Development of High Temperature/High Sensitivity Novel Chemical Resistive Sensor

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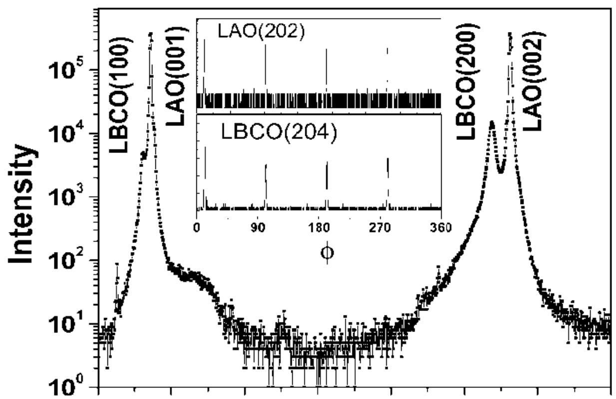
Grant Number: DE-FE0003780
Project Manager: Dr. Susan M. Maley
Performance Period: 09/01/2010-8/31/2013

- Introduction
- Mixed Ionic/Electronic Conductive $LnBaCo_2O_{5.5}$ Oxides
- Full Scale Chemical Sensor Development
- Summary

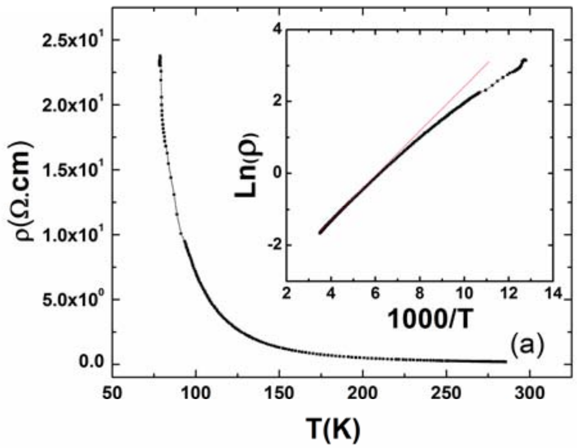
OBJECTIVES & GOALS

- **The objective of this research is:**
  - investigate and understand the mechanisms of mixed ionic electronic conductive $LaBaCo_2O_{5+\delta}$ highly epitaxial thin-films
  - establish the relationship between electrochemical properties and surface/interface microstructure of the mixed conductive thin films
  - determine the overall feasibility of the $LaBaCo_2O_{5.5+\delta}$ based novel electrochemical devices for sensing gases in high temperature applications.
- **The goals of this research are:**
  - resolving and optimizing fabrication issues of highly epitaxial $LaBaCo_2O_{5+\delta}$ single crystalline thin films
  - establishing relationship of processing—microstructure—sensing properties—stability of the $LaBaCo_2O_{5+\delta}$ thin film
  - understanding the kinetics and mechanisms of redox processes on the $LaBaCo_2O_{5.5+\delta}$ thin films
  - demonstrating the new concept high temperature, high sensitivity, and chemically stable devices for high temperature applications.

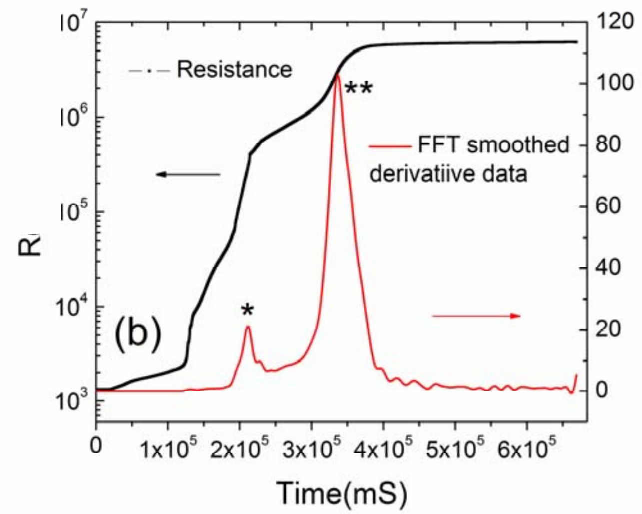
Why LBCO?



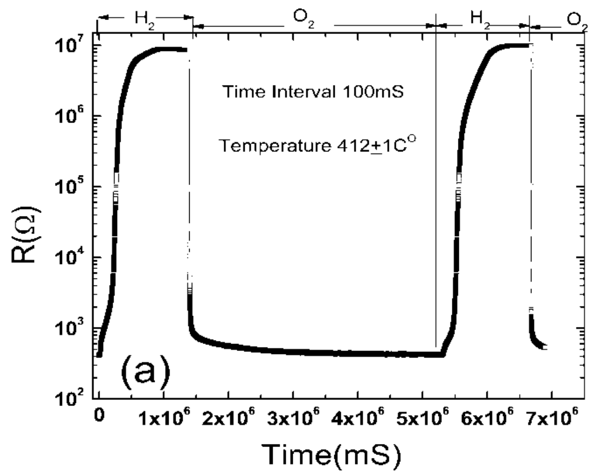
Intensity
LBCO(100)
LAO(001)
LBCO(200)
LAO(002)
LAO(202)
LBCO(204)
ϕ



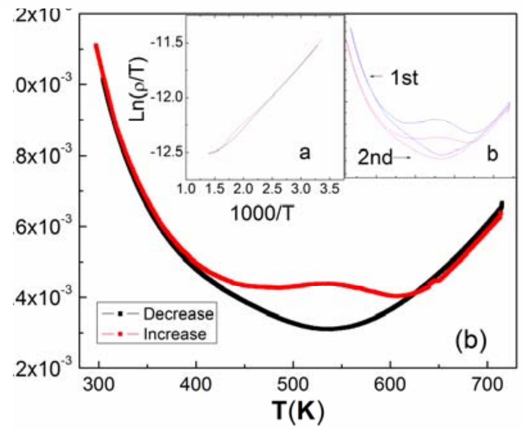
ρ(Ω.cm)
Ln(ρ)
1000/T
(a)
T(K)



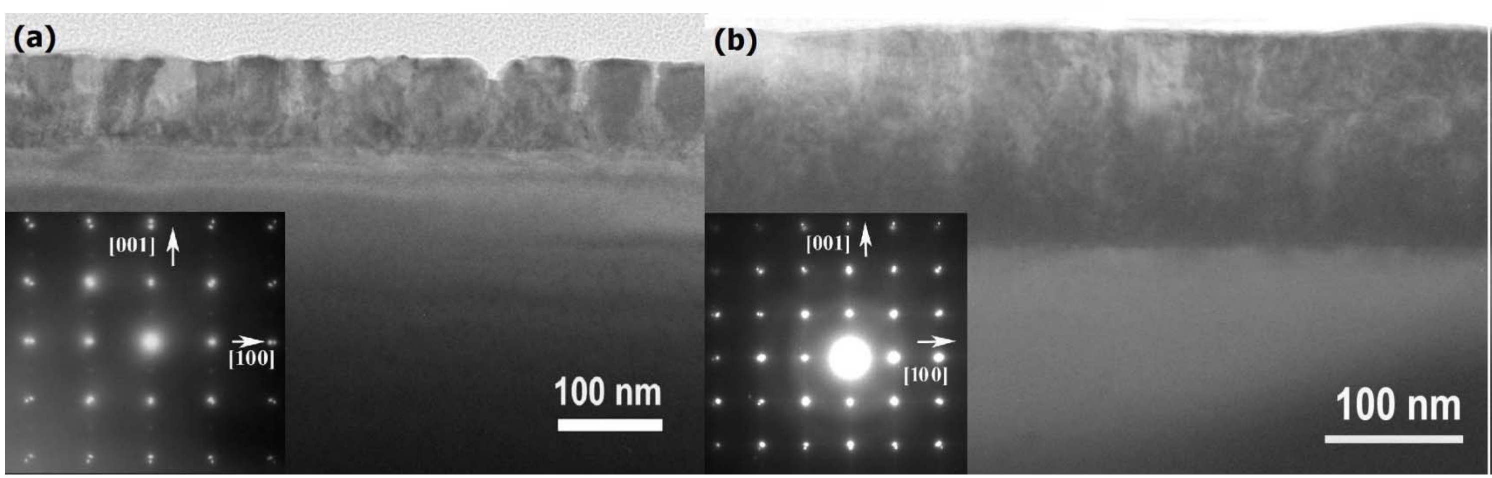
Resistance
FFT smoothed derivatiive data
(b)
R
Time(mS)



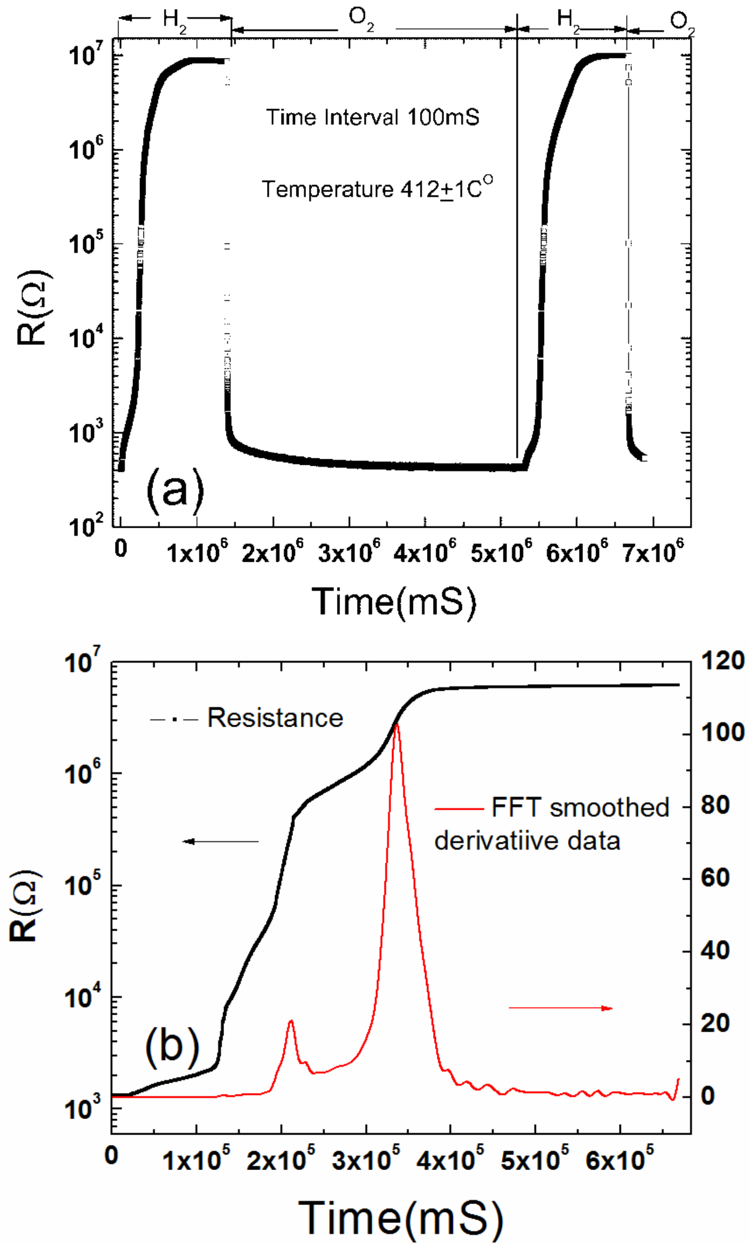
H2
O2
Time Interval 100mS
Temperature 412±1C°
R(Ω)
(a)
Time(mS)

