At critical polarization P_c, the tunnel barrier becomes triangular with ϕ_1 = 0.

S. Das *et al*., Nature Communications (2019)

Mechanically tunable quantum tunneling

- polarization-induced local metallization in SrTiO



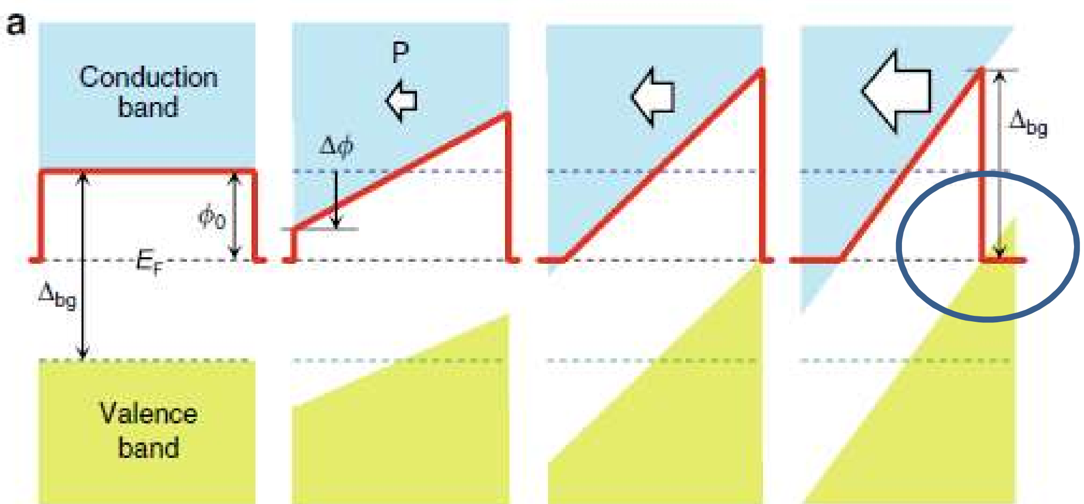
DFT with uniform displacement of
Ti atom by 0.2 A
(black line : non-polar SRO)

The gap shows a shift of energy bands due to polarization-induced electric field.

The cross metallizes the interfacial barrier layer region & concomitantly decreases the effective barrier width.

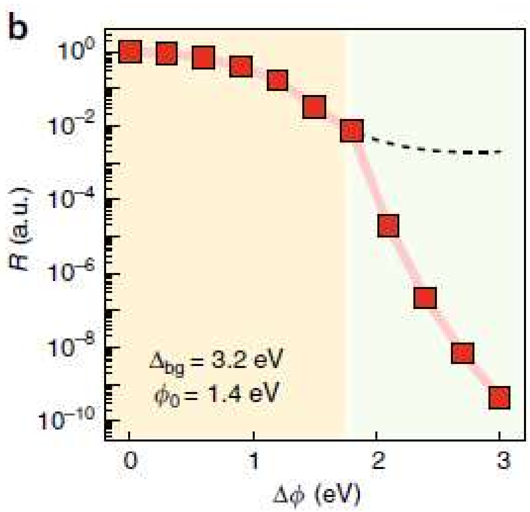
S. Das *et al*., Nature Communications (2019)

Colossal flexoresistance in dielectrics



highly conducting !

(~ 10^8 fold change)



