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Atomically-resolved imaging of surfaces of complex oxide thin films grown by laser-MBE

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Oak Ridge, TN, USA



 **OAK RIDGE**
National Laboratory

CENTER FOR
NANOPHASE
MATERIALS SCIENCES

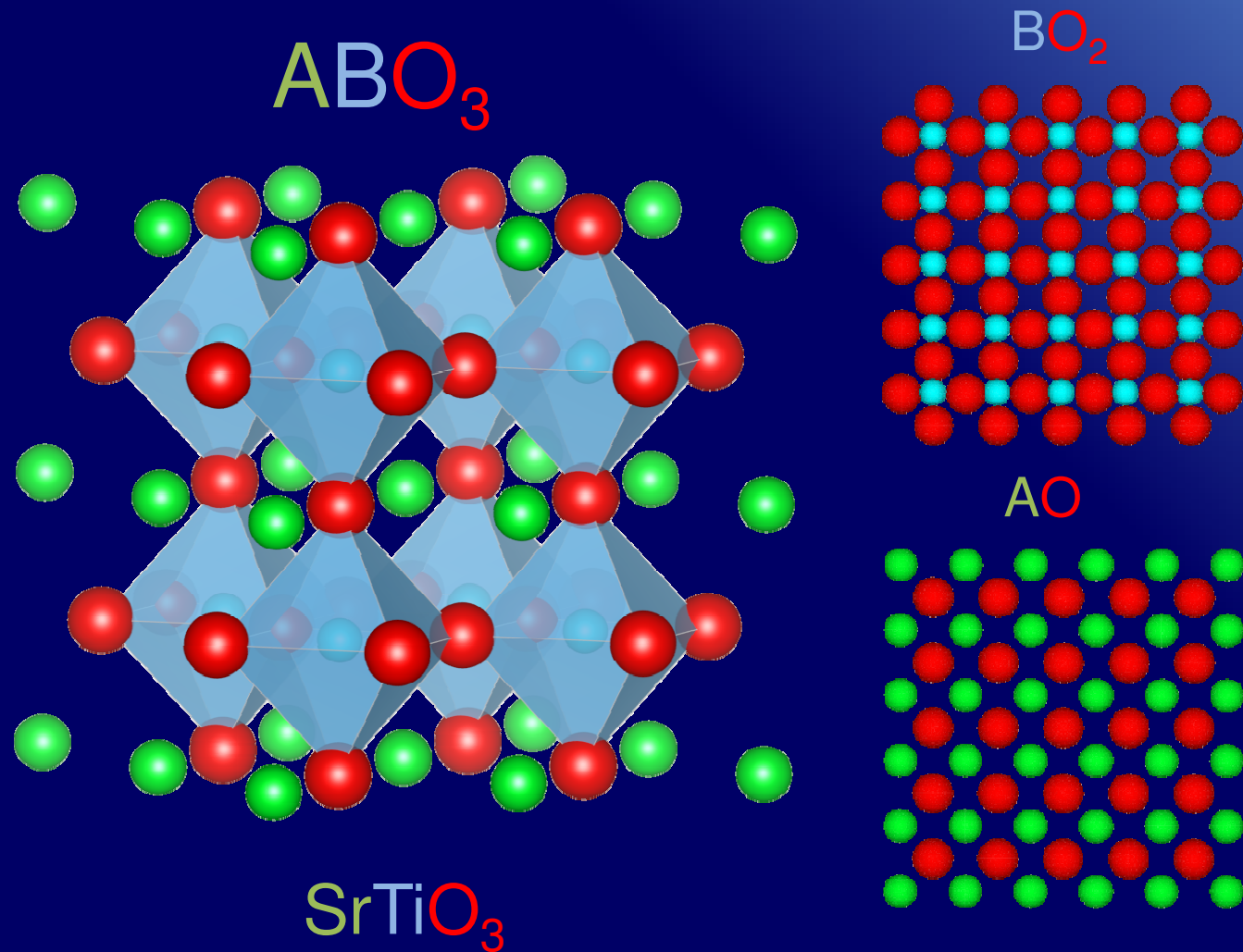
What complex oxides offer

- Superconductivity
- Colossal magnetoresistance
- Ferromagnetism (spintronics)
- Ferroelectricity
- Multiferroism
- Metal-insulator transitions
- Tunability by strain
- Memristive behavior

Can be integrated with existing Si-based technologies

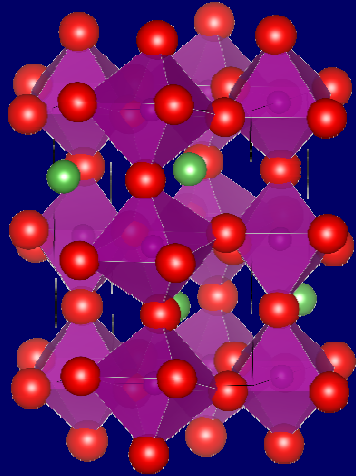
- Use in electrochemical energy storage and conversion systems

Perovskite structure



Epitaxial film growth

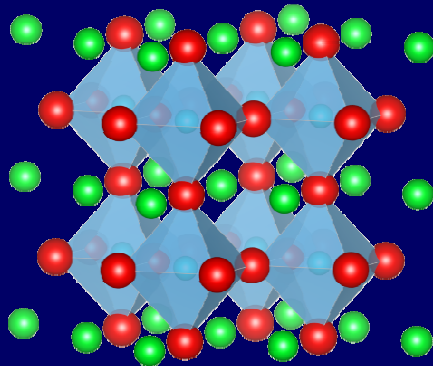
LaMnO₃



Growth at:
elevated substrate temperatures
650-850 °C &
in oxygen atmosphere 1-300 mTorr

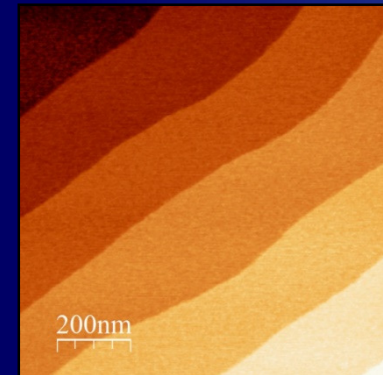
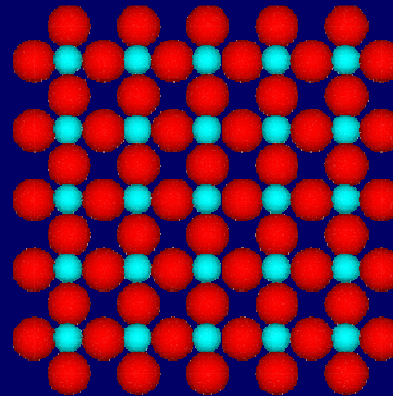
B-terminated single-crystalline substrate