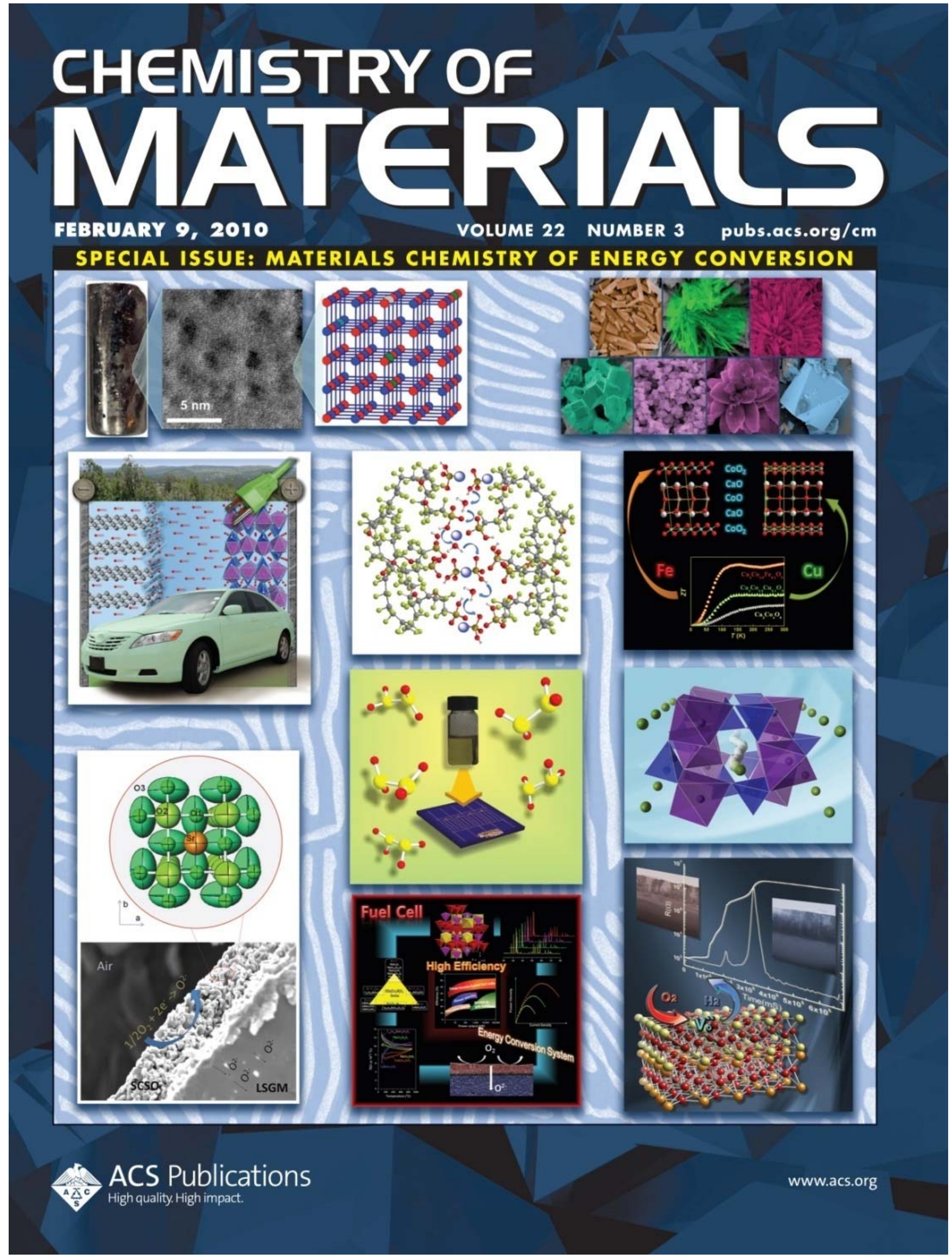


Ln(ρ/T)
1000/T
1st
2nd
a
b
Decrease
Increase
(b)
T(K)



(a)
[001]
[100]
100 nm
(b)
[001]
[100]
100 nm





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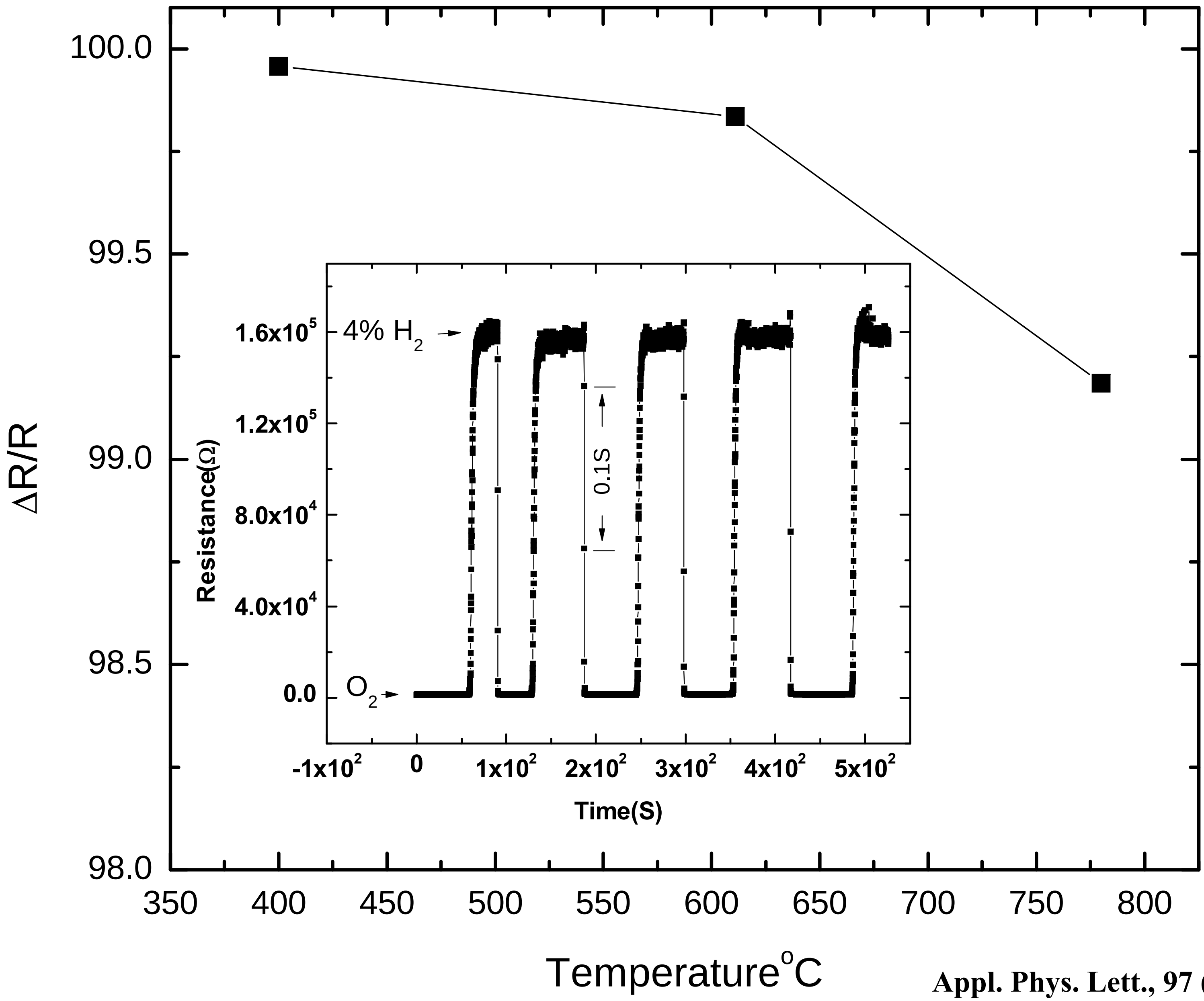
Nanoscale ordered cobaltite $LaBaCo_2O_6$ thin films



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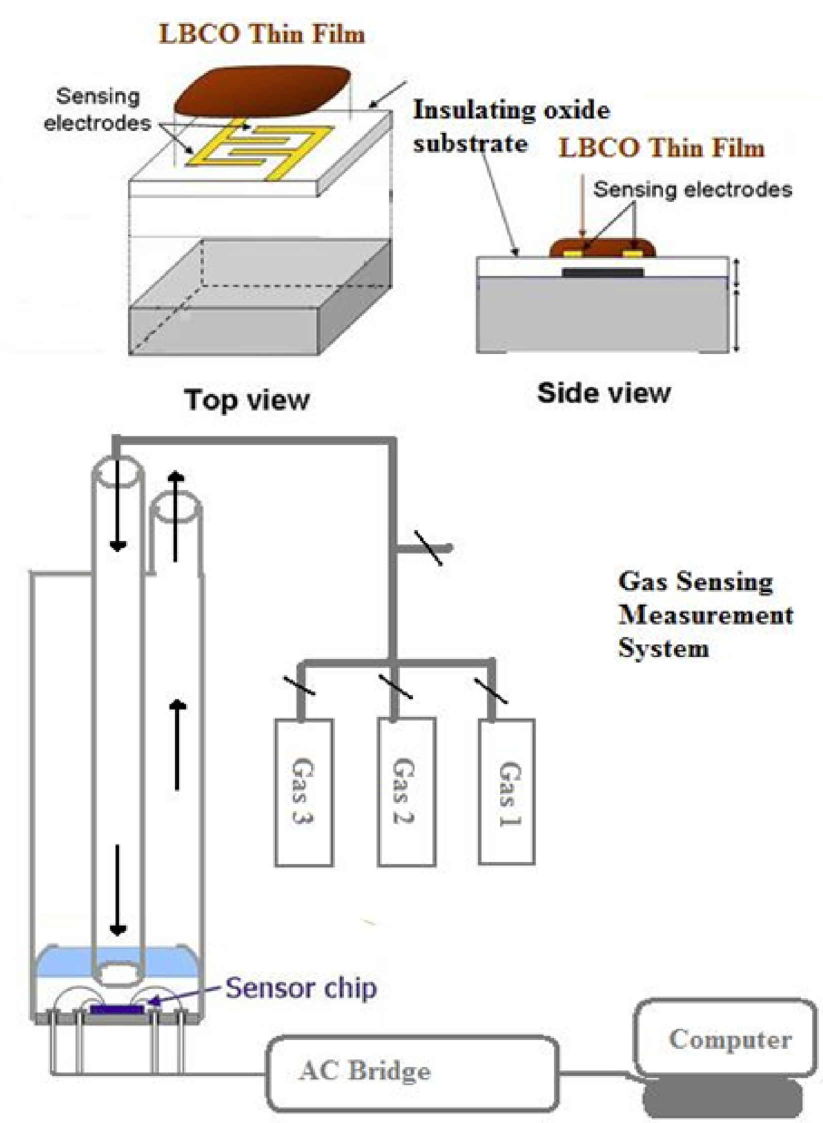
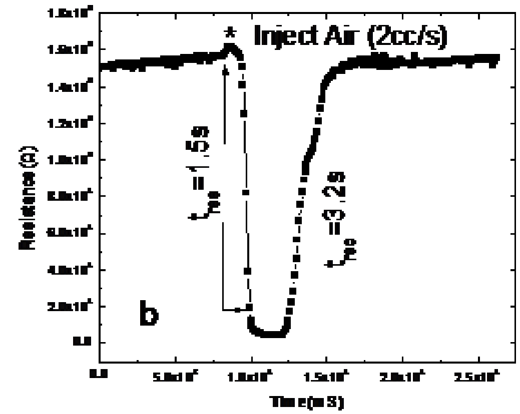
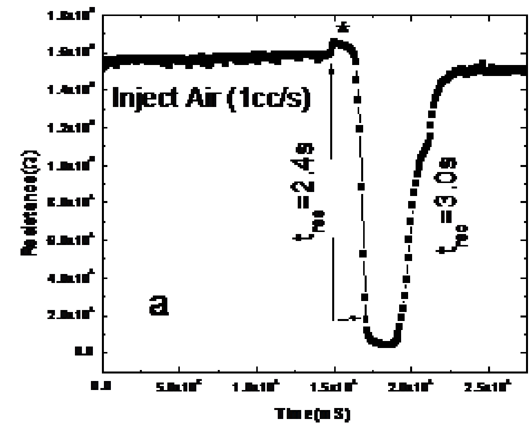
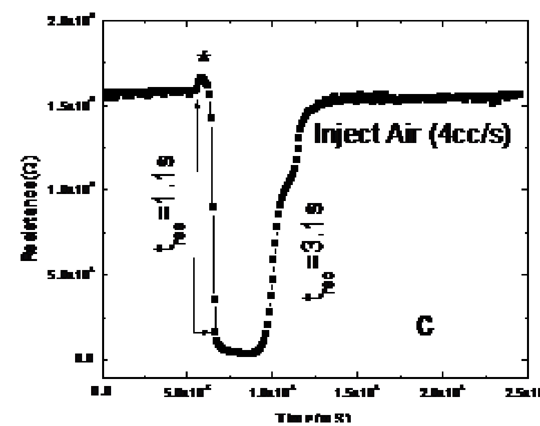
Sensor Structures





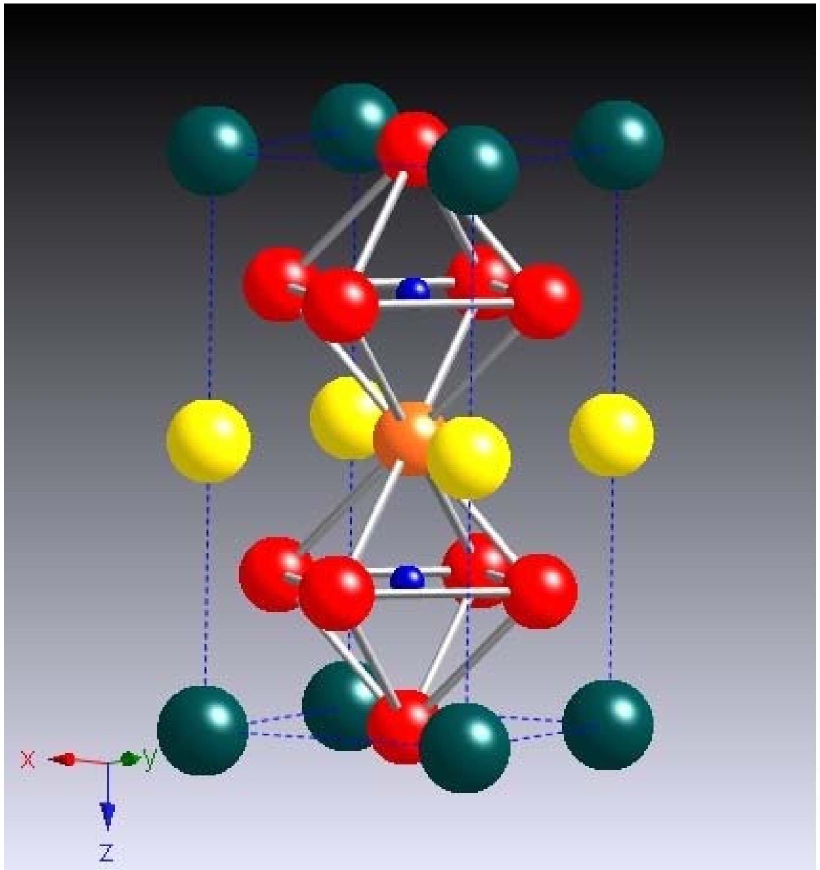




Oxygen Deficient Double Perovskite $(LnBa)Co_2O_{5+\delta}$ (Ln=Lanthanide)