Wentzel-Kramers-Brillouin (WKB) approximation

—— with valence band conduction

------ without v.b.c

S. M. Park *et al*., Nature Communications (2020)

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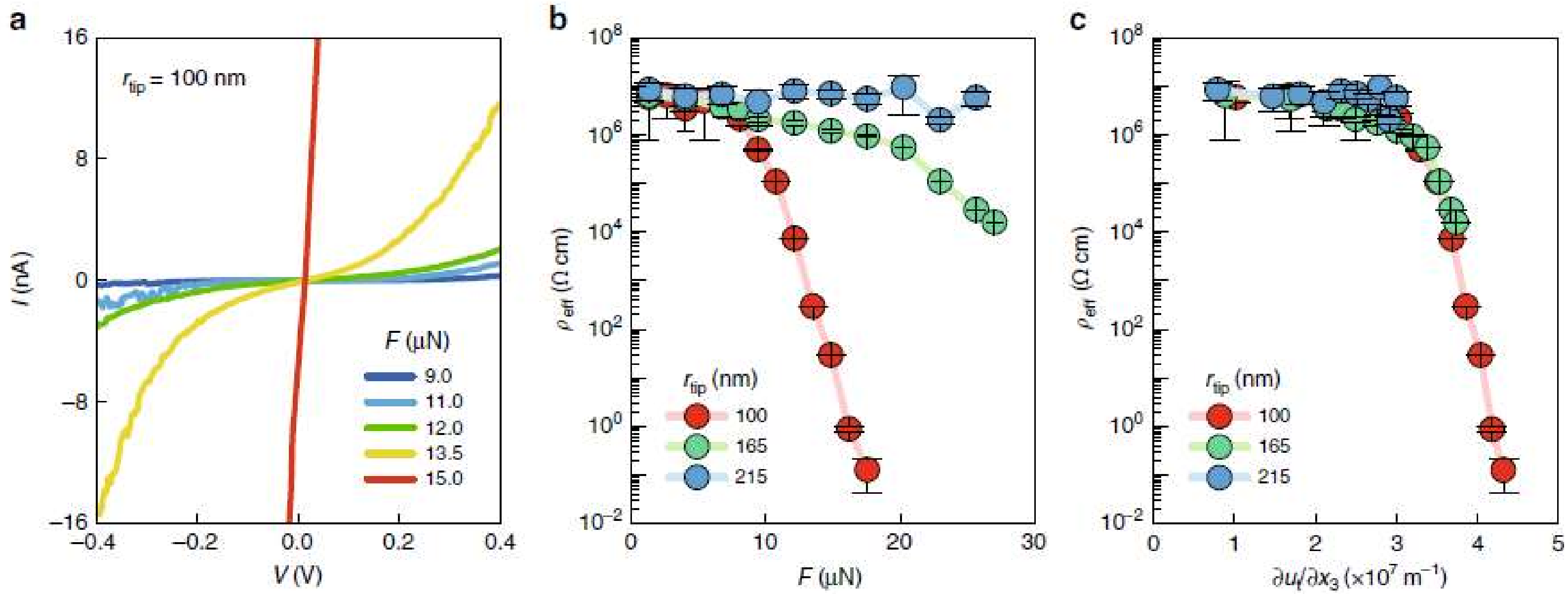
Colossal flexoresistance effect in ultrathin $SrTiO_3$



I – *V* nonlinear at low F
→ insulating states

I – *V* linear at high F
→ conducting state

Electrical-state switching in a large-bandgap dielectric leads to an extremely large change in the electrical resistivity (~ 10^8)

Scaling behavior with a strain gradient, demonstrating flexo-effects

S. M. Park *et al*., Nature Communications (2020)

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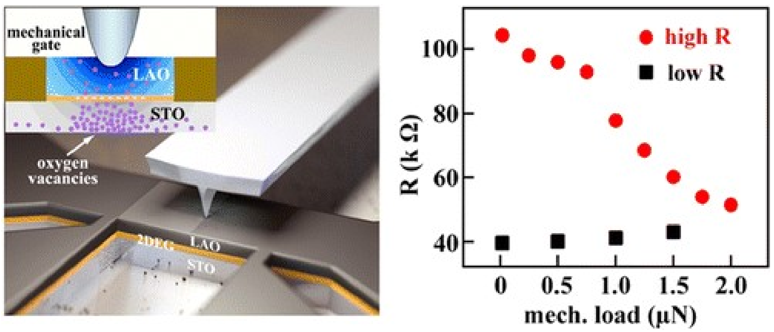
More examples on tip-induced flexoelectricity