SrTiO₃



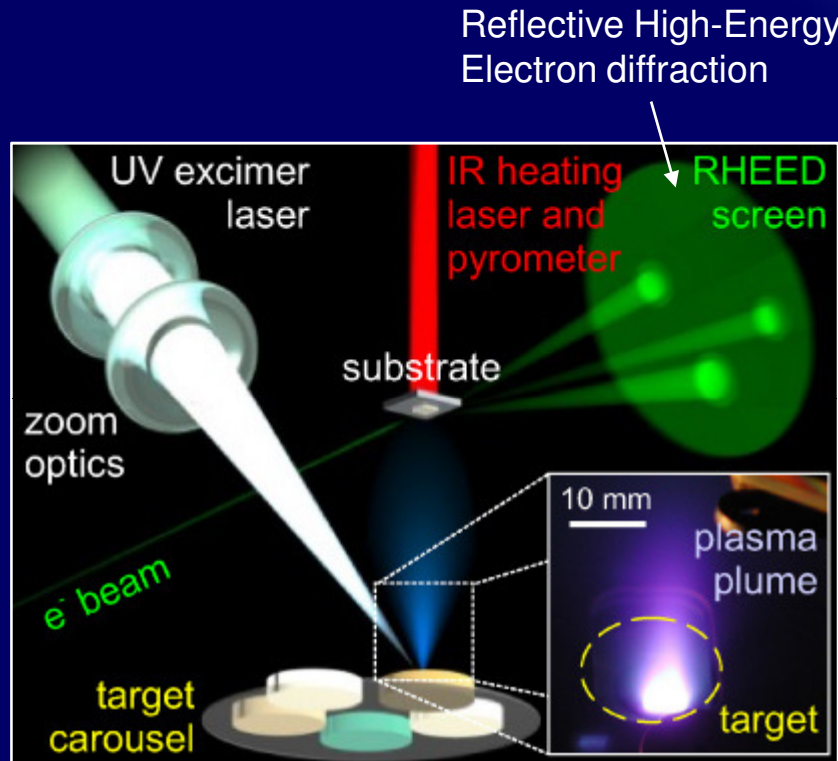
substrate

BO₂

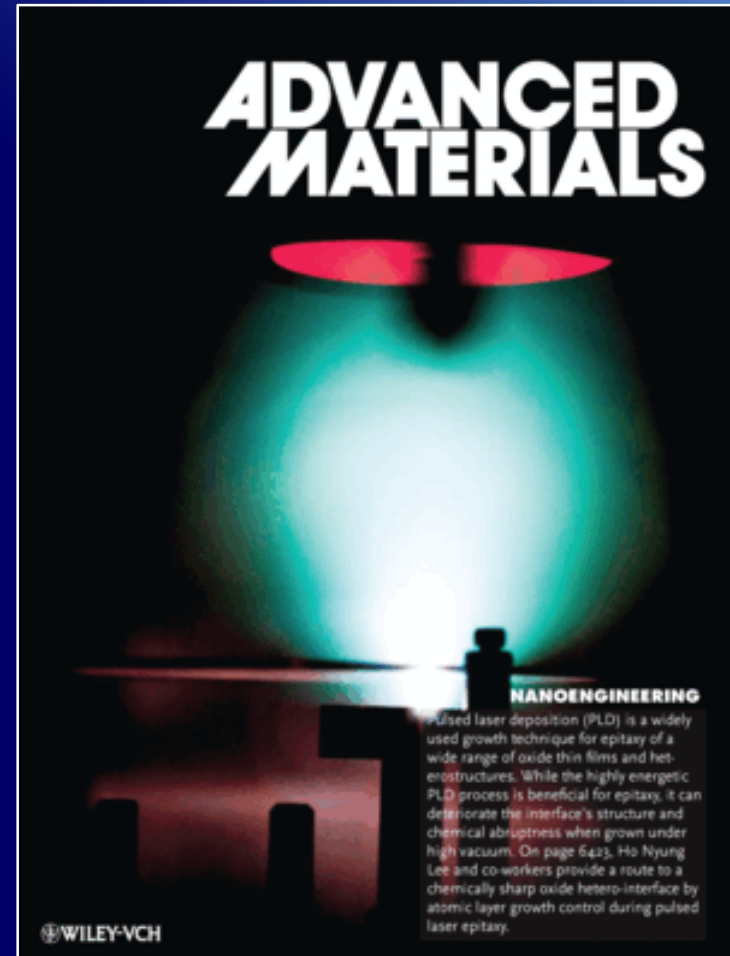


Atomic force
microscopy image

Pulsed Laser Deposition

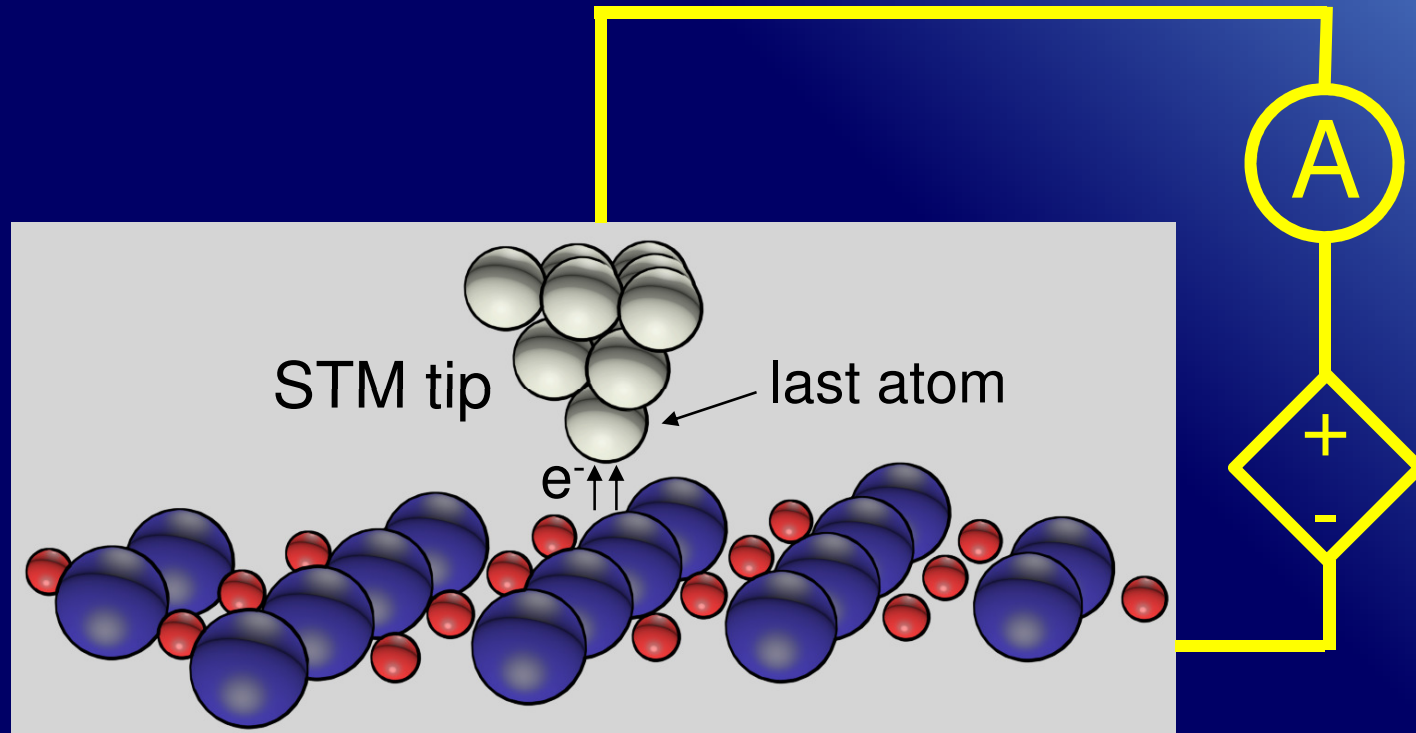


The schematic is from: M. Opel,
J. Phys. D 45, 033001 (2012)