Structure of $LnBaCo_2O_{5+\delta}$

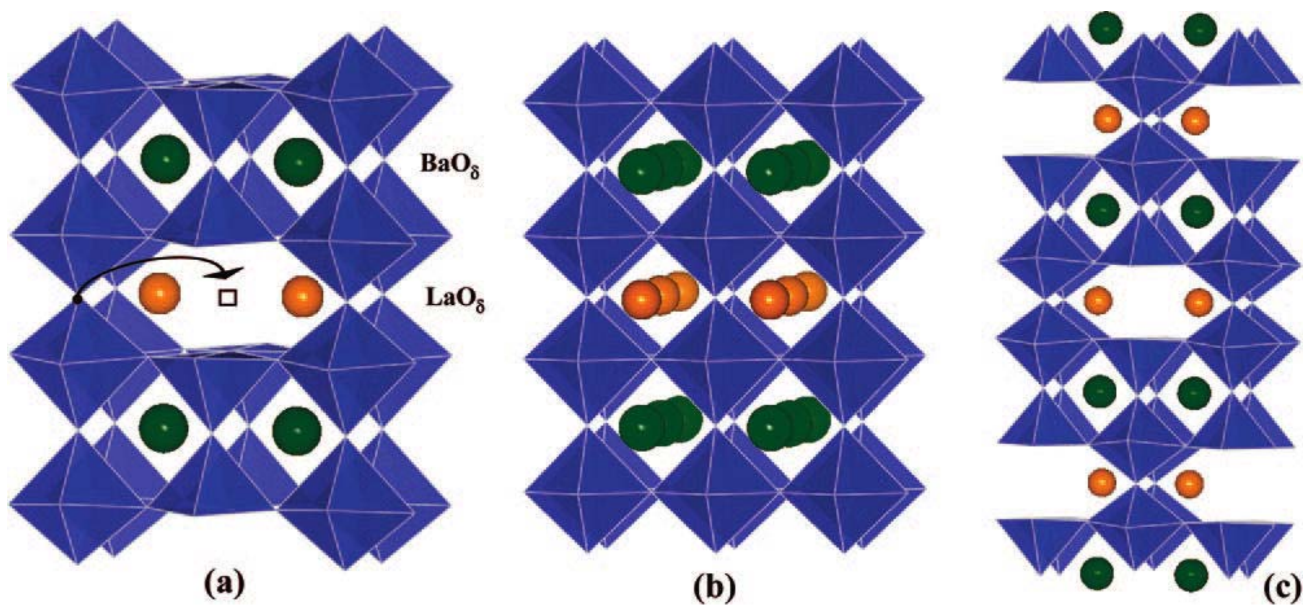
- **— Ba**
- **— Ln**
- **— Co**
- **— O (occupied)**
- **— O (partial occupied)**



(La, Ba)$Co_2O_{5+\delta}$

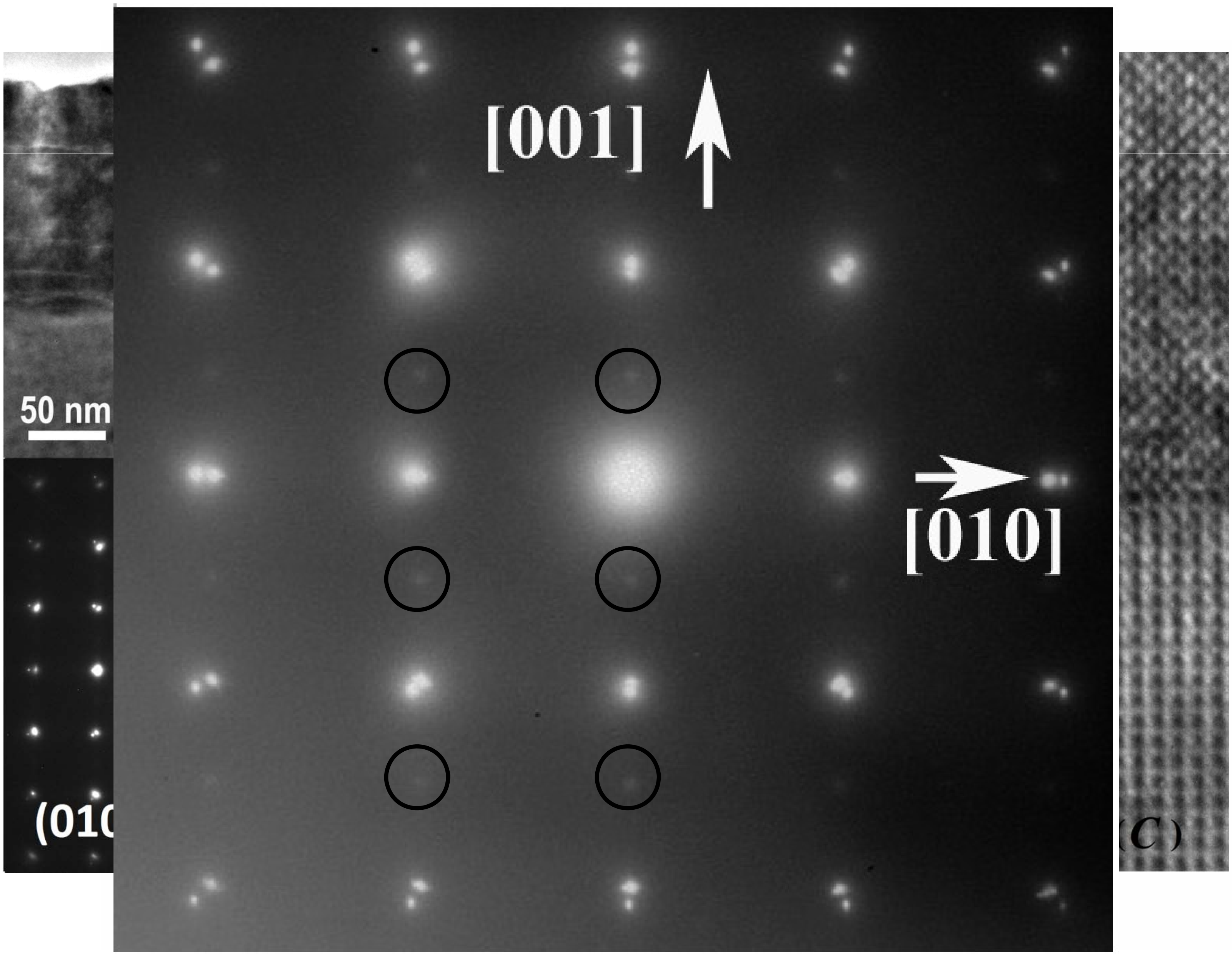
$$Co^{2+} : Co^{3+} = \left(\frac{1}{2} - \delta\right) : \left(\frac{1}{2} + \delta\right) ----- 0 \le \delta \le 0.5$$

$$Co^{4+} : Co^{3+} = \left(\delta - \frac{1}{2}\right) : \left(\frac{3}{2} - \delta\right) ----- 1 \ge \delta \ge 0.5$$



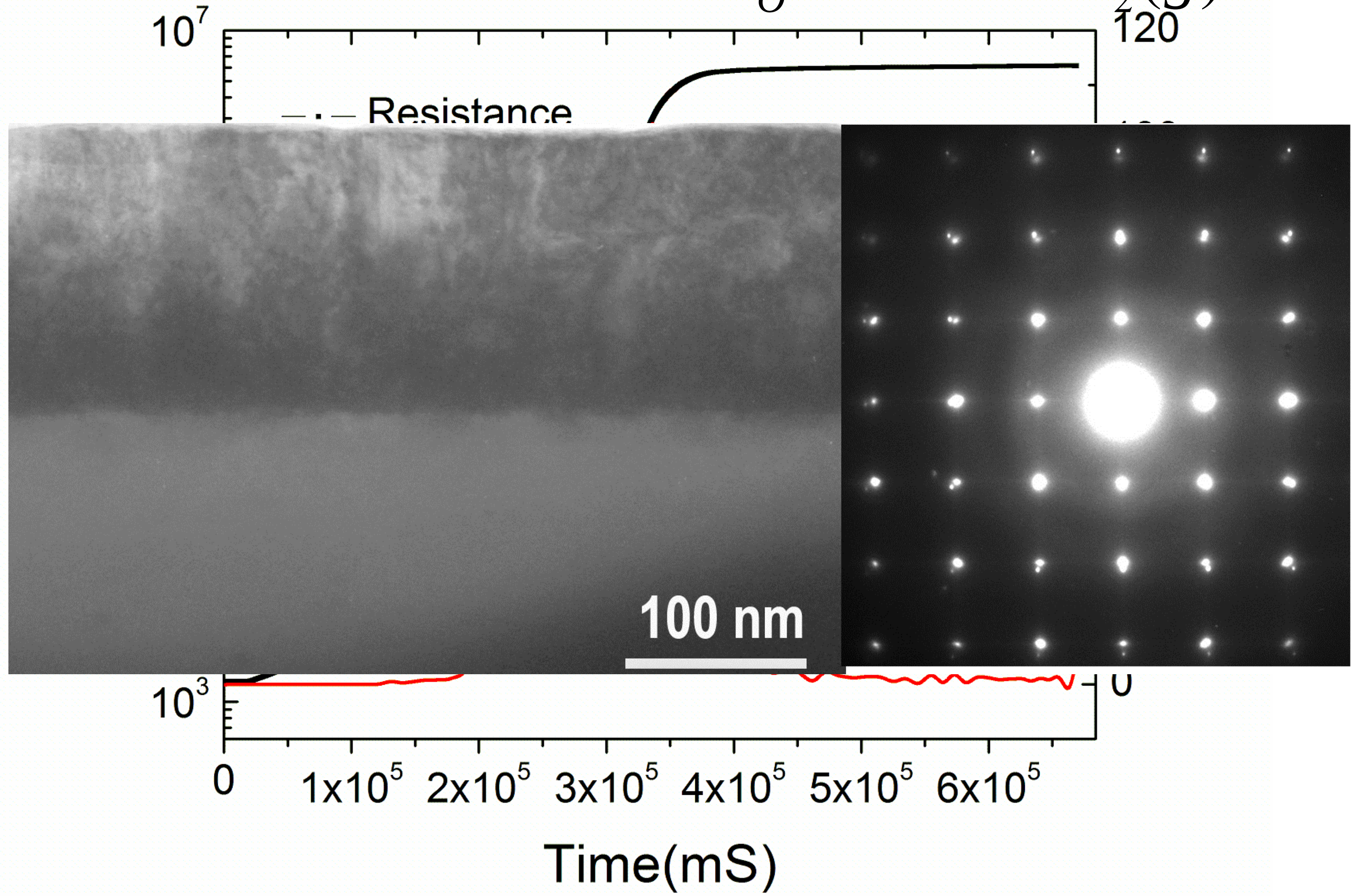
Rautama et , Chem. Mater., Vol. 21, No. 1, 2009.

