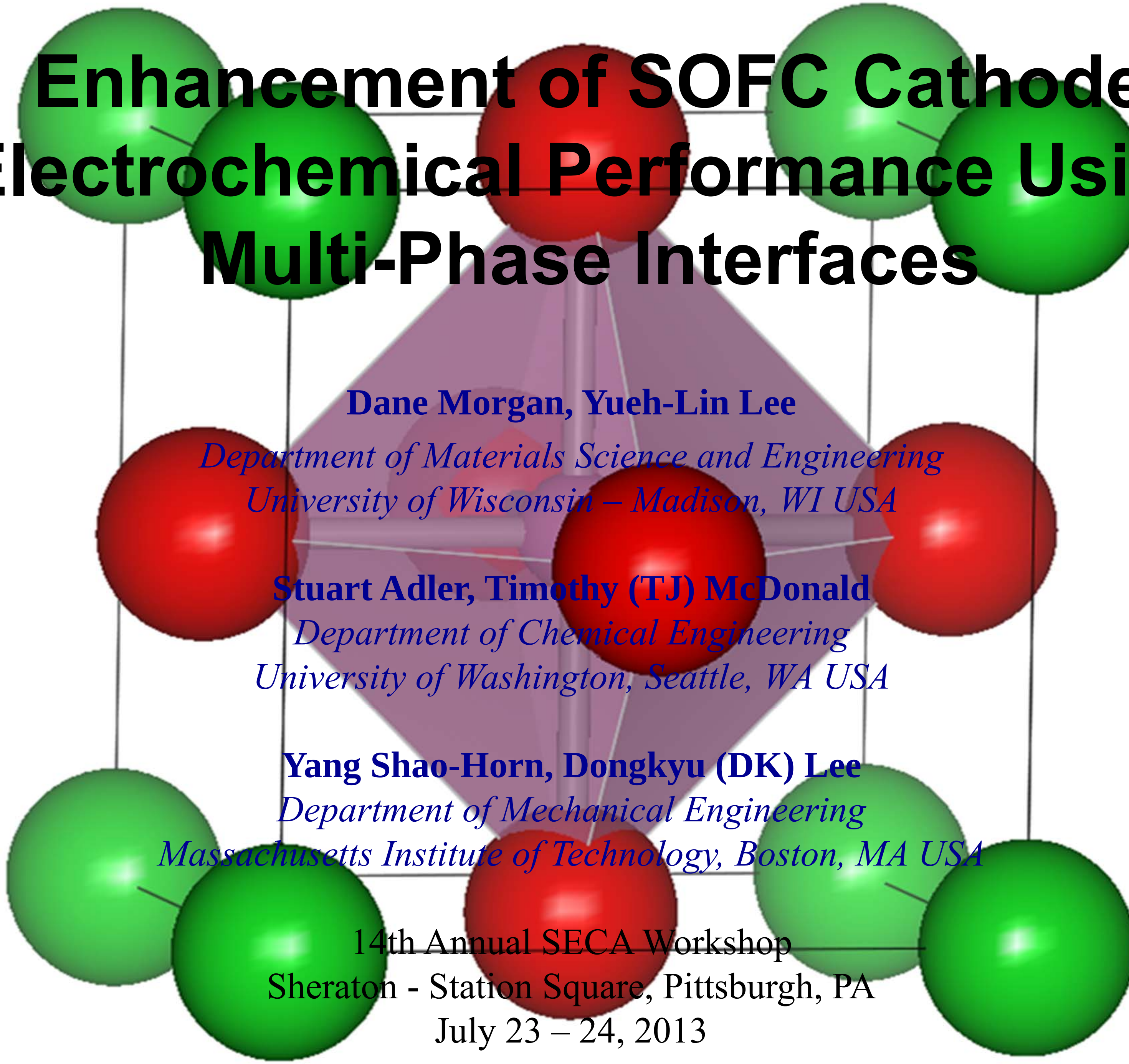

Enhancement of SOFC Cathode Electrochemical Performance Using Multi-Phase Interfaces

Dane Morgan, Yueh-Lin Lee

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Department of Mechanical Engineering

Massachusetts Institute of Technology, Boston, MA USA

14th Annual SECA Workshop

Sheraton - Station Square, Pittsburgh, PA

July 23 – 24, 2013

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External Collaborators

- Michael D. Biegalski, H.M. Christen (Oak Ridge National Laboratory)
- Paul Fuoss, Edith Perret, Brian Ingram, Mitch Hopper, Kee-Chul Chang (Argonne National Laboratory)
- Paul Salvador (Carnegie Melon University)
- Briggs White (NETL)

Computing Support

NSF Supercomputing

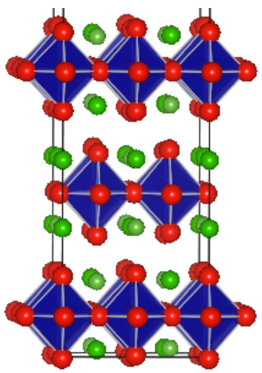
National Energy Research Scientific Computing Center

Oak Ridge National Laboratory

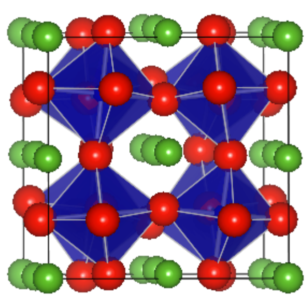
This material is based upon work supported by the Department of Energy under Award Number DE-FE0009435).

Oxide Heterointerface for SOFC Cathodes

Interface of two oxides: Enhances ORR kinetics by orders of magnitude compared to individual phases[1-4]



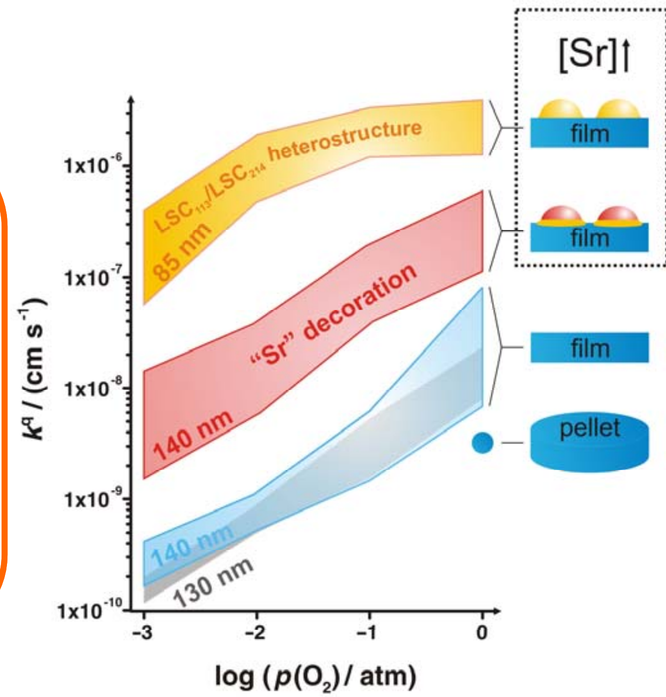
LSC-214: K_2NiF_4 type $AO\text{-}AO\text{-}BO_2$ stacking, coating



LSC-113: ABO_3 Perovskite ($AO\text{-}BO_2$ stacking) Cathode Material

Enhances ORR kinetics at 500-600° C

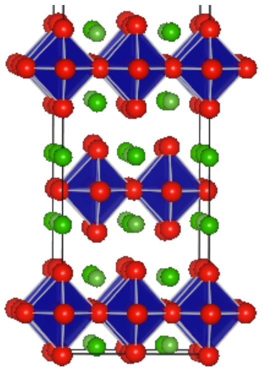
Novel Heterostructure



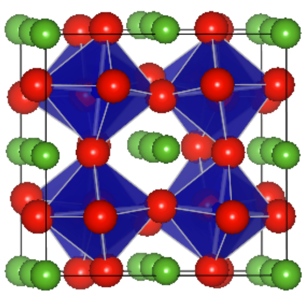
[1] E. J. Crumlin, et al., *The Journal of Physical Chemistry Letters, 1, 3149-3155.*
[2] M. Sase, et al., *Journal of The Electrochemical Society, 2008, 155, B793-B797.*
[3] M. Sase, et al., *Solid State Ionics, 2008, 178, 1843-1852.*
[4] K. Yashiro, et al., *Electrochem. Solid State Lett., 2009, 12, B135-B137.*

Oxide Heterointerface for SOFC Cathodes

Interface of two oxides: Enhances ORR kinetics by orders of magnitude compared to individual phases[1-4]



LSC-214: K_2NiF_4 type
AO-AO-BO_2 stacking, coating



LSC-113: ABO_3 Perovskite
(AO-BO_2 stacking)
Cathode Material

1. **How does this interfacial enhancement work?**
2. **Can it be extended to XYZ-214/LSCF-113 interfaces?**
3. **Can we make more active, more stable cathodes with these interfaces?**

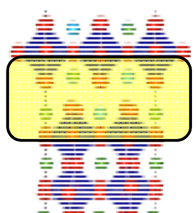
[1] E. J. Crumlin, et al., *The Journal of Physical Chemistry Letters, 1, 3149-3155.*
[2] M. Sase, et al., *Journal of The Electrochemical Society, 2008, 155, B793-B797.*
[3] M. Sase, et al., *Solid State Ionics, 2008, 178, 1843-1852.*
[4] K. Yashiro, et al., *Electrochem. Solid State Lett., 2009, 12, B135-B137.*

Project Overview

LSC-214/LSCF-113 Films

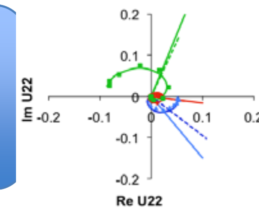
Yang Shao-Horn (MIT)
Present work: LSC-214/LSC-113 and LSC-214/LSCF-113

Ab initio Energetics Thermokinetic Modeling



Dane Morgan (U Wisc.)
Present work: Sr Thermodynamics in LSC, LSCF

NLEIS + Rate modeling, LSC-214/LSCF-113 porous electrodes



Stuart Adler (U Wash.)
Present work: (NLEIS) on LSC_{113}, $LSCF_{113}$

Overall Conclusions

- LSC_{214} enhances $LSCF_{113}$ (~3x) far less than LSC_{113} (~100x)
- $LSCF_{113}$ has a more stable and Sr rich surface than LSC_{113}
  - Supported by aspects of AFM, Auger, DFT, NLEIS
- LSC_{214} changes Sr stability of LSC_{113} more than $LSCF_{113}$ and may enhance LSC_{113} performance by stabilization of Sr rich interface
  - Supported by AFM, Auger, COBRA, DFT

What Are Our Compositions?

- LSC_{113} = $(La_{0.8}Sr_{0.2})CoO_3$
- $LSCF_{113}$ = $(La_{0.6}Sr_{0.4})(Co_{0.2}Fe_{0.8})O_3$
- LSC_{214} = $(La_{0.5}Sr_{0.5})_2CoO4$

Project Overview

LSC-214/LSCF-113 Films

LSC-214

LSCF-113

Yang Shao-Horn (MIT)
Present work: LSC-214/LSC-113 and LSC-214/LSCF-113

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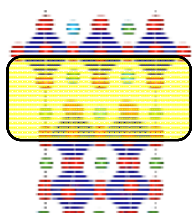

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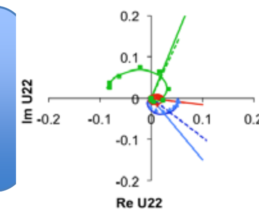

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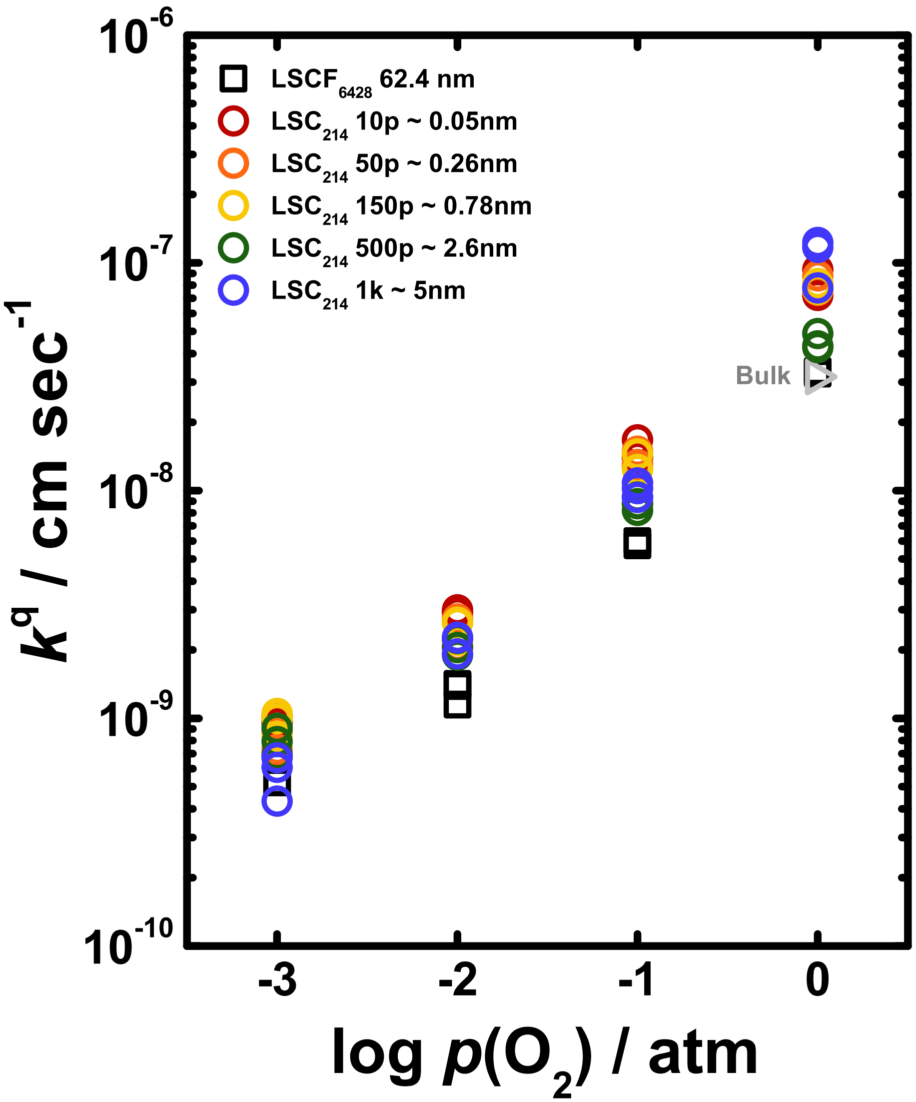
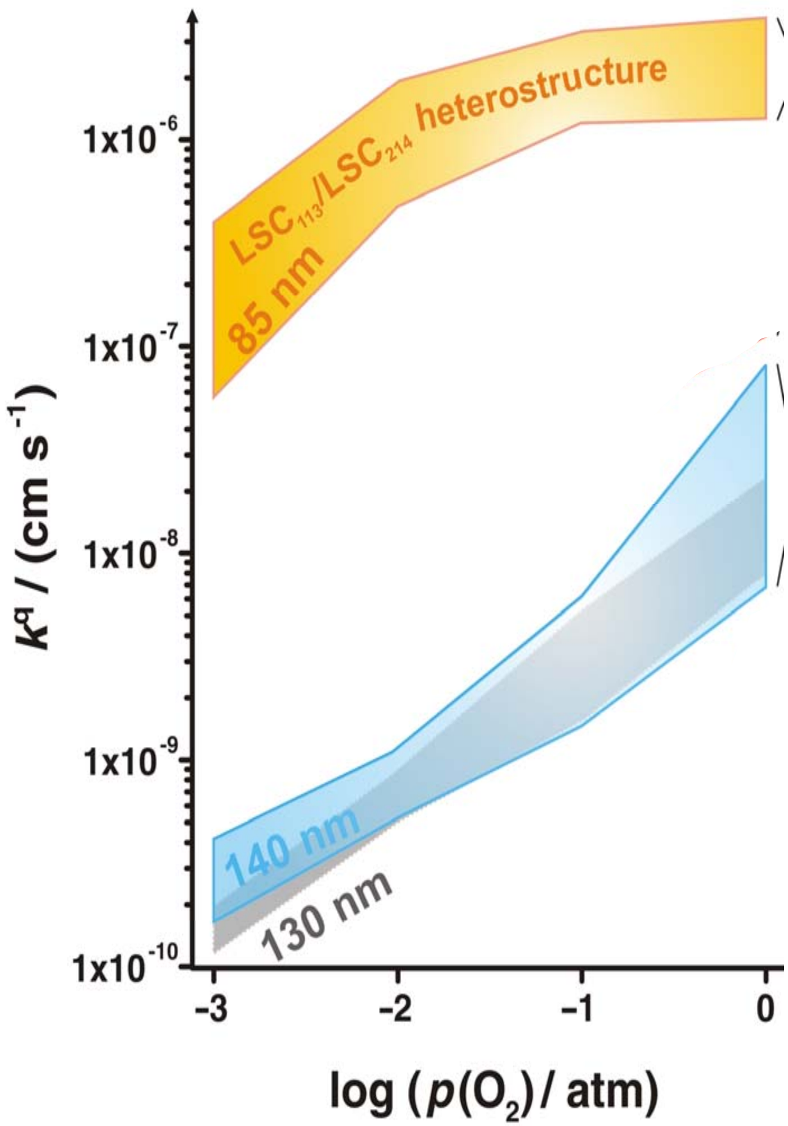
Surface Exchange Kinetics

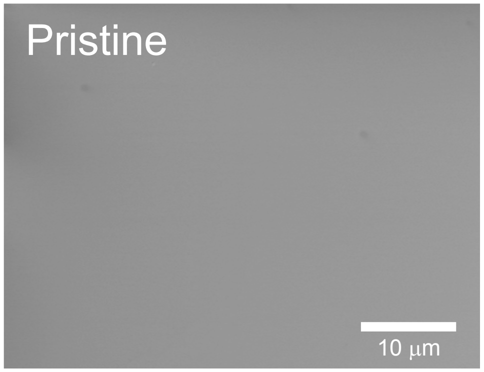


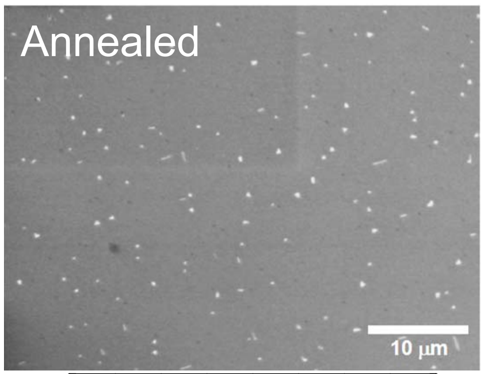


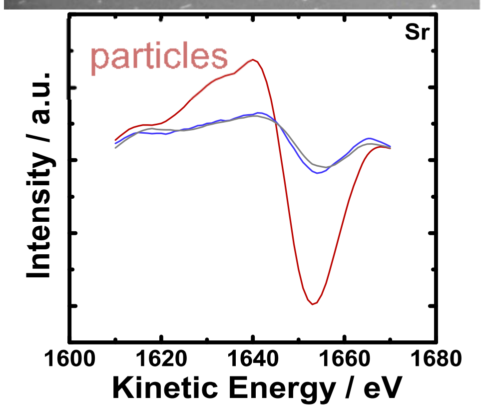
- LSC$_{214}$ decoration can slightly enhance the surface exchange rate (k^q) of LSCF
- LSC$_{214}$ decorated LSCF shows comparable k^q with LSC$_{214}$

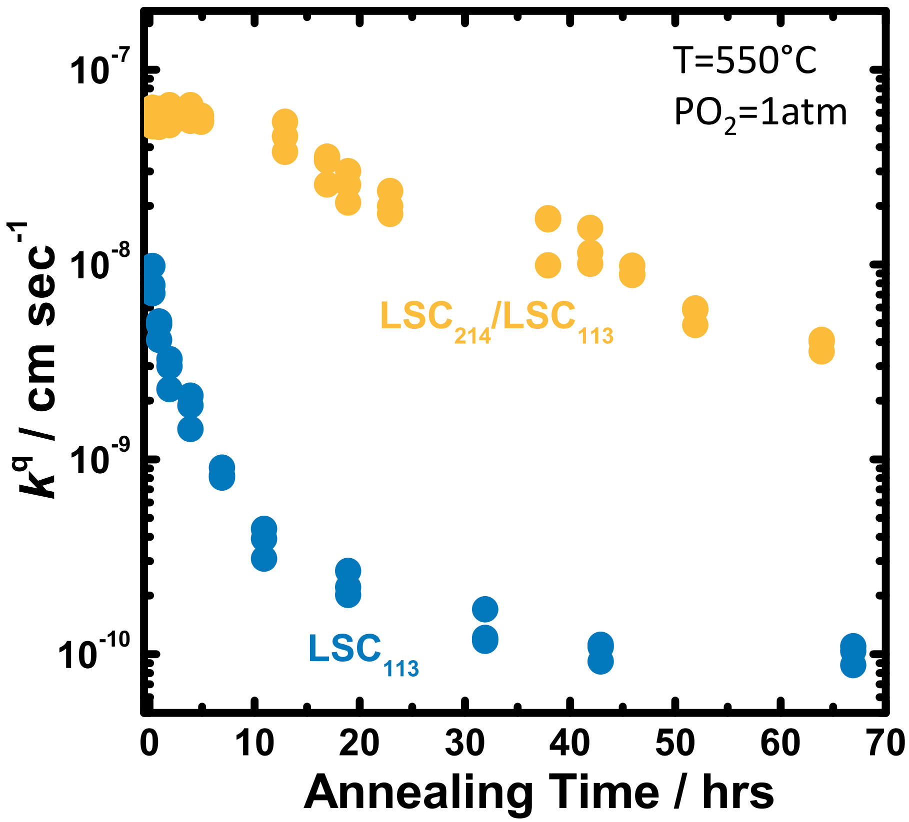
Auger Electron Spectroscopy of LSC_{113} and $LSC_{113/214}$ on GDC/YSZ (001)

LSC_{113}

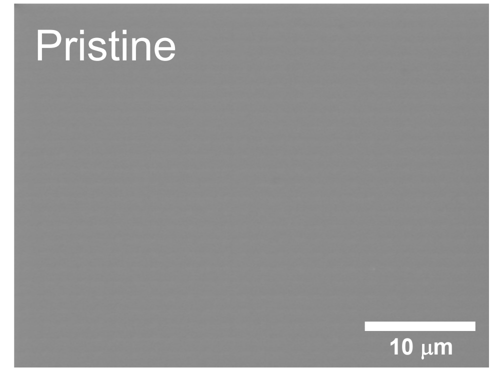


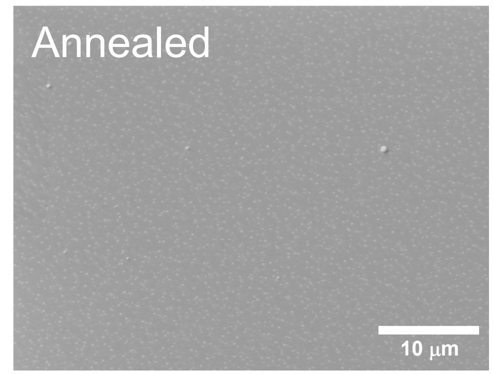


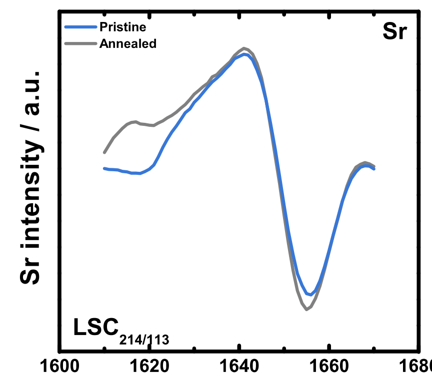




$LSC_{113/214}$







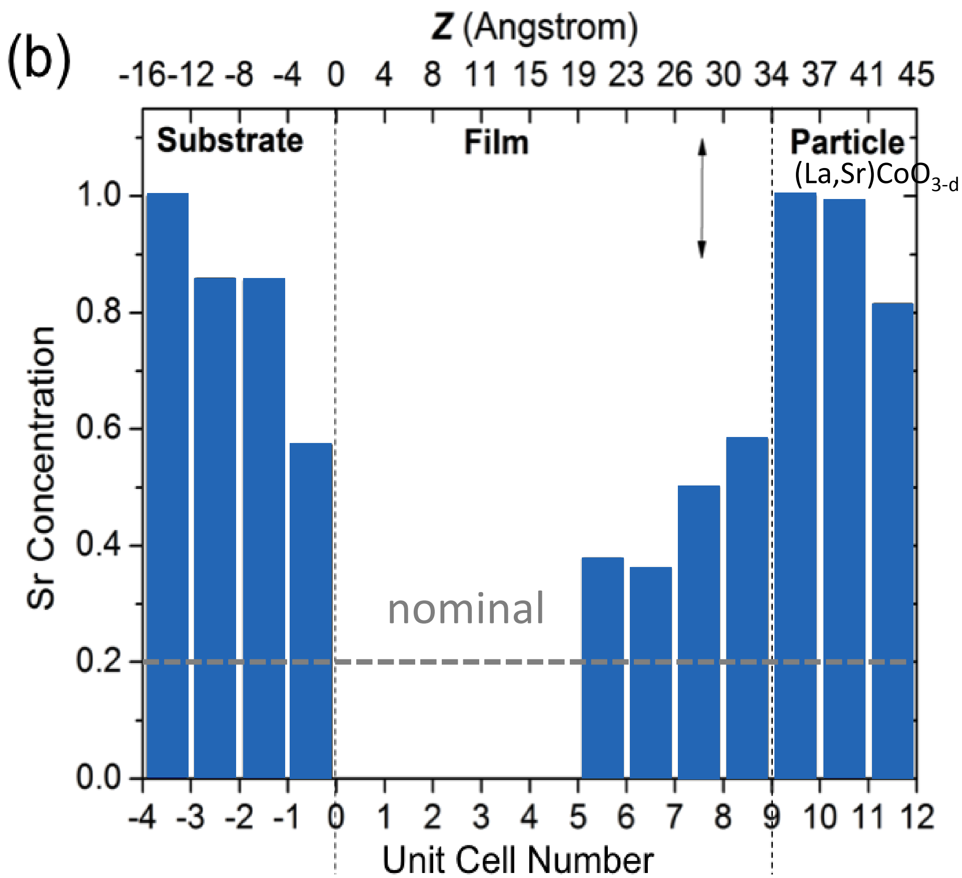
Feng et al., JPCL 2013 and D Lee*, YL Lee* et al, *Manuscript In Preparation*

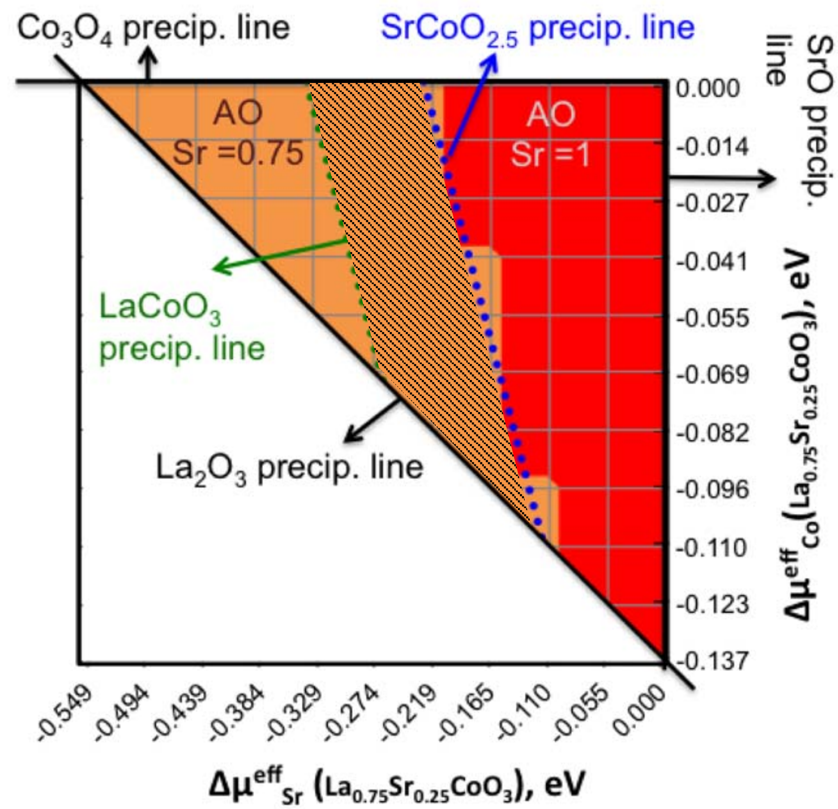
Sr Occupancy in LSC_{113} Surface

Coherent Brag Rod Analysis (COBRA)