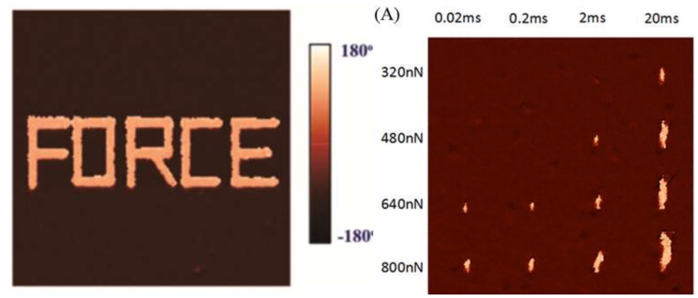
Mechanical gating

H. Lu *et al.*, Nano Letters. (2014)



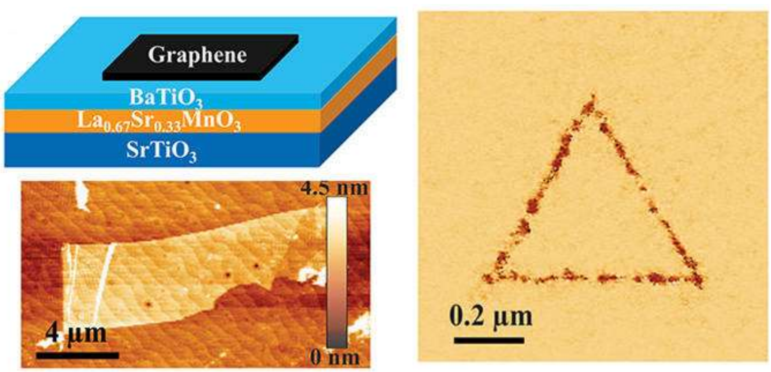
Writing domains in P(VDF-TrFE) **polymer**

Chen et al., APL 2015



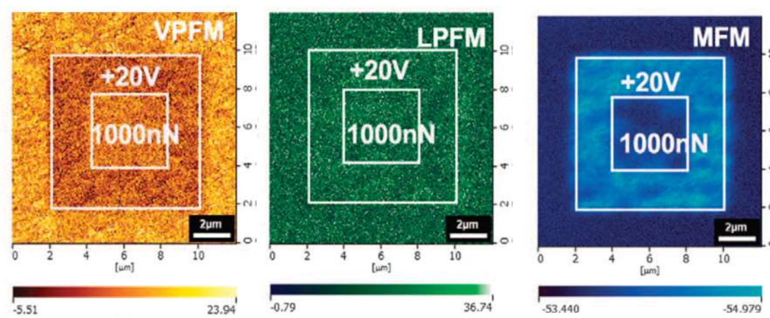
Writing domains under Graphene top electrode