Adv. Mat. cover Dec 2012, connected
to a paper from an ORNL group

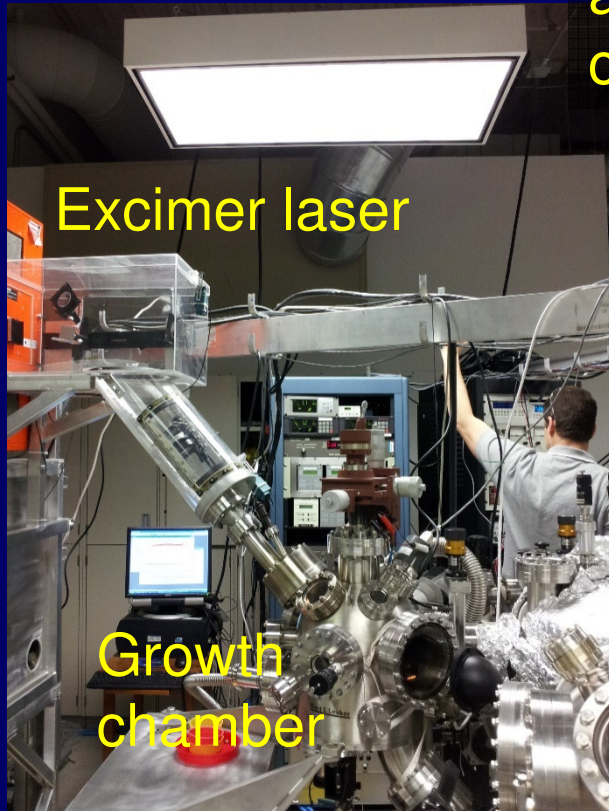
Scanning tunneling microscopy



- *Atomic resolution is generally very difficult to achieve on PLD-grown films*

NanoTransport system at CNMS

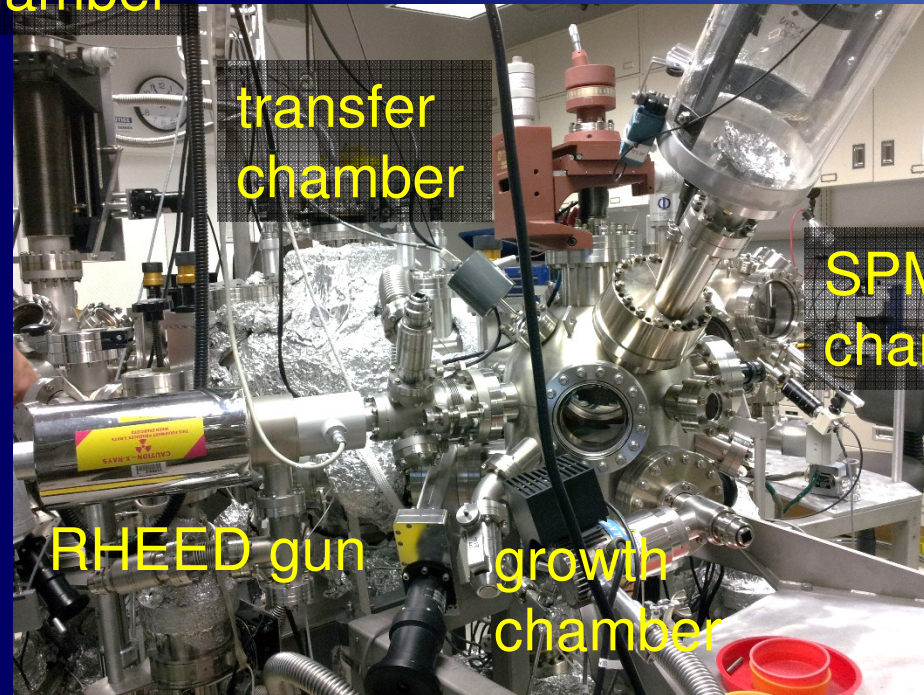
<http://www.cnms.ornl.gov>



Excimer laser

Growth chamber

analysis chamber



transfer chamber

RHEED gun

growth chamber

SPM chamber

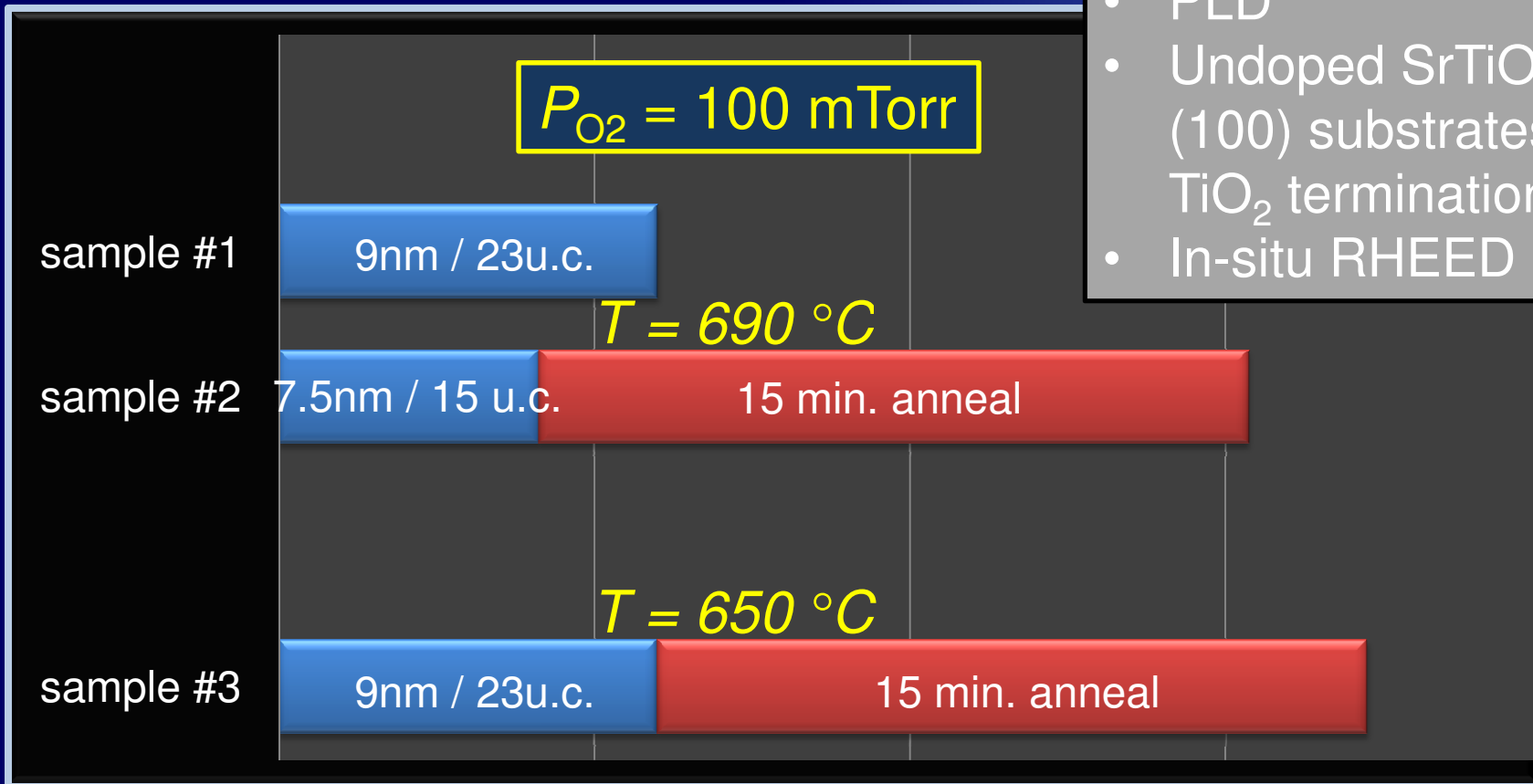
STM
AFM
in
UHV



Widely used as a metal electrode

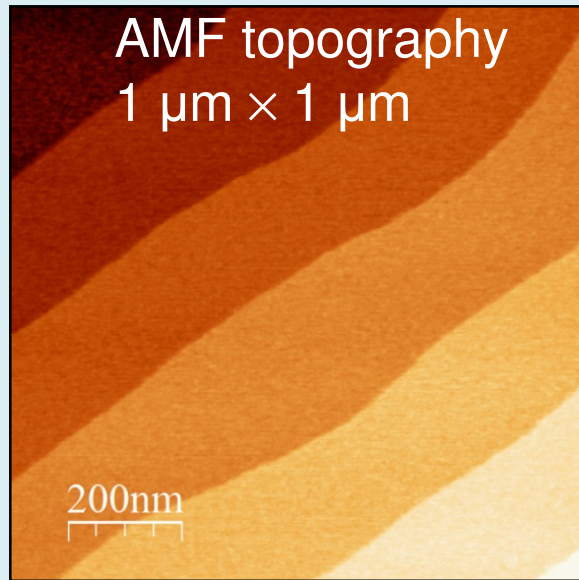
Samples

- PLD
- Undoped SrTiO₃ (100) substrates / TiO₂ termination
- In-situ RHEED



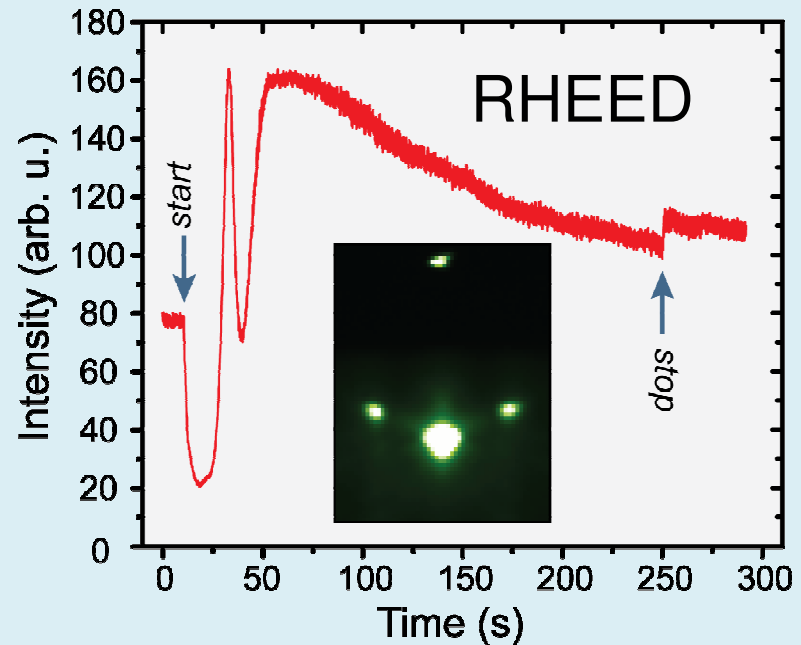
Transfer to the STM chamber right after film growth

Film growth



RMS Roughness = 55 pm

Substrate selection:
RMS roughness < 70 pm

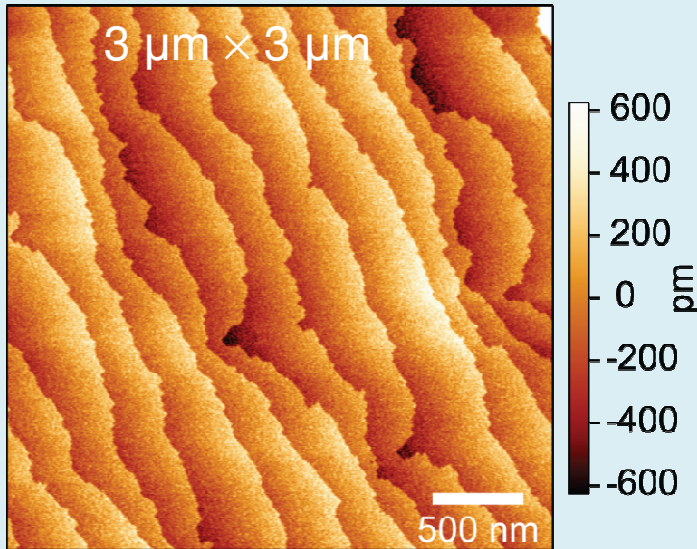


Growth modes:

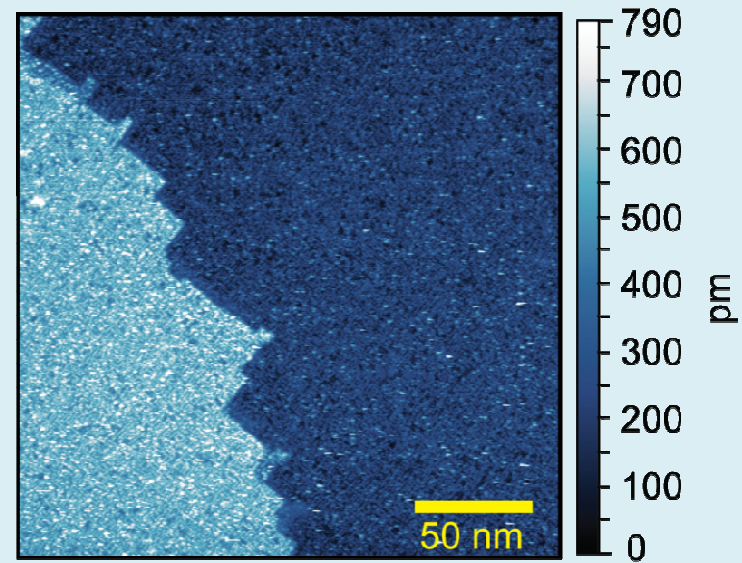
- 690 $^{\circ}\text{C}$ – step-flow
- 650 $^{\circ}\text{C}$ – step-flow/island-formation

Film surface: larger scale

ex-situ AFM (topography)

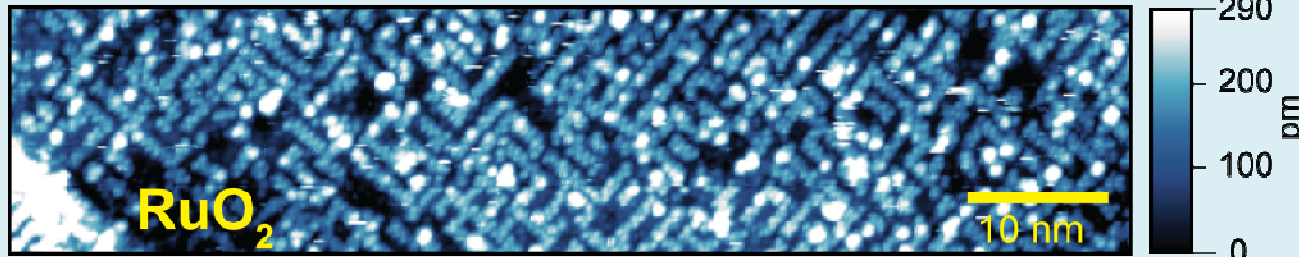


in-situ STM



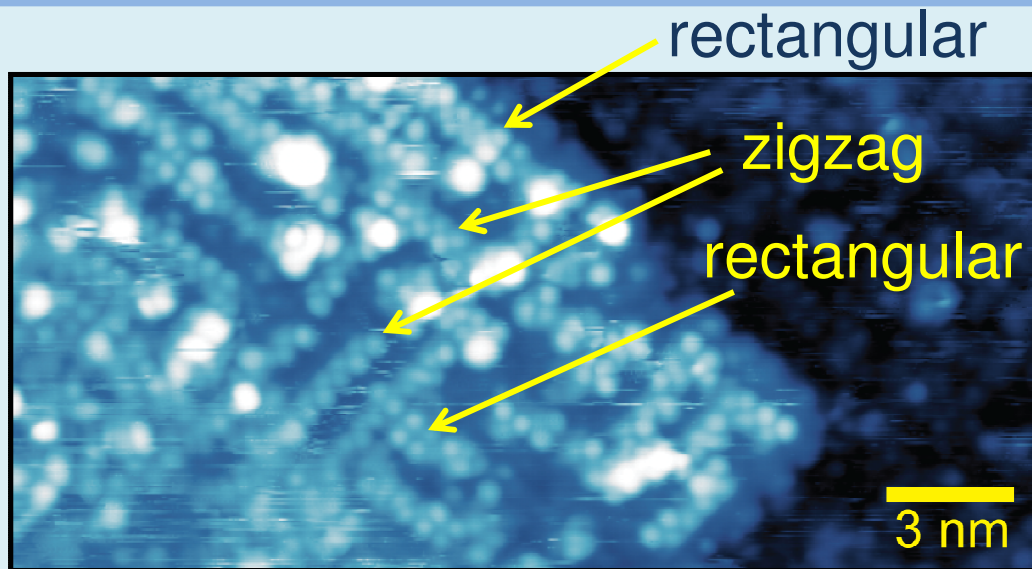
690 °C, no anneal

Competing surface patterns



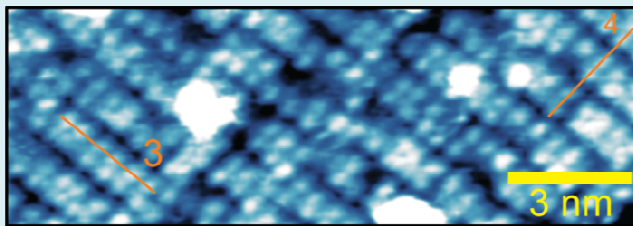
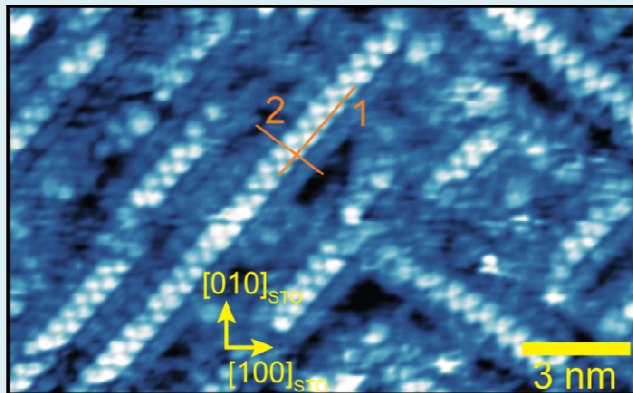
SrO

690 °C,
no ann.

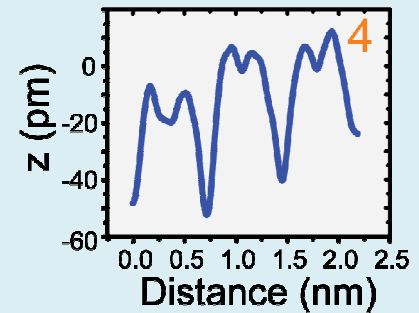
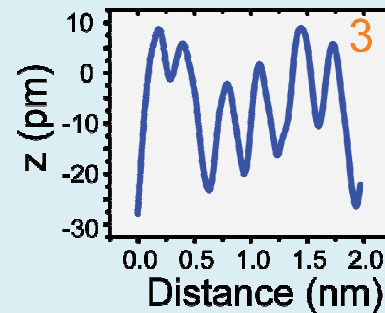
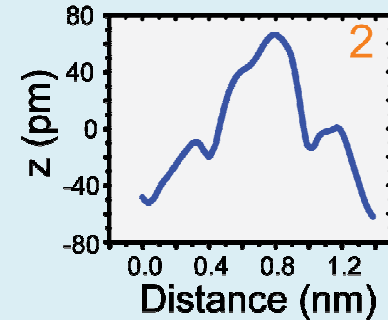
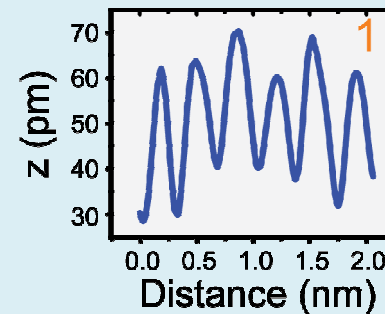


Zigzag and rectangular patterns

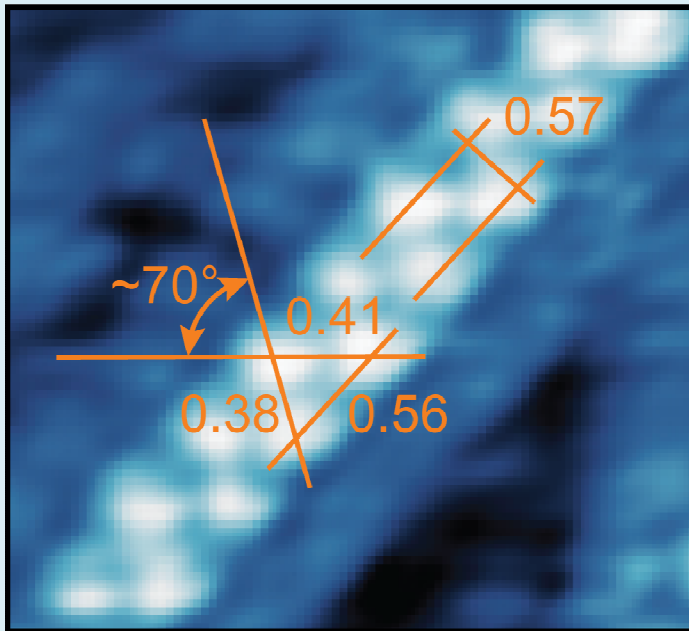
650 °C, ann.