550°C, PO_2=1atm

Consistent with DFT (~0.75 Sr)

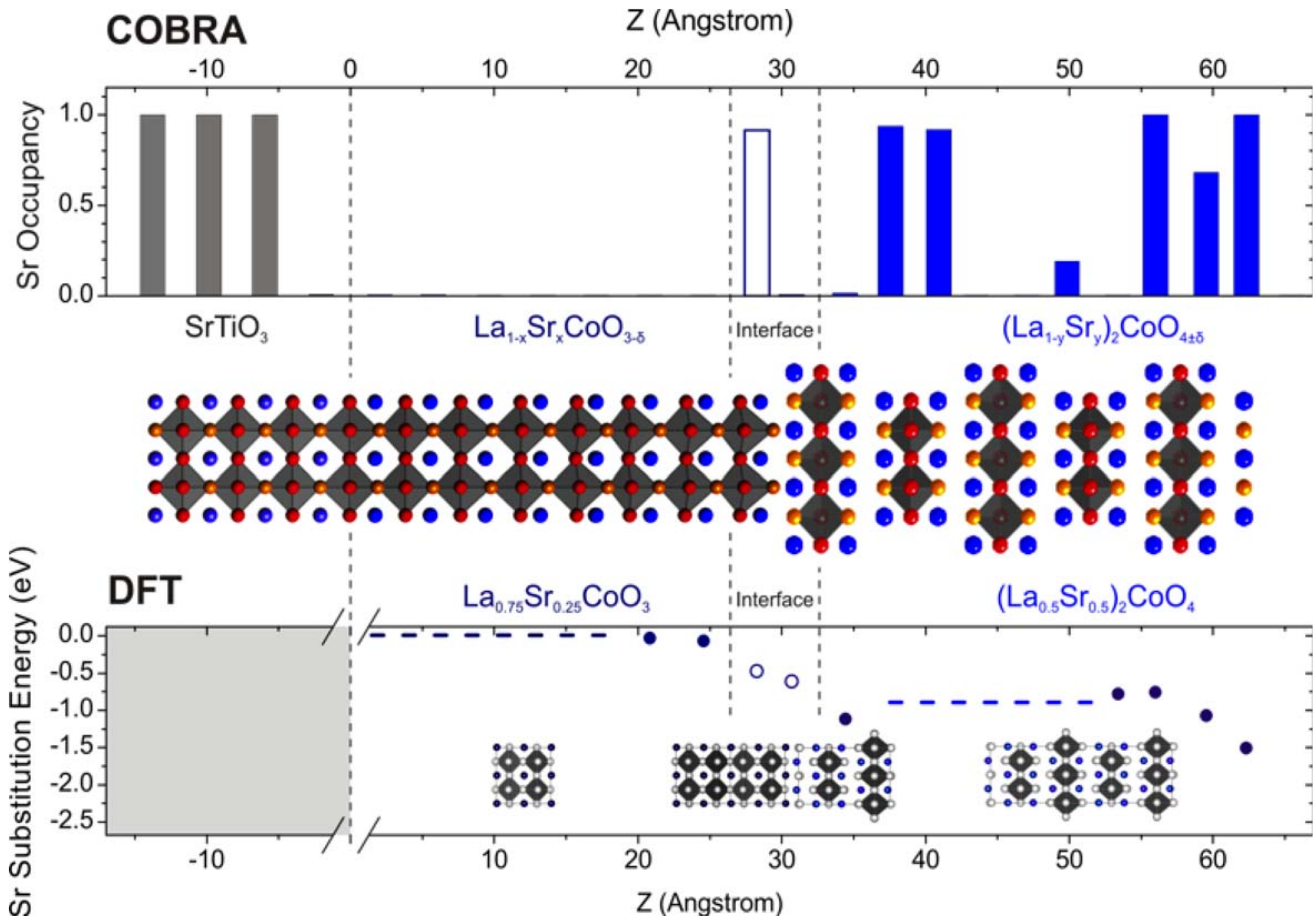




Feng et al., Energy Environ. Sci. 2014; Feng et al., J Phys. Chem. Lett. 2014; Lee, et al. in preparation 2014

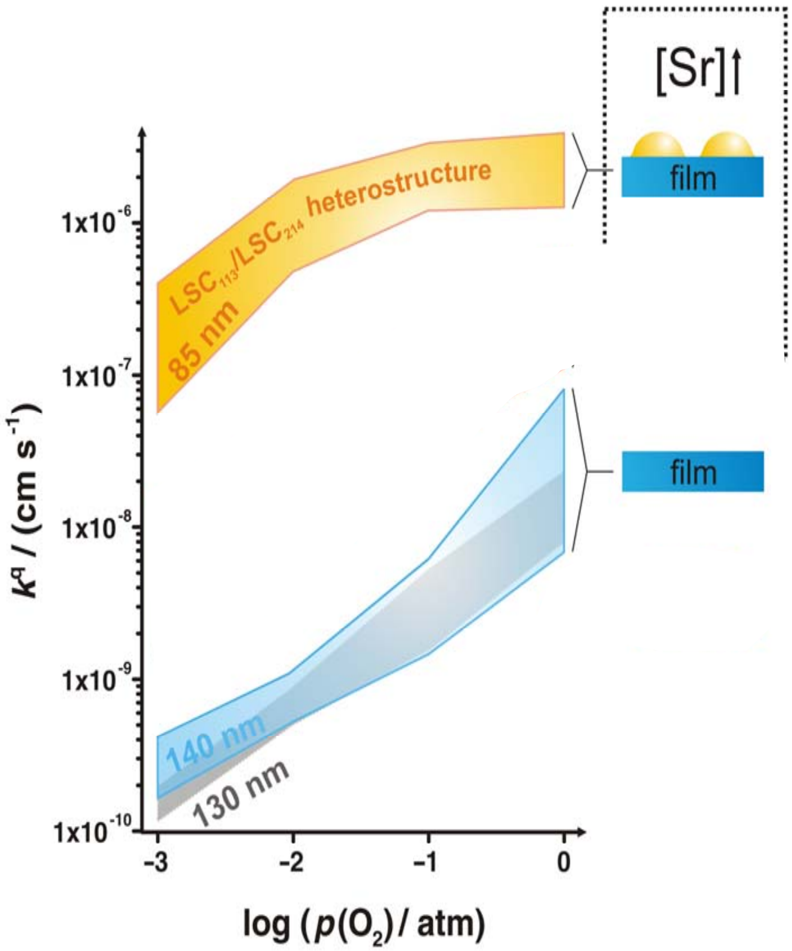
LSC_{113} has about 0.6-0.8 Sr in top (La,Sr)O [001] layer

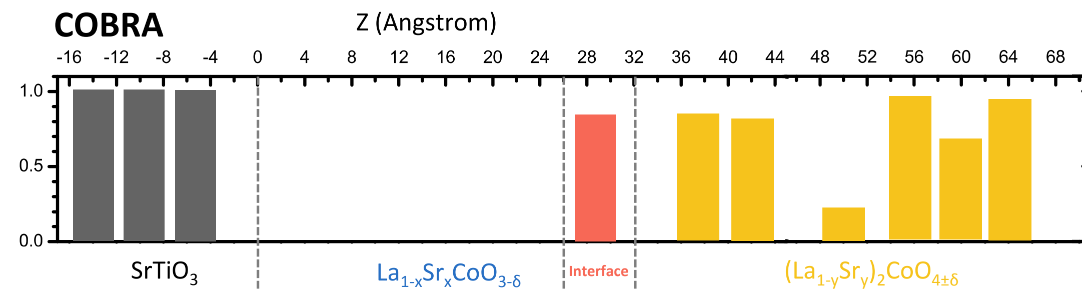
Sr Occupancy in LSC_{214}/LSC_{113} Interface



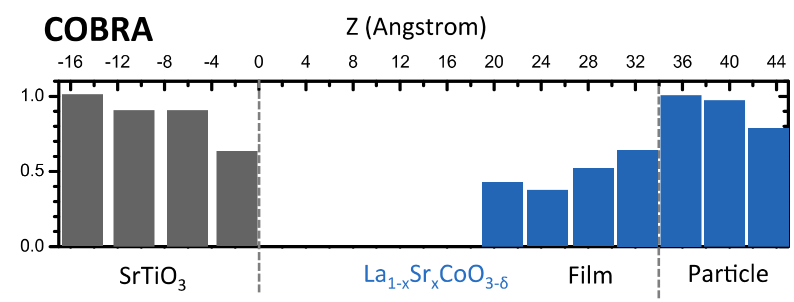
Sr in interface and LSC_{214} film and depleted from LSC_{113}

Surface Sr Segregation =>Enhanced Activity of $LSC_{113/214}$





More active, more stable



Less active, less stable

Feng et al., Energy Environ. Sci. 2014; Feng et al., J Phys. Chem. Lett. 2014

Sr Occupancy in LSCF$_{113}$ Surface

550°C, PO_2=1atm

$\Delta\mu^{eff}_{Fe}(LSCF_{113}) = -0.24$ eV vs. $\mu^{eff}_{Fe}(Fe_2O_3)$

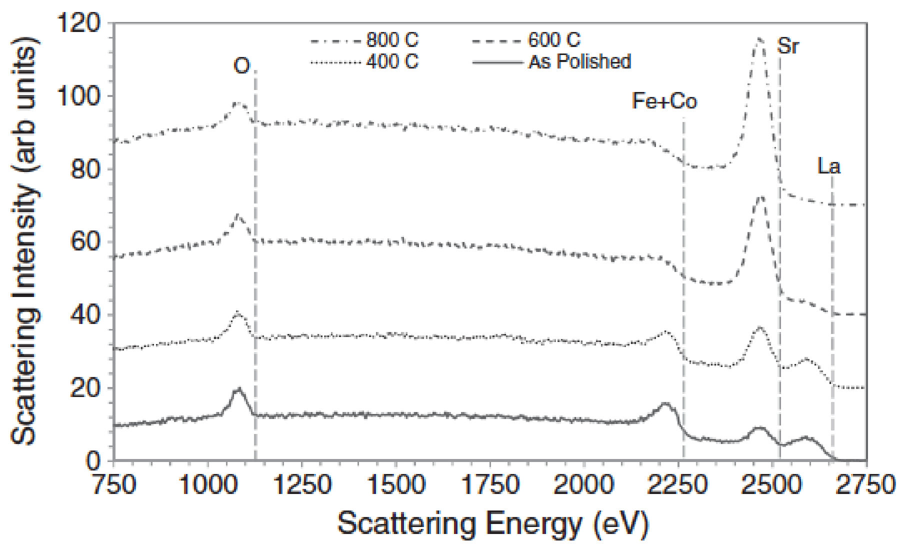
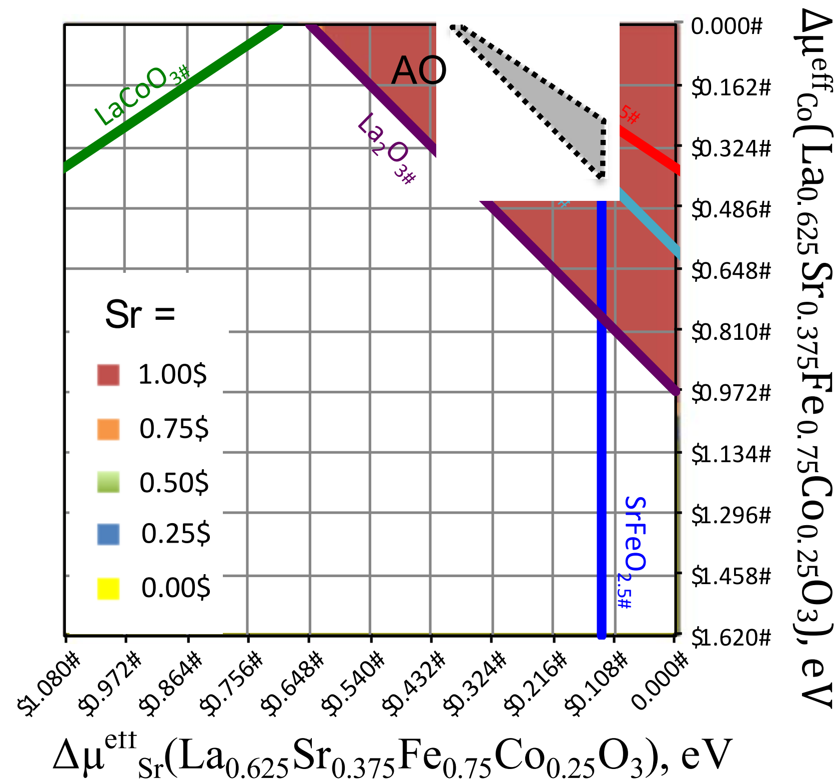


Fig. 1. LEIS spectra (3 keV $^4He^+$ ions) for LSCF annealed in air for 8 h at various temperatures. Vertical lines indicate theoretical scattering edges for two-body scattering.

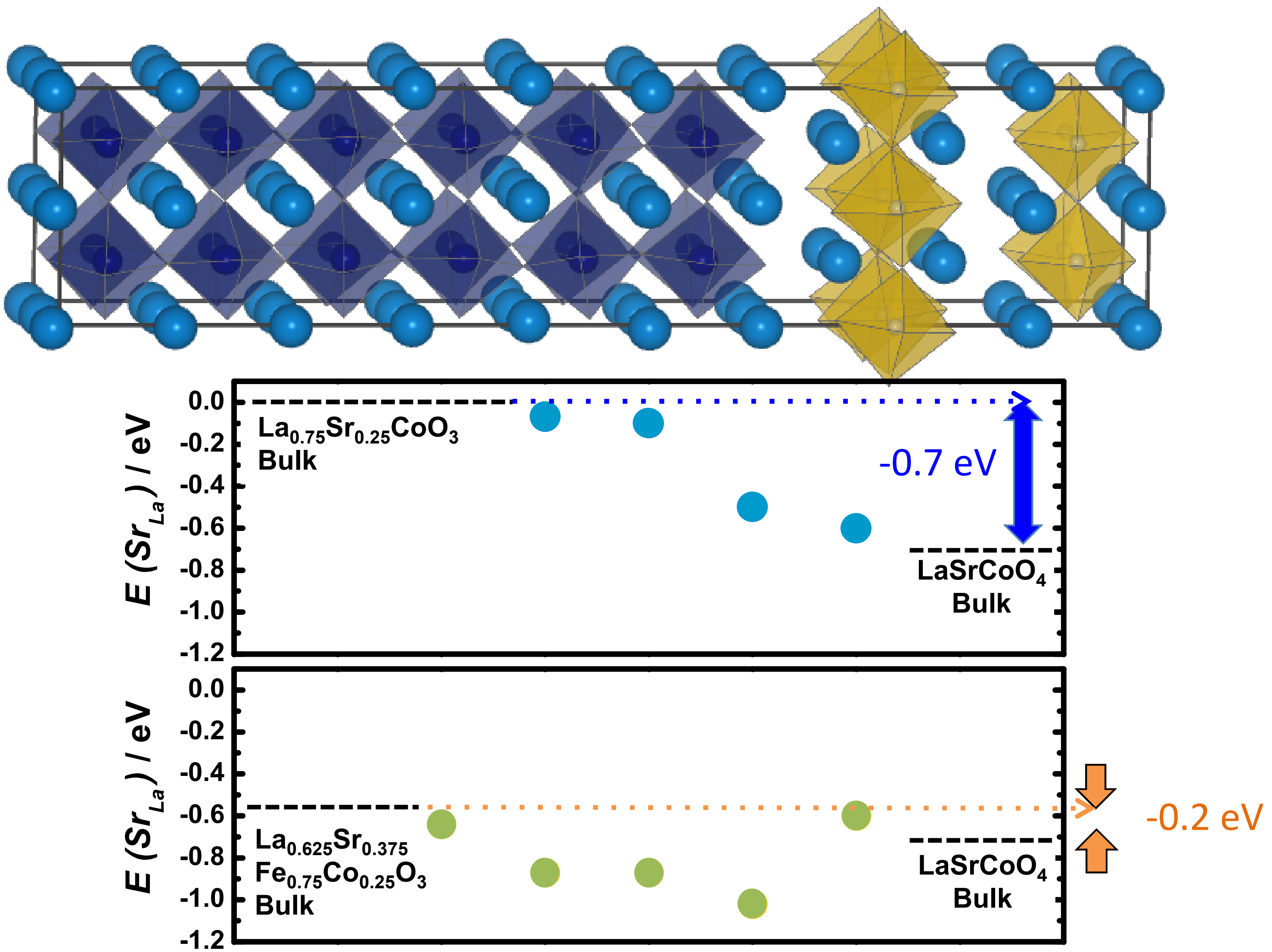
Druce SSI 2014



Lee et al. in prep

- *Ab initio* analysis predicts LSCF$_{113}$ (001) AO surface with surface layer Sr conc. 100% is stable
- Agreement between *ab initio* thermodynamic analysis and the Low Energy Ion Scattering (LEIS) measurement

Sr Occupancy in $LSC_{214}/LSCF_{113}$ Interface



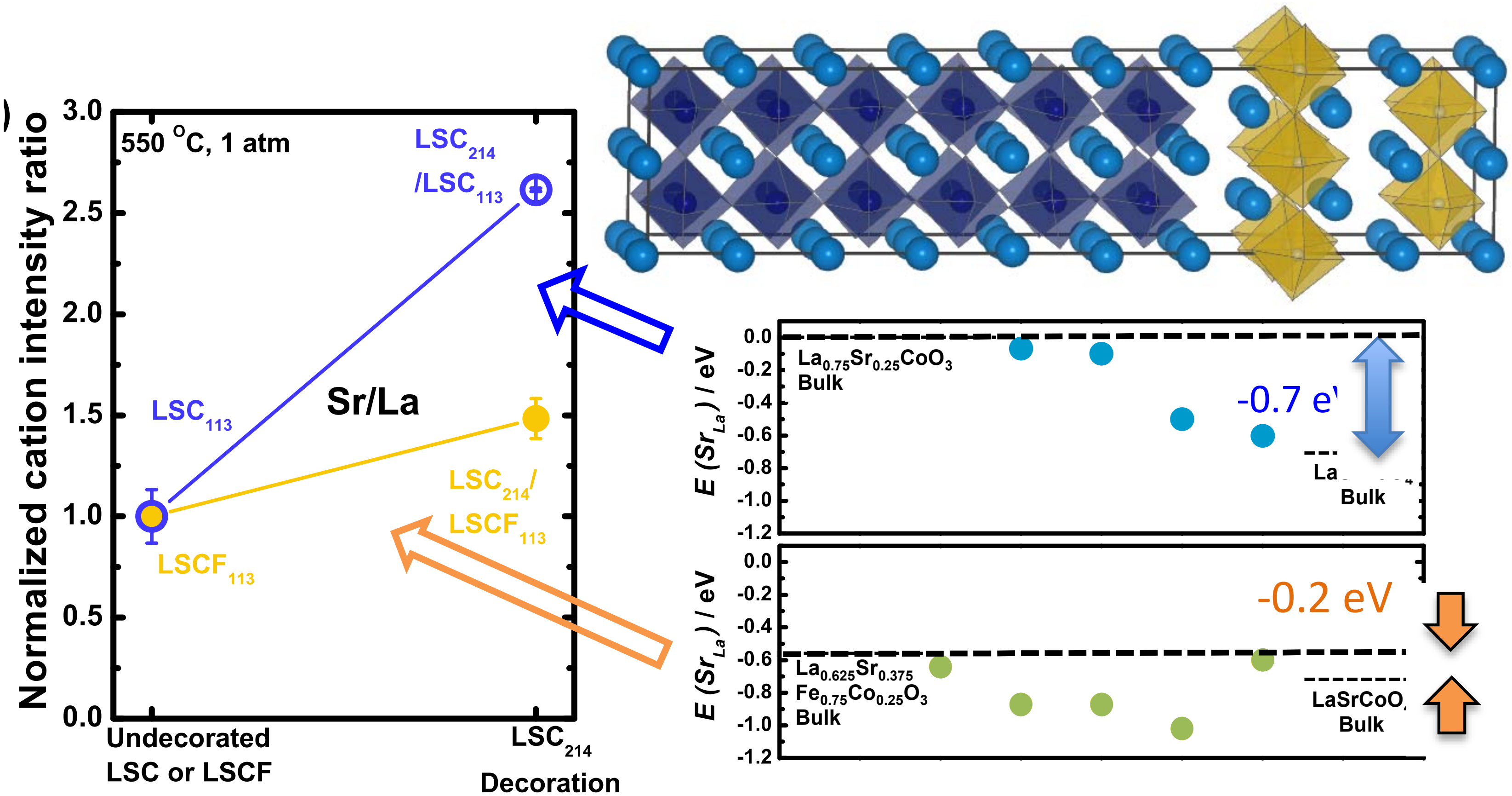
Strong enhancement

Weak enhancement

Gadre, PCCP, 2012; Lee et al. in prep

Ab initio analysis predicts $LSCF_{113}$ more stable vs. Sr reaction with LSC_{214} than LSC_{113}

Sr Occupancy in $LSC_{214}/LSCF_{113}$ Interface



Strong enhancement

Weak enhancement

Gadre, PCCP, 2012
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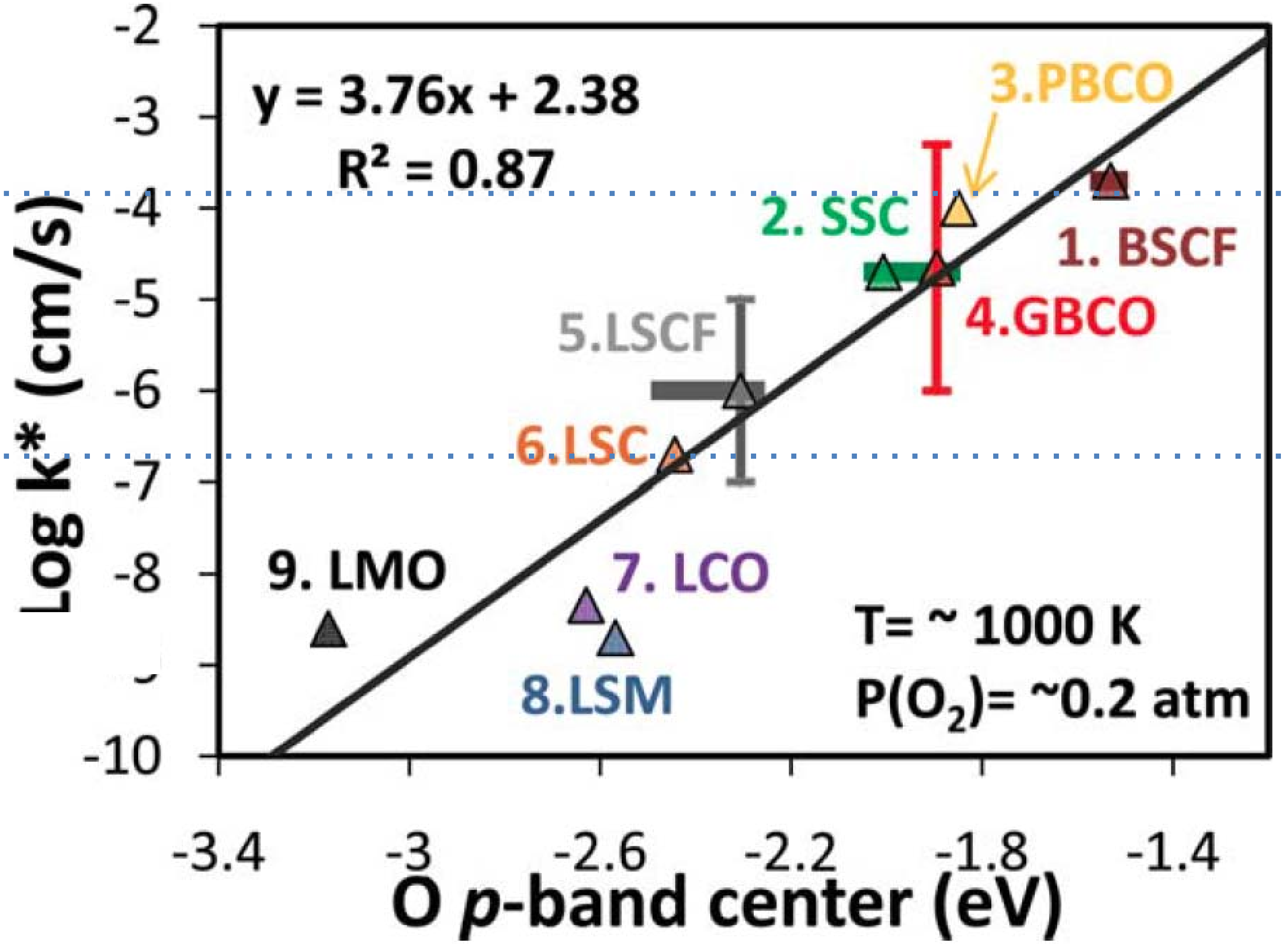
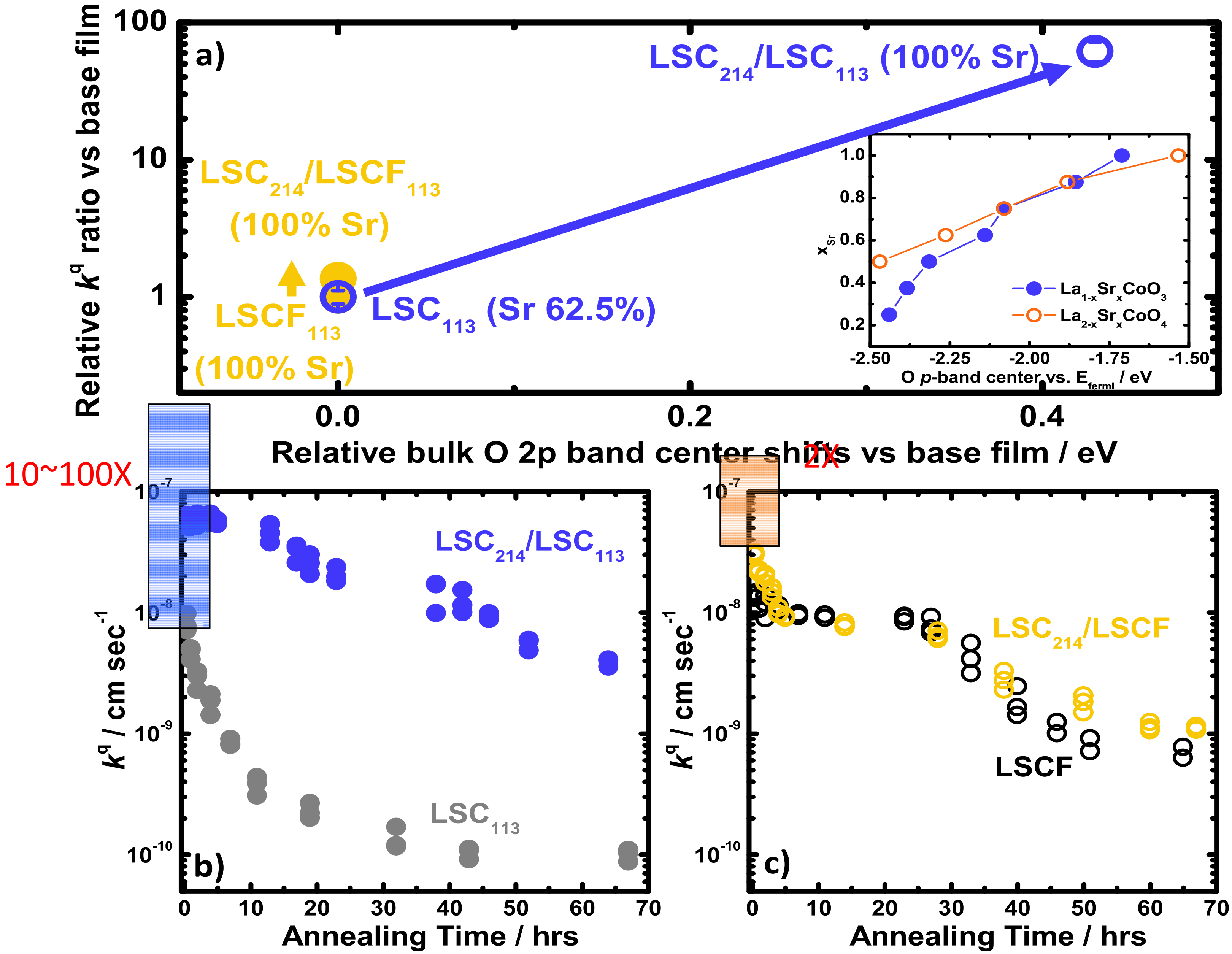
Ab initio analysis predicts $LSCF_{113}$ more stable vs. Sr reaction with LSC_{214} than LSC_{113}

P-band Correlation for SOFC Oxygen Reduction



Lee, Rossmeisl, Shao-Horn, Morgan, EES 2011

P-band Correlation Consistent with Interfacial Enhancements



Summary

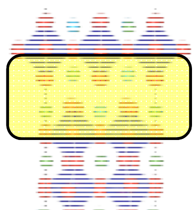
- Coating with LSC_{214} enhances LSC_{113} much more than $LSCF_{113}$.
- Ab initio and COBRA surface stability analysis suggests
  - Unsaturated surface layer Sr content (60~75%) for LSC_{113} within the bulk stability region
  - Saturated Sr content (100%) for $LSCF_{113}$ within the bulk stability region
- LSC_{214} decoration → Introduces Sr/La chemical potential perturbation near surface for LSC_{113} more than $LSCF_{113}$
  - Strong thermodynamic driving force (-0.7~-0.9 eV) for Sr_{La} interdiffusion between LSC_{113} and LSC_{214}
  - Little thermodynamic driving force for Sr_{La} interdiffusion (-0.2 eV) between $LSCF_{113}$ and LSC_{214}
  - Sr segregation with LSC_{214} decoration observed for LSC_{113} but not $LSCF_{113,}$ consistent with DFT. May be origin of enhanced performance!
  - Longer-term (10h-70h) surface exchange kinetics may couple with formation of surface Sr secondary phases and surface Sr concentrations making it sensitive to Sr segregation induced by LSC_{214}.