$LaBaCo_2O_{5+\delta}$ Thin Film on (001)$LaAlO_3$



Transport Properties in 4%H_2 / N_2

$$2O^{2-} + 2Co^{3+} + Co^{4+} \leftrightarrow 2V_O^{\bullet\bullet} + 3Co^{2+} + O_2(g)$$



Transport Properties in O_2

$LaCoO_3$

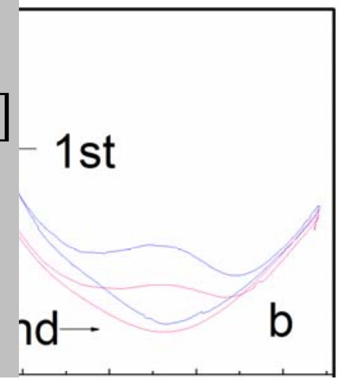
$2Co^{3+} \leftrightarrow Co^{2+} + Co^{4+}$

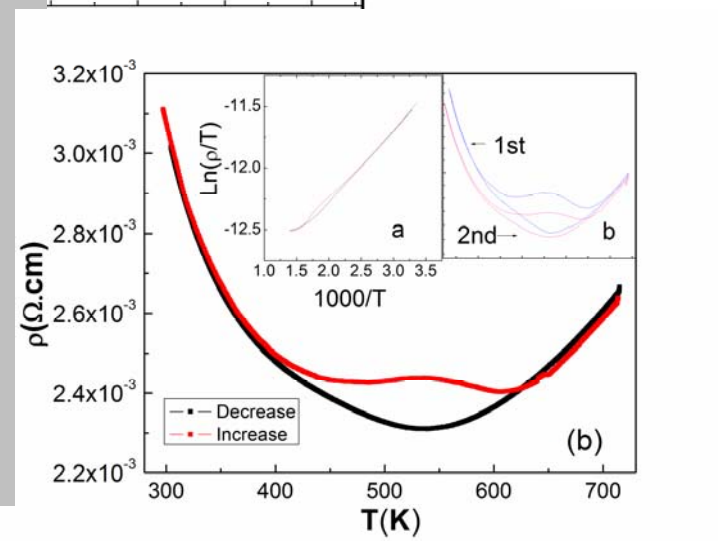
$Ba^{2+} \Leftrightarrow (La^{3+}Ba^{2+})(Co^{3+}Co^{4+})O_{5+\delta} \mapsto [Co^{4+}] + [V_O]$

$$O^{2-} + 2Co^{4+} \leftrightarrow V_O + 2Co^{3+} + \frac{1}{2}O_2$$

$$O_{ad}{}^{2-} \leftrightarrow 2e^{-} + \frac{1}{2}O_2$$

$T\uparrow \Rightarrow [e^{-}]\uparrow \& [h]\downarrow$





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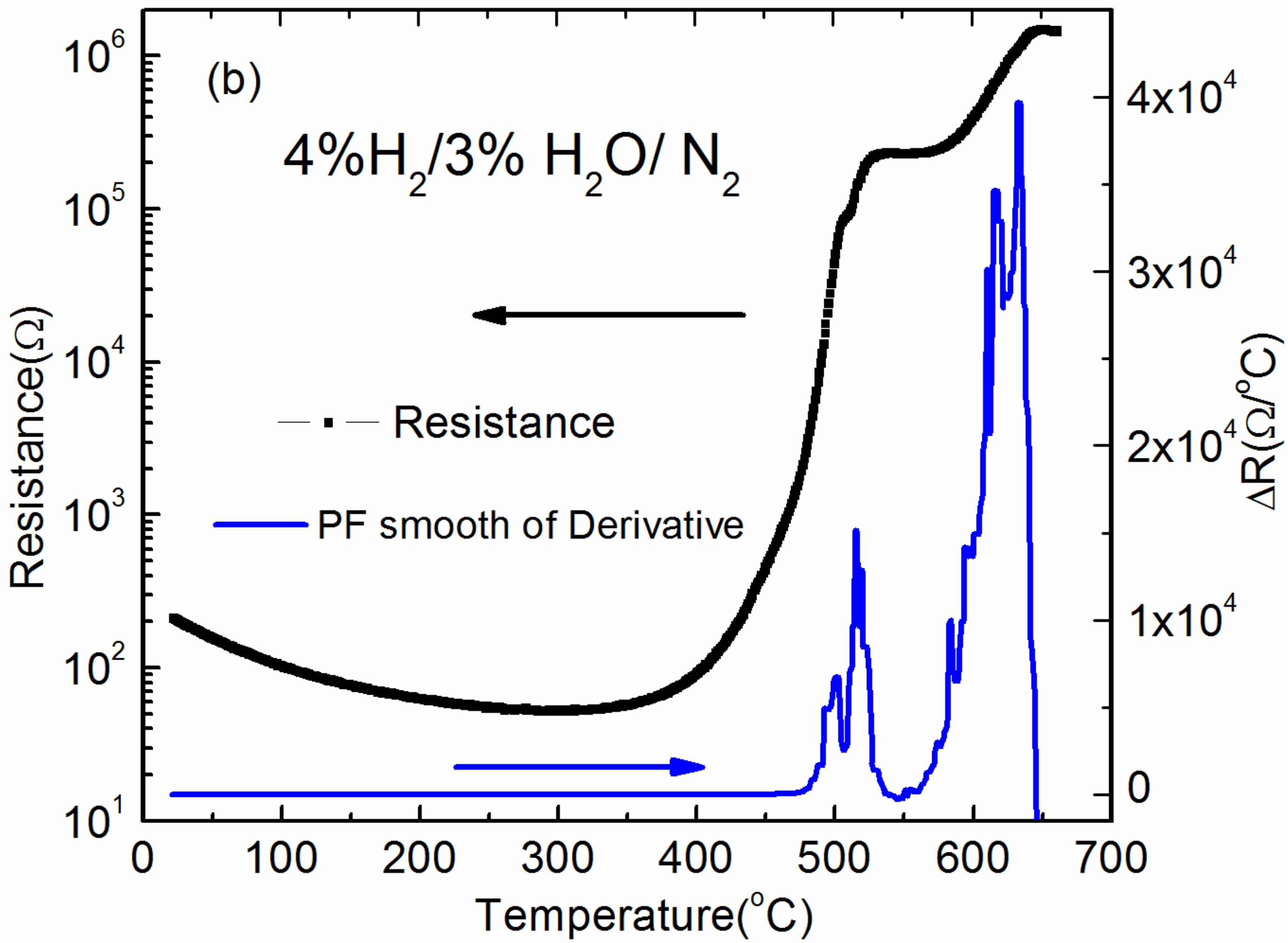
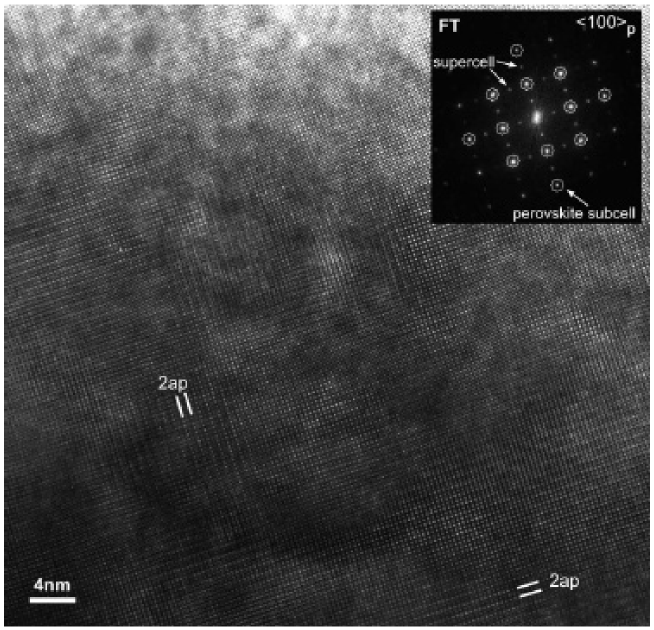
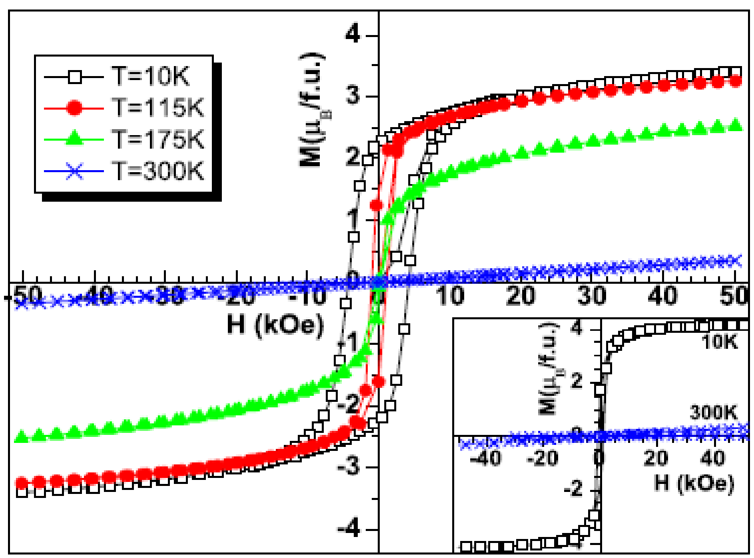
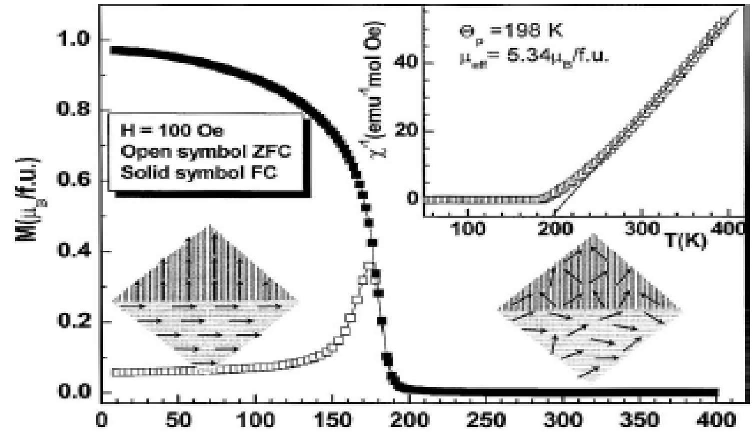
Transport Properties in 4%H_2 / N_2

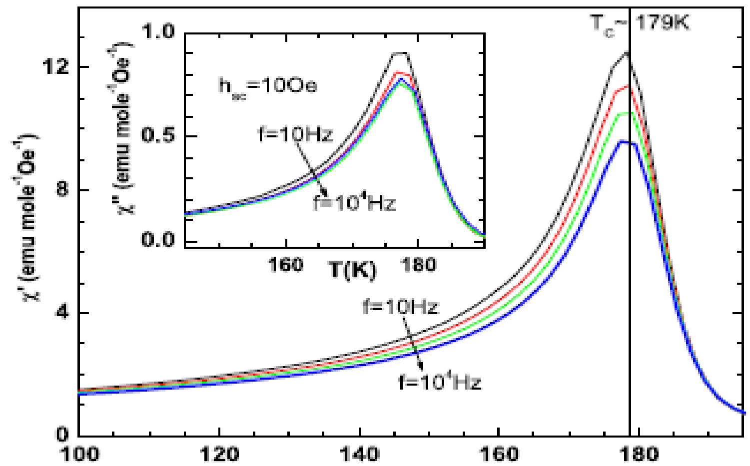


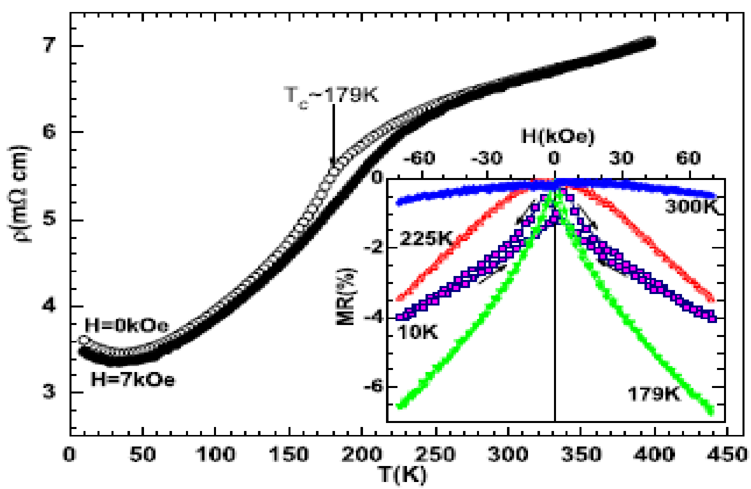
Nanoscale ordered cobaltite $LaBaCo_2O_6$