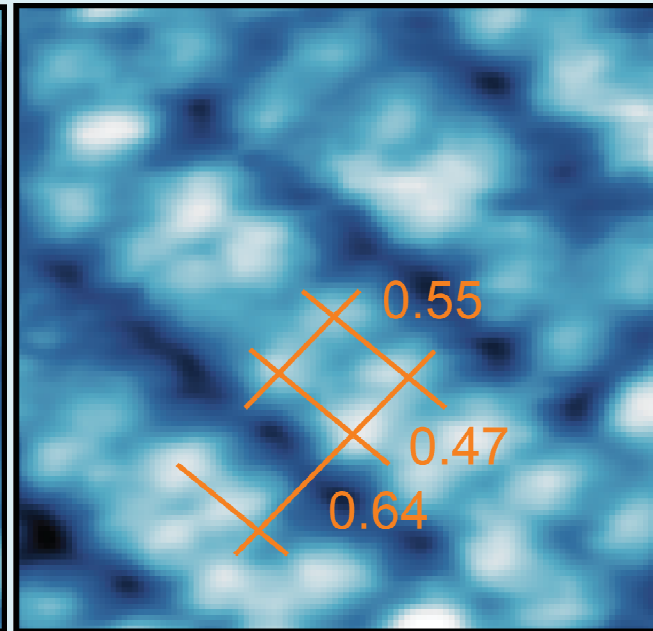
690 °C, ann.