Project Overview

LSC-214/LSCF-113 Films

Yang Shao-Horn (MIT)
Present work: LSC-214/LSC-113 and LSC-214/LSCF-113

Ab initio Energetics Thermokinetic Modeling



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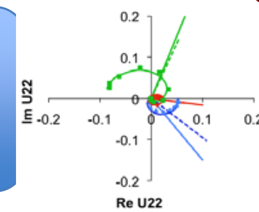


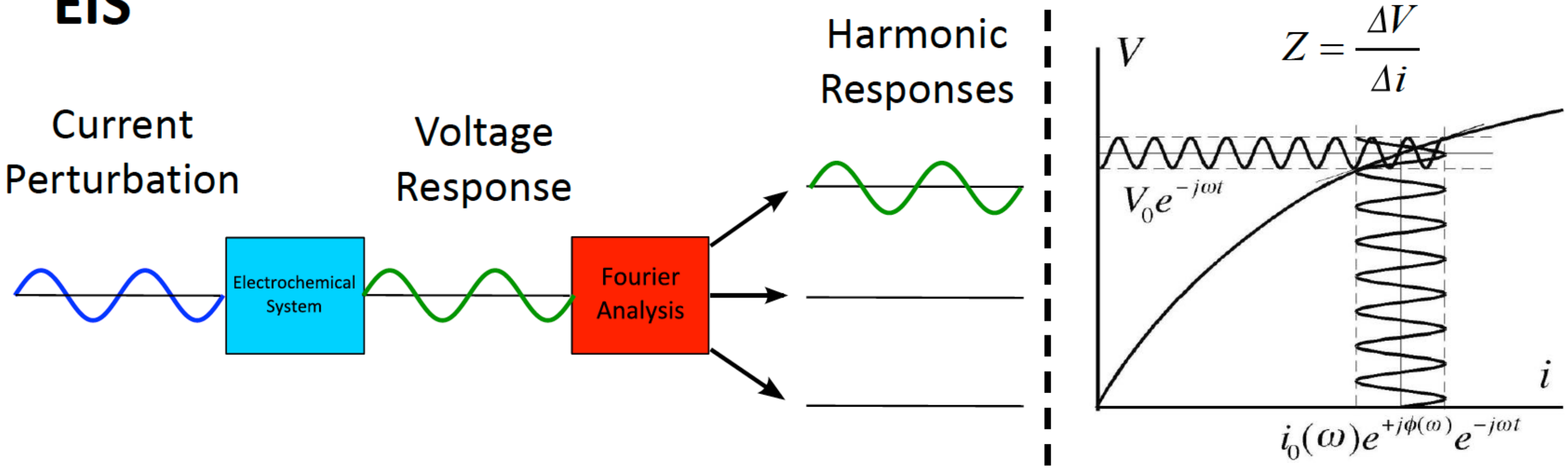
Stuart Adler (U Wash.)
Present work: (NLEIS) on LSC_{113}, $LSCF_{113}$

Non-Linear Impedance Spectroscopy (NLEIS) on LSC_{113}, $LSCF_{113}$

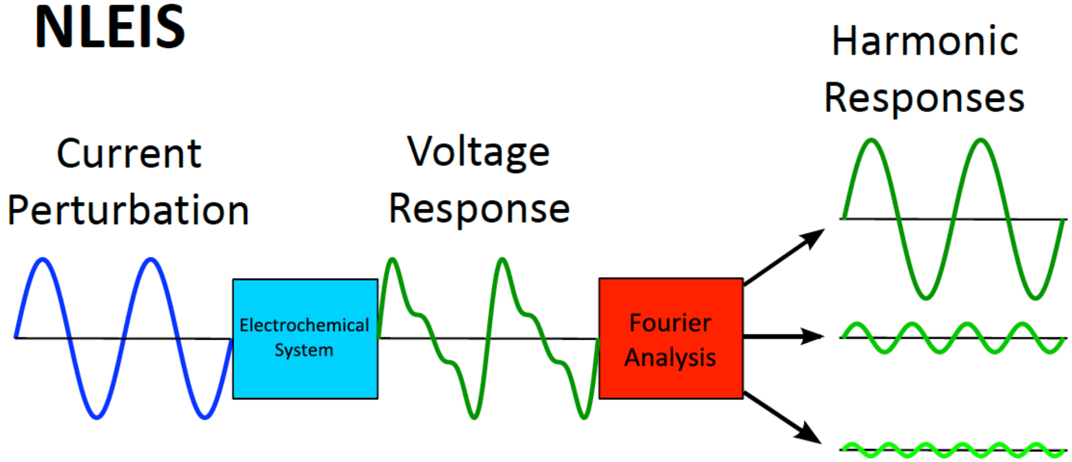
Adler (Univ. Washington)

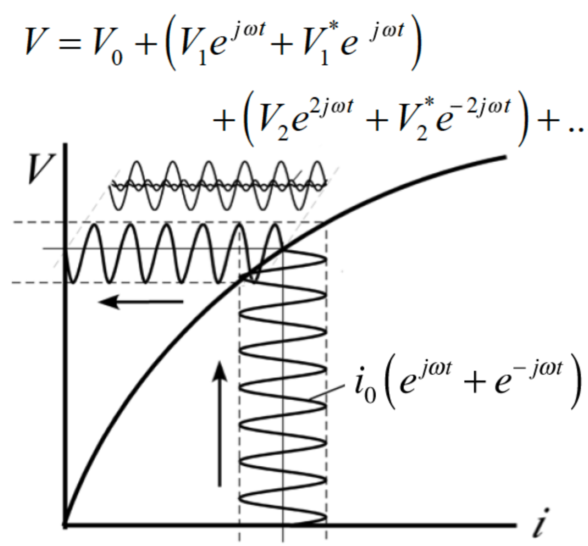
Electrochemical Measurements

EIS



NLEIS

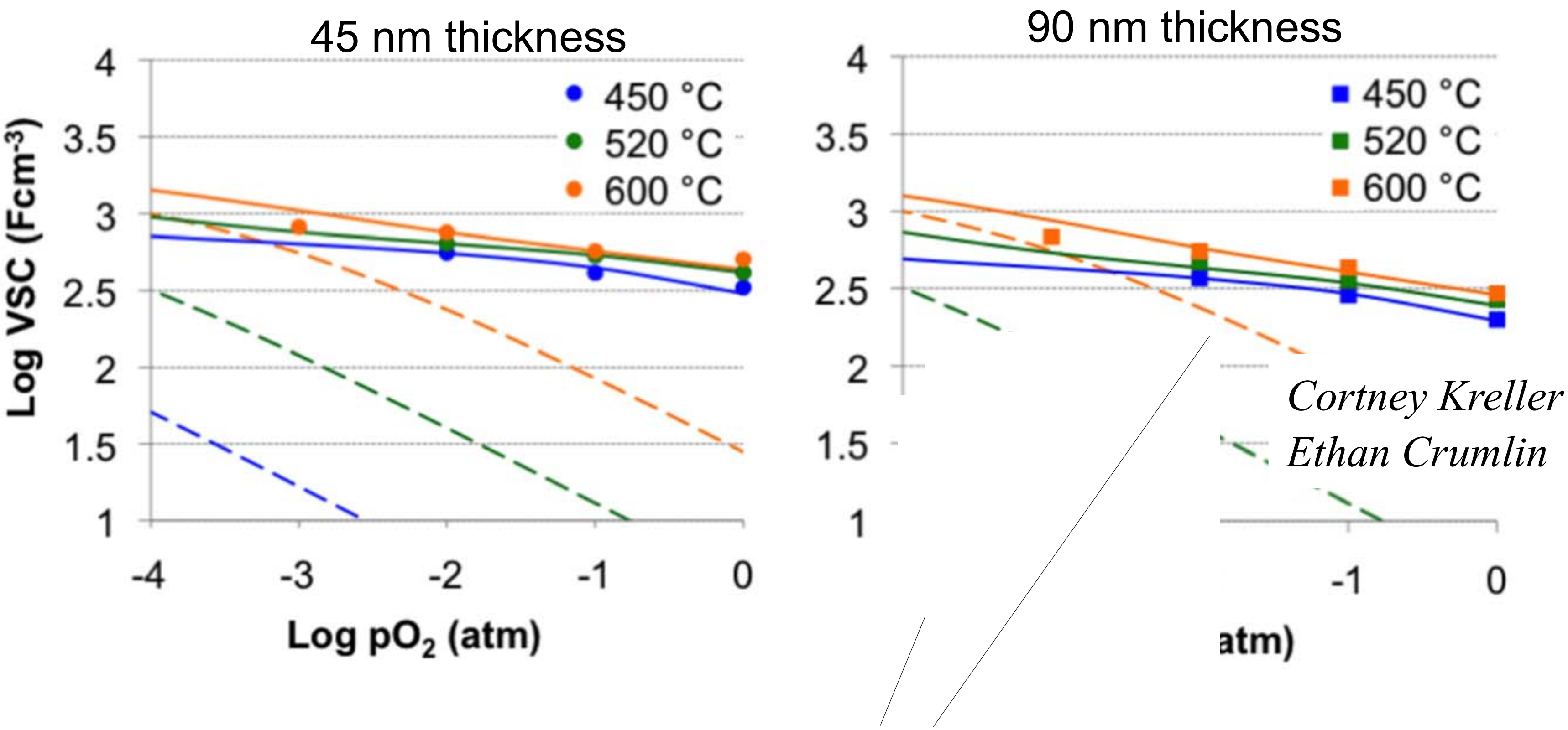




NLEIS insensitive very sensitive to kinetic/transport and thermodynamic properties

Example: $(La_{0.8}Sr_{0.2})CoO_3$ thin films

Volume-Specific Capacitance (VSC) of LSC thin films vs. pO2 and thickness

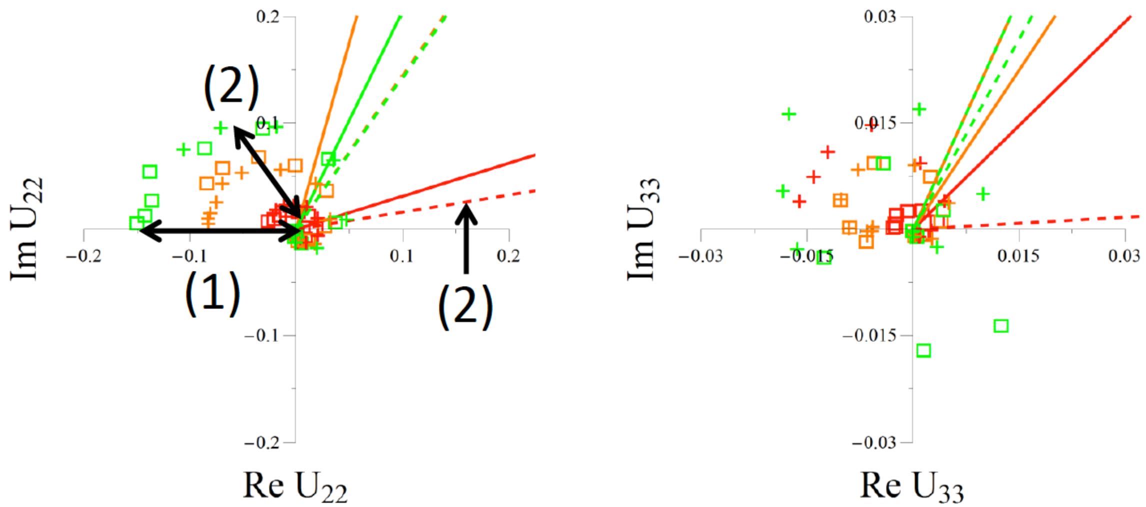


Cortney Kreller
Ethan Crumlin

Predicted from bulk model (Kawada, et al. JES '02)

Example: $(La_{0.8}Sr_{0.2})CoO_3$ thin films

NLEIS response of 34 nm LSC-82 thin film vs. pO2



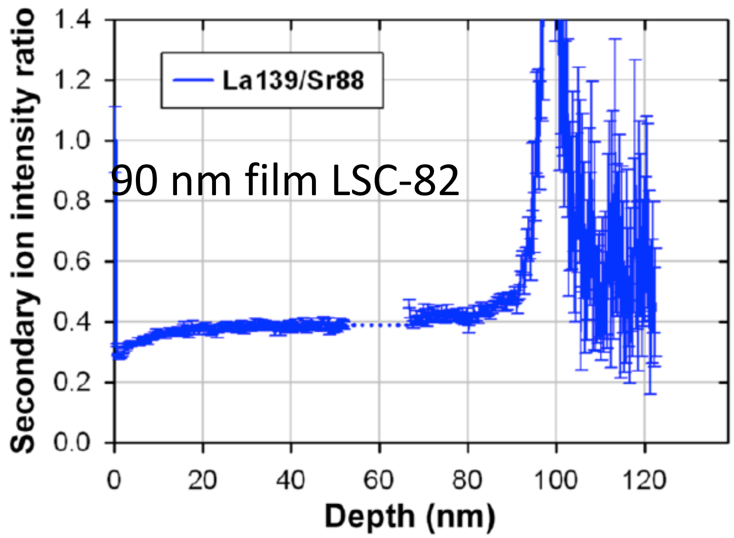
(1) = Thermodynamics of surface and the surface exchange reaction mechanism

(2) = Thermodynamics of bulk

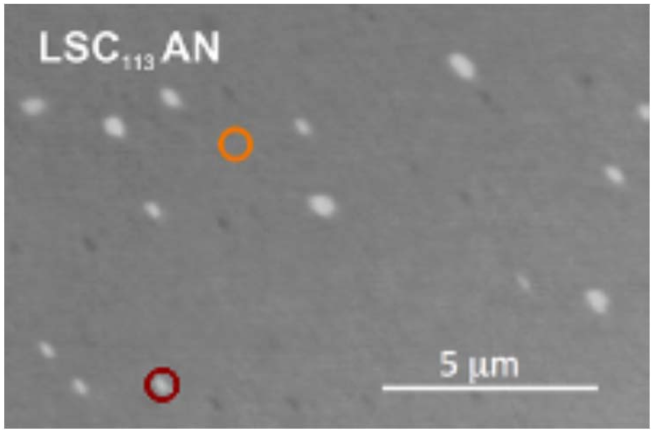
- Results completely inconsistent with bulk thermodynamic properties of LSC-82.
- Hard to rationalize based on ***any*** reasonable rate law and properties under the assumption that the film is single phase perovskite with uniform strontium content.

Example: $(La_{0.8}Sr_{0.2})CoO_3$ thin films

Films exhibit Sr stratification both perpendicular and lateral to interface.



SIMS depth profile on 90nm film
Richard Chater and John Kilner, Imperial College



Crumlin, et al. (MIT)
SEM

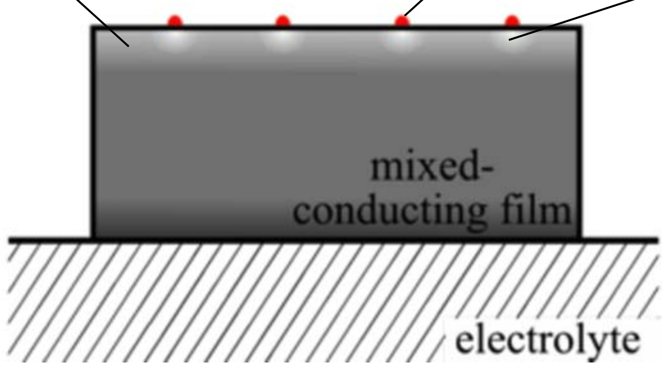
Example: $(La_{0.8}Sr_{0.2})CoO_3$ thin films

Revised model (*T.J. McDonald*):

general Sr enrichment at surface.

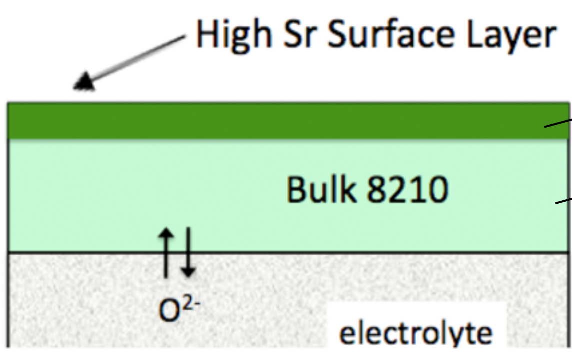
$x_s^{(2)}$

Sr-rich secondary phase(s) on surface



extra enrichment near precipitates

$x_s^{(1)}$



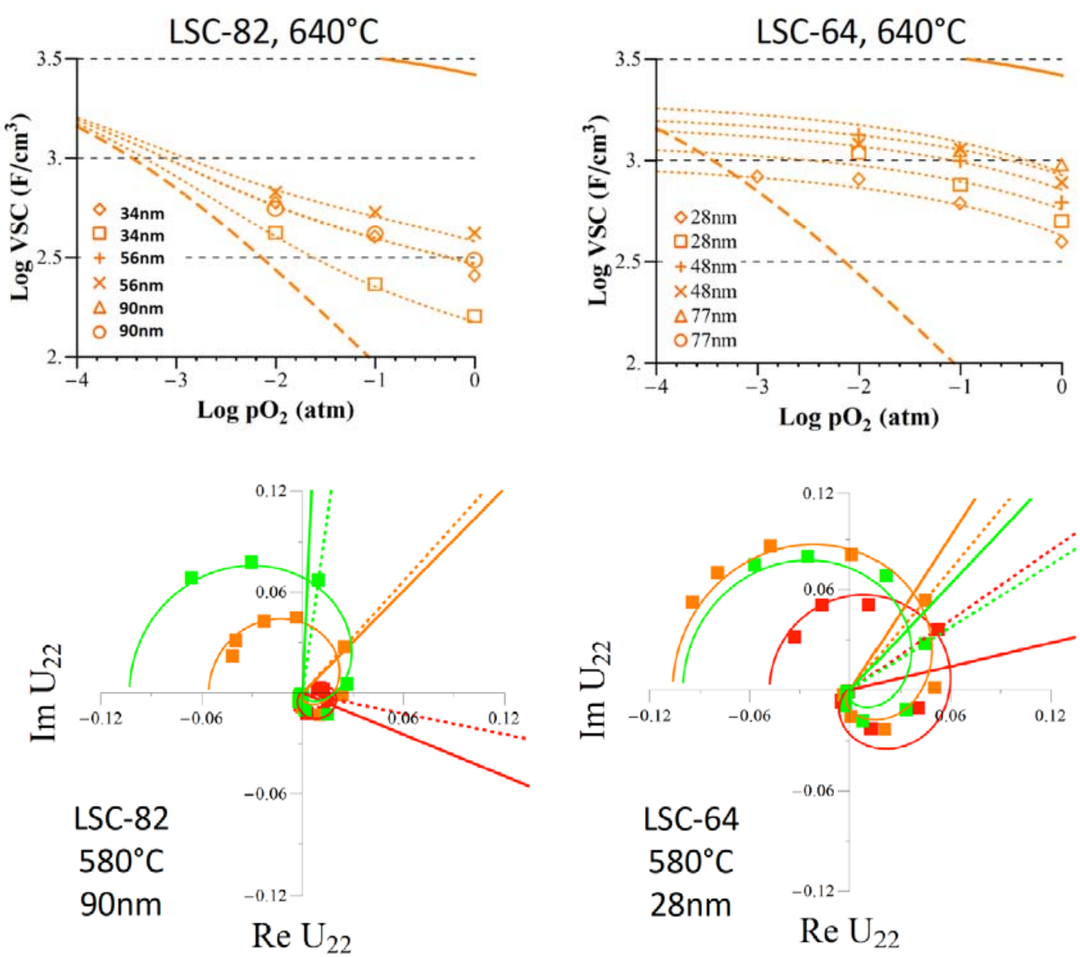
$$\left(L_{SL}\frac{d\delta_{SL}}{dt}+L_{bulk}\frac{d\delta_{bulk}}{dt}\right)\frac{c_0}{3} = -\frac{\tilde{i}\cos(\tilde{\omega}t)}{2F}$$

$$-2\gamma\Re_{01}\left[1-e^{\frac{-\Lambda}{\lambda RT}}\right]-2(1-\gamma)\Re_{02}\left[1-e^{\frac{-\Lambda}{\lambda RT}}\right]$$

Forward rate law $\Re_0 = k(T)p_{O_2}\delta^2$ depends on local vacancy defect concentration (δ) in the surface layer.

Example: $(La_{0.8}Sr_{0.2})CoO_3$ thin films

Dual Surface, Altered Bulk Model



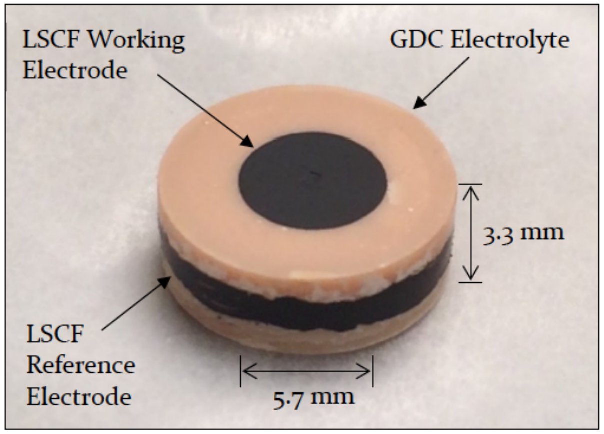
Conclusions

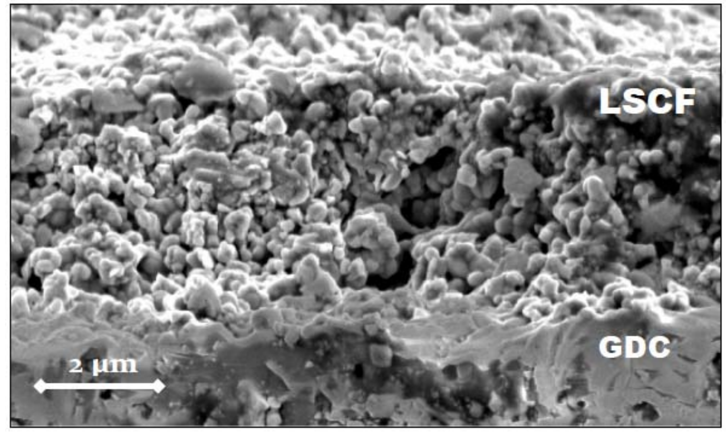
- Capacitance and harmonic response agree well.
- Implies Sr segregation is laterally inhomogeneous.
- O_2-active material for all films has properties of LSC (113) with $x_s^{(1)} \sim 0.45$.

Speculation

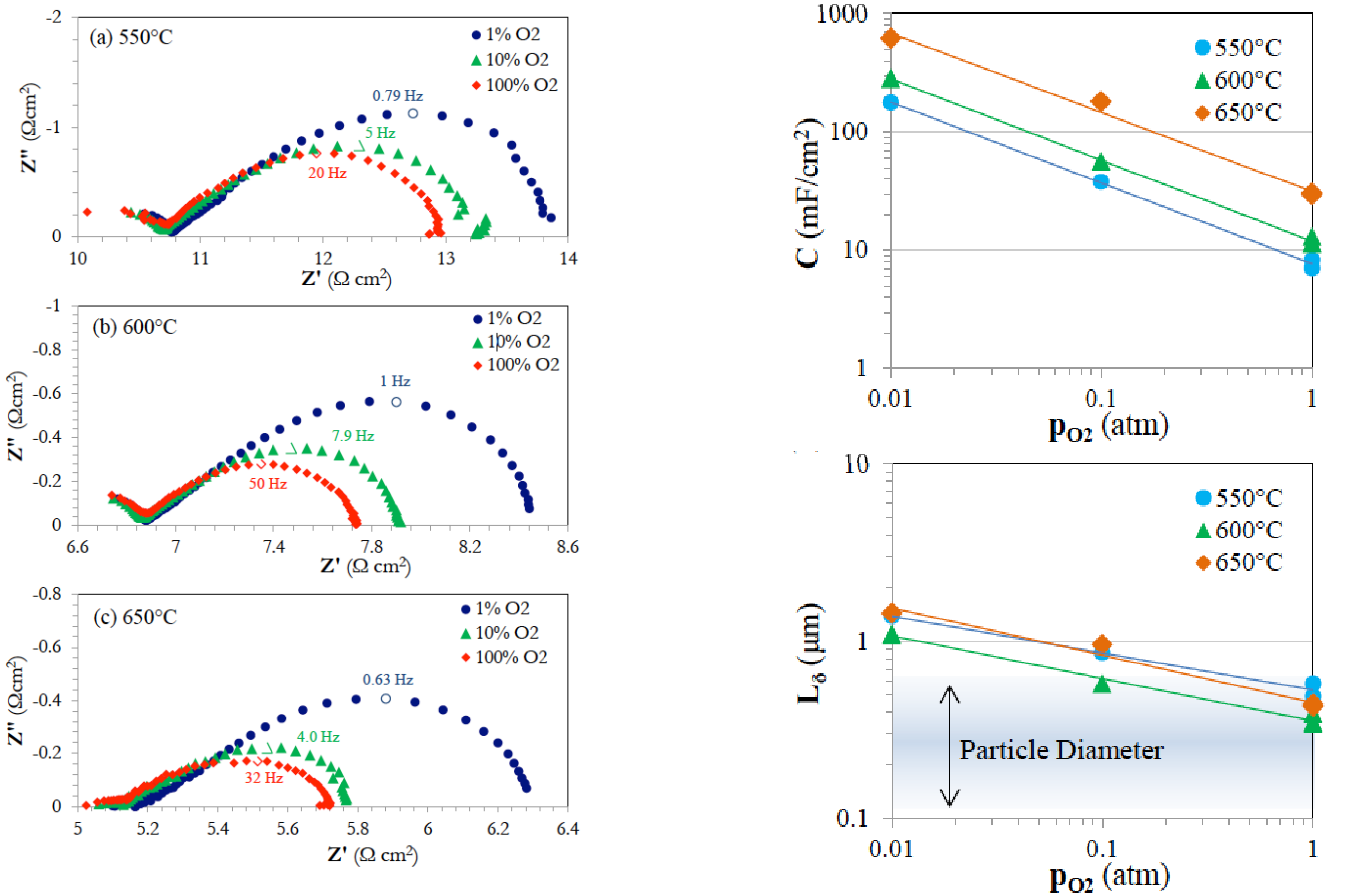
These films all show precipitation of secondary phases. Could the active material be associated with two-phase saturation/precipitation?