Lu et al., Nano Letter 2016



Mechanical control of magnetism

Jia et al., NPG Asia Materials 2017



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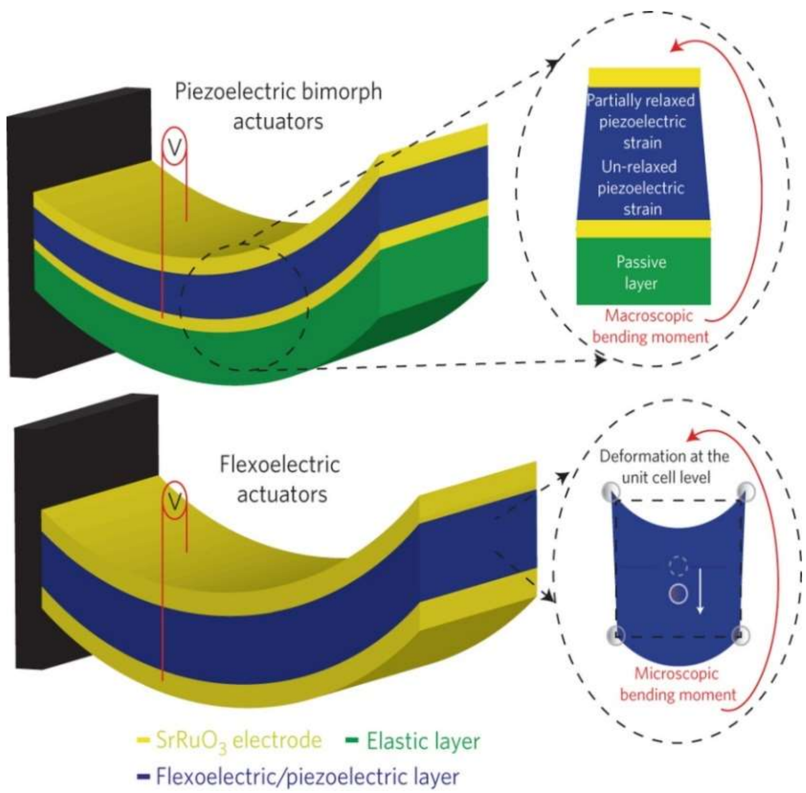
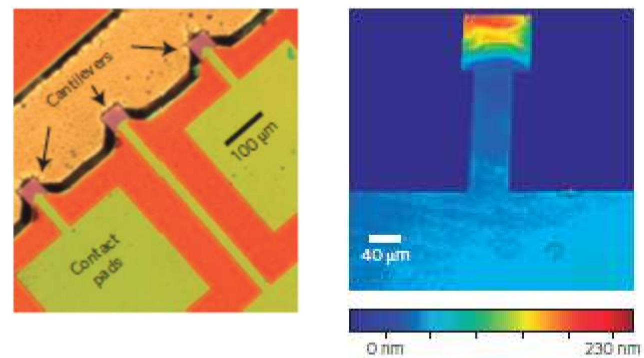
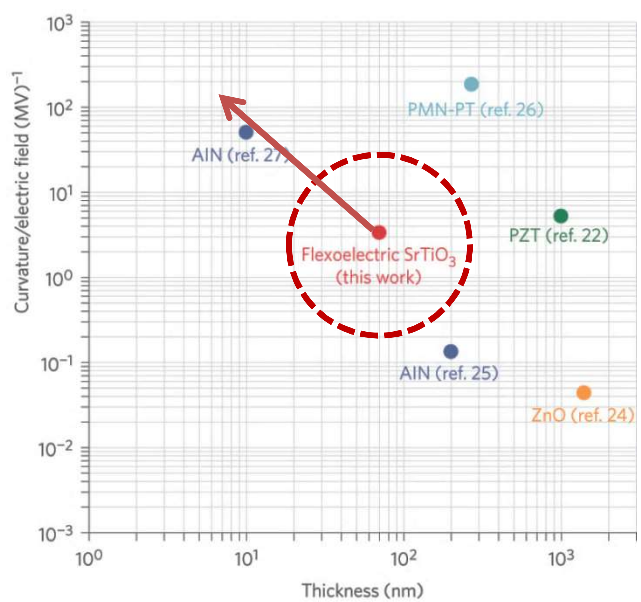
3. Potential applications

4. Summary

Possible applications: flexoelectric MEMS on Si



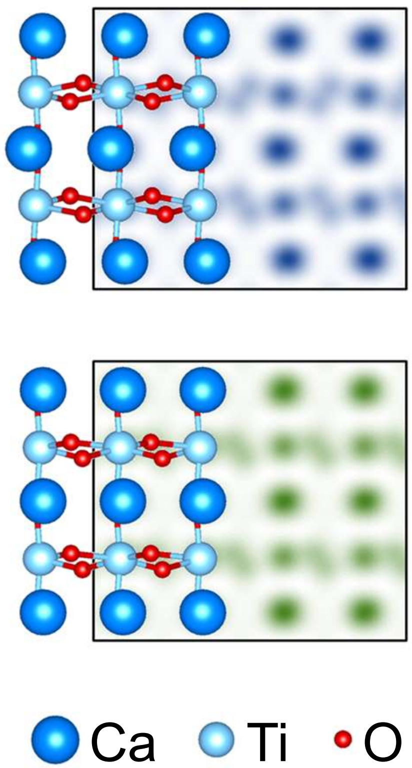




U. Bhaskar *et al*. Nature Nanotech. (2016)

Metastable ferroelectric thin films

room-temperature ferroelectricity in $CaTiO_3$ (111) films



Bulk $CaTiO_3$:

orthorhombic (P_{nma} with $a^-b^+a^-$)

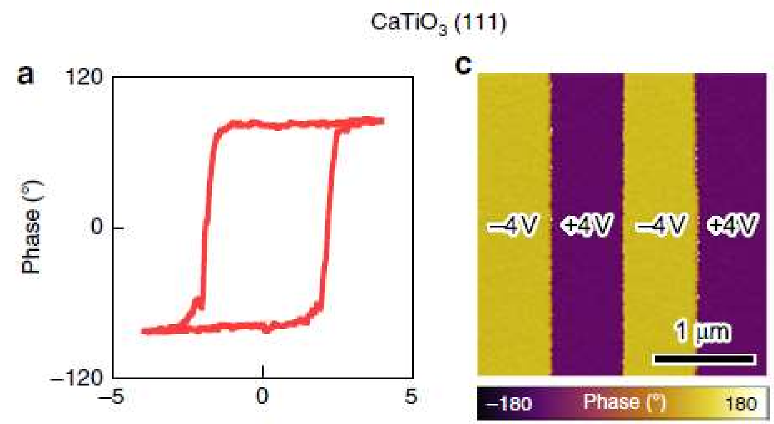
paraelectric

Epi-stabilized $CaTiO_3$ (111) films on $LaAlO_3$

rhombohedral ($R3c$ with $a^-a^-a^-$)