Zigzag and rectangular patterns

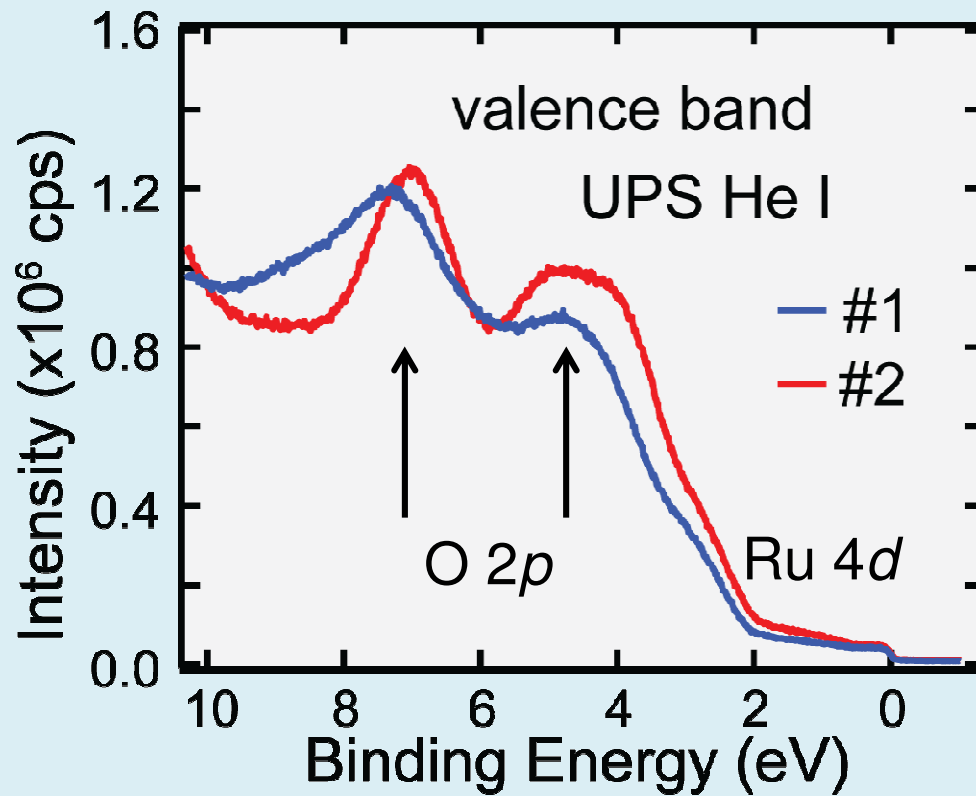


zigzag



rectangular

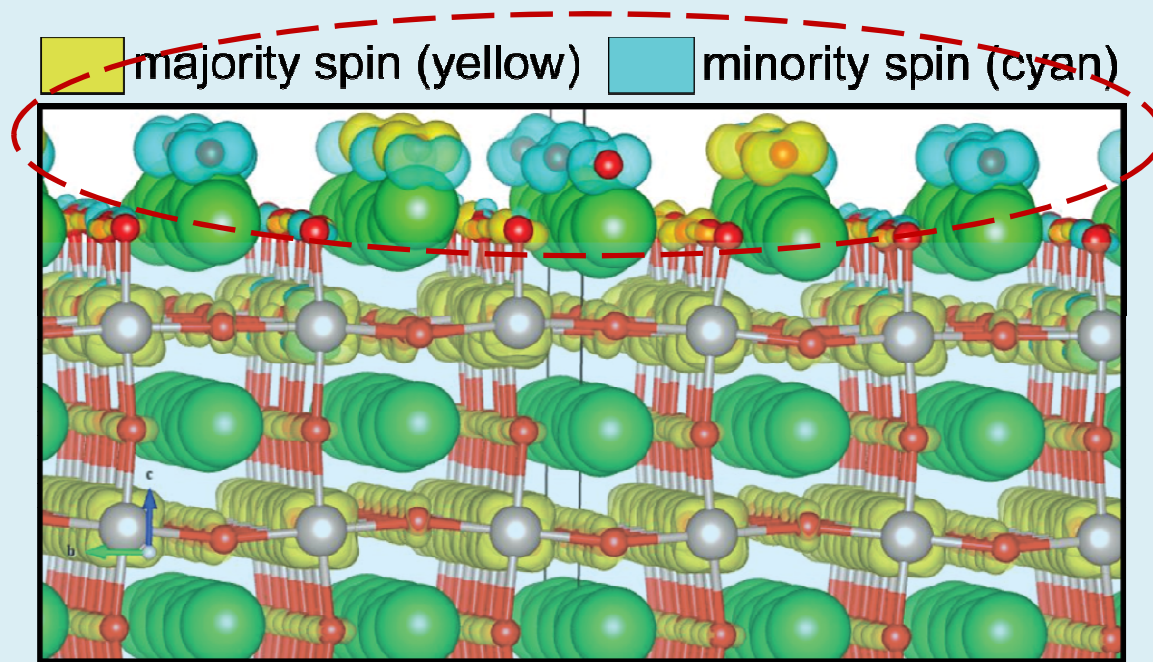
In-situ UPS



#2:
rectangular

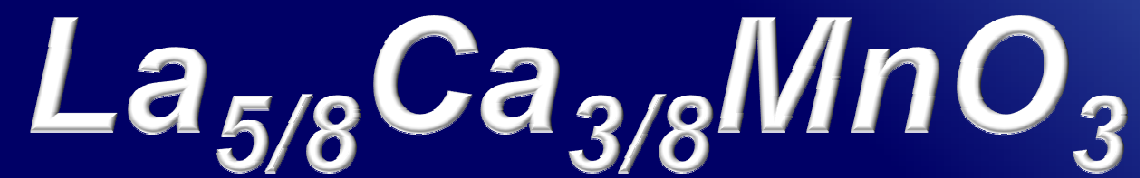
#1:
zigzag

Surface spin-glass behavior



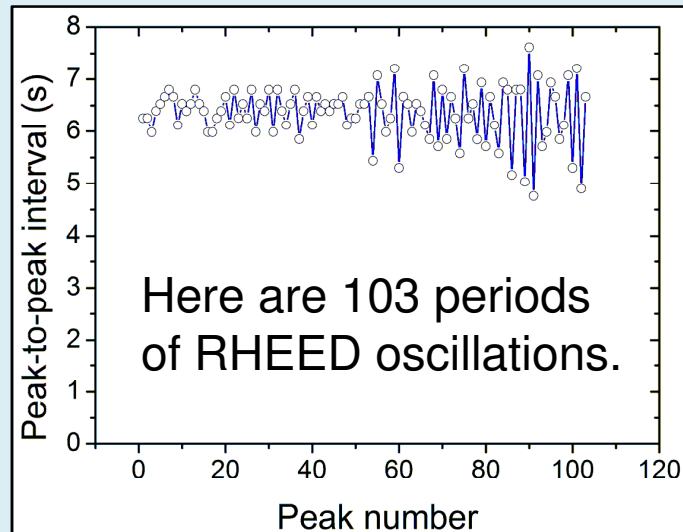
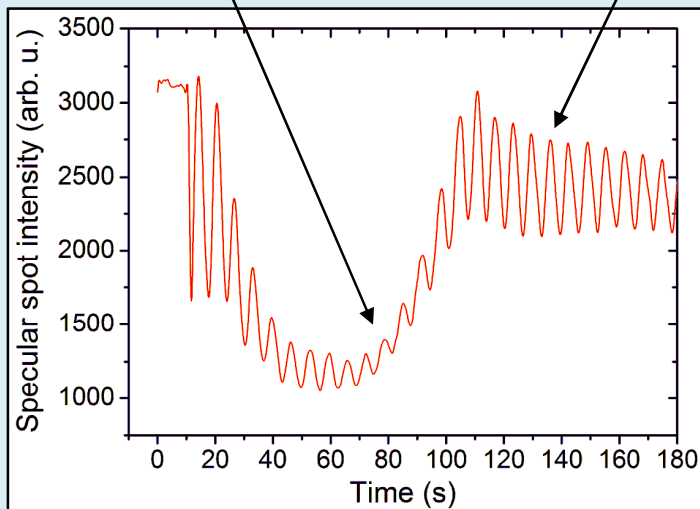
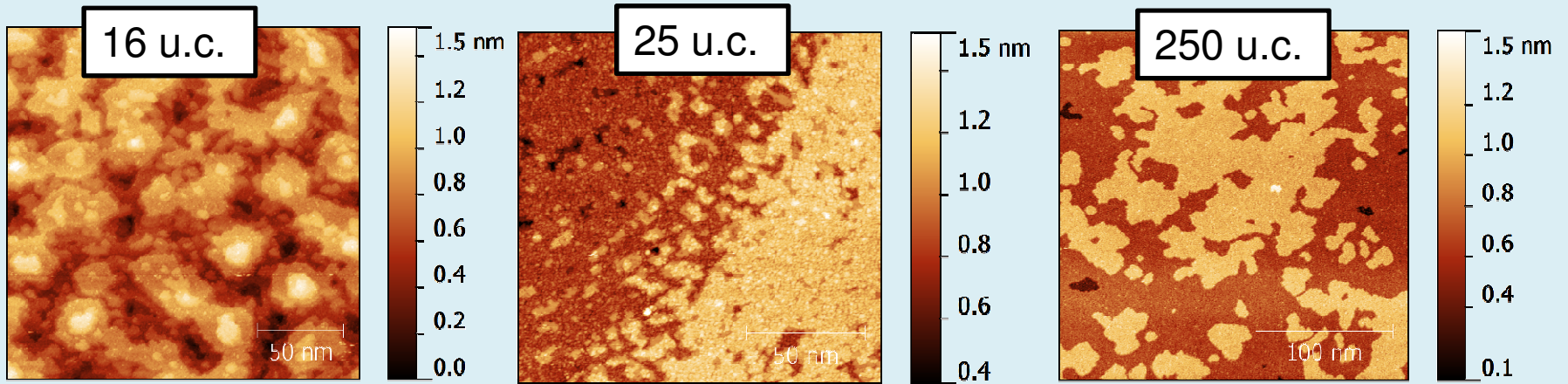
Isosurfaces of magnetization densities

- Randomly oriented local spins from O_{ad} p -orbitals

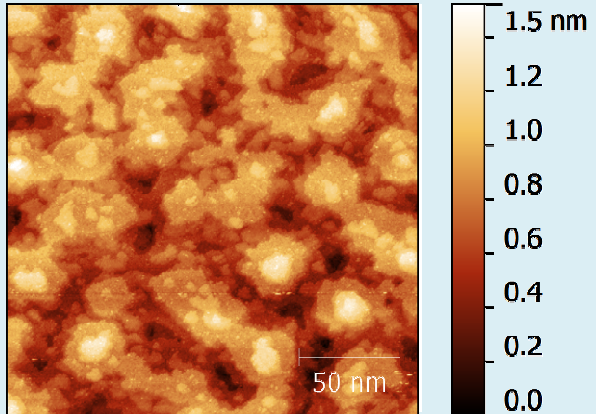


Classical *Colossal magnetoresistance* material

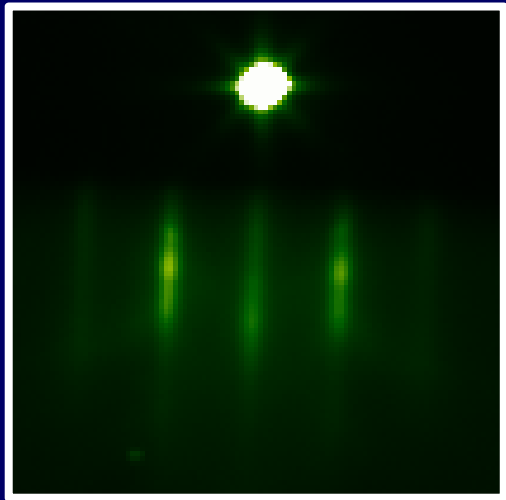
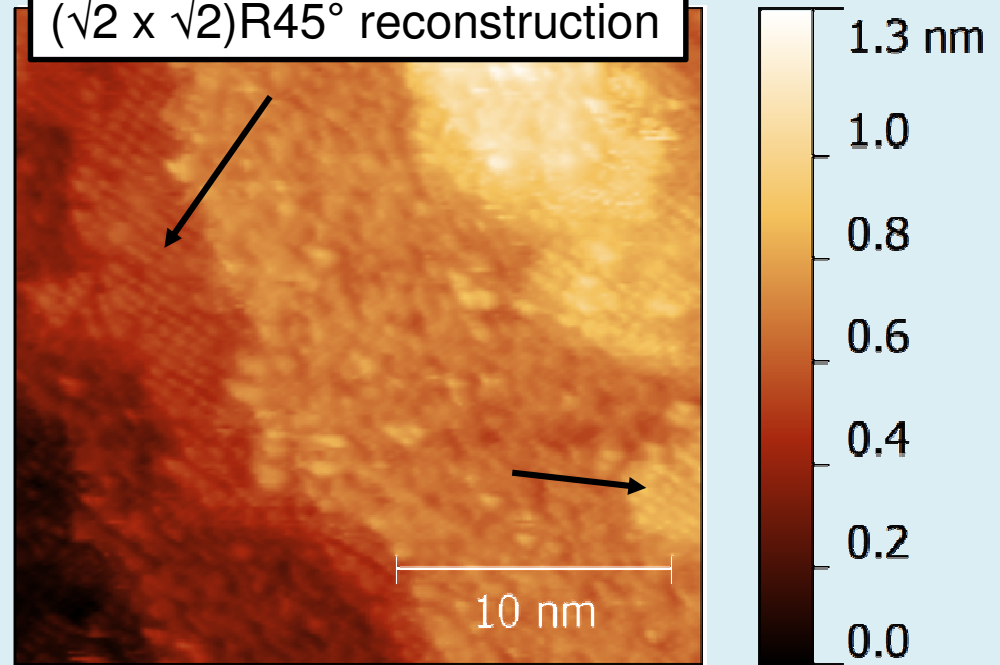
Evolution of surface morphology