Porous LSCF


LSCF Working
Electrode
GDC Electrolyte
3.3 mm
LSCF
Reference
Electrode
5.7 mm

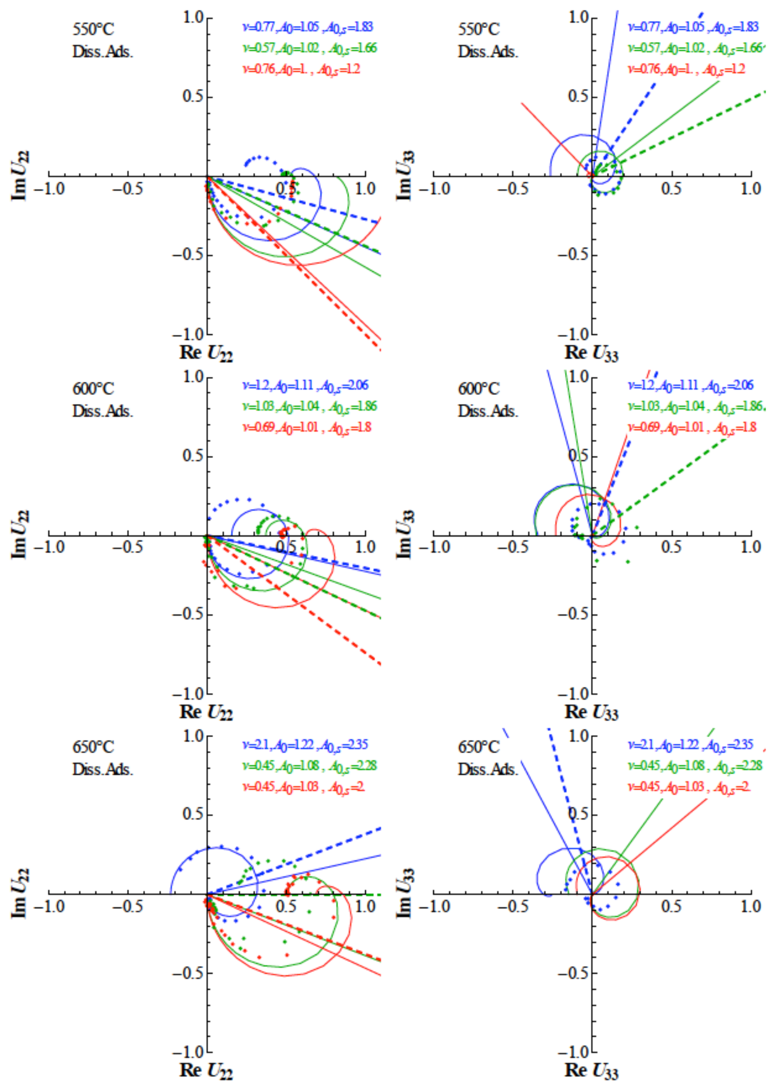

LSCF
GDC
2 μm

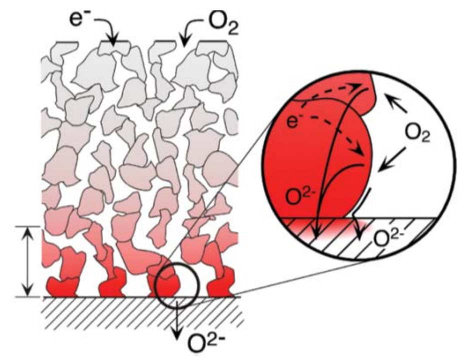
Porous LSCF - EIS



- Decreasing *C* with p_{O2} reflects loss of vacancies and shorter utilization length.
- Justifies use of 1-D macrohomogeneous model for EIS and NLEIS analysis.

Porous LSCF - NLEIS





- No models fit perfectly, suggesting inhomogeneous properties.
- Impossible explain results without increased reducibility of surface relative to bulk (may be due to Sr enrichment at surface)
- Transport rates too fast to be consistent with bulk diffusion alone – Implies significant surface diffusion.
- Kinetics appear to be 1st order in pO2, and somewhere between 1st and 2nd order in vacancy concentration.

Overall Conclusions

- LSC_{214} enhances $LSCF_{113}$ (~3x) far less than LSC_{113} (~100x)
- $LSCF_{113}$ has a more stable and Sr rich surface than LSC_{113}
  - Supported by aspects of AFM, Auger, DFT, NLEIS
- LSC_{214} changes Sr stability of LSC_{113} more than $LSCF_{113}$ and may enhance LSC_{113} performance by stabilization of Sr rich interface
  - Supported by AFM, Auger, COBRA, DFT

Future Work

- Investigate other 214 decoration candidates to achieve the enhanced surface activity (e.g. $(La,Sr)_2NiO_4$, $(La_{0.25}Sr_{0.75})CoO_4$)
- Investigate the short- and long-term degradation of $LSCF_{113}$ and $LSC_{214}/LSCF_{113}$ and relate to surface properties

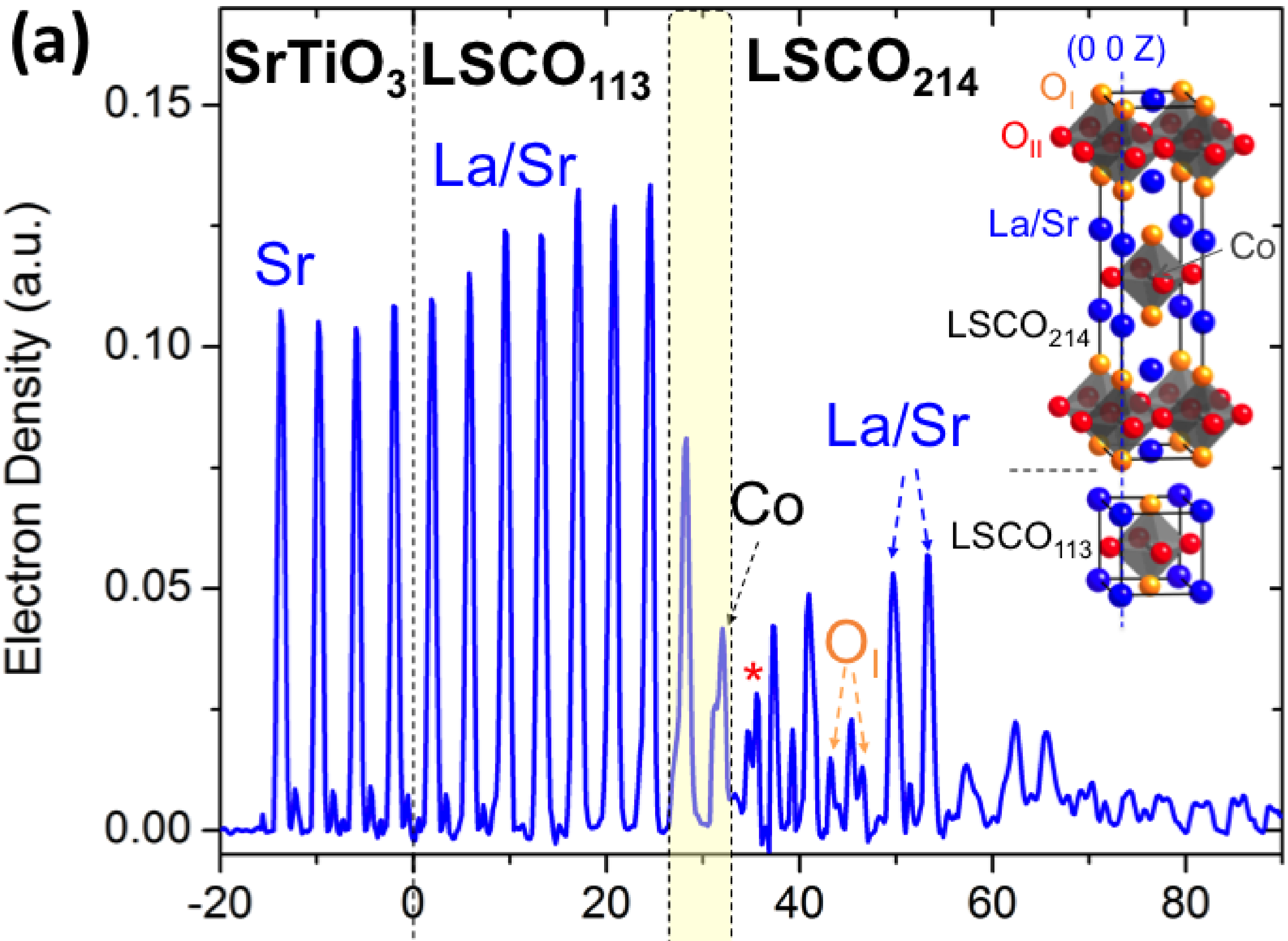
- Film growth + Physical characterization (MIT)
- Ab initio stability /reaction energies (Univ. Wisconsin)
- NLEIS + Modeling (Washington Univ.)

END

Thank you for your attention

Backup for Yang

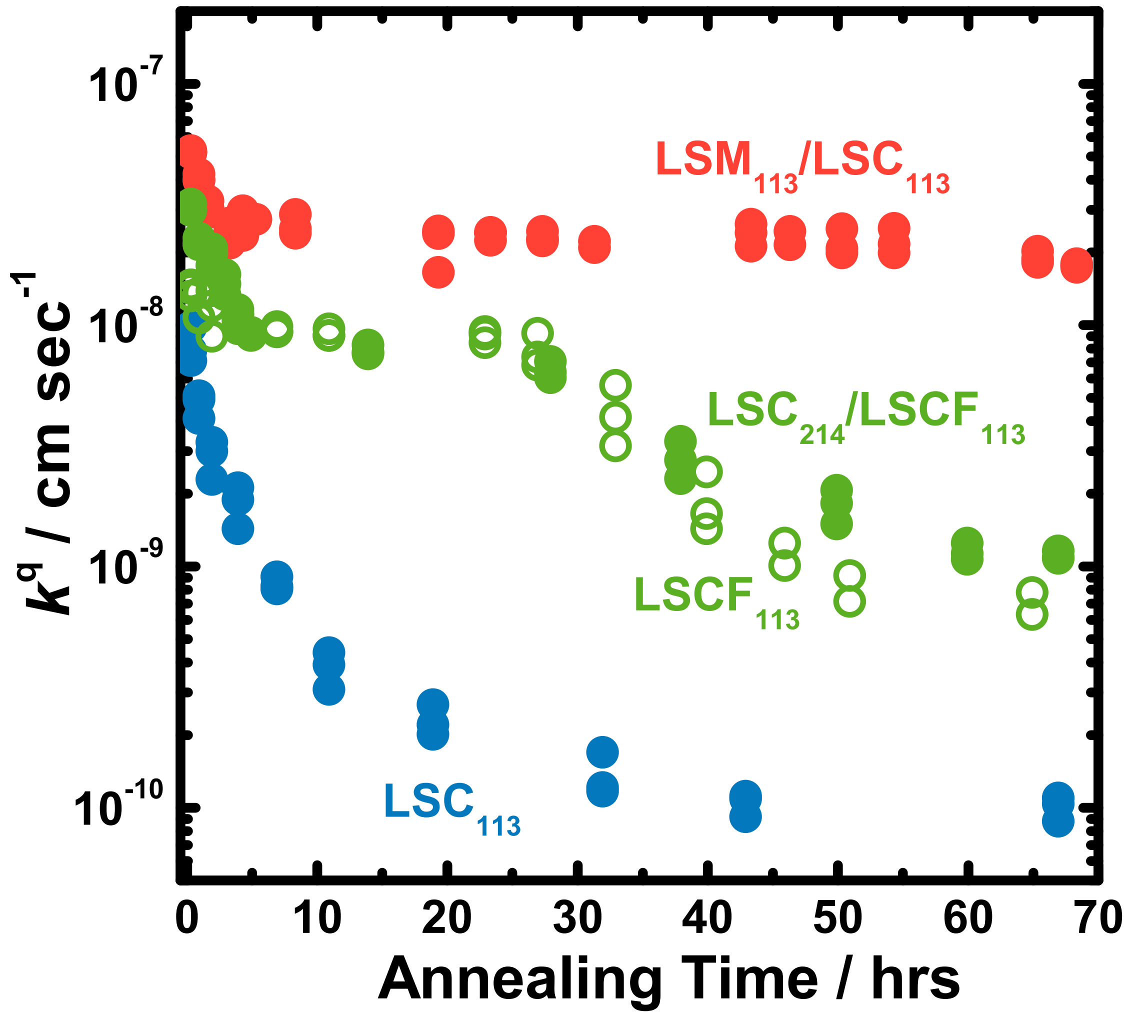
$La_{0.5}Sr_{0.5}Co_2O_4$ (LSC_{214}) decorated LSC_{113} on STO



Z. Feng, Y. Yacoby, W. T. Hong, et al. *JPCL* 2014

Understanding Oxide Surface Chemistry Critical to Activity and Stability

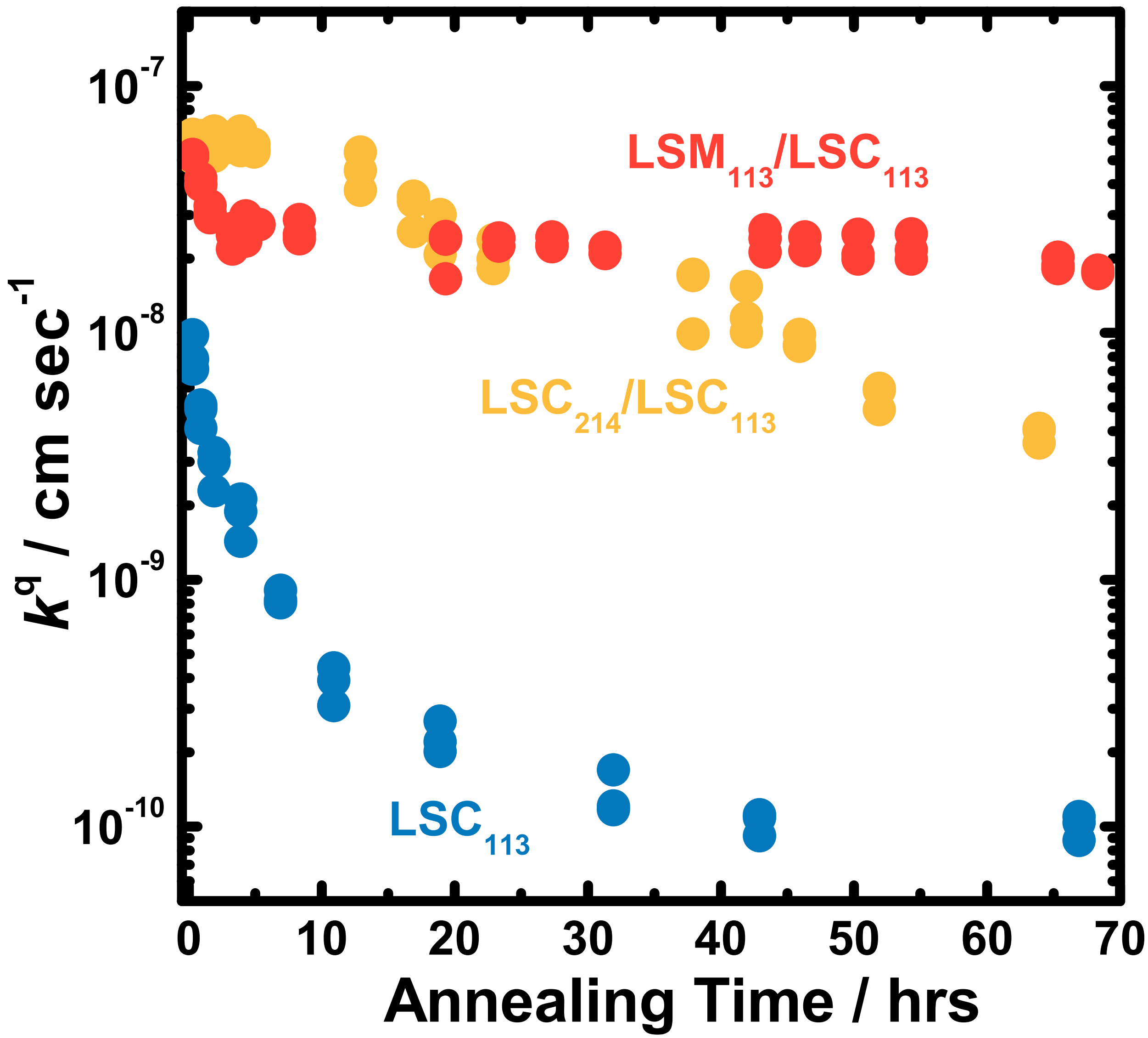
T=550°C
PO_2=1atm



D Lee et al., *JPCC submitted*

LSM Decoration Enhances Surface Stability

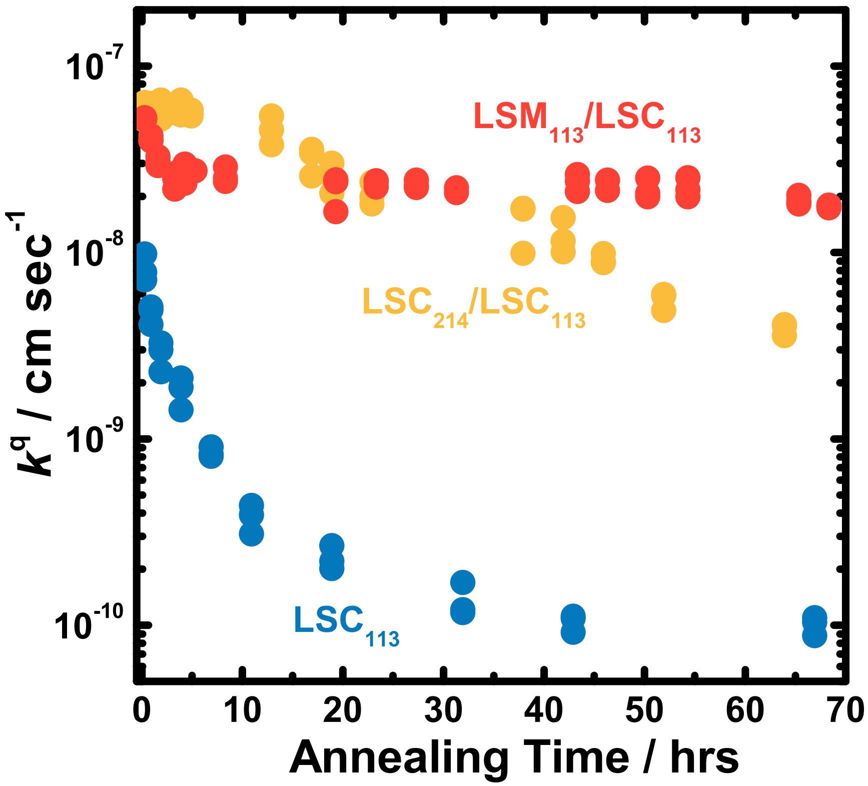
T=550°C
PO_2=1atm

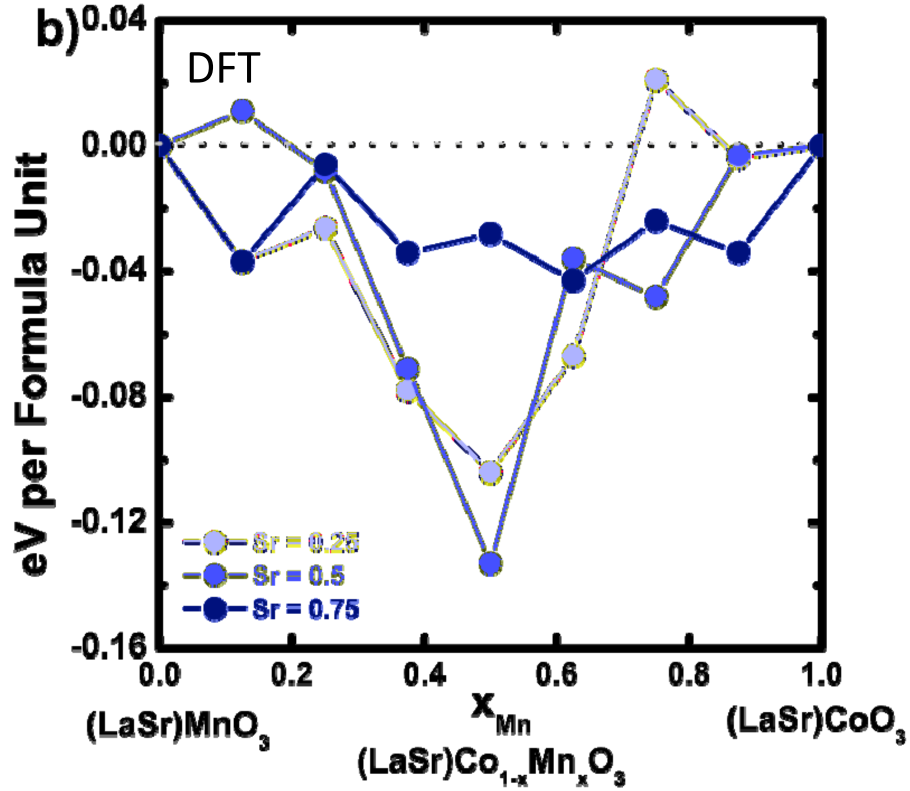


D Lee et al., *JPCC submitted*

LSM Decoration Enhances Surface Stability

T=550°C
PO_2=1atm

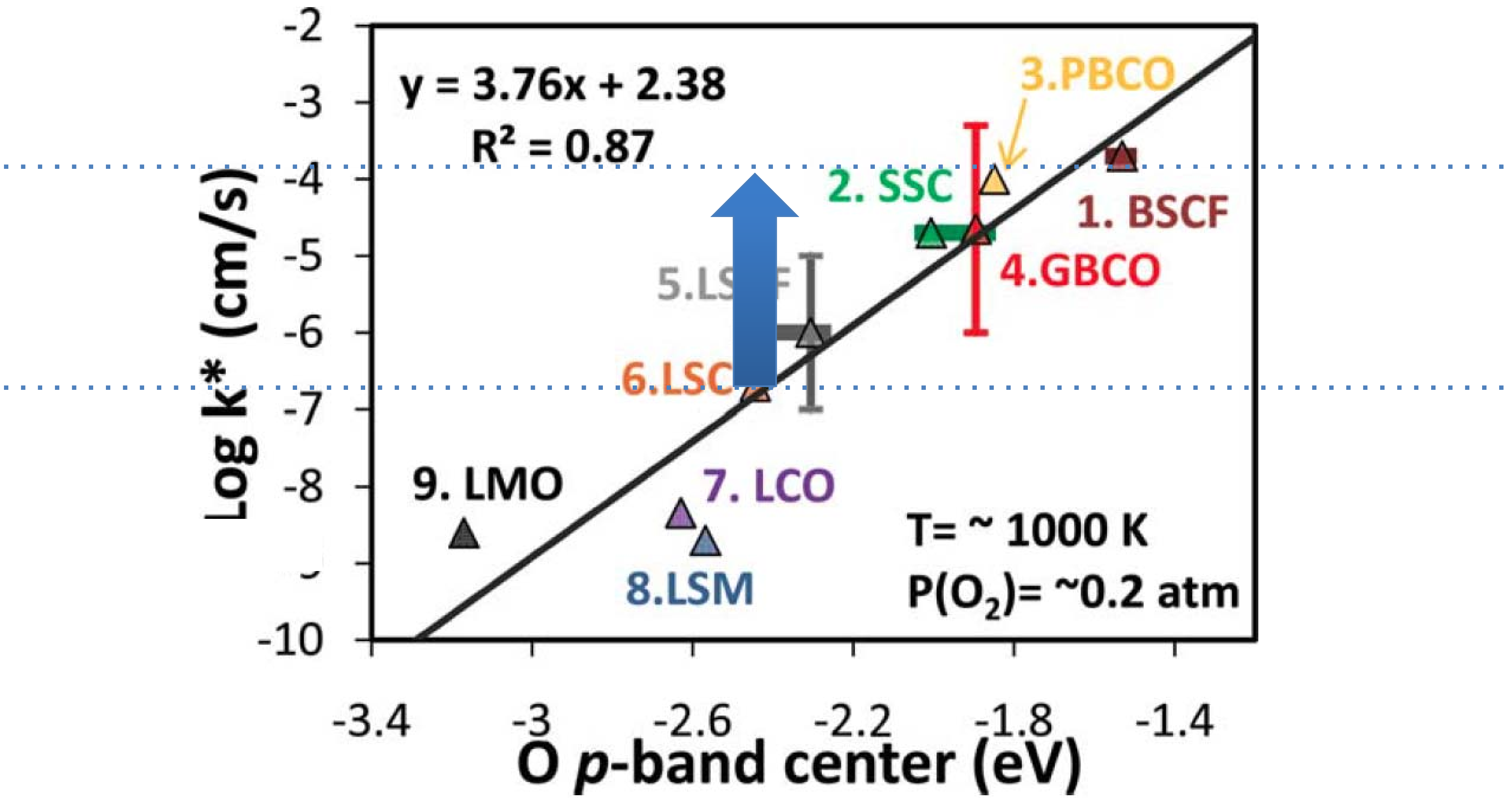




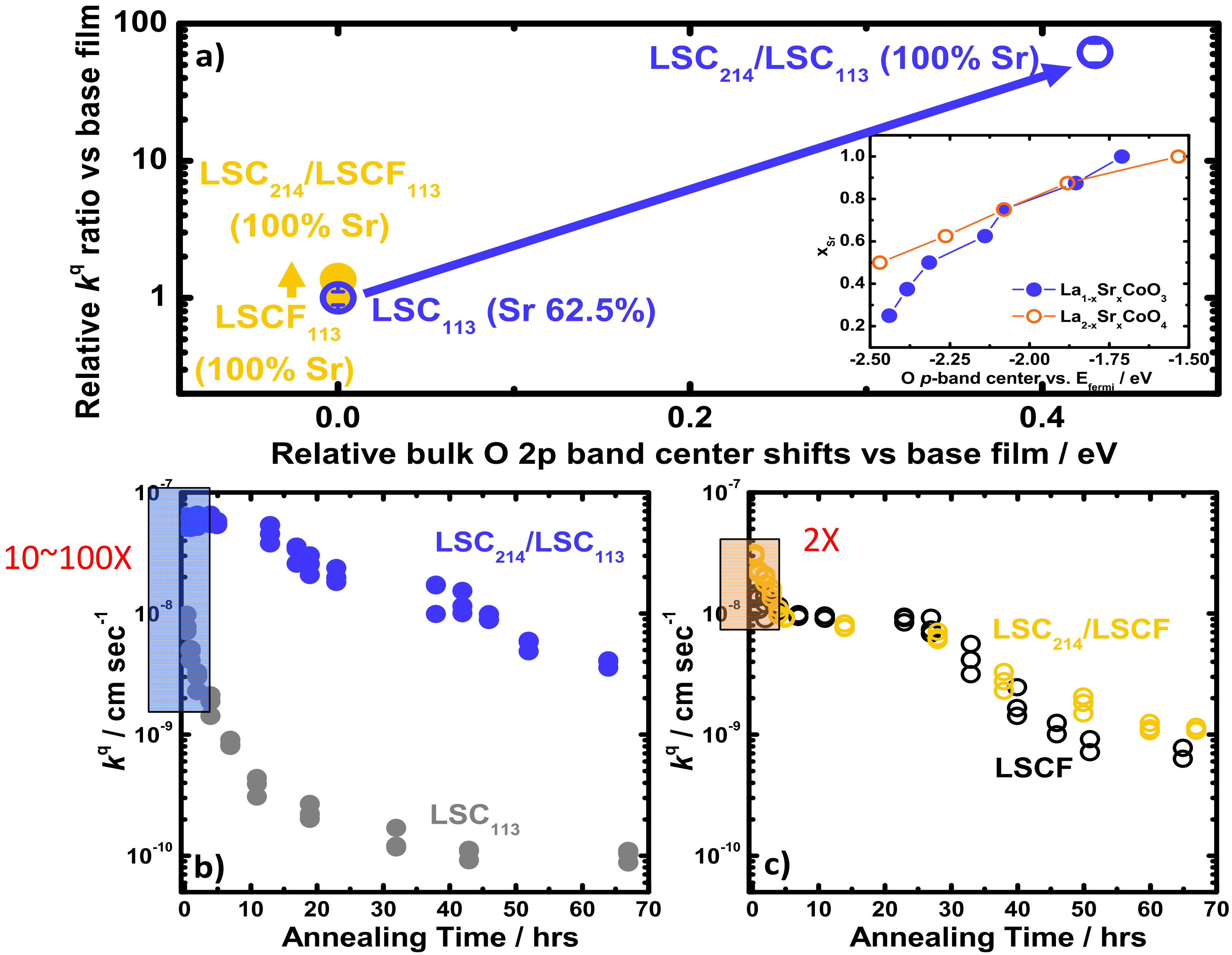
- Mn incorporation into in LSC may drive surface stabilization, enhancing activity and durability.
- Role of Sr unclear.