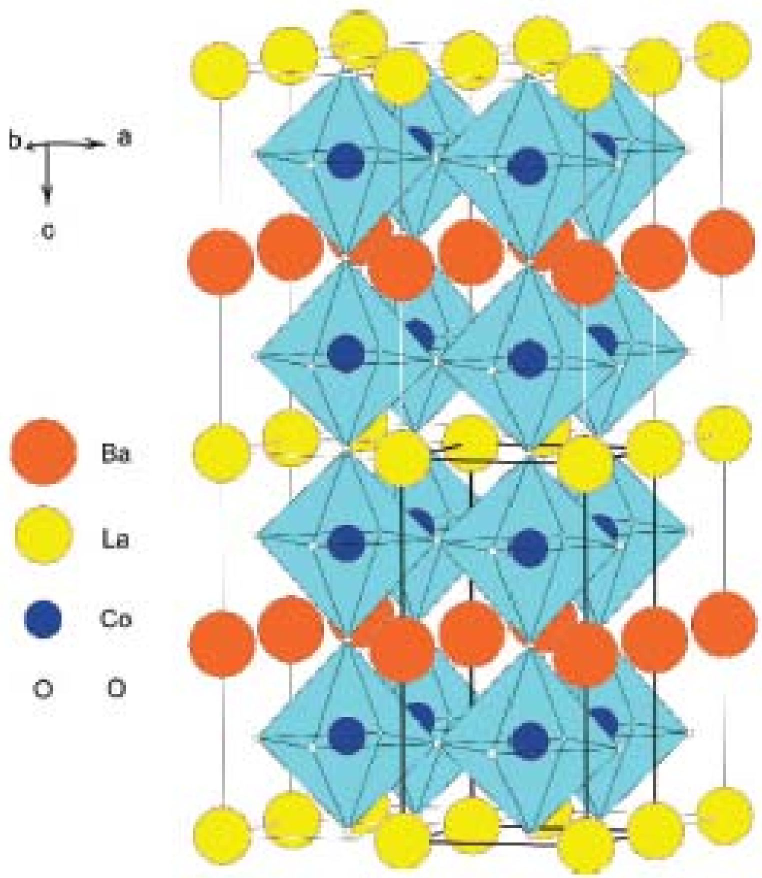


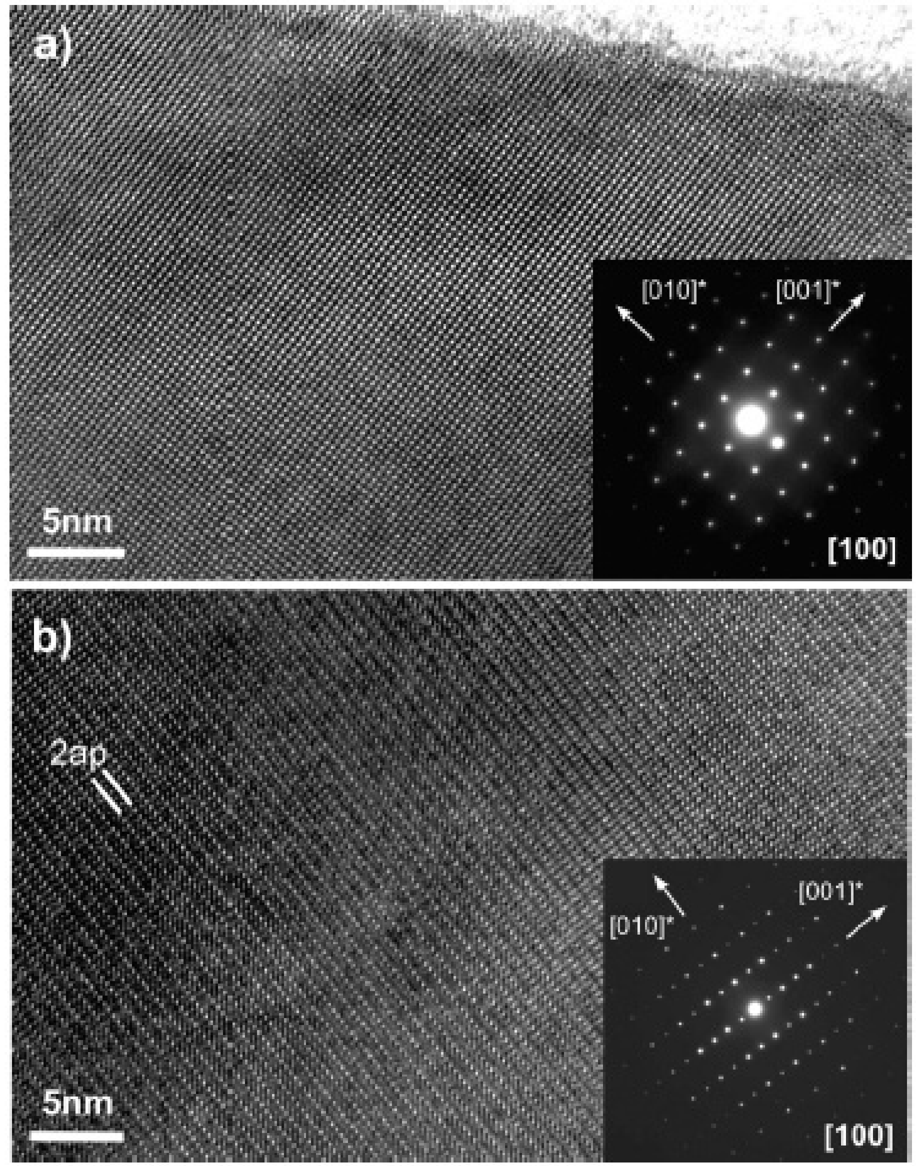




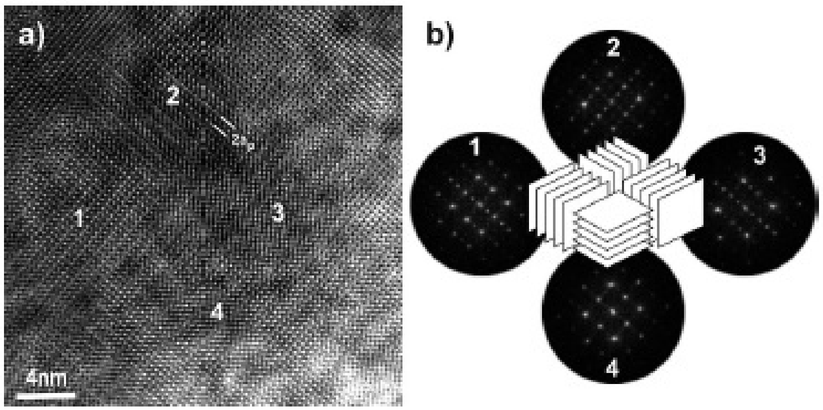


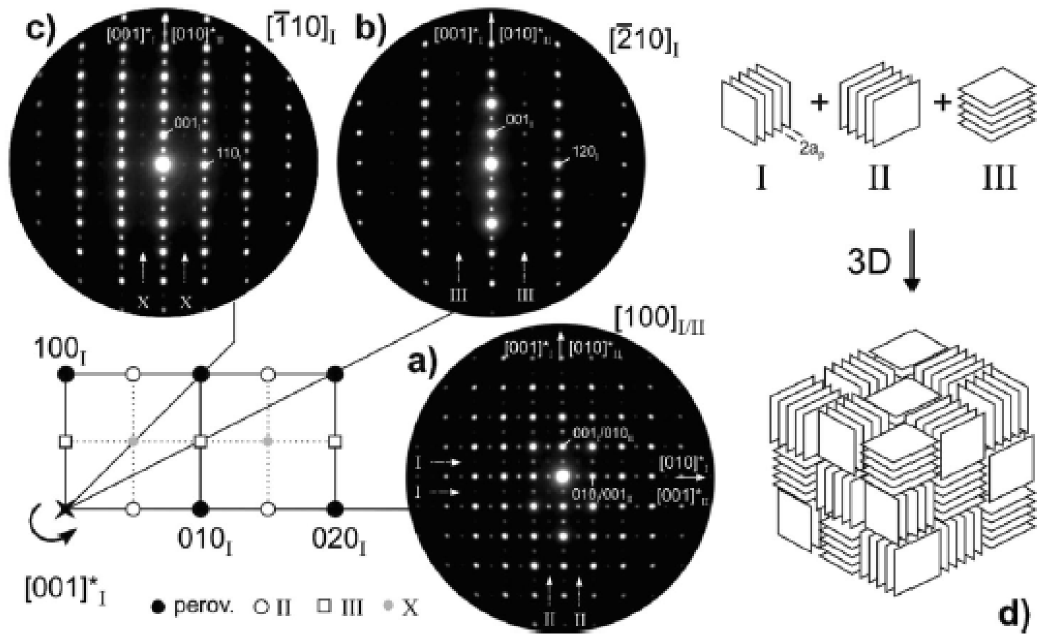
Nanoscale ordered cobaltite $LaBaCo_2O_6$

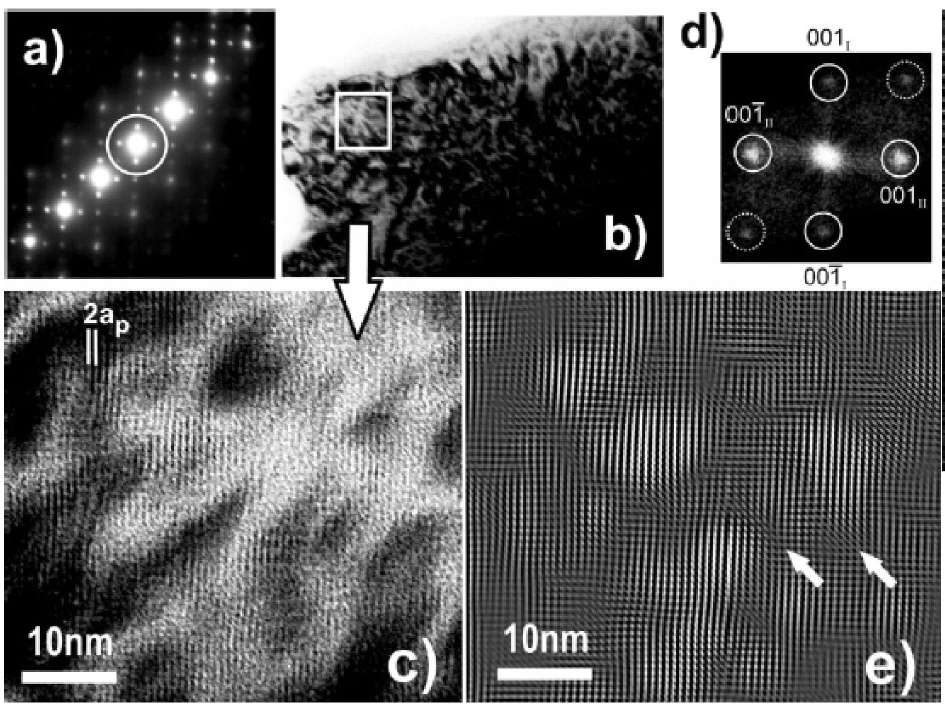


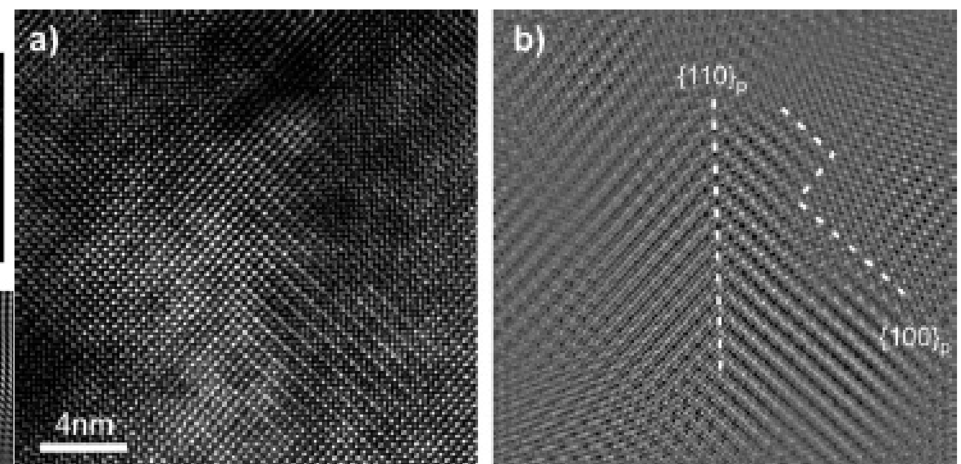


Nanoscale ordered cobaltite $LaBaCo_2O_6$









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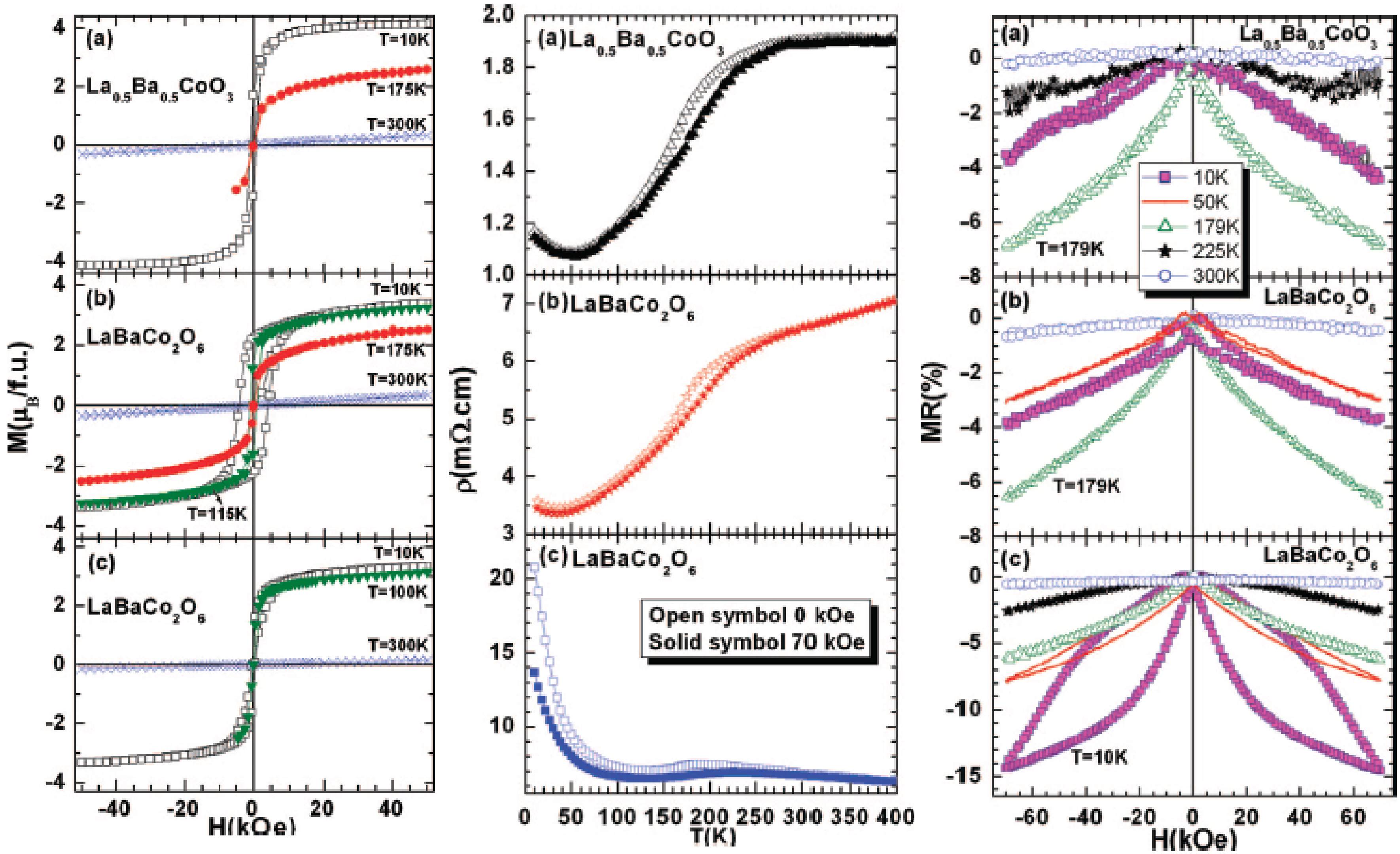
Physical Properties of cobaltite $LaBaCo_2O_6$