16 u.c. on SrTiO₃



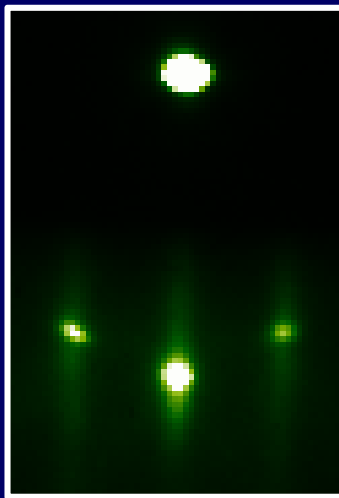
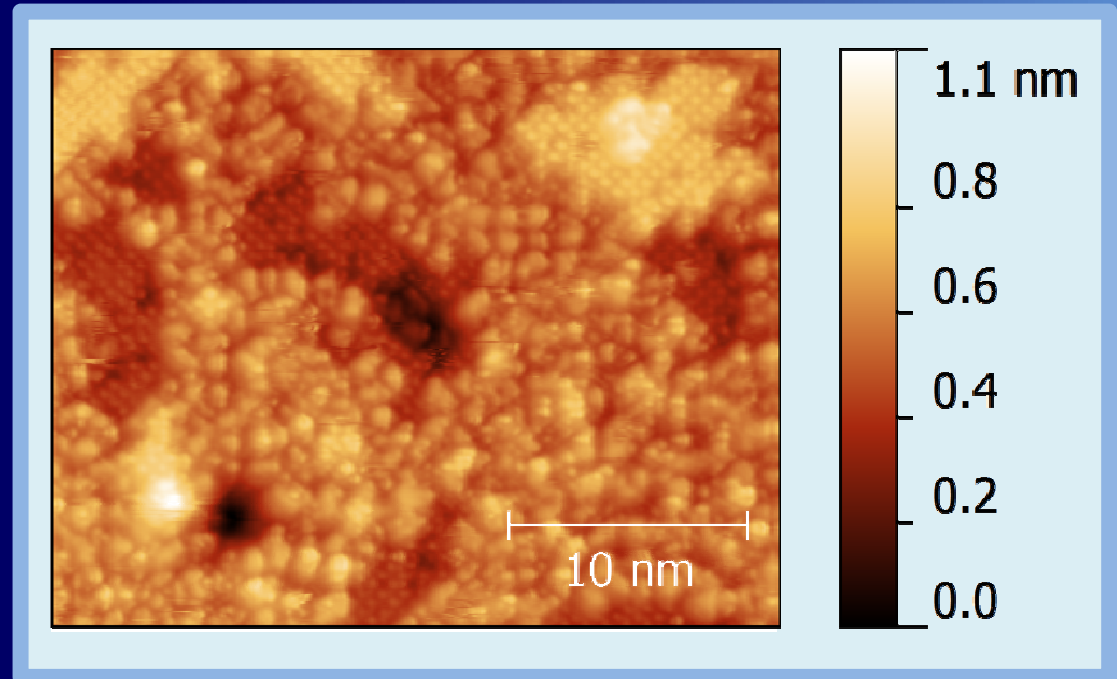
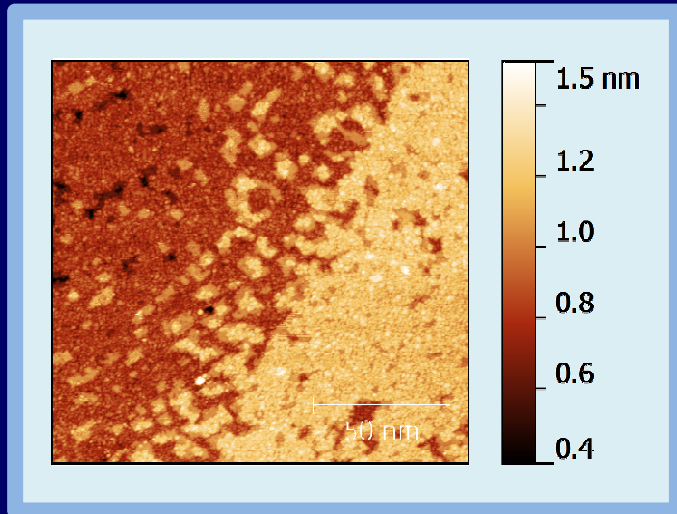
$(\sqrt{2} \times \sqrt{2})R45^\circ$ reconstruction



- Up to 7 u.c. layers can be seen.
- Two terminations: *ordered* and *disordered*.

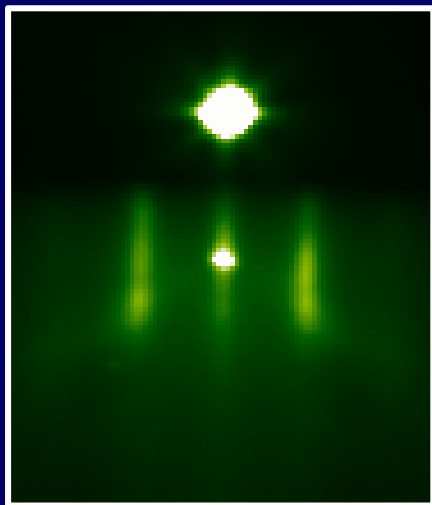
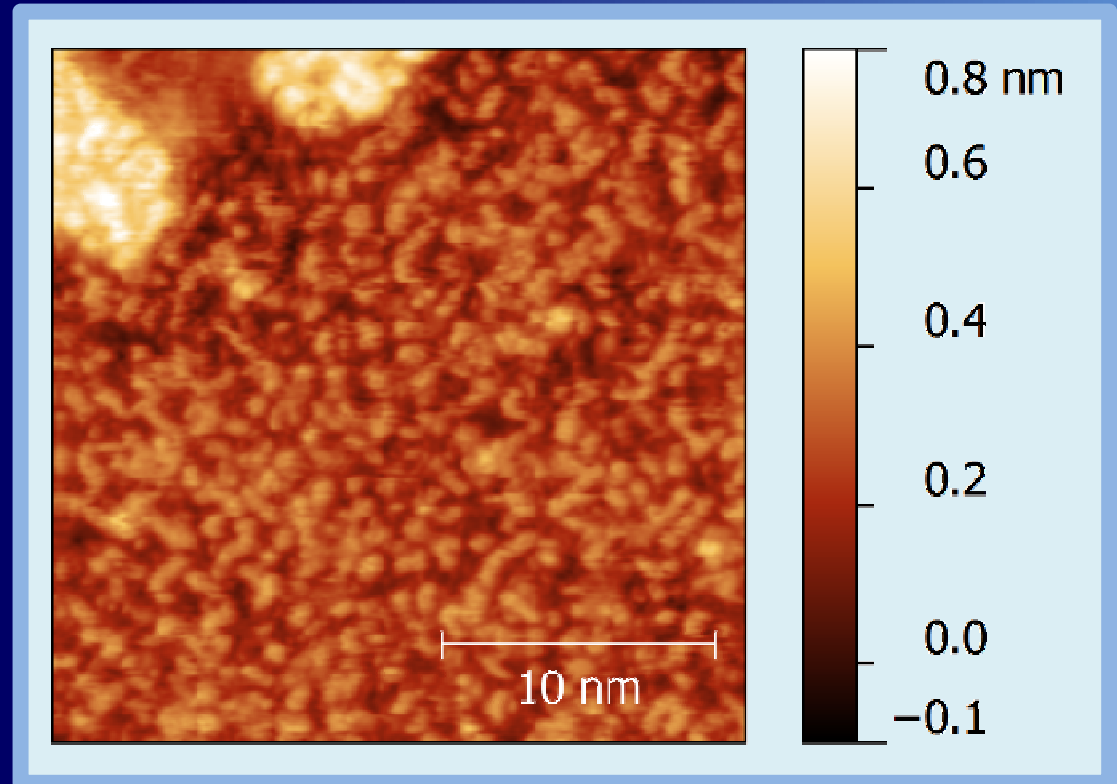
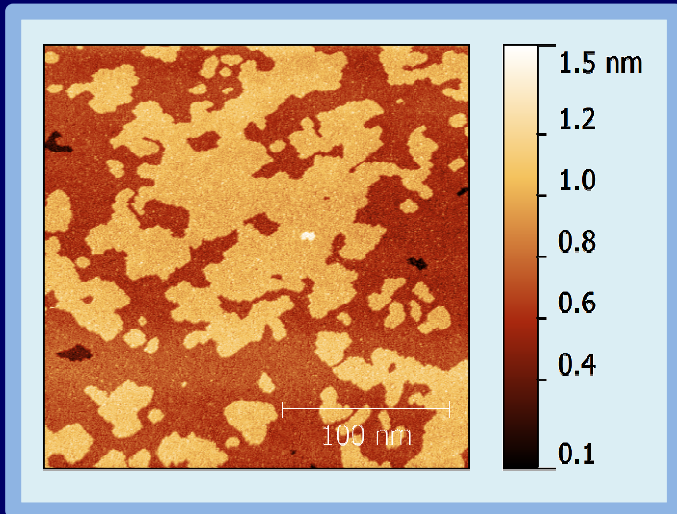
$V_t = -1.8$ V, $I_t = 40-90$ pA, $T = 297$ K

25 u.c. on SrTiO₃



- Appear after anneals in oxygen (at 400 °C for 30 min.) as well as after fast cooling (150 °C/min.) to room temperature.
- No significant change after anneals in vacuum at 450 °C for 30 min.