D Lee et al., *JPCC submitted*

O_2 electrocatalysis on perovskites at high temperatures



Lee, Rossmeisl, Shao-Horn, Morgan, EES 2011

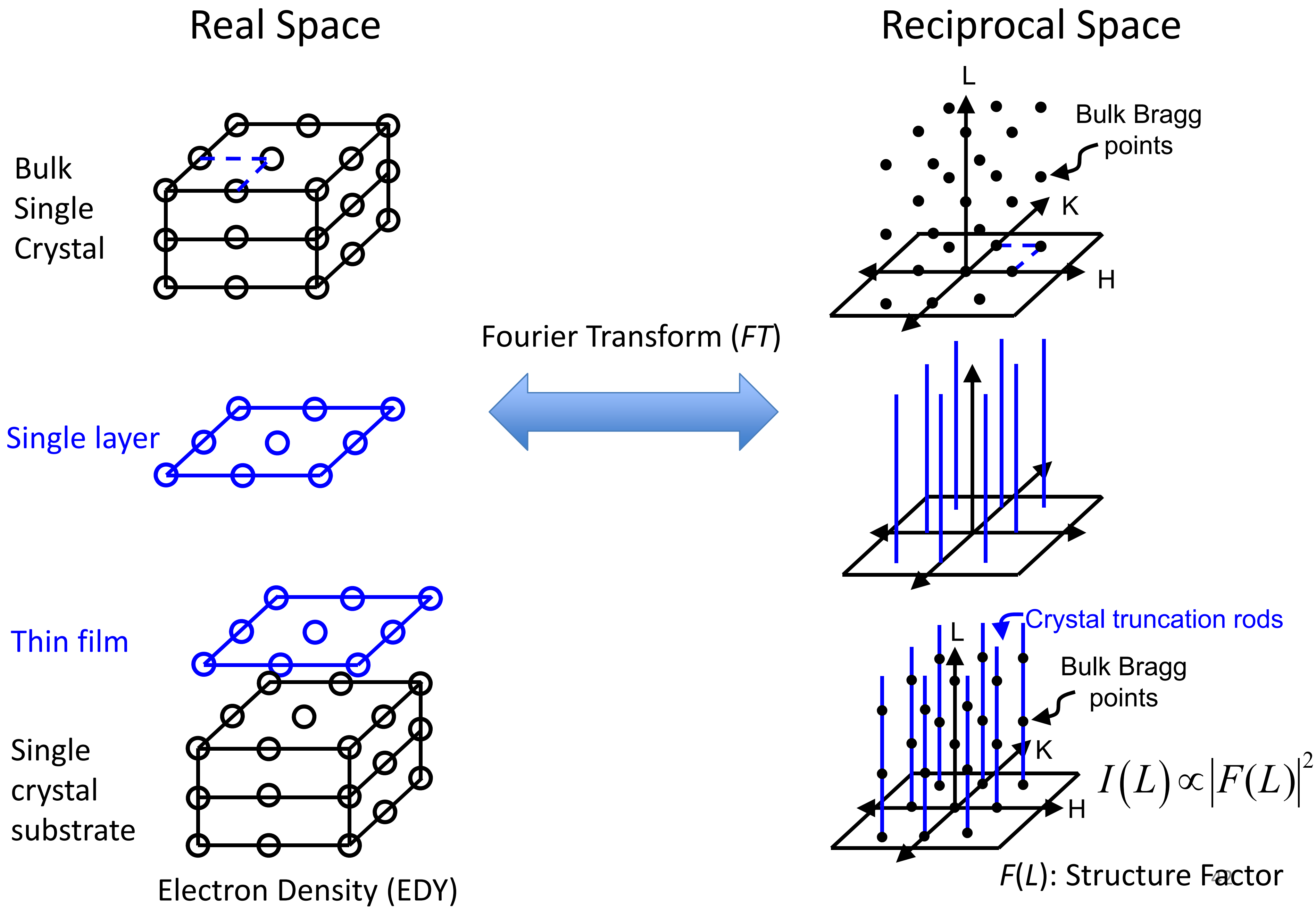


a)
LSC214/LSC113 (100% Sr)
LSC214/LSCF113
(100% Sr)
LSCF113
(100% Sr)
LSC113 (Sr 62.5%)
Relative k^q ratio vs base film
Relative bulk O 2p band center shifts vs base film / eV
La1-xSrxCoO3
La2-xSrxCoO4
xSr
O p-band center vs. Efermi / eV
10~100X
LSC214/LSC113
LSC113
b)
k^q / cm sec^-1
Annealing Time / hrs
2X
LSC214/LSCF
LSCF
c)
k^q / cm sec^-1
Annealing Time / hrs

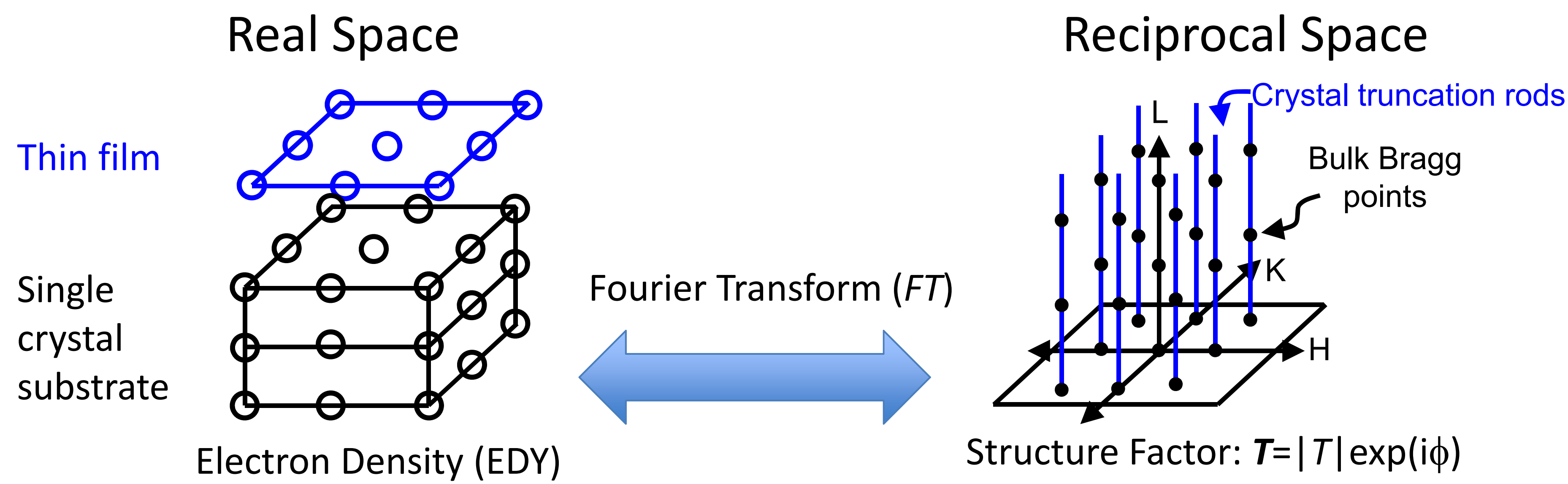
Outline

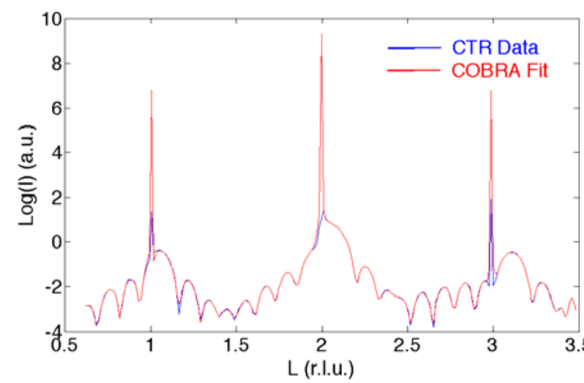
- *Layer-by-layer chemical distribution and oxygen disorder in oxides catalysts*

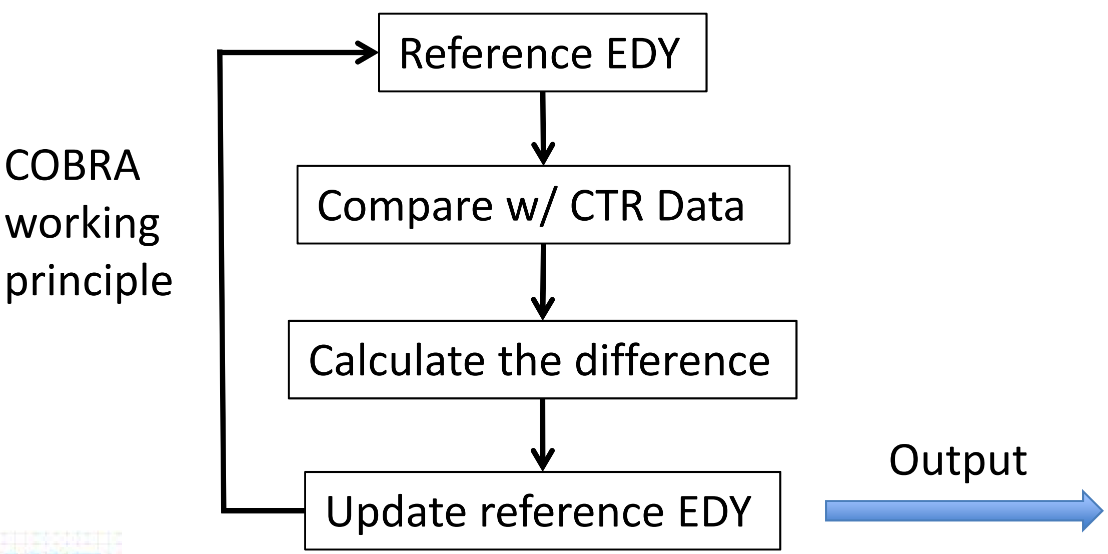
Crystal Truncation Rod (CTR)

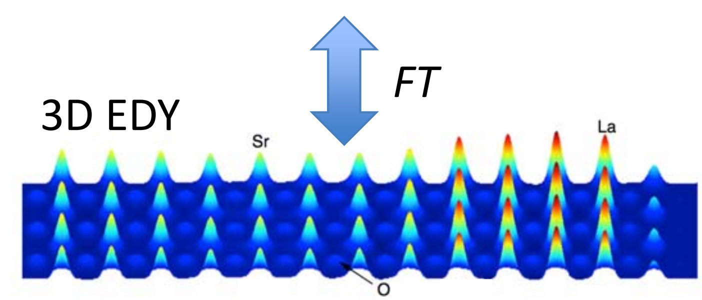


CTR and Coherent Bragg Rod Analysis (COBRA)





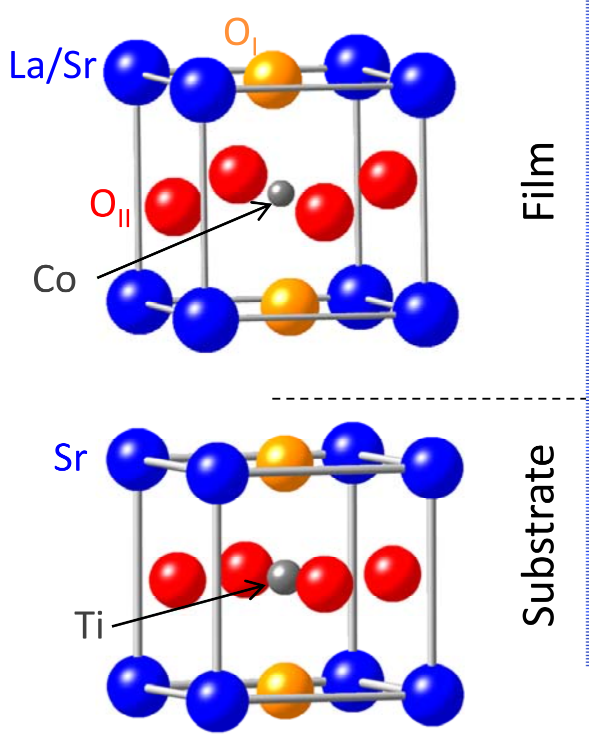


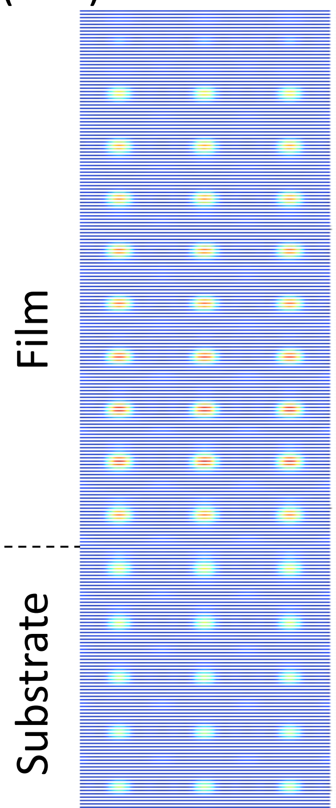


Z. Feng, et al., Energ. Environ. Sci., 2014, 7, 1166-1174

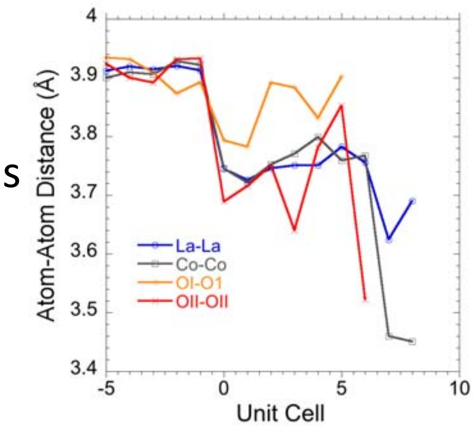
Information we can obtain

4 nm $La_{0.8}Sr_{0.2}CoO_{3-\delta}$/STO(001)

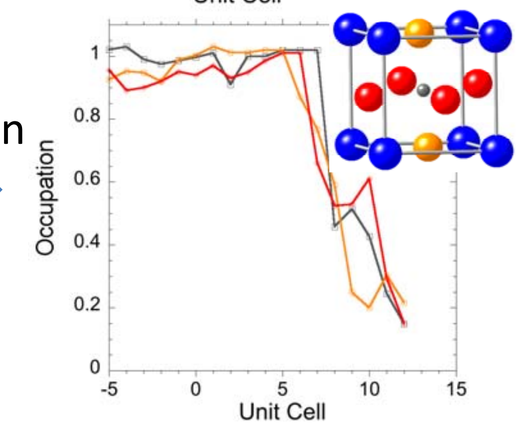




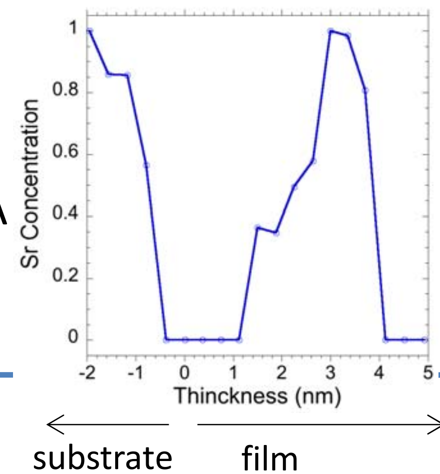
Atomic positions



Element occupation



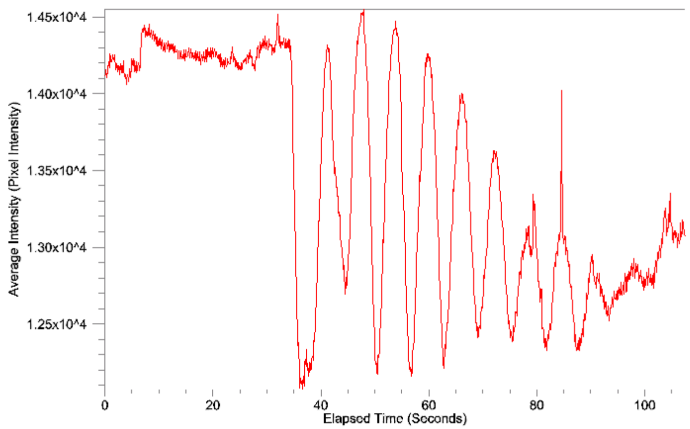
Differential COBRA
Sr depth profile



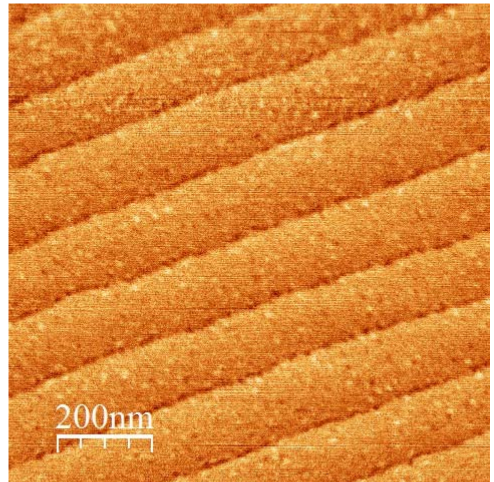
Z. Feng, et al., Energ. Environ. Sci., 2014, 7, 1166-1174

LSC$_{113}$ 8020 (4 nm) Model systems: layer-by-layer growth

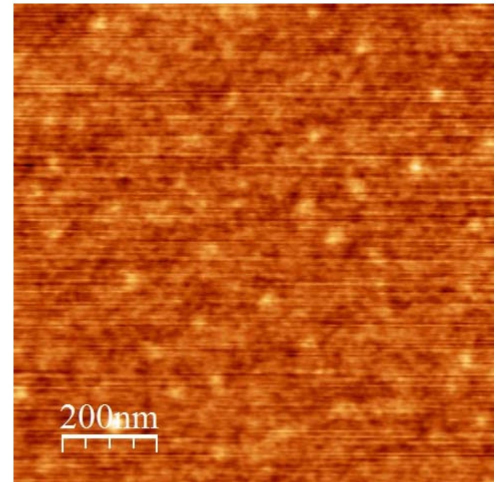
RHEED



As-deposited



Annealed

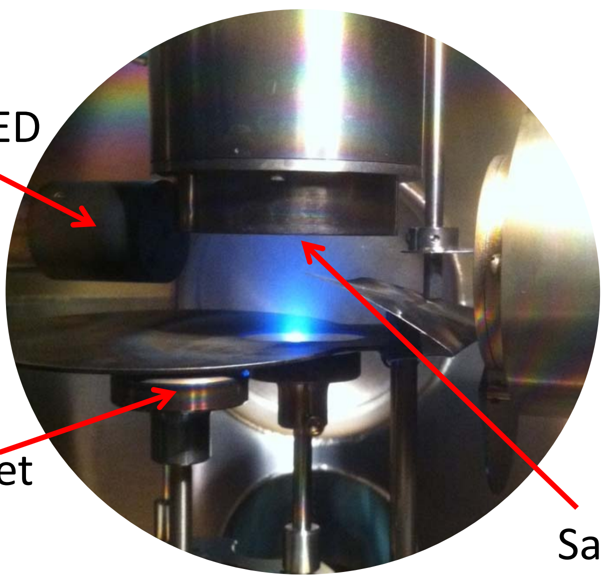


COBRA

COBRA

Compare

RHEED



Target

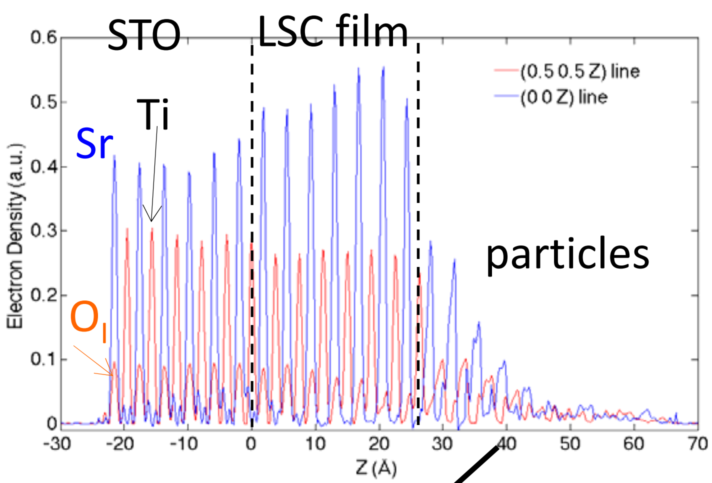
Sample

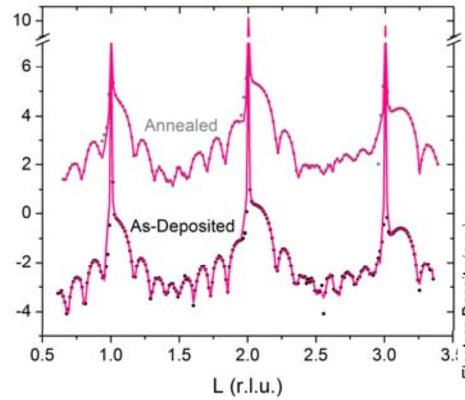
Annealing was performed at 550 °C with 400 Torr pure O_2 condition for 1 hour.