Ordering Nano-Ordering Disordering



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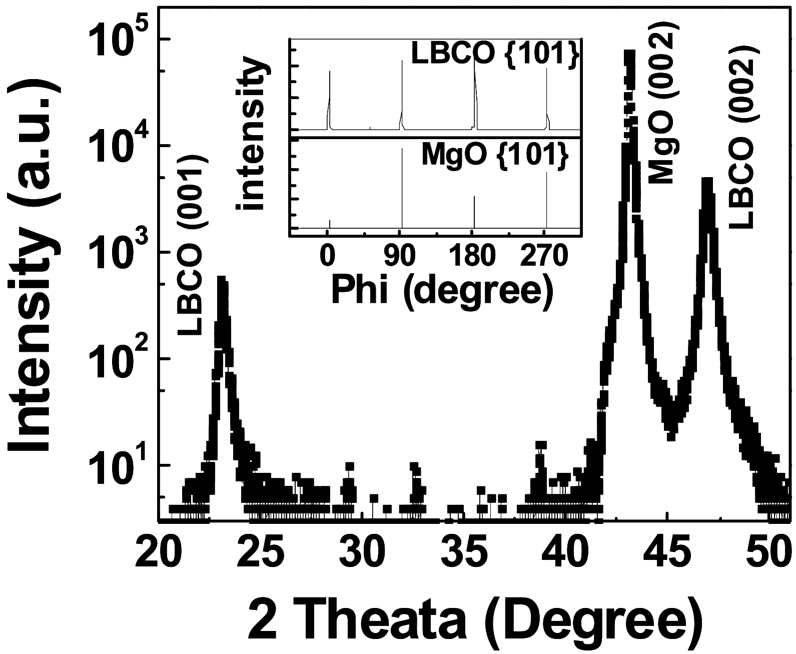
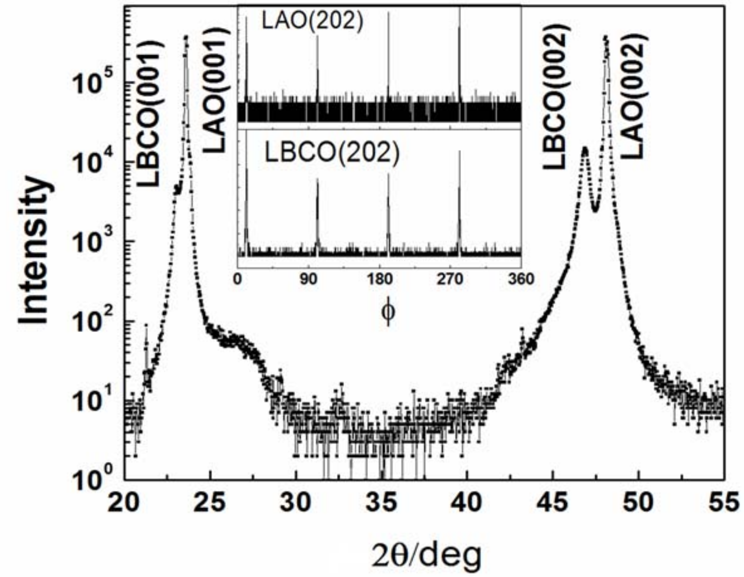
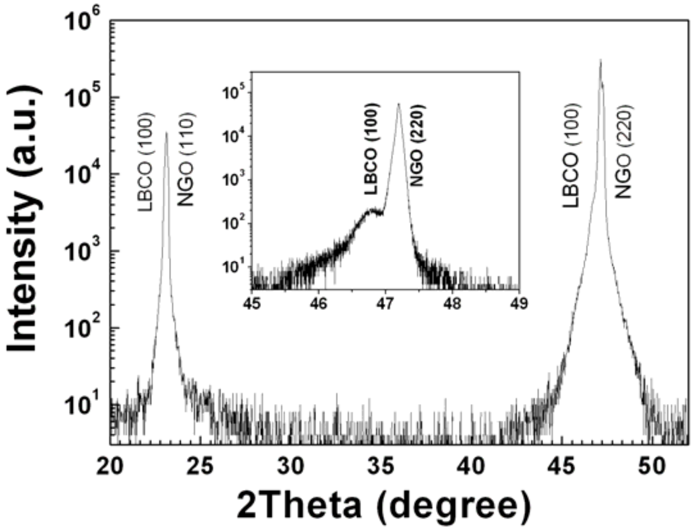


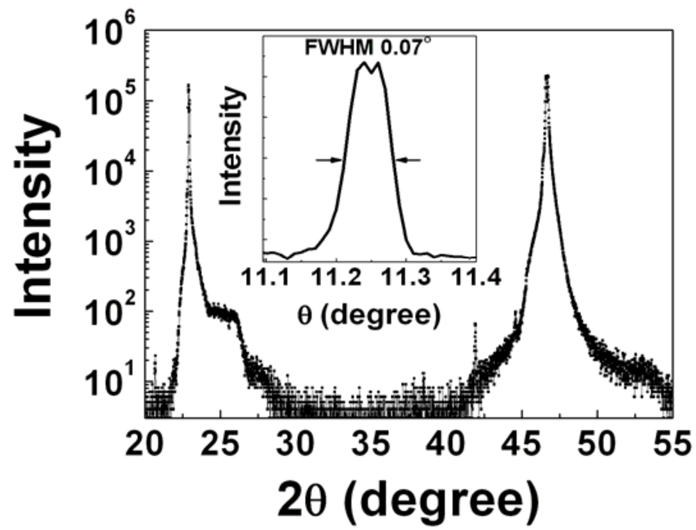
Intensity (a.u.)
2 Theata (Degree)
LBCO (001)
MgO (002)
LBCO (002)
LBCO {101}
MgO {101}
intensity
Phi (degree)



Intensity
2θ/deg
LBCO(001)
LAO(001)
LBCO(002)
LAO(002)
LAO(202)
LBCO(202)
ϕ

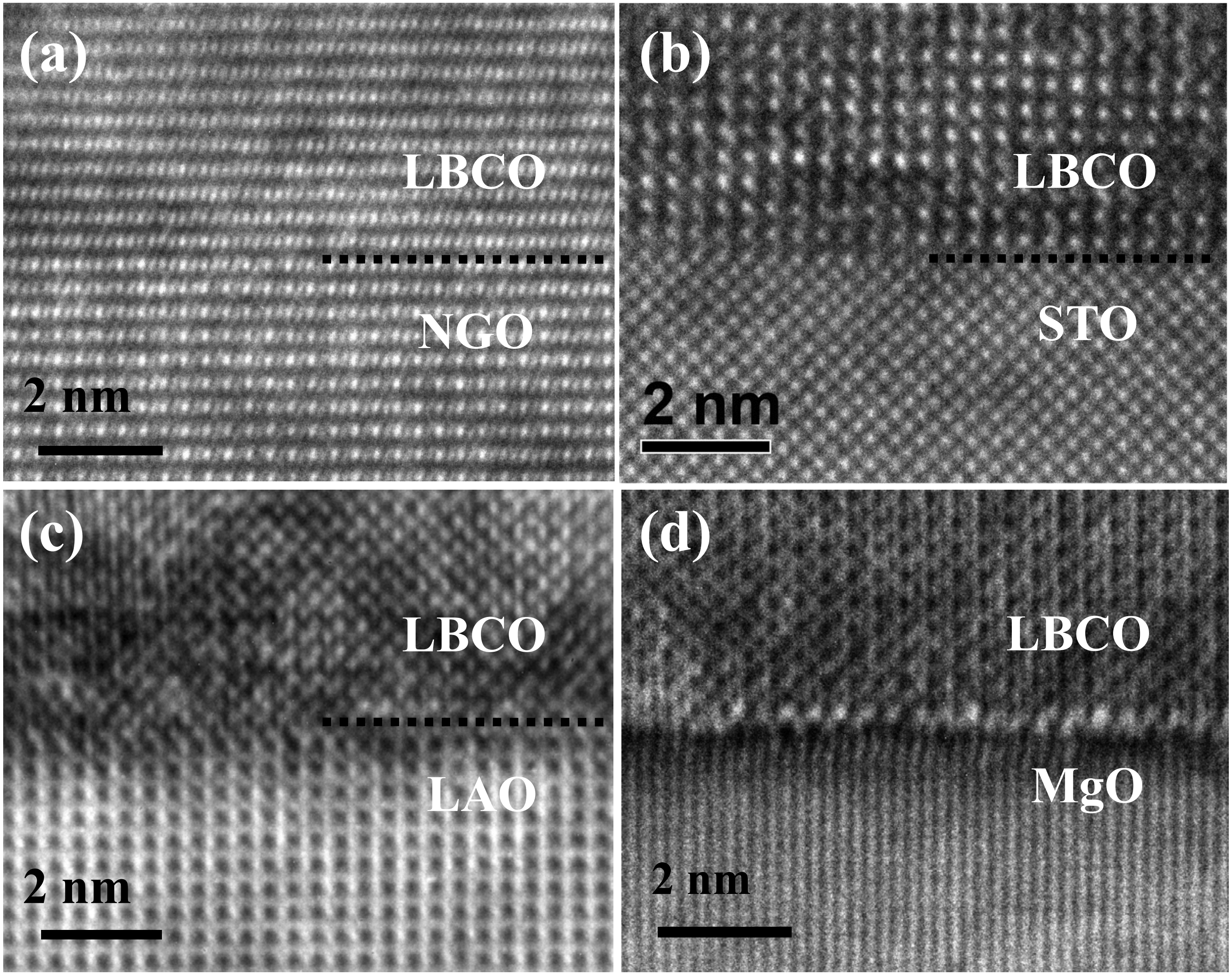


Intensity (a.u.)
2Theta (degree)
LBCO (100)
NGO (110)
LBCO (100)
NGO (220)
LBCO (100)
NGO (220)

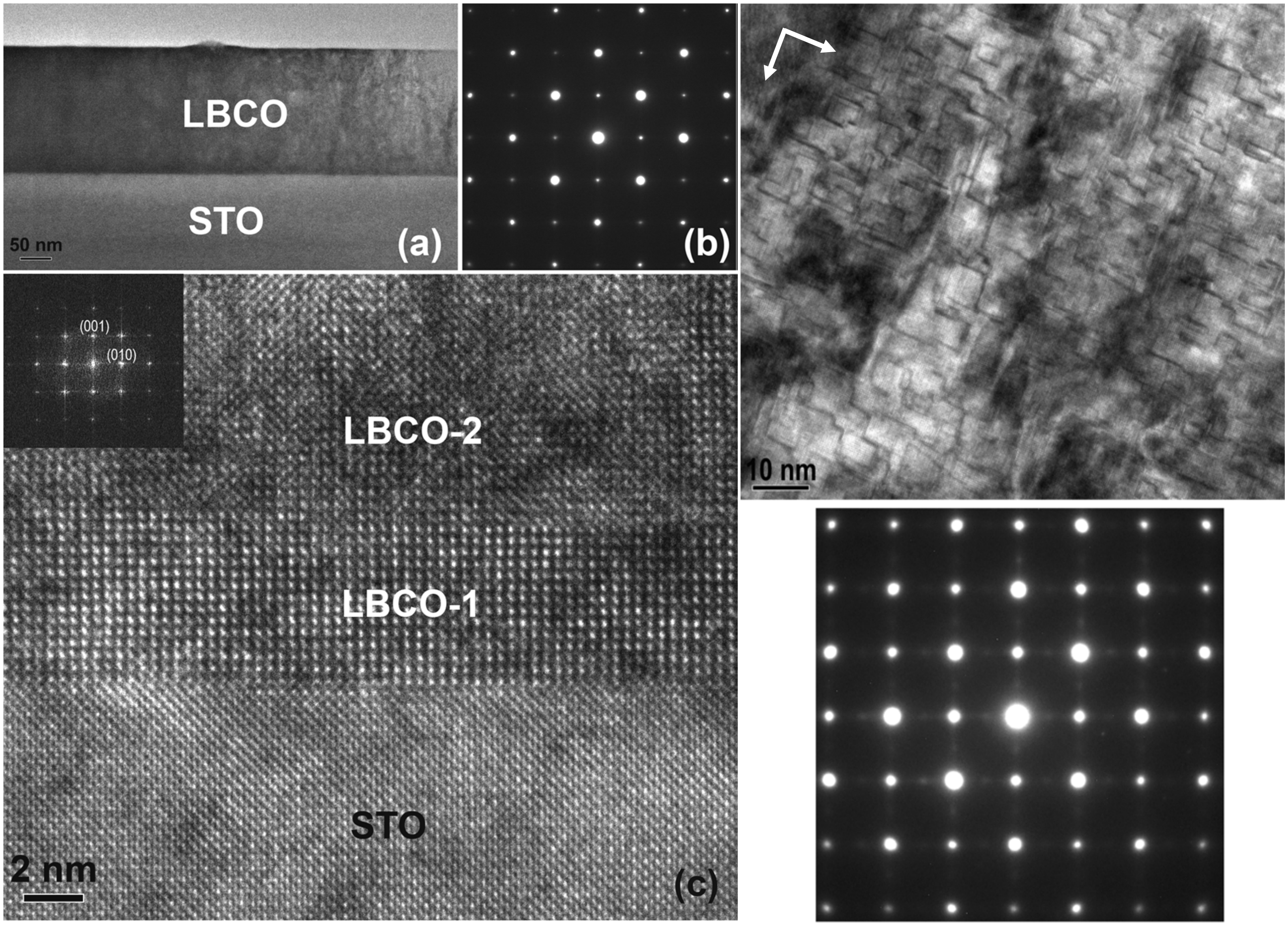


Intensity
2θ (degree)
FWHM 0.07°
Intensity
θ (degree)