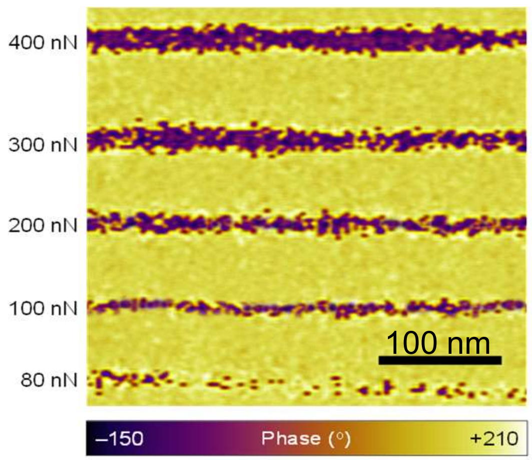
metastable ferroelectric at RT

J. R. Kim *et al.*, Nature Communications (2020)



Metastable ferroelectric thin films

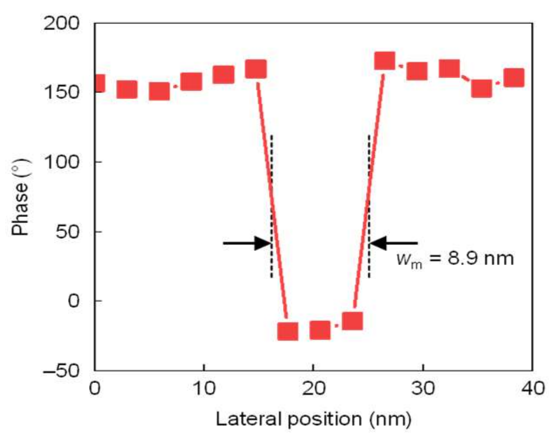
Epi-stabilized ferroelectric $CaTiO_3$ (111) films on $LaAlO_3$

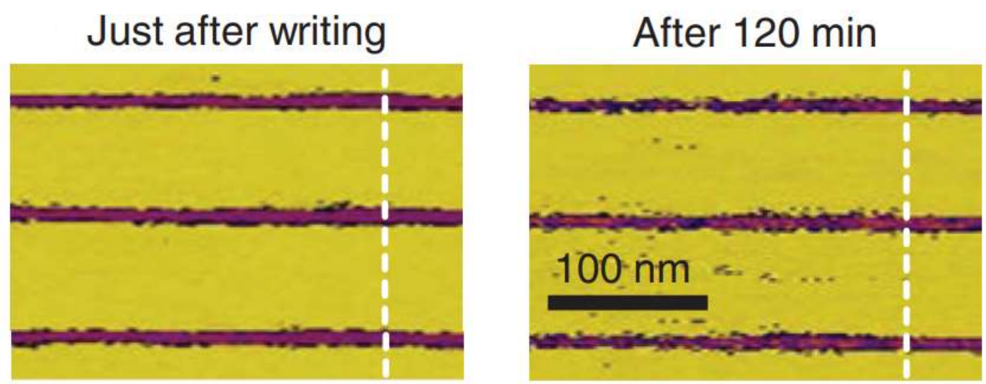


- Mechanical switching → nanodomains of <10 nm width

- Quite stable with time

- Ultrahigh data storage density ($\geq$1 Tbit cm^{-2})