Z. Feng, et al., Energ. Environ. Sci., 2014, 7, 1166-1174

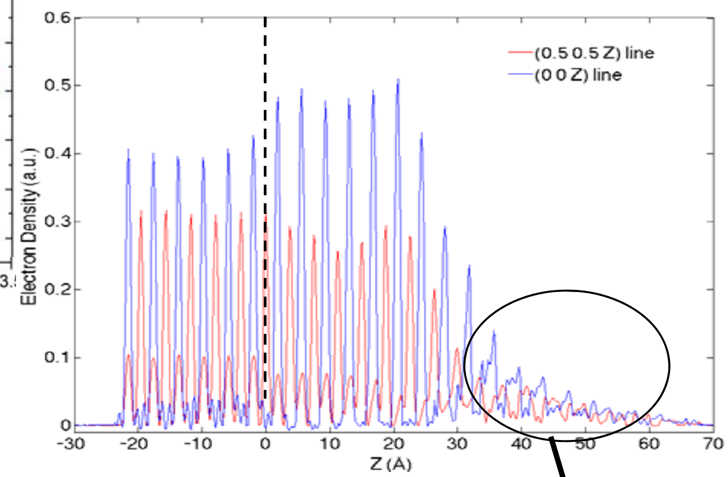
COBRA results

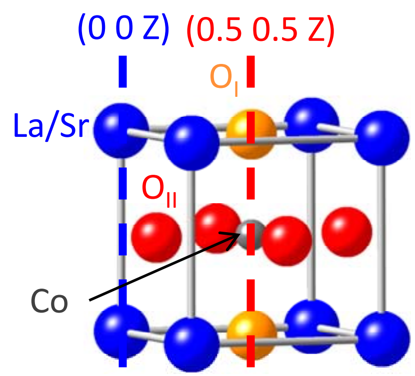
As-deposited

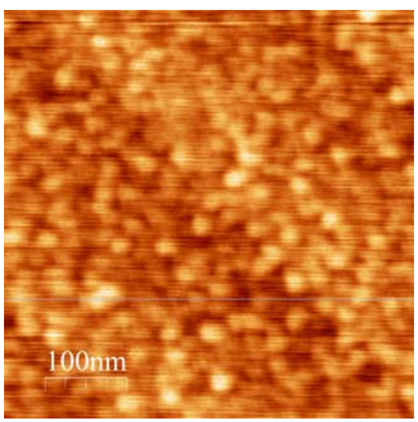


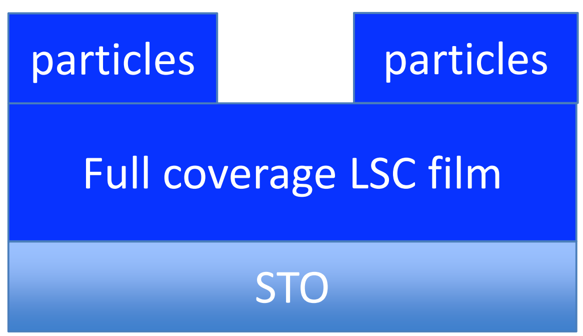


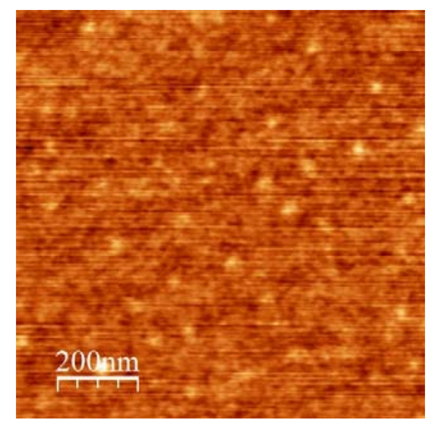
Annealed











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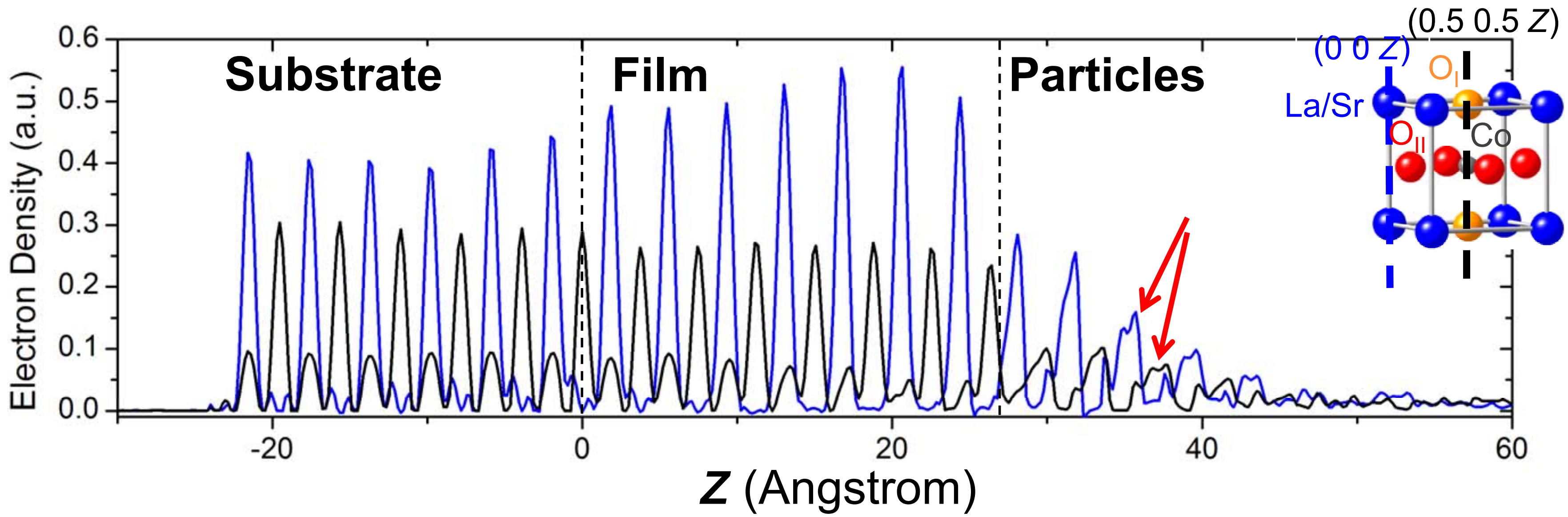
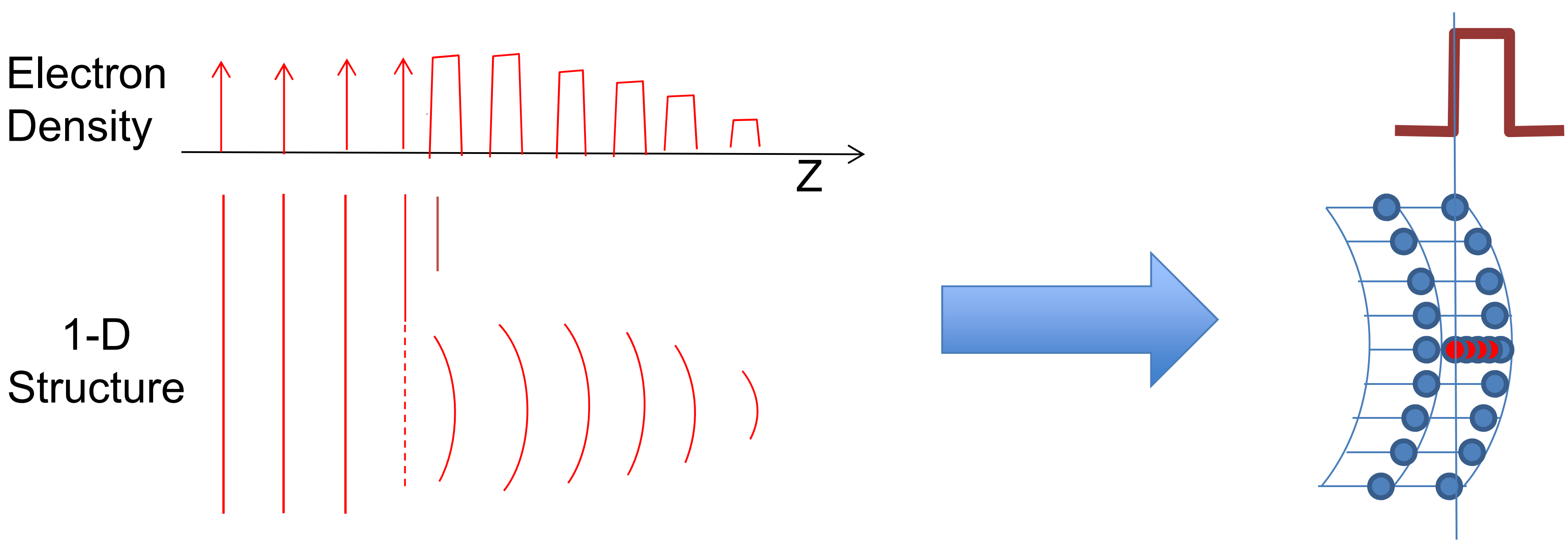
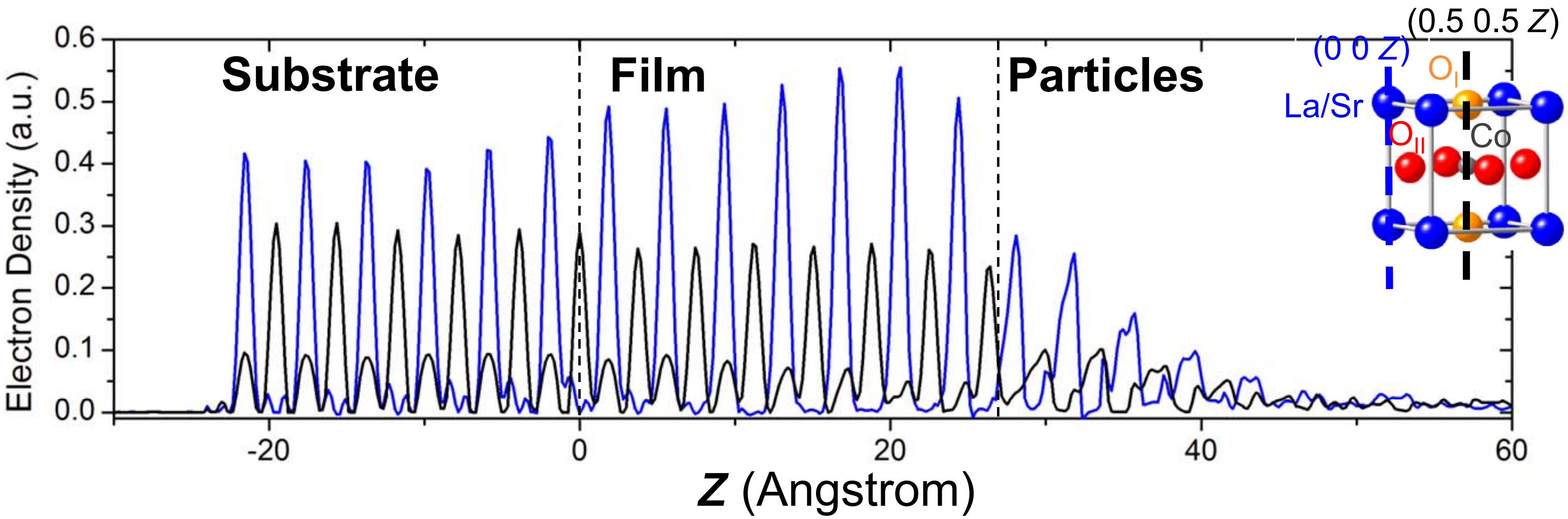
Surface Particle Structure

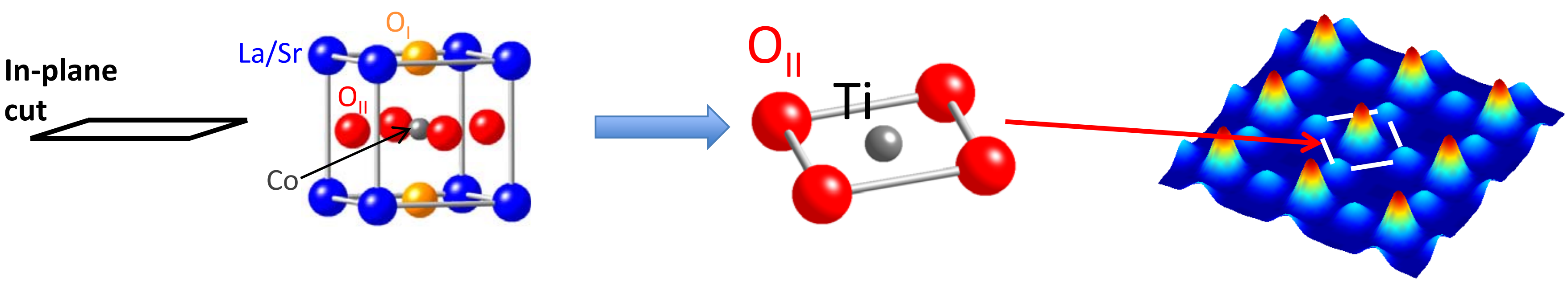




Z. Feng, et al., Energ. Environ. Sci., 2014, 7, 1166-1174

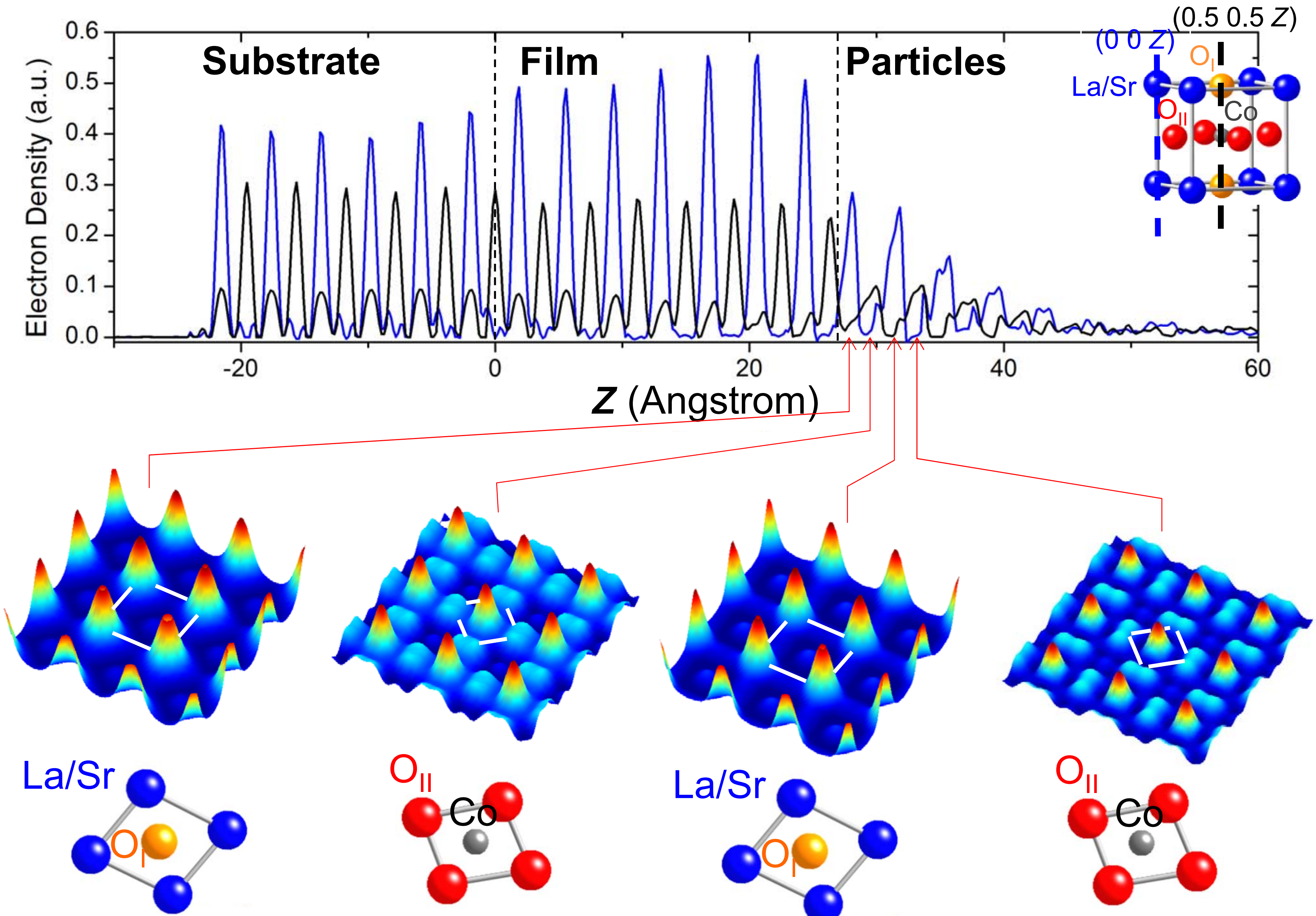
Surface Particle Structure



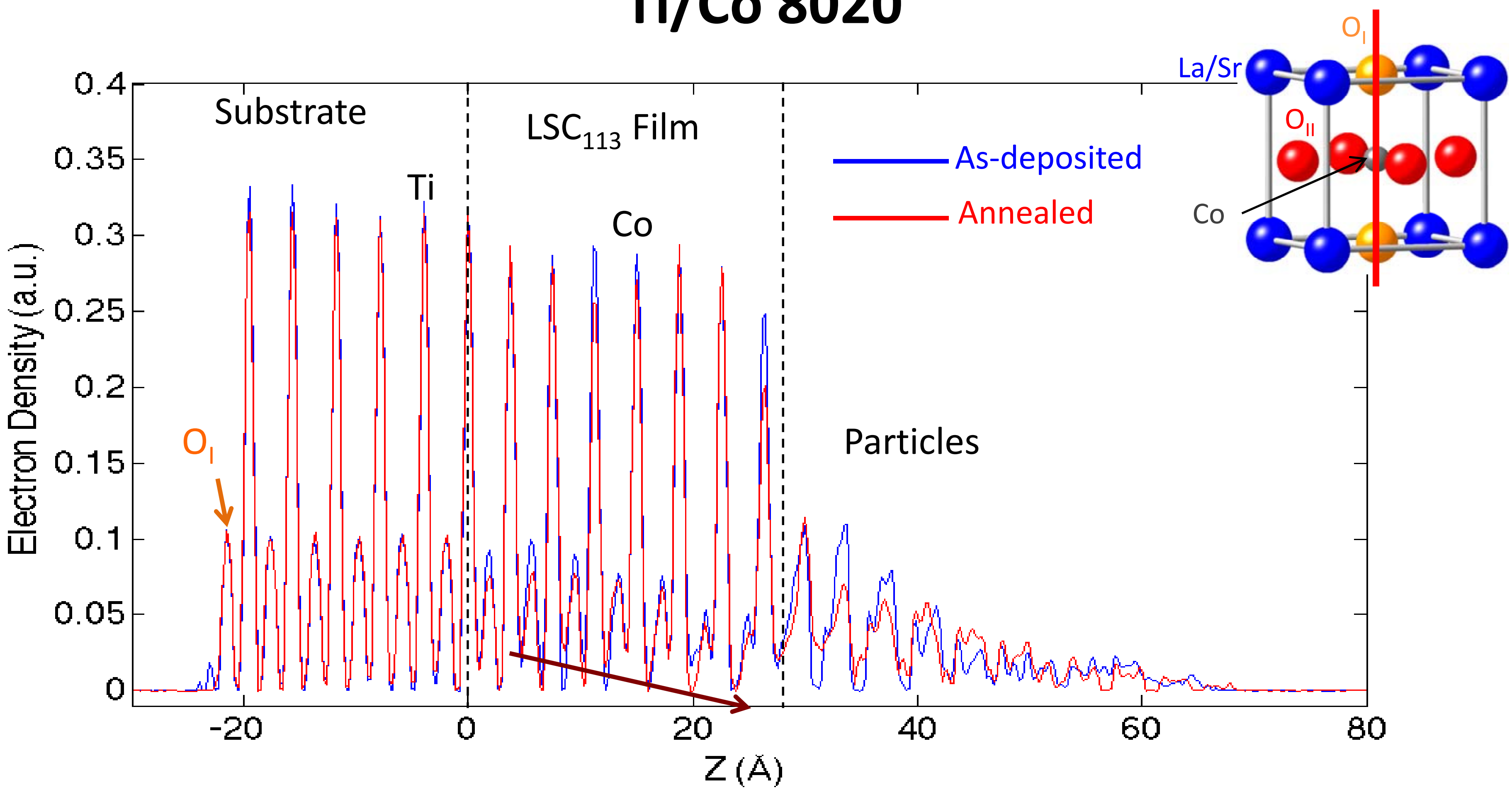


Z. Feng, et al., Energ. Environ. Sci., 2014, 7, 1166-1174

Surface Particle Structure

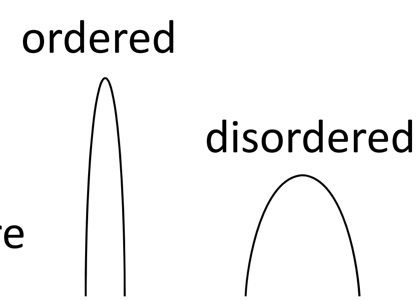


Ti/Co 8020



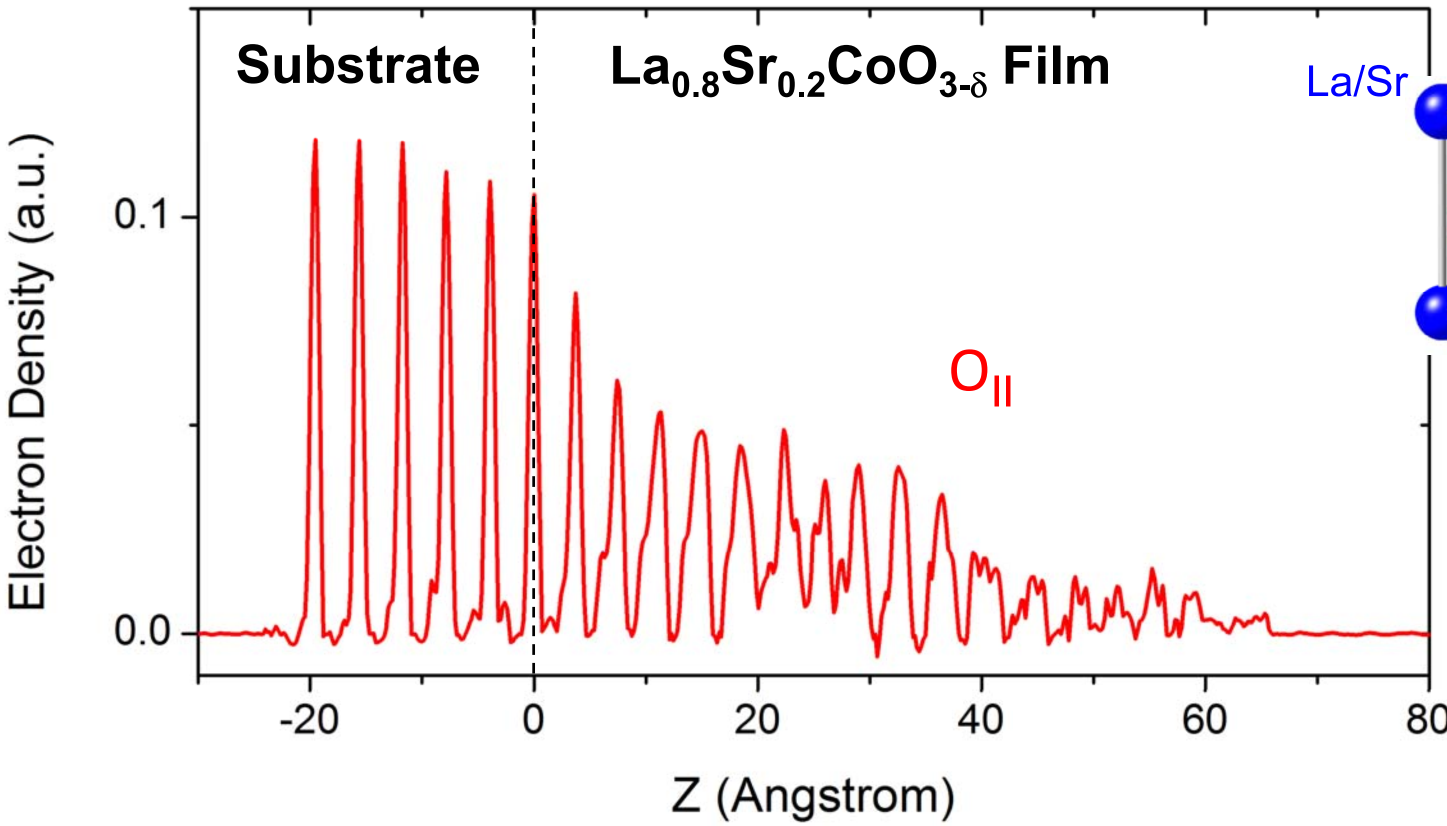
Apical oxygen (O$_I$) peaks in film becomes broader and broader

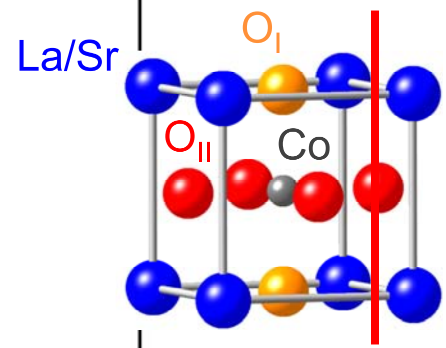
position less coherent to FCC ideal structure

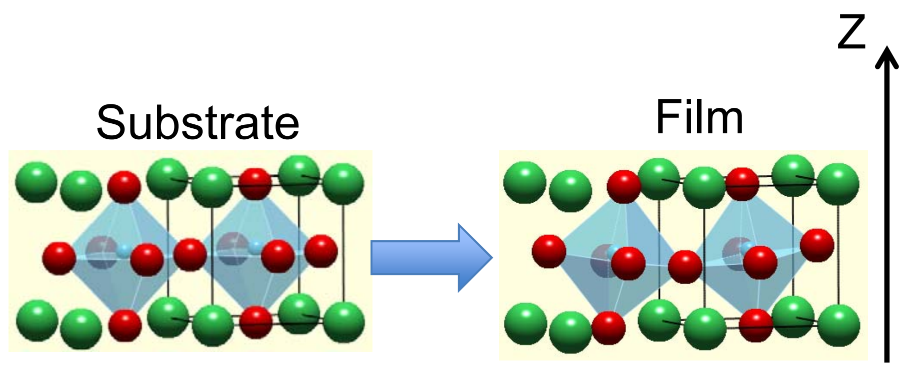


Z. Feng, et al., Energ. Environ. Sci., 2014, 7, 1166-1174

Oxygen Order-Disorder Transition





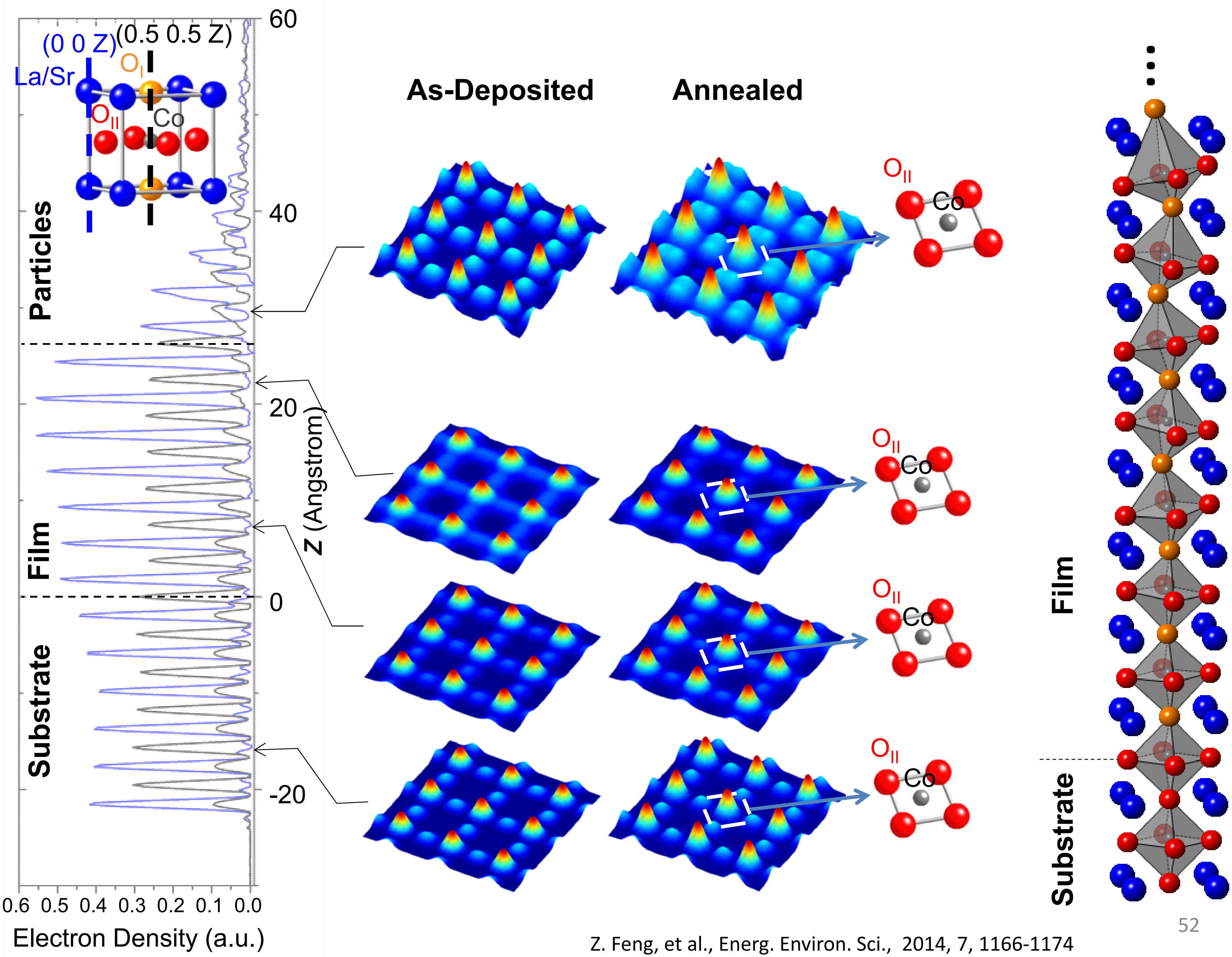


Sharp change between substrate and film

⇓

stronger octahedral distortion

Z. Feng, et al., Energ. Environ. Sci., 2014, 7, 1166-1174



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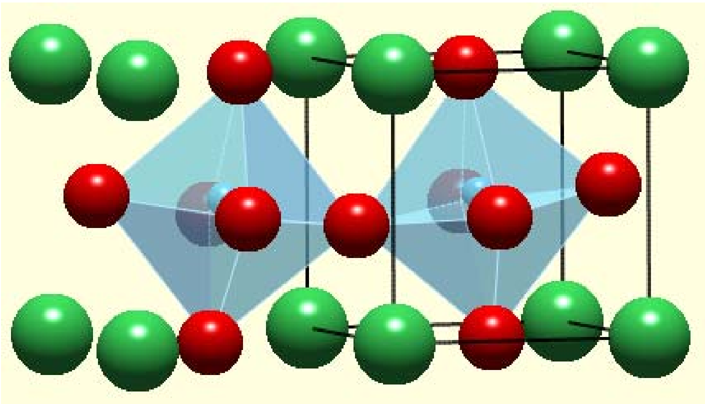
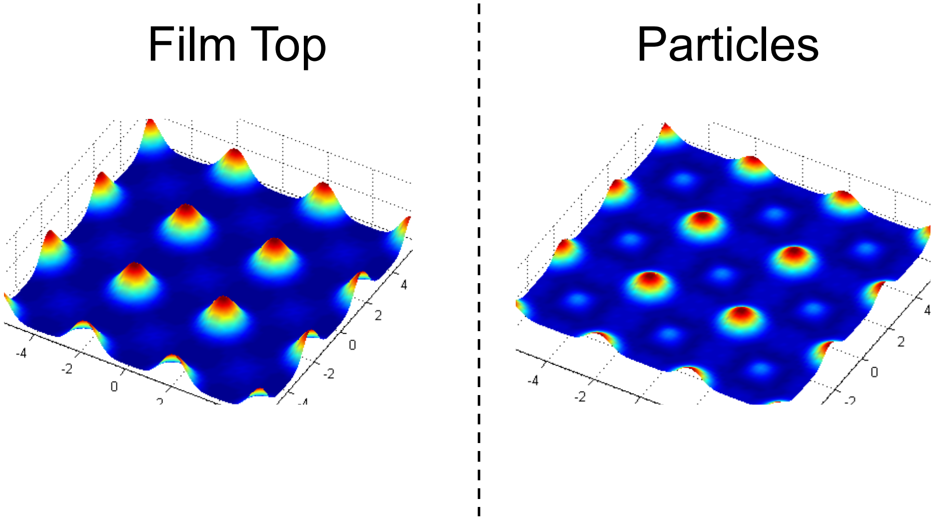
Connections to oxygen electrocatalysis