Nanoscale ordered cobaltite LaBaCo2O6



Highly epitaxial nanoscale ordered cobaltite $LaBaCo_2O_6$ thin films



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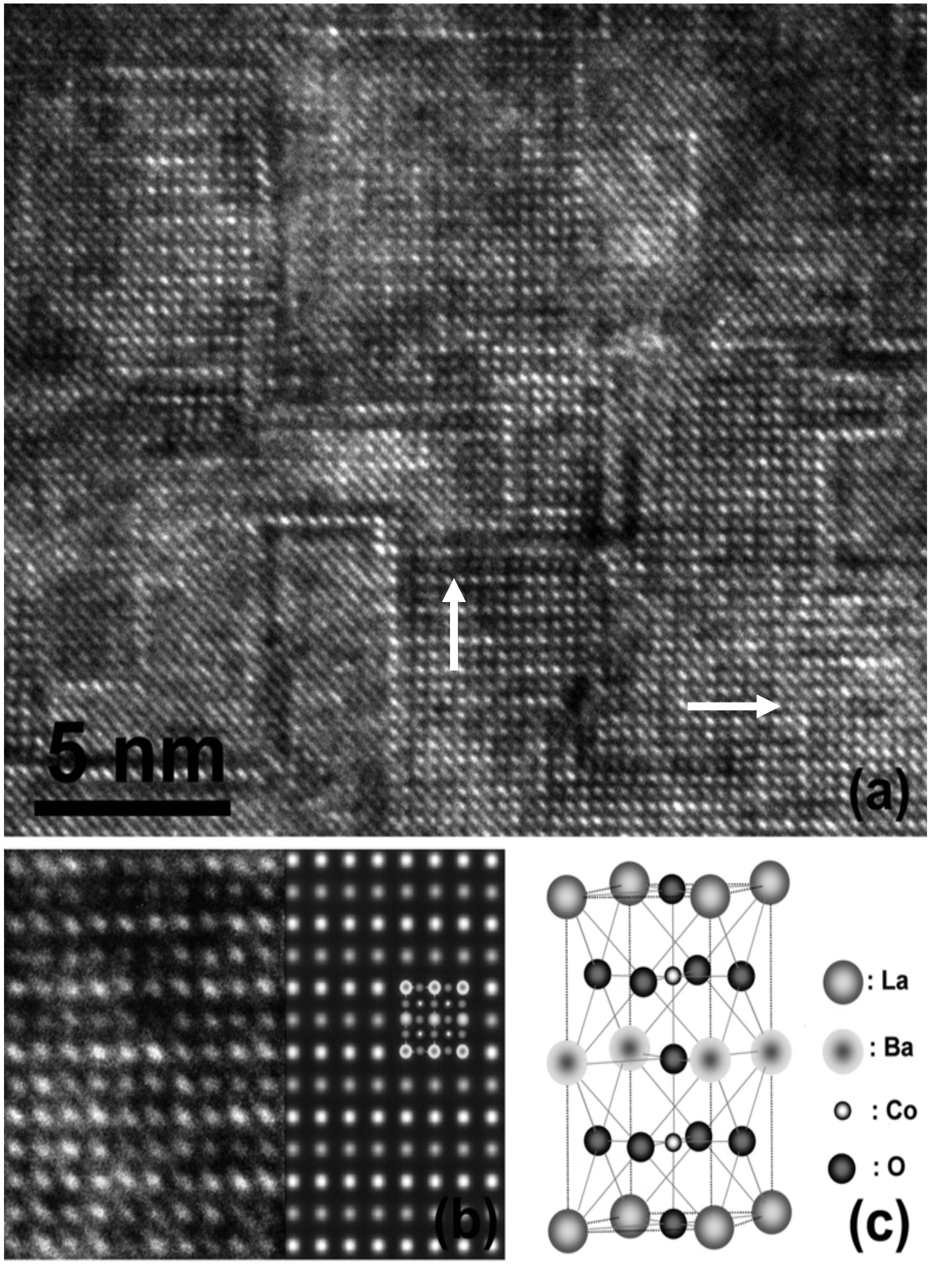
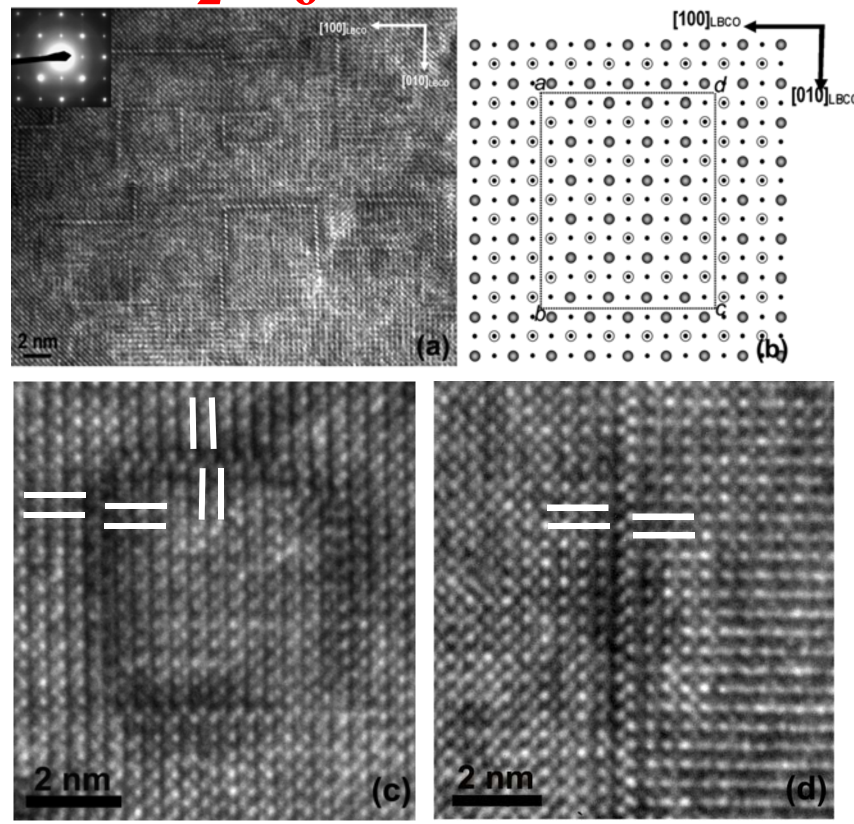
Nanoscale ordered $LaBaCo_2O_6$ thin films