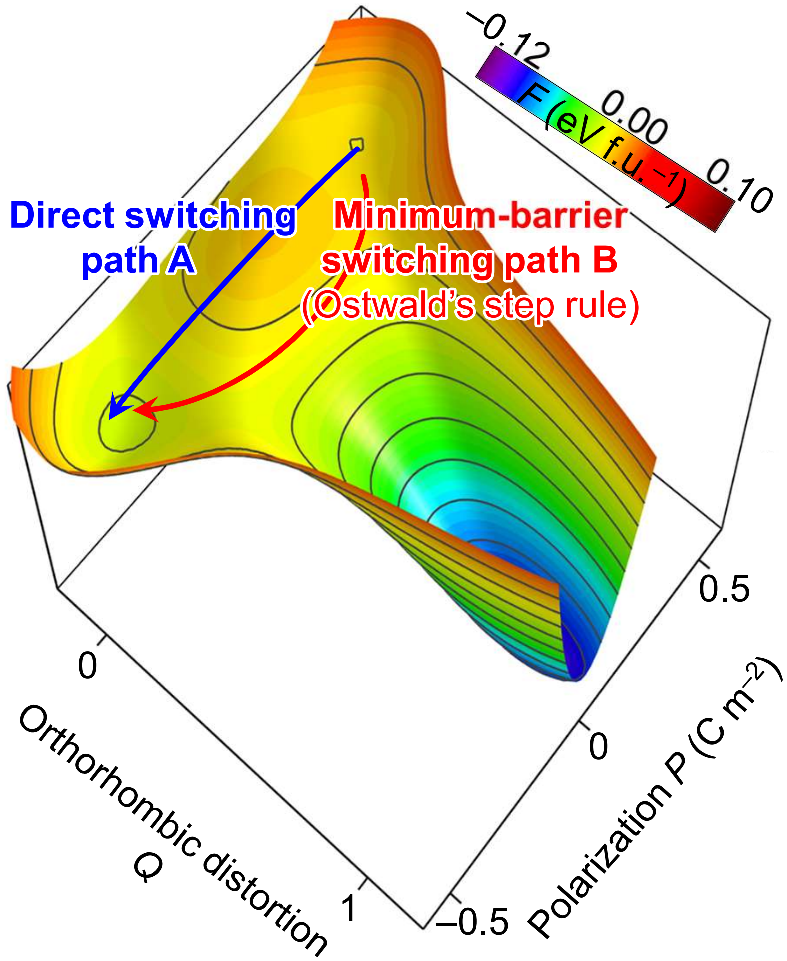


J. H. Lee *et al*., Phys. Rev. Lett. (2022)

J. H. Lee et al. PRL (2022)

Metastable ferroelectric thin films

Epi-stabilized ferroelectric $CaTiO_3$ (111) films on $LaAlO_3$



J. H. Lee *et al.*, Phys. Rev. Lett. (2022)

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Summary

Flexoelectricity comes from the electromechanical coupling between charge and lattice (strain gradient). It is ubiquitous: i.e. it can occur in any dielectric material. Its effect can increase with decrease of sample size.

Strain gradient inside an epitaxial film can be systematically controlled by varying P_{O2} ,T_D and so on. Such internally developed strain gradient can affect many physical properties, including ferroelectric domain formation & switching dynamics, polarization rotation, defect pinning and related transport, photocurrent, and so on.

We can generate the strain gradient by applying pressure with an AFM tip. With such external forces, we can control mechanical ferroelectric switching and & its pathways, concentration of oxygen vacancies, mechanical tunneling and gating, metal-insulator transition, and so on.

Studies on flexoelectricity will provide opportunities to resolve numerous unresolved issues/puzzles occurring in nanoscale and to tune functional properties of many nano-materials.

Acknowledgement

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Prof. Sang Mo Yang (Currently @ Sogang Univ.)

Prof. Seung Chul Chae

Prof. T. H. Kim (Univ of Ulsan)

Dr. Eunkyo Ko (Stanford Univ.)

Dr. Sung-min Park (Samsung)

Dr. B. C. Jeon (Samsung)

Prof. Seyong Park (Soonsil Uni.)

Prof. J. –G. Yoon (Univ. of Suwon)

Dr. Jeong-Rae Kim (Caltech)

Penn State Univ.

Prof. Long-Qing Chen

Dr. Bo Wang (Phase-field simulations of strain and polarization profile)

Univ. of Nebraska-Lincoln

Prof. Evgeny Tsymbal

Dr. Lingling Tao (Tight binding calculation)

Dr. Tula Paudel (Density functional theory calculations)

Thank you very much! (~ 33 years in SNU)

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