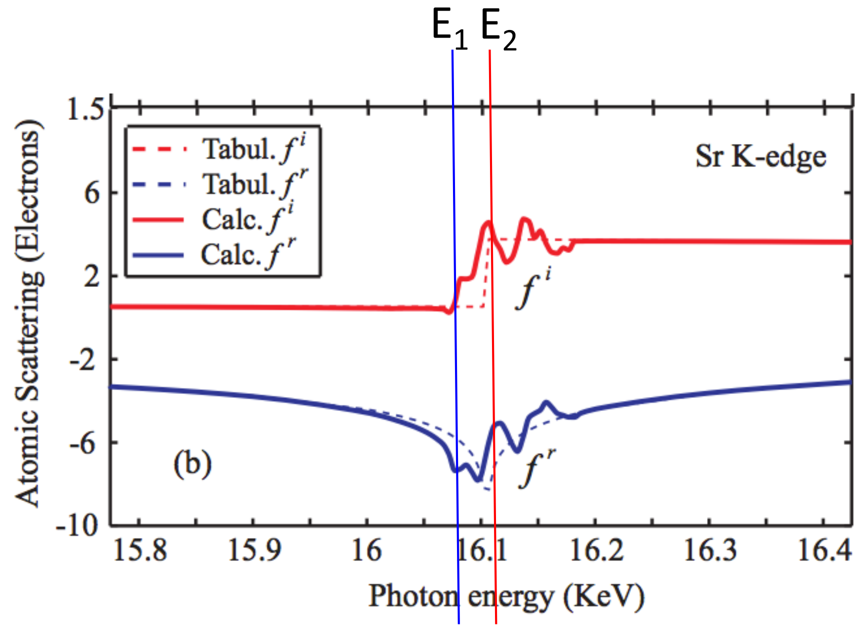
- Oxygen become more and more disordered → stronger octahedral distortion
- Order—Disorder –Order transition→ interface is important/active for incorporating and diffusing oxygen → high ORR activity



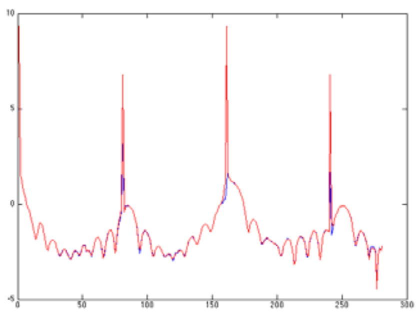


Differential COBRA

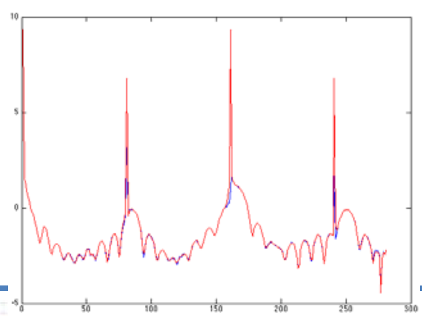
Energy dependent Atomic scattering factor



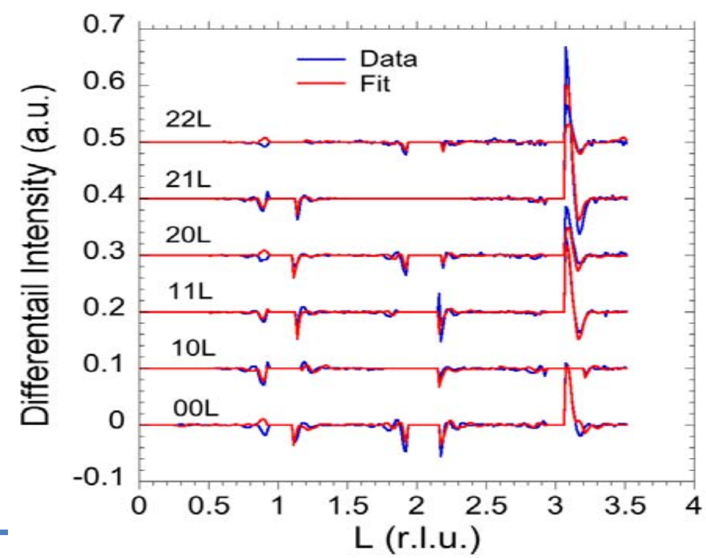
E_1



E_2

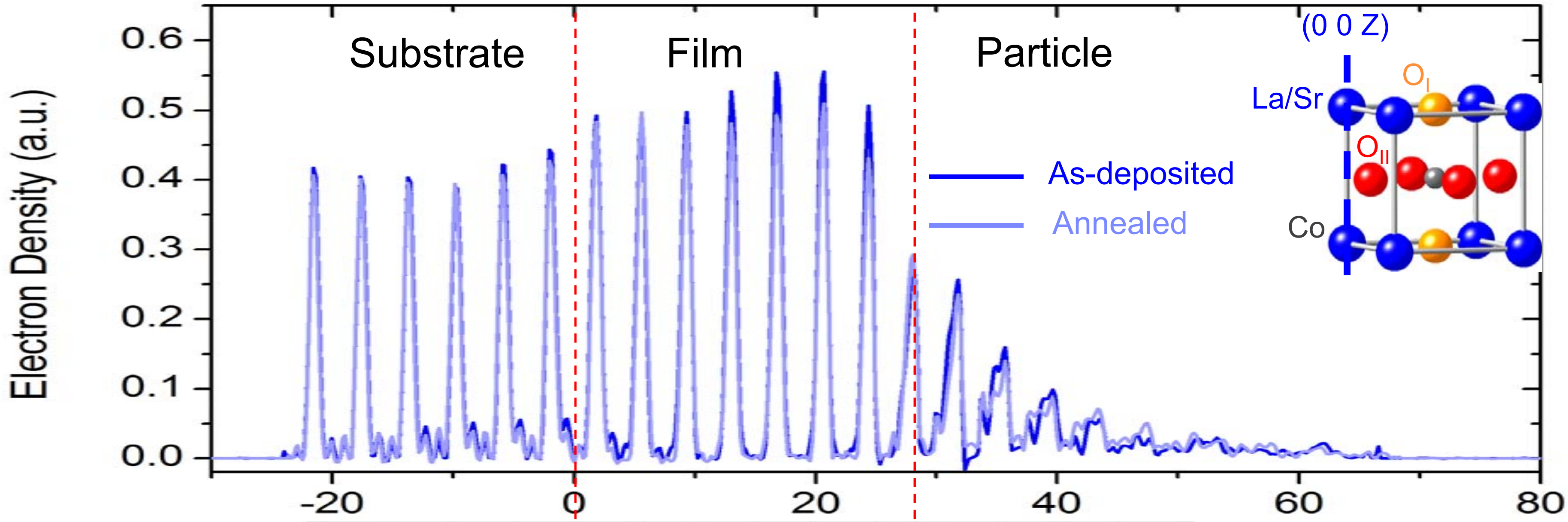


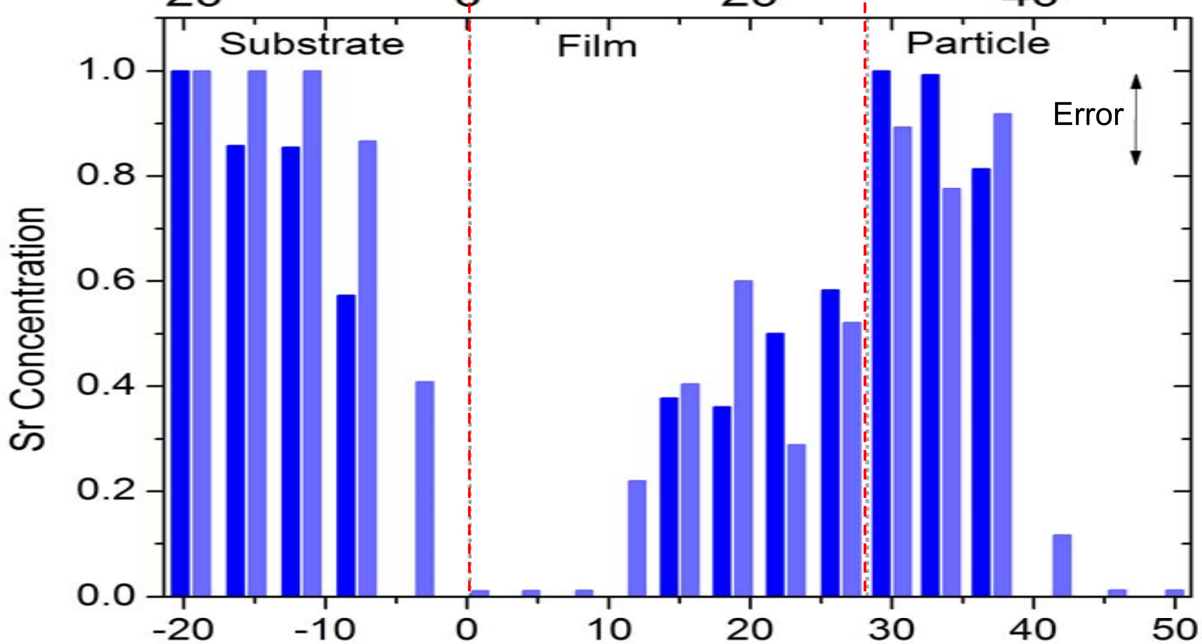
Difference



Z. Feng, et al., Energ. Environ. Sci., 2014, 7, 1166-1174

Sr depth-dependent distribution, 1st Experimental Evidence!





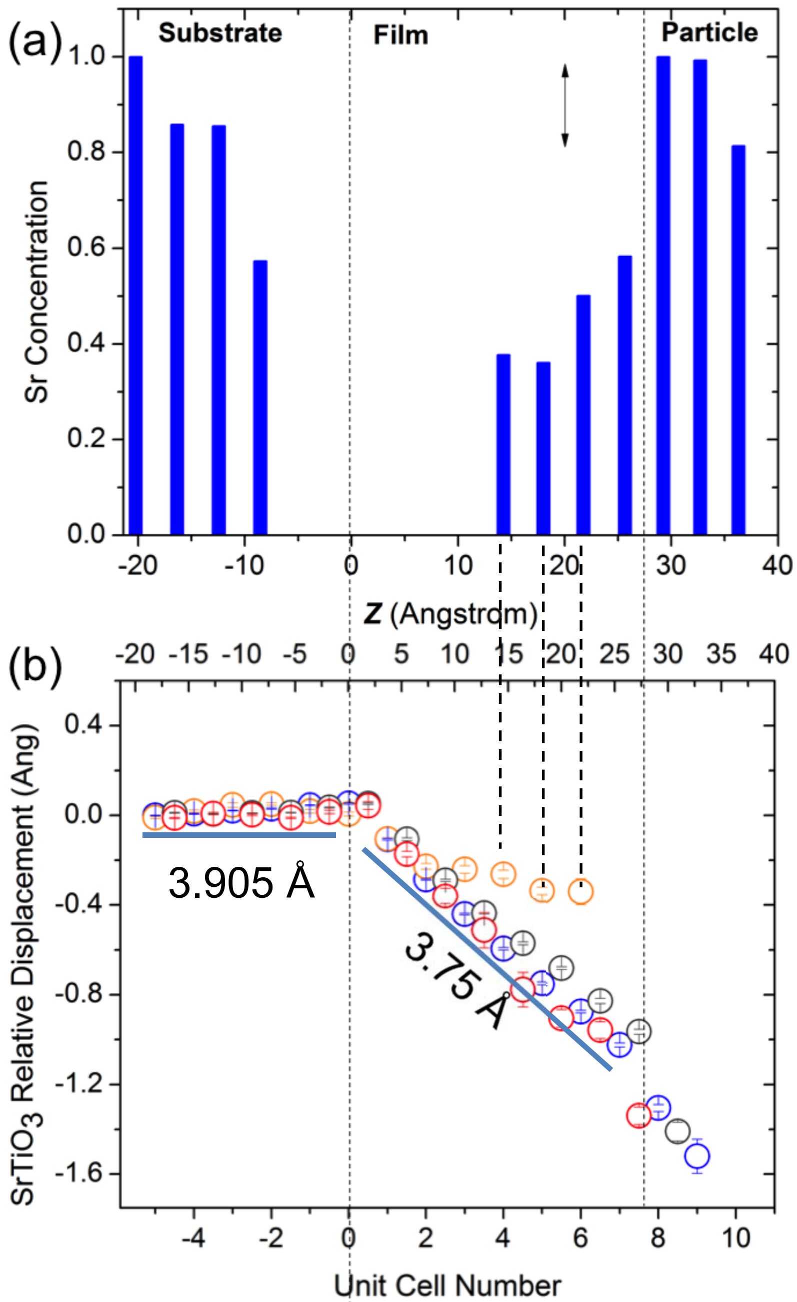
Schneider et al, *APL*, 2010

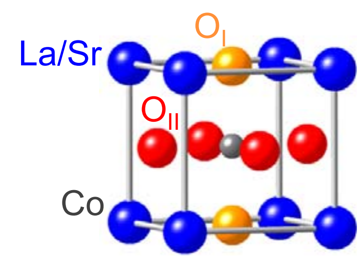
Z (Angstrom)

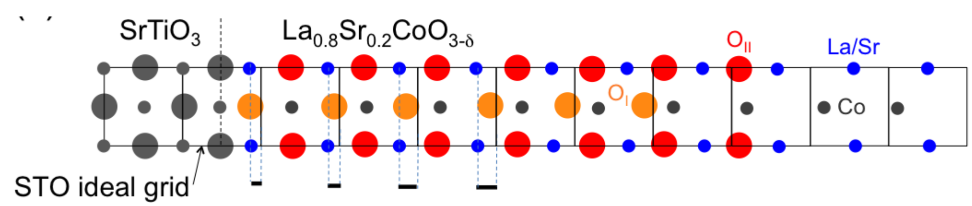
Z. Feng, et al., Energ. Environ. Sci., 2014, 7, 1166-1174

- Strong depletion of Sr at film/substrate interface→ substrate oxygen source
- Particles are Sr rich
- Substrate as O source

Apical Oxygen Displacement



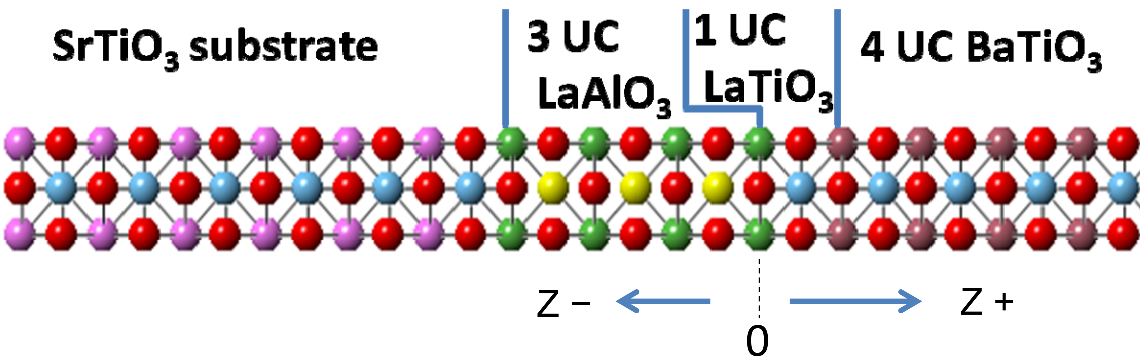


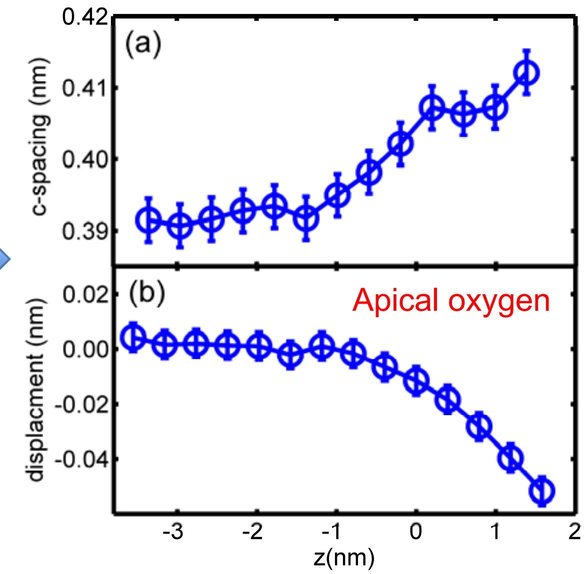


Z. Feng, et al., Energ. Environ. Sci., 2014, 7, 1166-1174

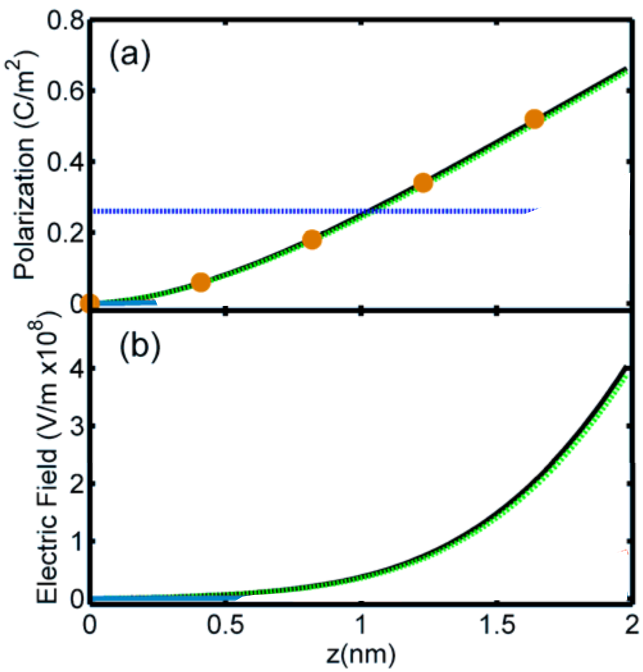
Sr Inhomogeneity and Apical Oxygen Displacement

Kumah et. al., APL Materials, 2013, 1, 62107

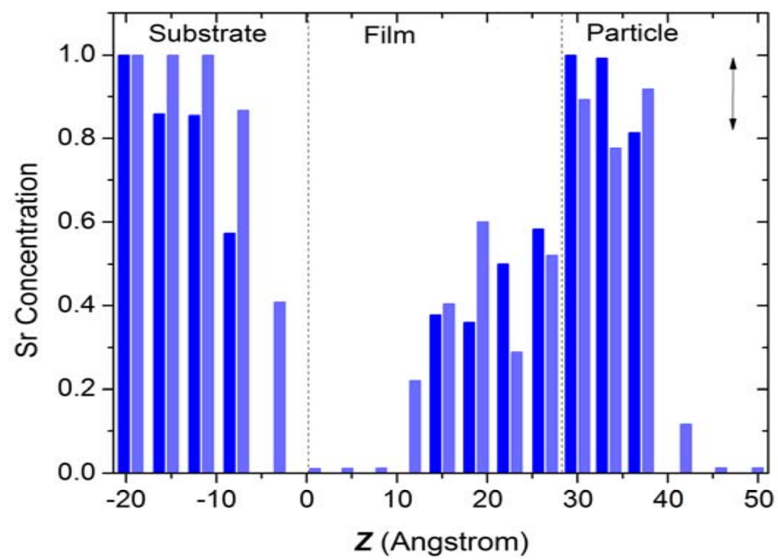




La^{3+} replace Ba^{2+} → electrical field



Sr^{2+} replace La^{3+}



DFT: Sr rich coupled w/ oxygen vacancies

Z. Feng, et al., Energ. Environ. Sci., 2014, 7, 1166-1174

Summary: LSC_{113}/STO Model System

- **Atomic Structure:**

Oxygen order—disorder—order transition → Octahedral distortion/rotation and active interface for ORR

Apical oxygen displacement → Electric fields (intermixing)

- **Chemistry:**

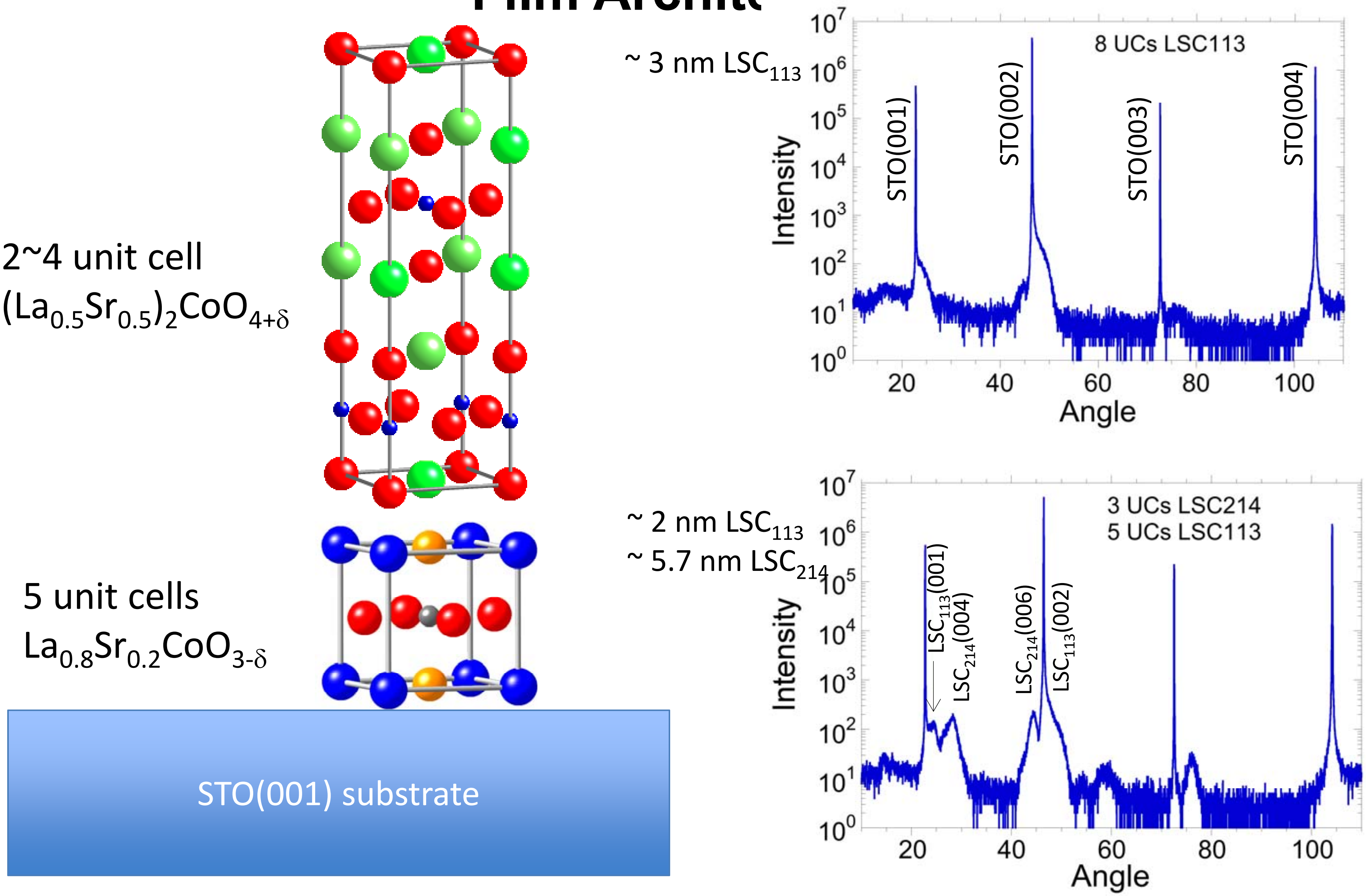
Inhomogeneous Sr depth dependence →

1. Octahedral distortion
2. Substrate as oxygen source
3. Oxygen vacancy concentration

Outline

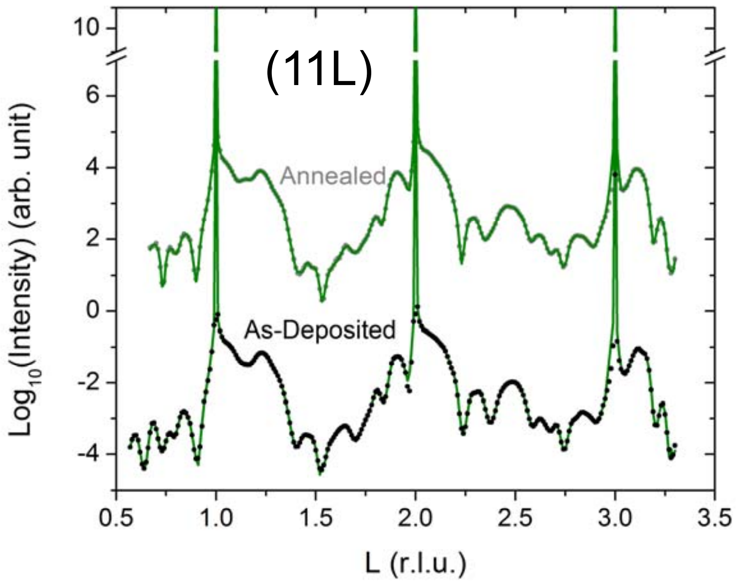
- *$(La_{0.5}Sr_{0.5})_2CoO_{4+\delta}/La_{0.8}Sr_{0.2}CoO_{3-\delta}/STO$ heterostructured systems*