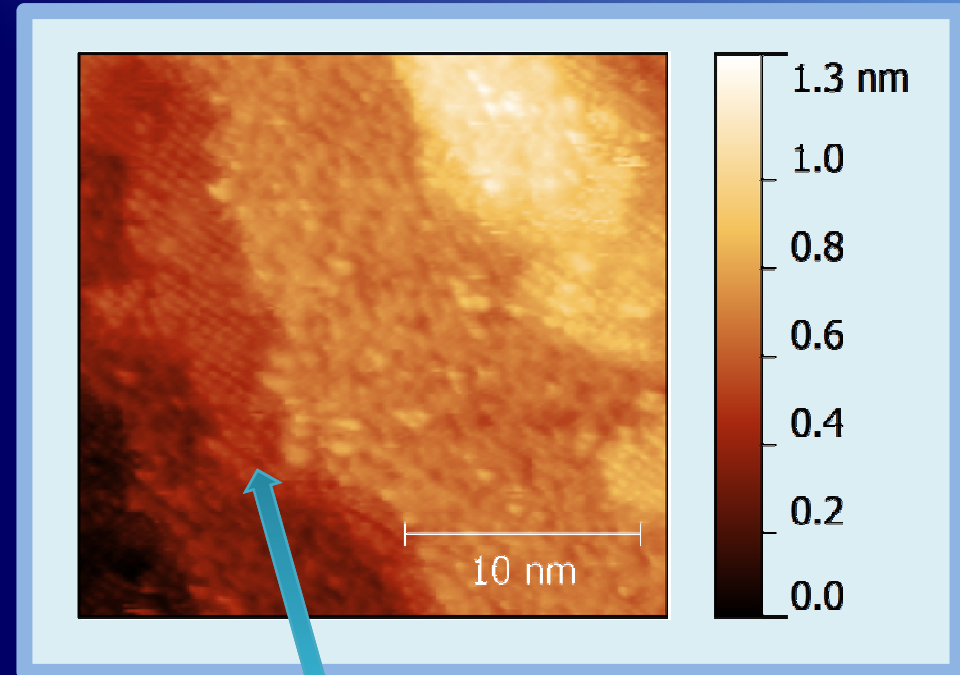
250 u.c. on SrTiO₃



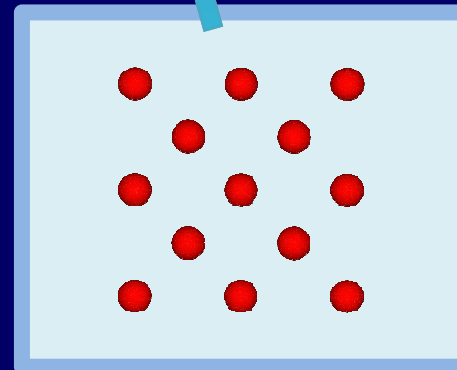
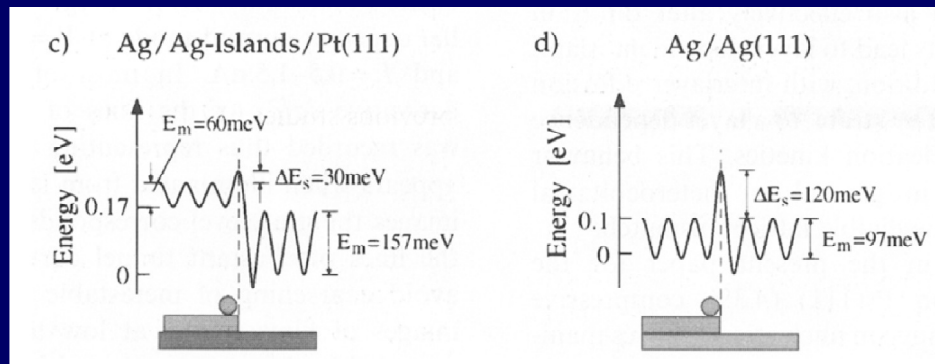
- Up to 3 u.c. layers can be seen.
- One termination: *disordered*.
- Nearly perfect layer-by-layer growth.
- X-ray: film is coherently strained to STO.

Interpretation of the growth evolution

- Dependence of the barrier for interlayer adatom transport (Ehrlich-Schwoebel barrier) on strain.
- Tensile strain increases the barrier.

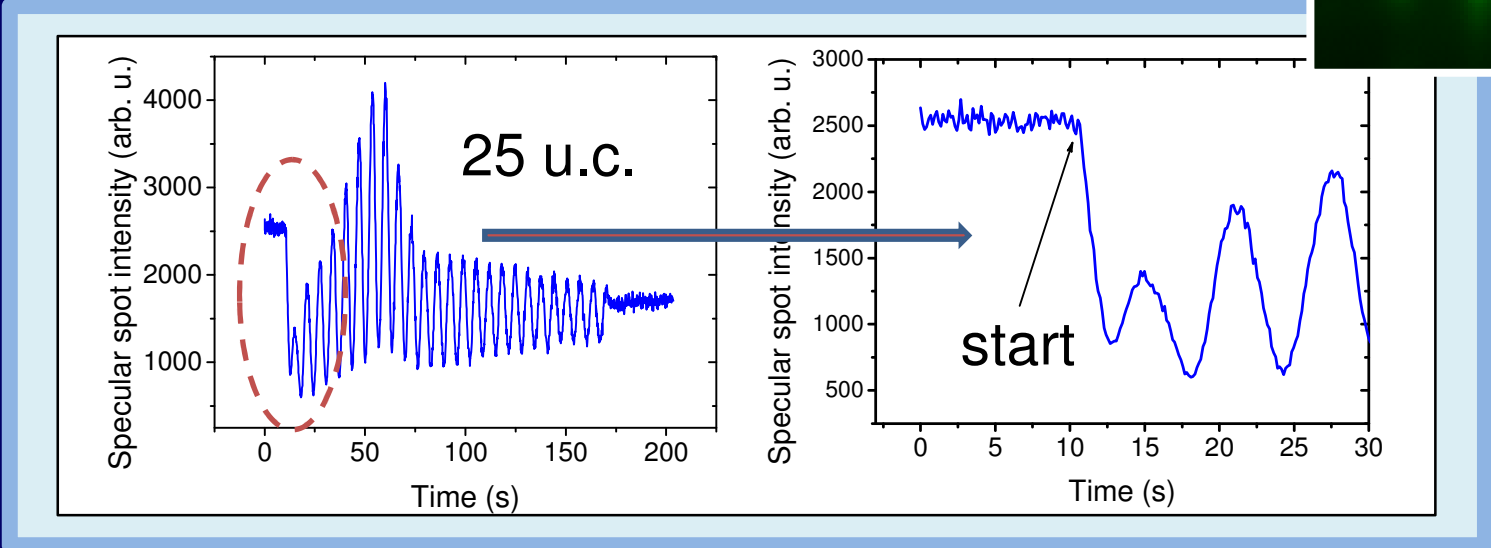
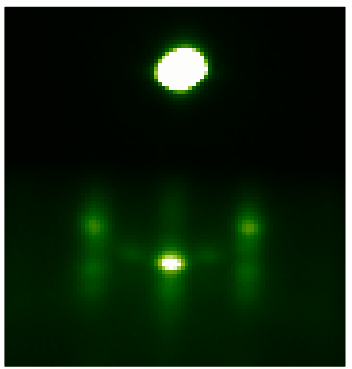
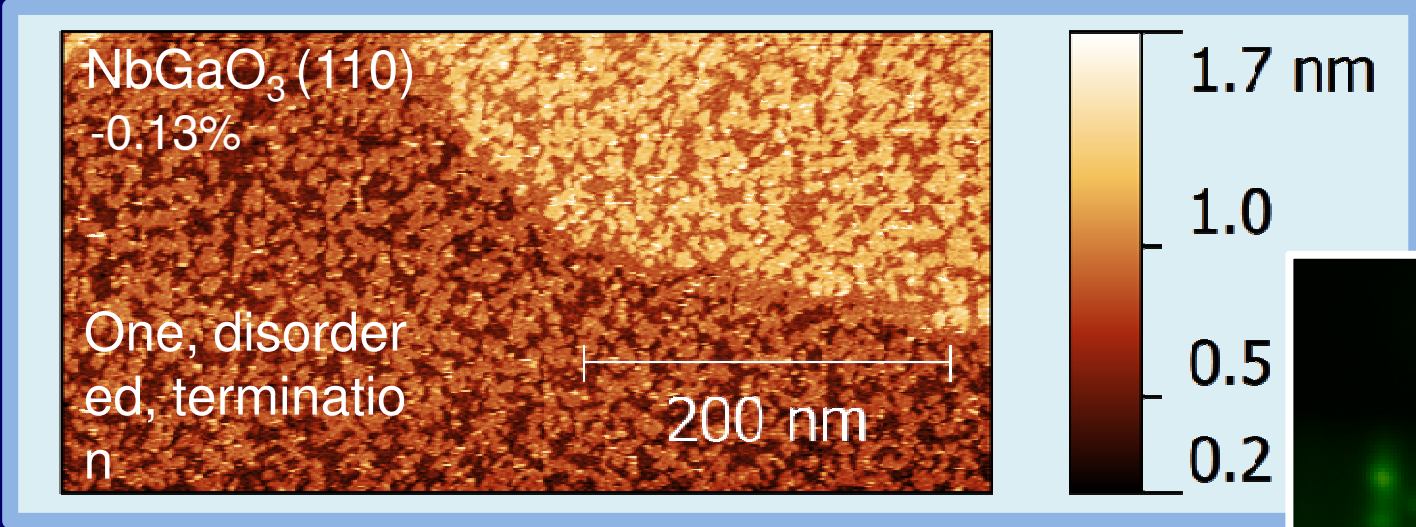


Ag-on-Pt (111) as an example



$\frac{1}{2}$ of oxygen atoms is visible on A-termination

25 u.c. on B-terminated NbGaO₃ – single termination



Acknowledgement

Funding:

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Mike Biegalski



Wolter Siemons



Art Baddorf



Sergei Kalinin

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