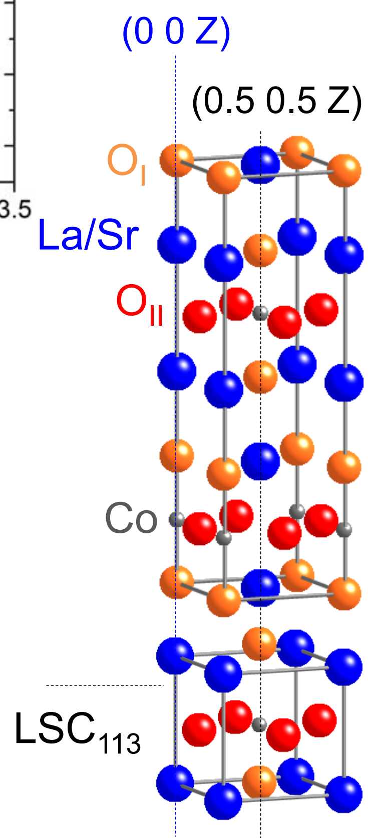
Film Architecture

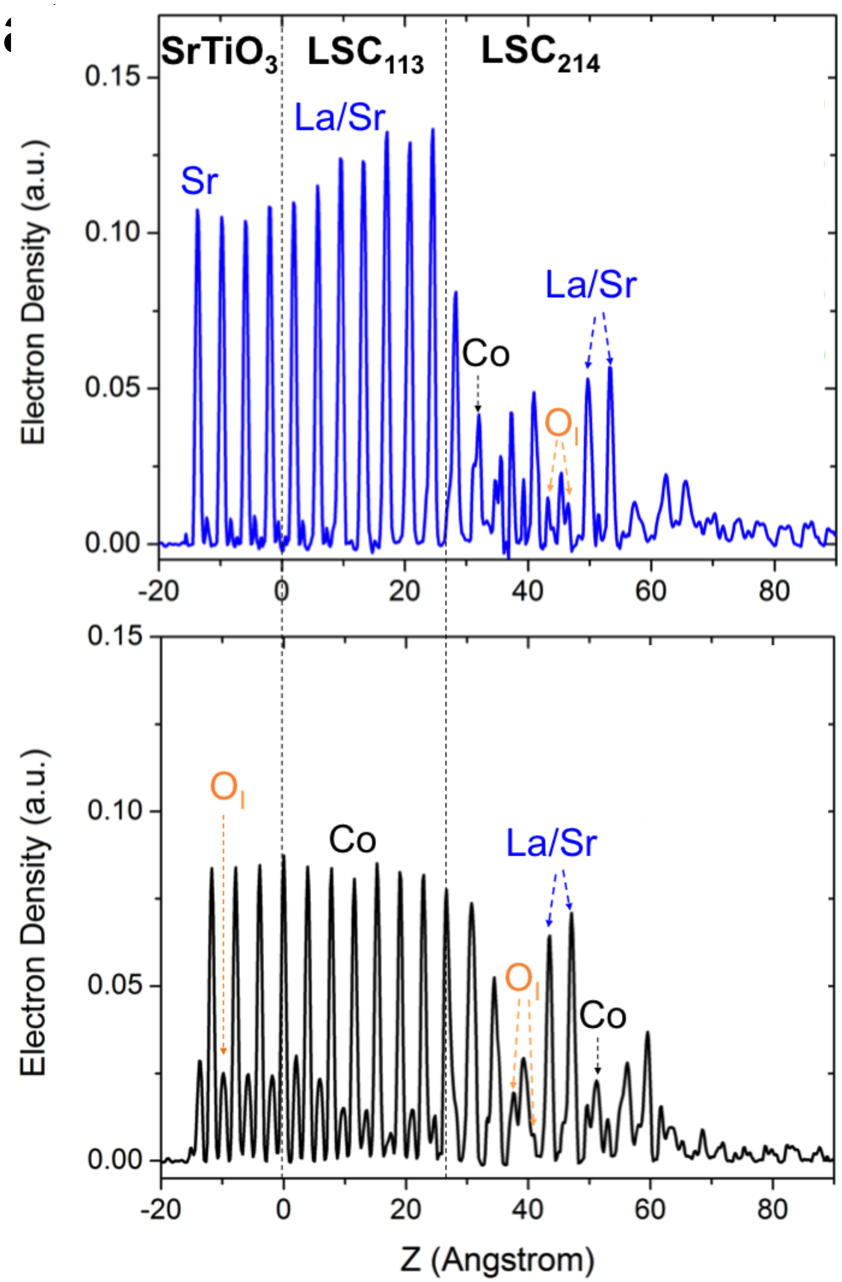


Z. Feng, et al., J. Phys. Chem. Lett., 2014, 5, 1027-1034

COBRA da

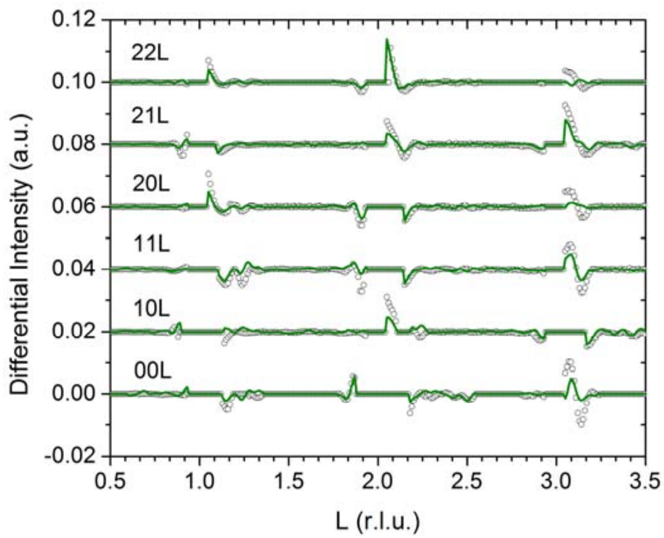


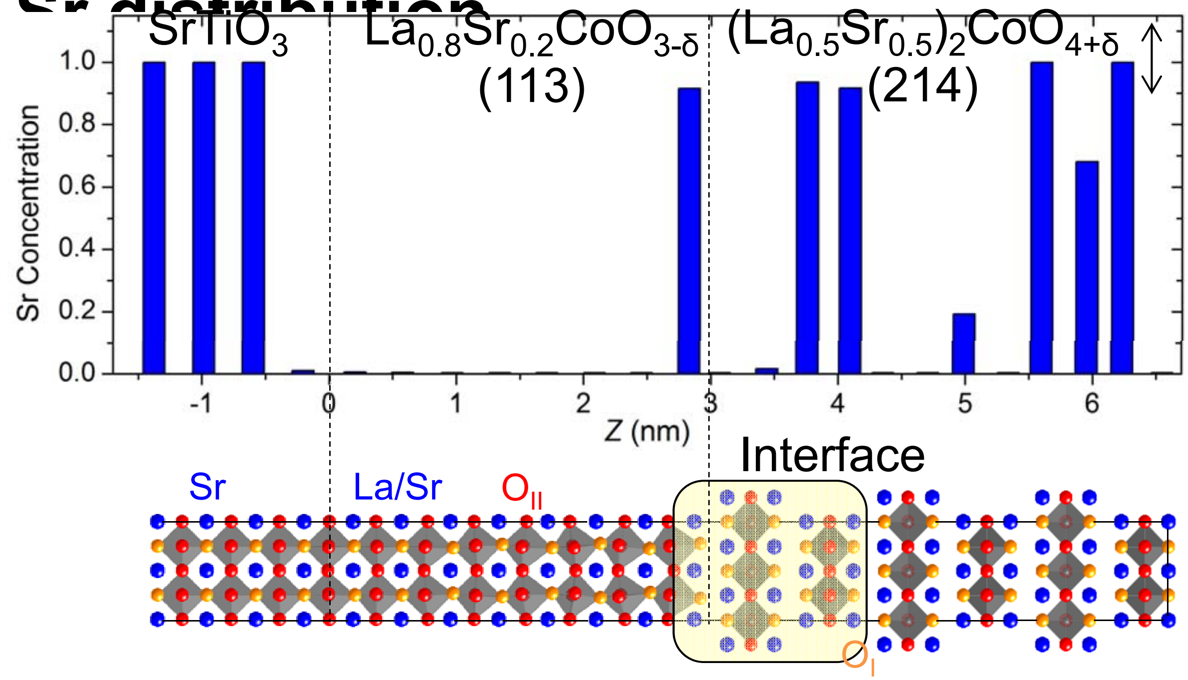




Z. Feng, et al., J. Phys. Chem. Lett., 2014, 5, 1027-1034

Sr distribution





- Sr concentrates on 113/214 interface and 214 surface (Sr-rich particles)
- Sr is depleted in 113 bulk film.
- Non-uniformed Sr layer occupation in one LSC_{214} unit cell.

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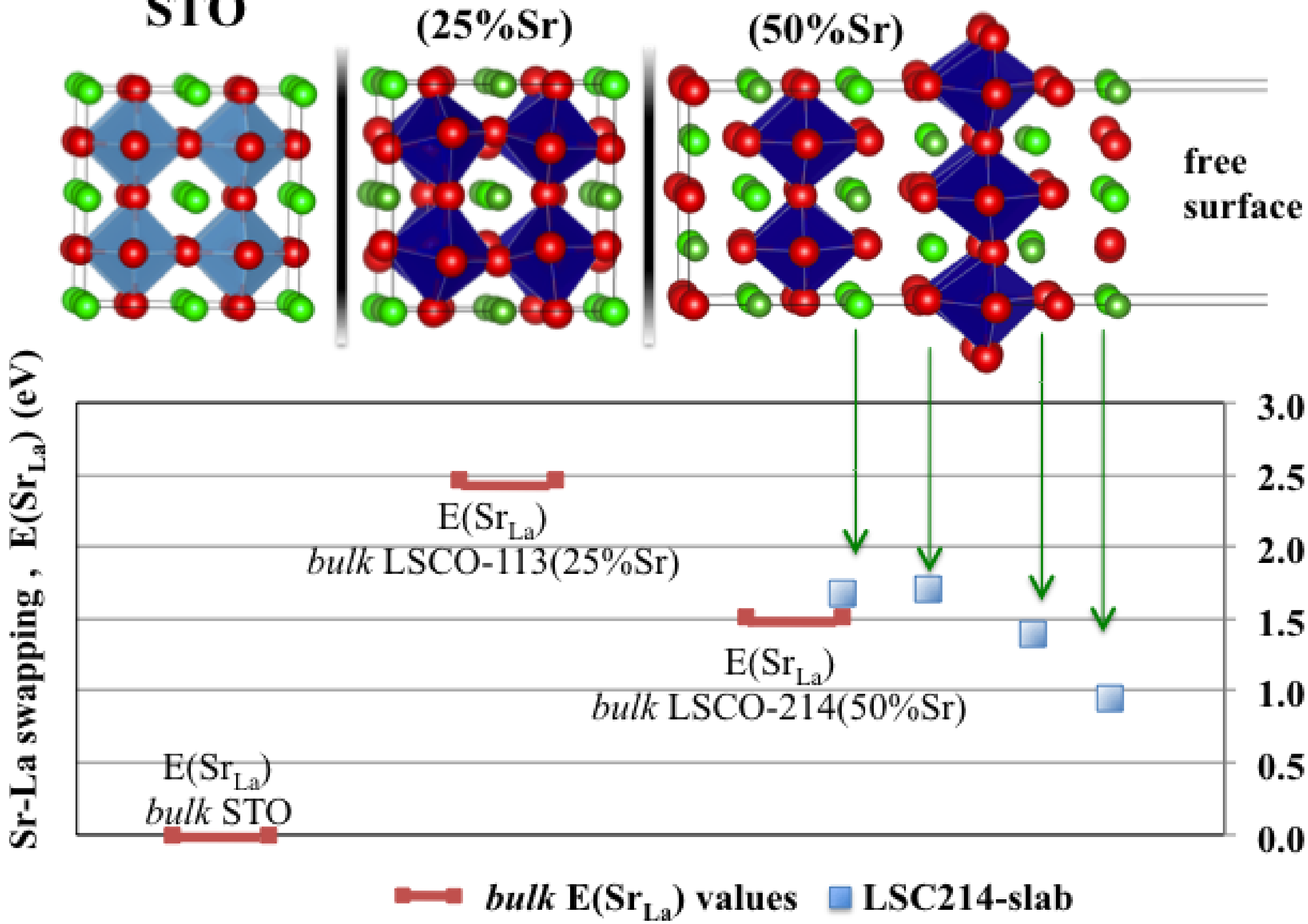

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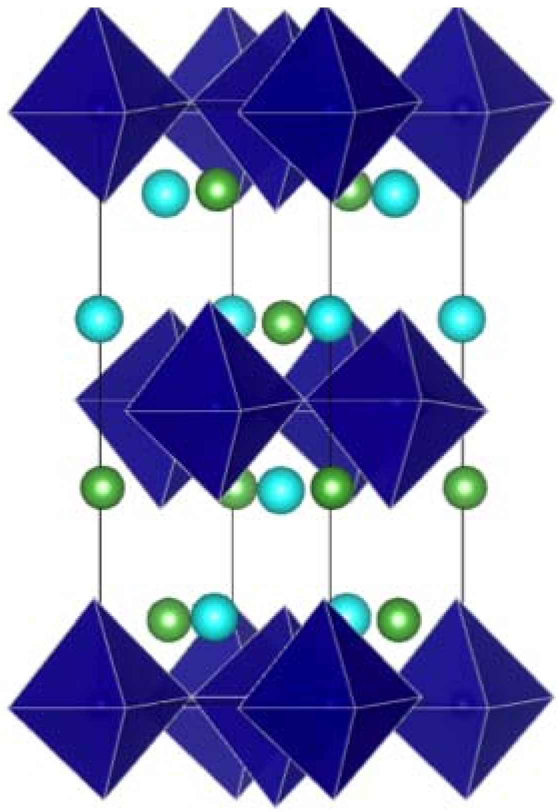
DFT to explain

La

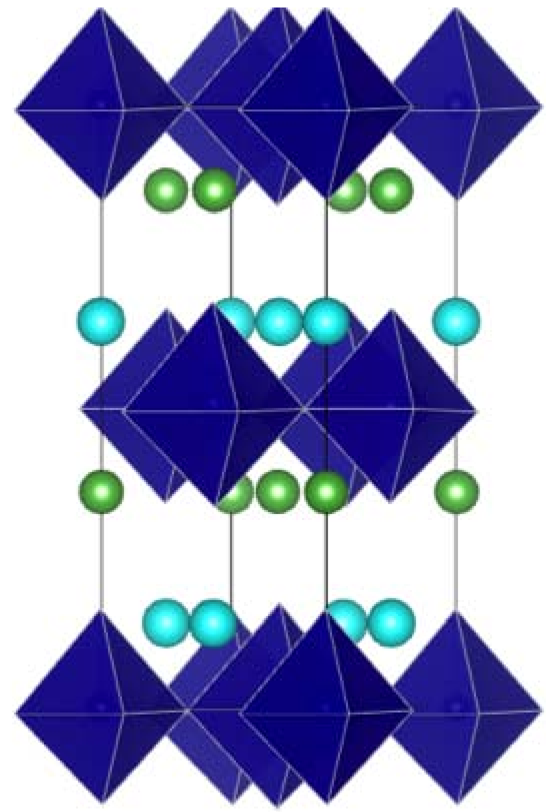
Sr

50% Sr 50%La in each AO layer



E0 = 0 eV (Reference)

Alternating LaO-SrO layer



E0 = -0.021 eV/FU (relaxed)
E0 = -0.037 eV/FU (fixed to STO lat const)

Z. Feng, et al., J. Phys. Chem. Lett., 2014, 5, 1027-1034

Summary

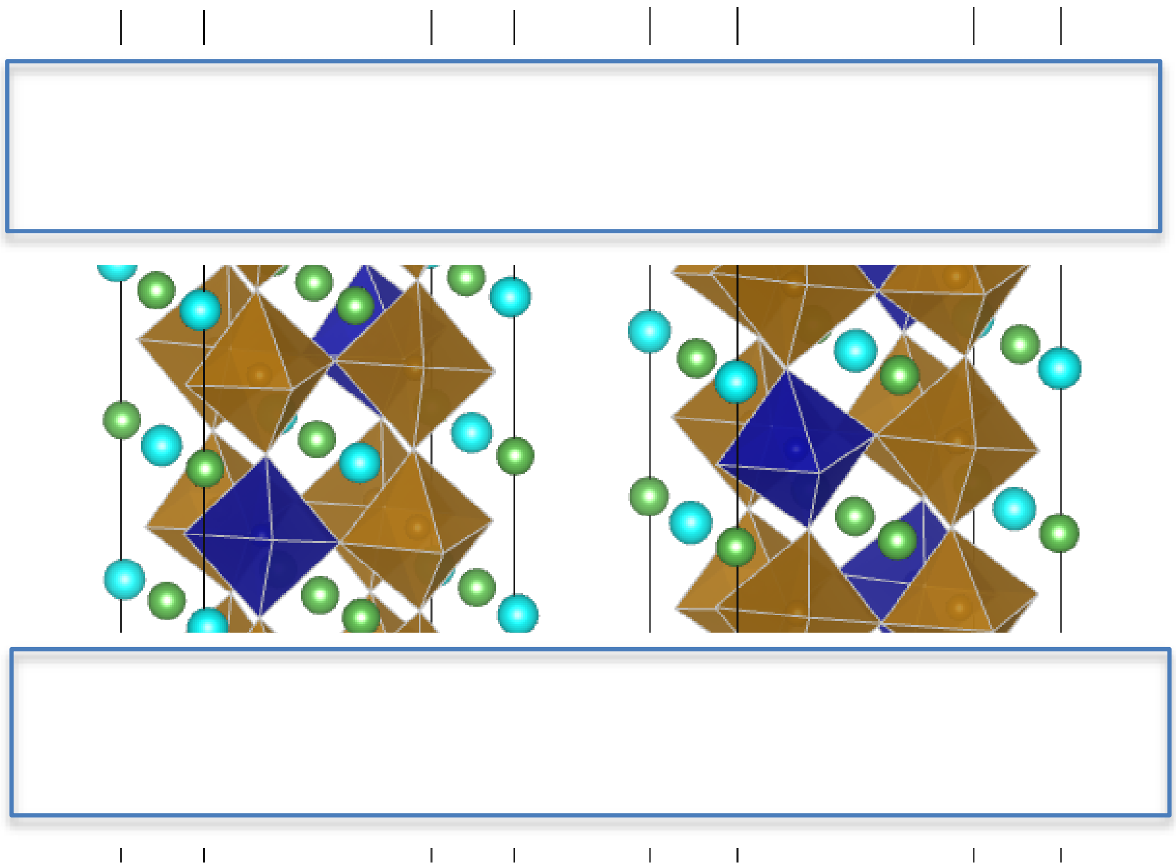
- Electrochemical Interface is important for Energy Storage and Conversion Systems

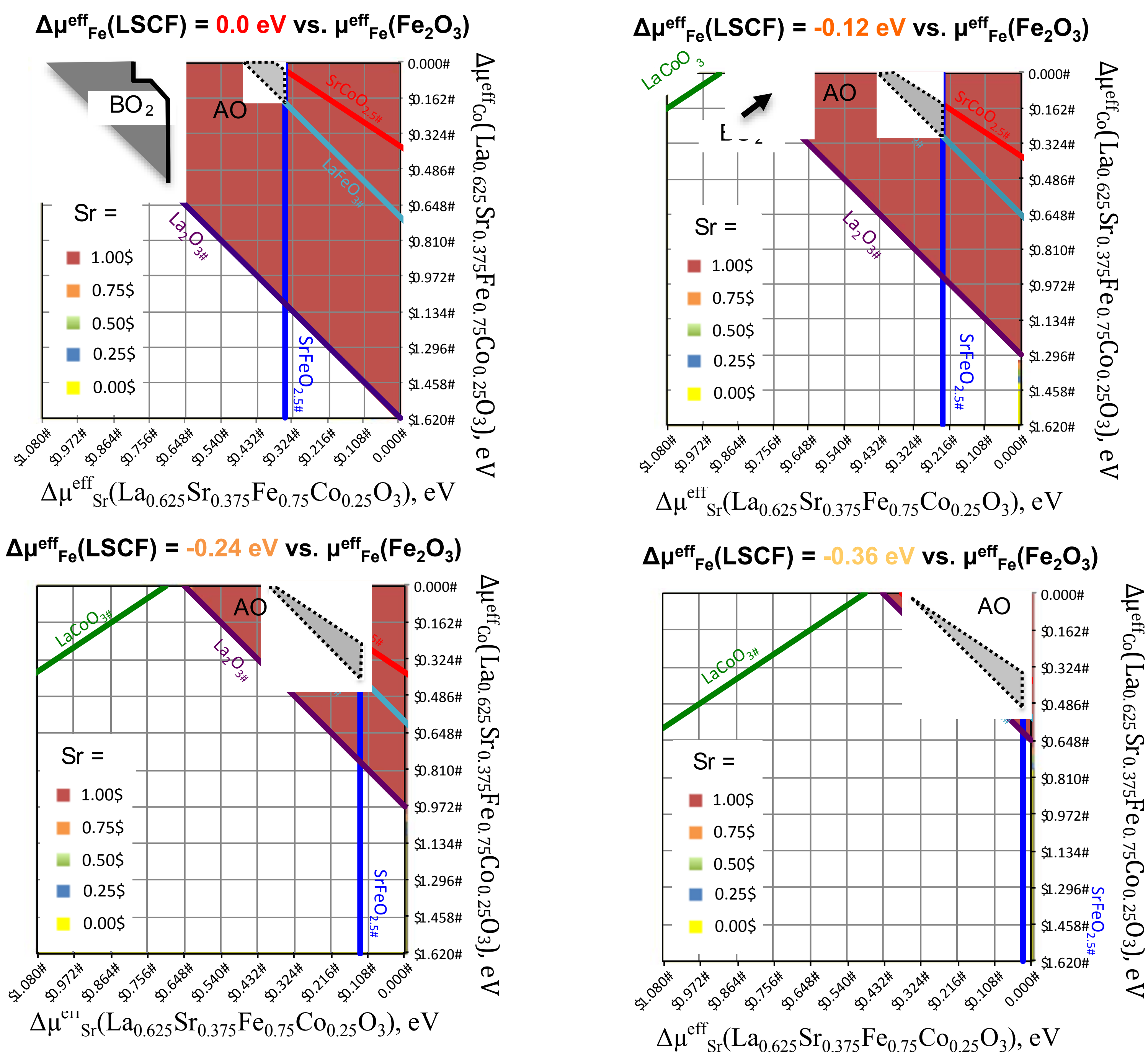
- COBRA is unique and sensitive to obtain atomic and chemical information.

- Anomalous Sr distribution is associated with its oxygen deviation (octahedral distortion) and is related to catalytic properties.

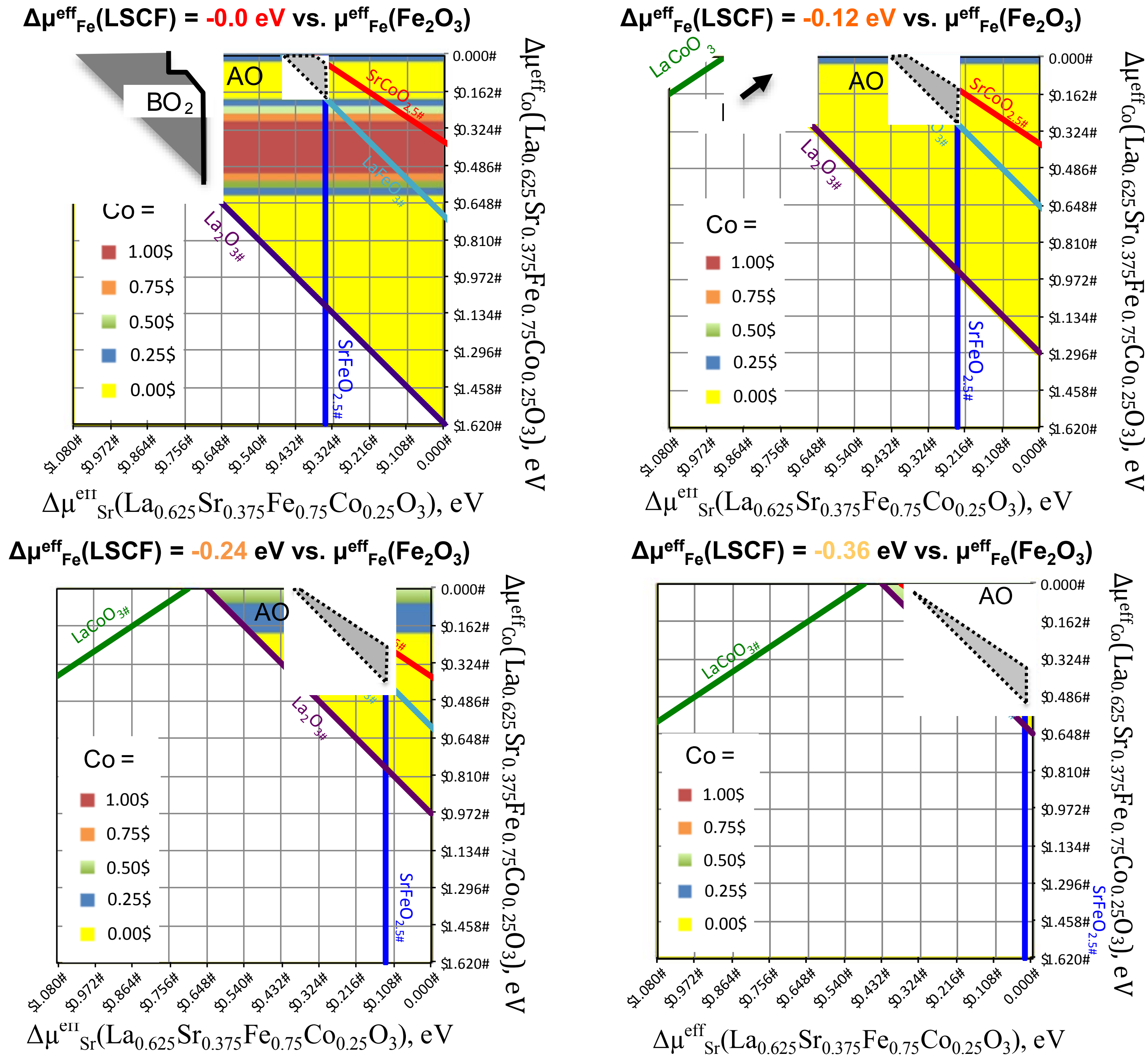
Backup for Dane

LSC_{113} and $LSCF_{113}$ Slab model





Δμeff Fe(LSCF) = 0.0 eV vs. μeff Fe(Fe2O3)
Δμeff Fe(LSCF) = -0.12 eV vs. μeff Fe(Fe2O3)
Δμeff Fe(LSCF) = -0.24 eV vs. μeff Fe(Fe2O3)
Δμeff Fe(LSCF) = -0.36 eV vs. μeff Fe(Fe2O3)
BO2
AO
SrCoO2.5
LaFeO3
La2O3
SrFeO2.5
LaCoO3
Sr =
1.00
0.75
0.50
0.25
0.00
Δμeff Sr(La0.625Sr0.375Fe0.75Co0.25O3), eV
Δμeff Co(La0.625Sr0.375Fe0.75Co0.25O3), eV

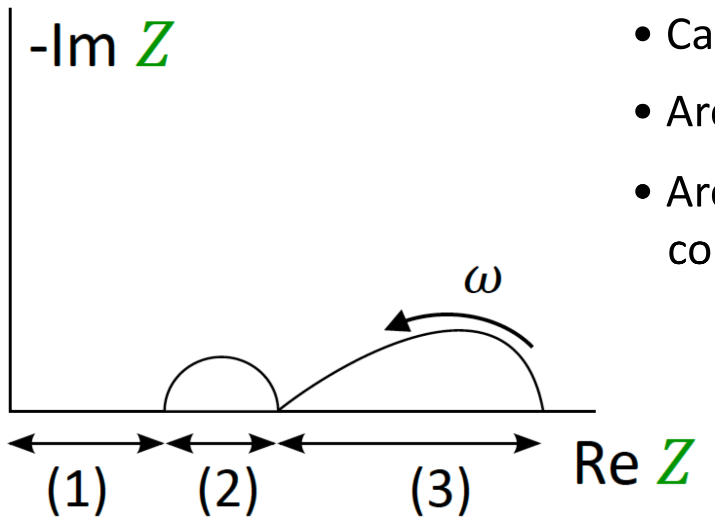


Δμ^eff_Fe(LSCF) = -0.0 eV vs. μ^eff_Fe(Fe2O3)
Δμ^eff_Fe(LSCF) = -0.12 eV vs. μ^eff_Fe(Fe2O3)
Δμ^eff_Fe(LSCF) = -0.24 eV vs. μ^eff_Fe(Fe2O3)
Δμ^eff_Fe(LSCF) = -0.36 eV vs. μ^eff_Fe(Fe2O3)
AO
BO2
SrCoO2.5
LaFeO3
La2O3
SrFeO2.5
LaCoO3
Co =
1.00
0.75
0.50
0.25
0.00
Δμ^eff_Sr(La0.625Sr0.375Fe0.75Co0.25O3), eV
Δμ^eff_Co(La0.625Sr0.375Fe0.75Co0.25O3), eV

Backup for Stu

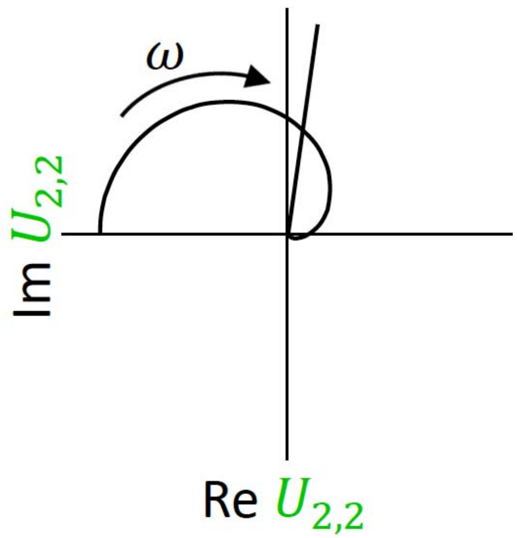
Electrochemical Measurements

EIS



- Can separate series rates by timescale.
- Arc resistance related to absolute rates.
- Arc capacitance related to defect concentrations.

NLEIS



- Insensitive to absolute rates (scaled out).
- Sensitive to nonlinearities in rate laws.
  - kinetic/transport mechanisms
  - surface thermodynamic properties
  - bulk thermodynamic properties