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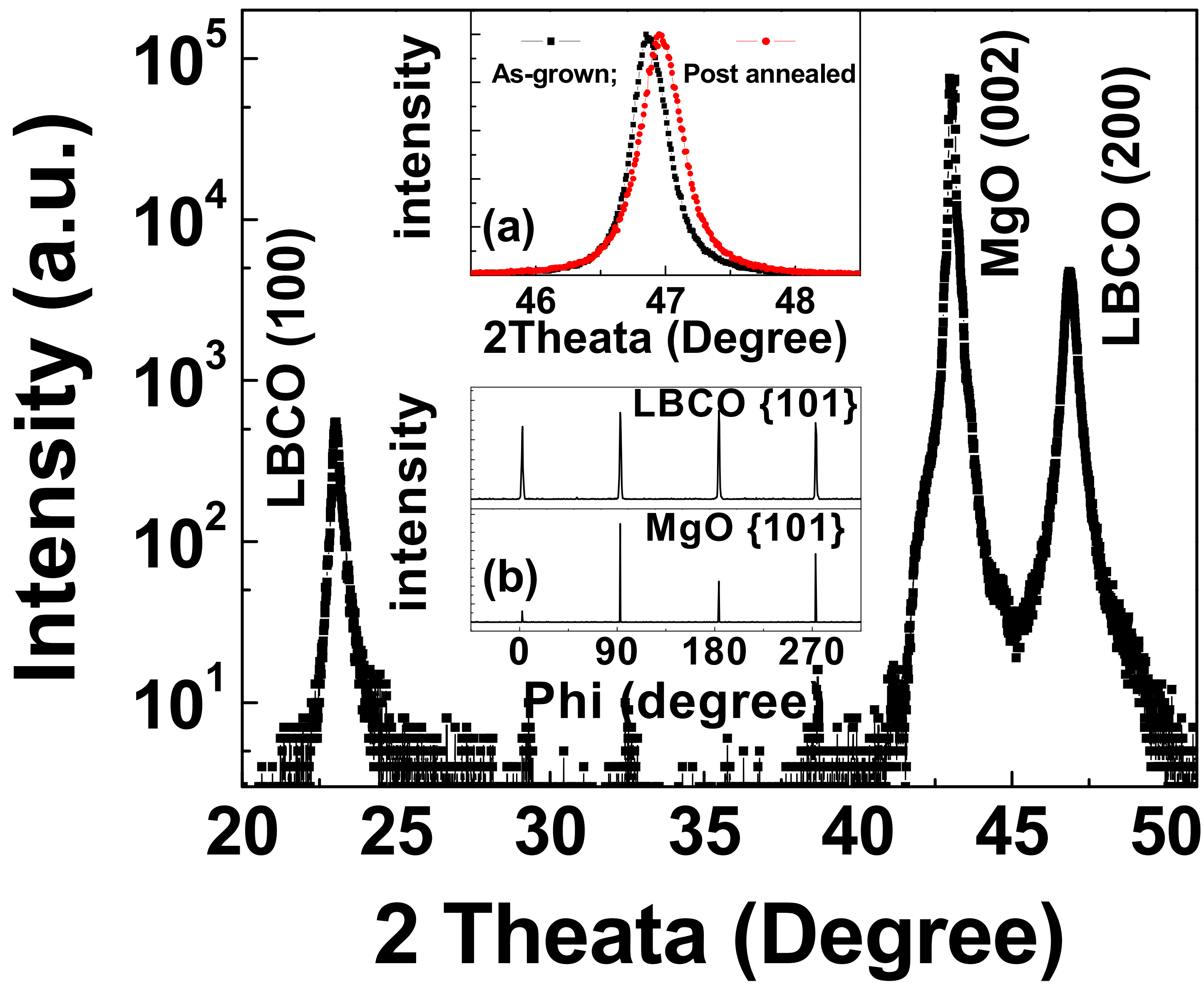
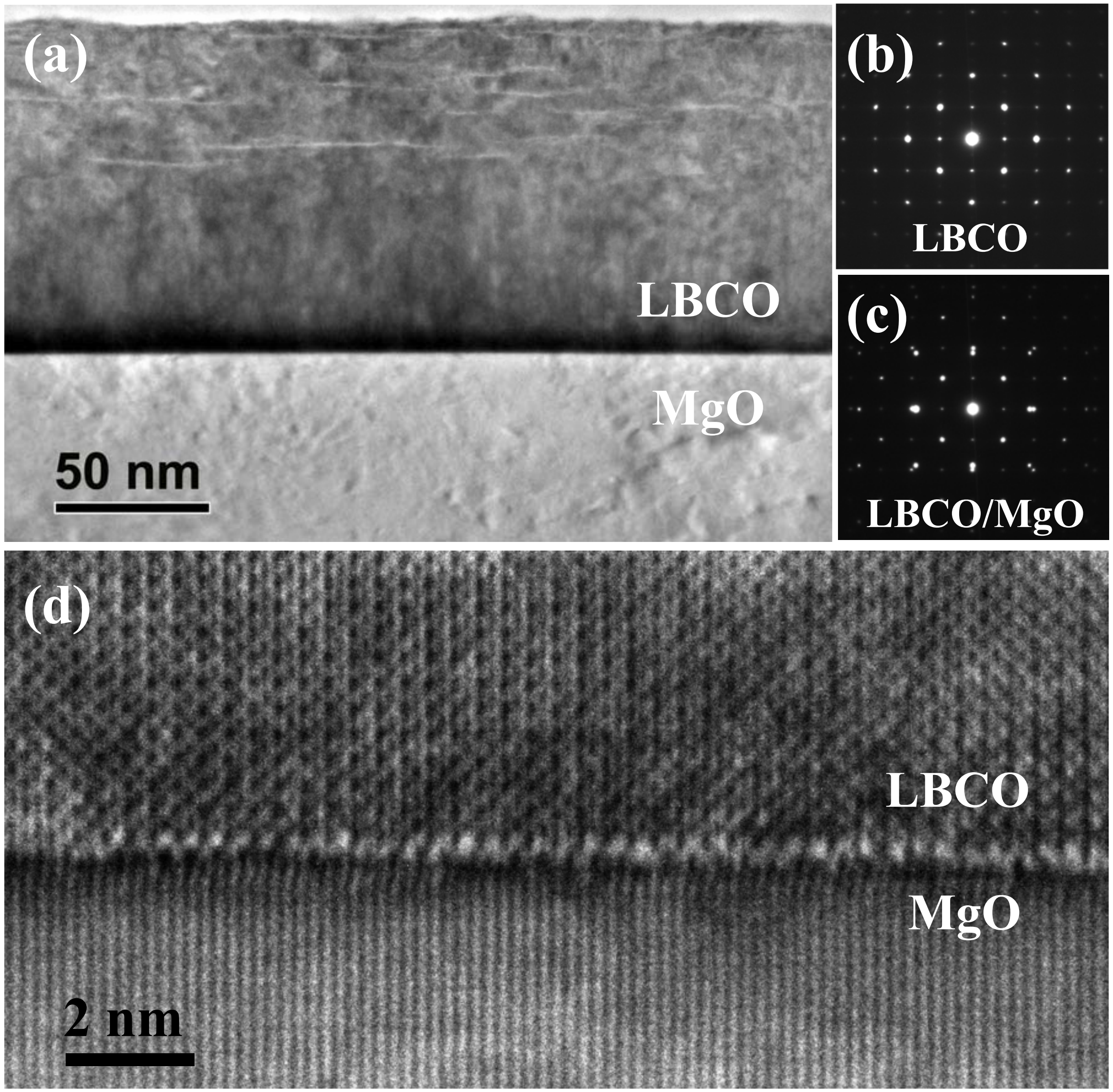
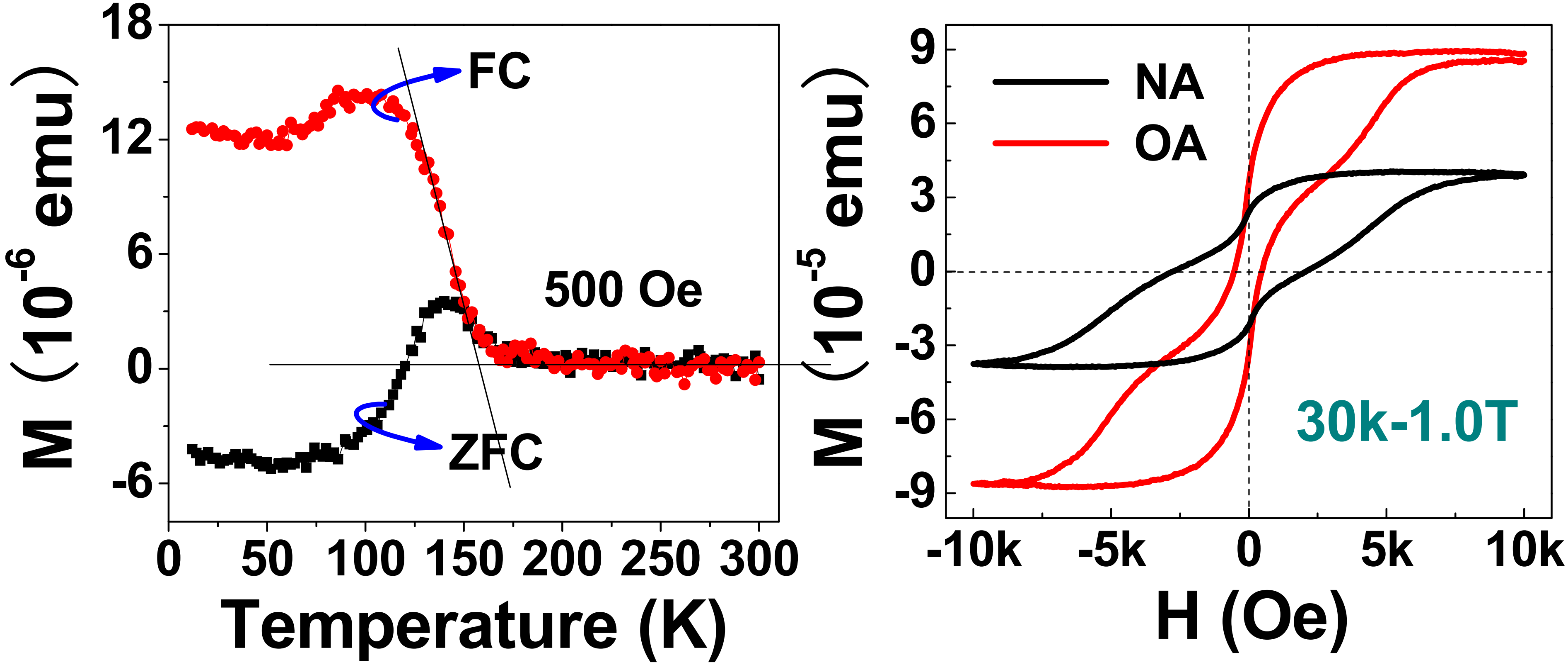
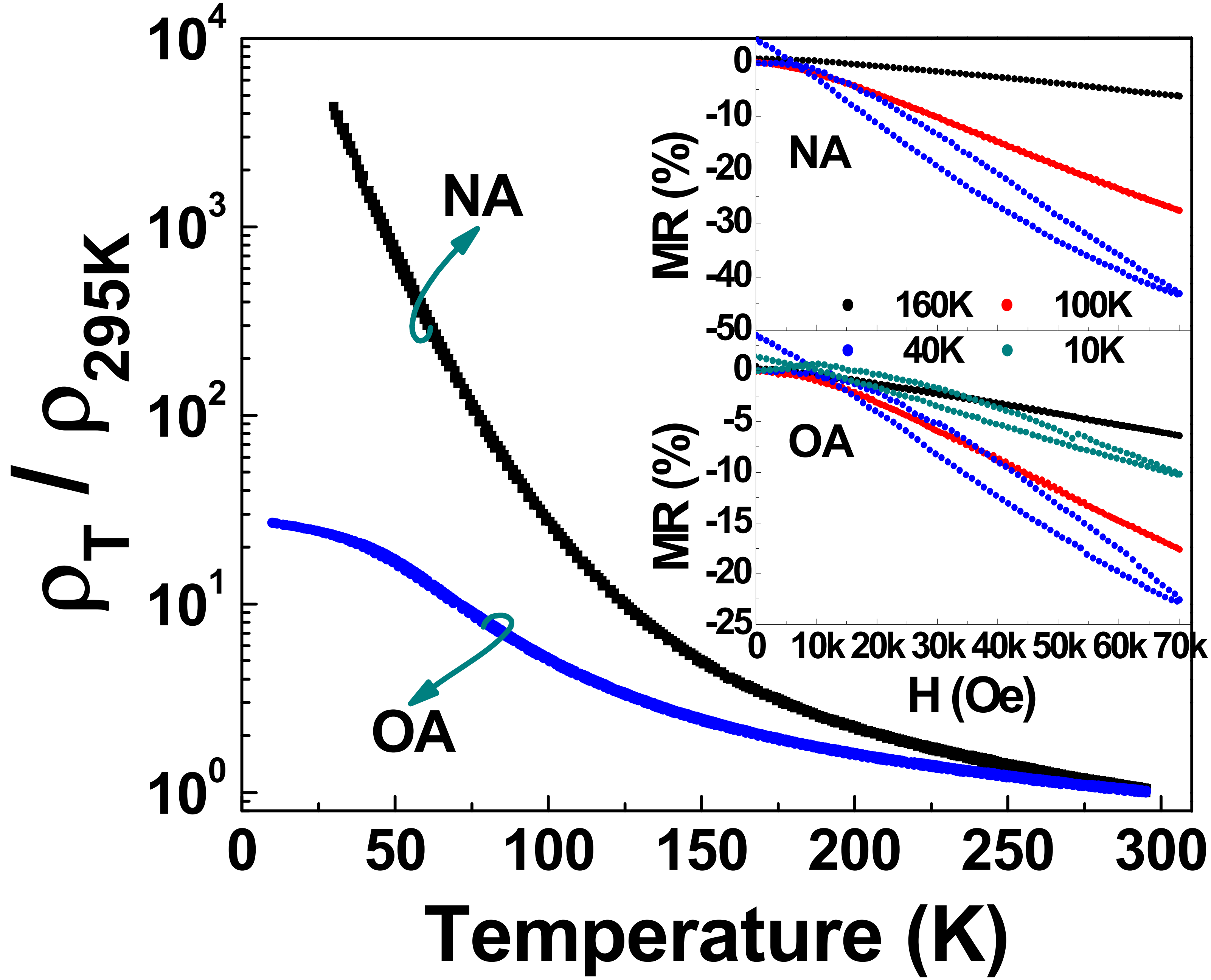


Intensity (a.u.)
10^5
10^4
10^3
10^2
10^1
LBCO (100)
MgO (002)
LBCO (200)
20
25
30
35
40
45
50
2 Theata (Degree)
intensity
As-grown;
Post annealed
(a)
46
47
48
2Theata (Degree)
intensity
LBCO {101}
MgO {101}
(b)
0
90
180
270
Phi (degree)

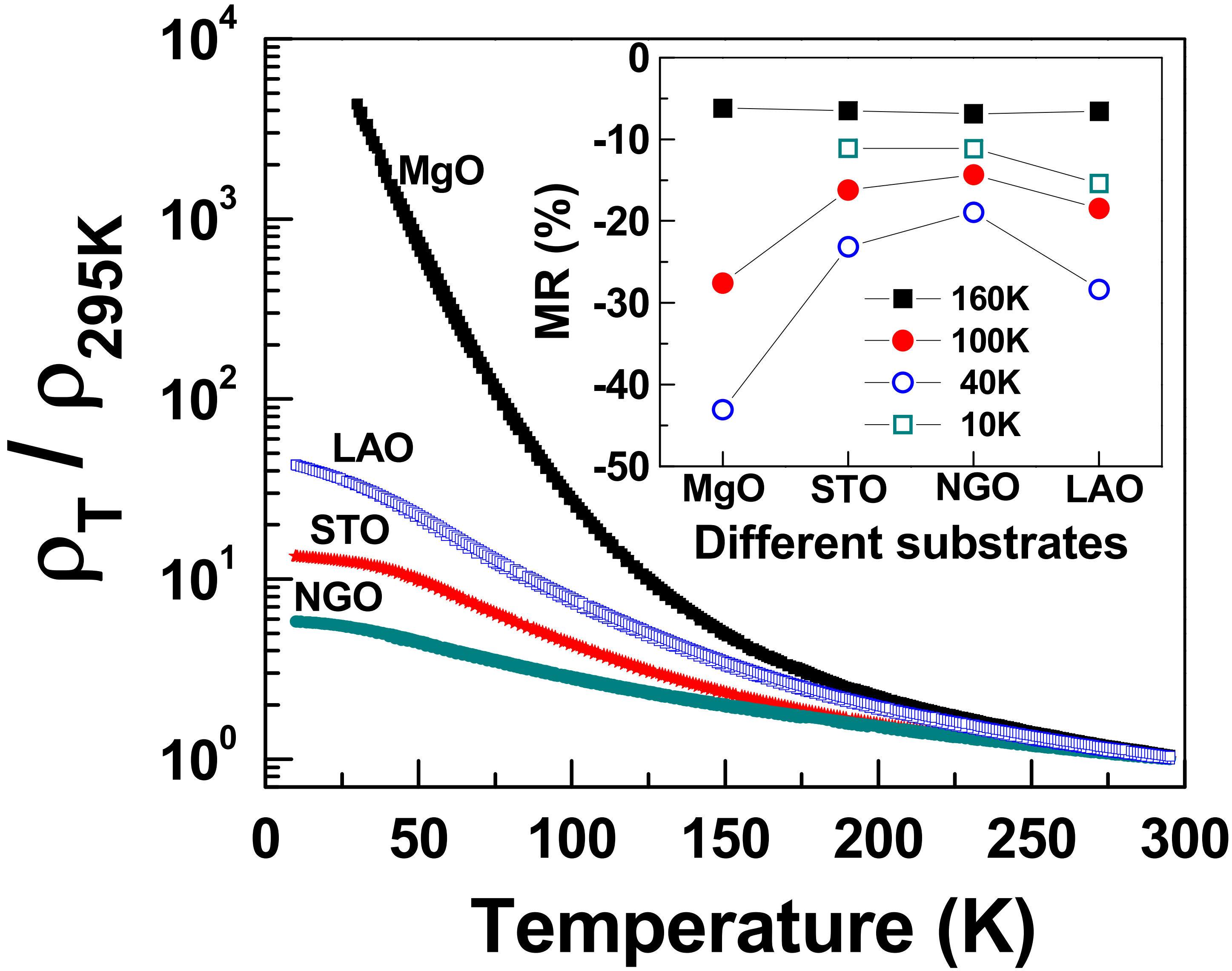

(a)
LBCO
MgO
50 nm
(b)
LBCO
(c)
LBCO/MgO
(d)
LBCO
MgO
2 nm



M (10^-6 emu)
18
12
6
0
-6
FC
ZFC
500 Oe
0
50
100
150
200
250
300
Temperature (K)
M (10^-5 emu)
9
6
3
0
-3
-6
-9
NA
OA
30k-1.0T
-10k
-5k
0
5k
10k
H (Oe)

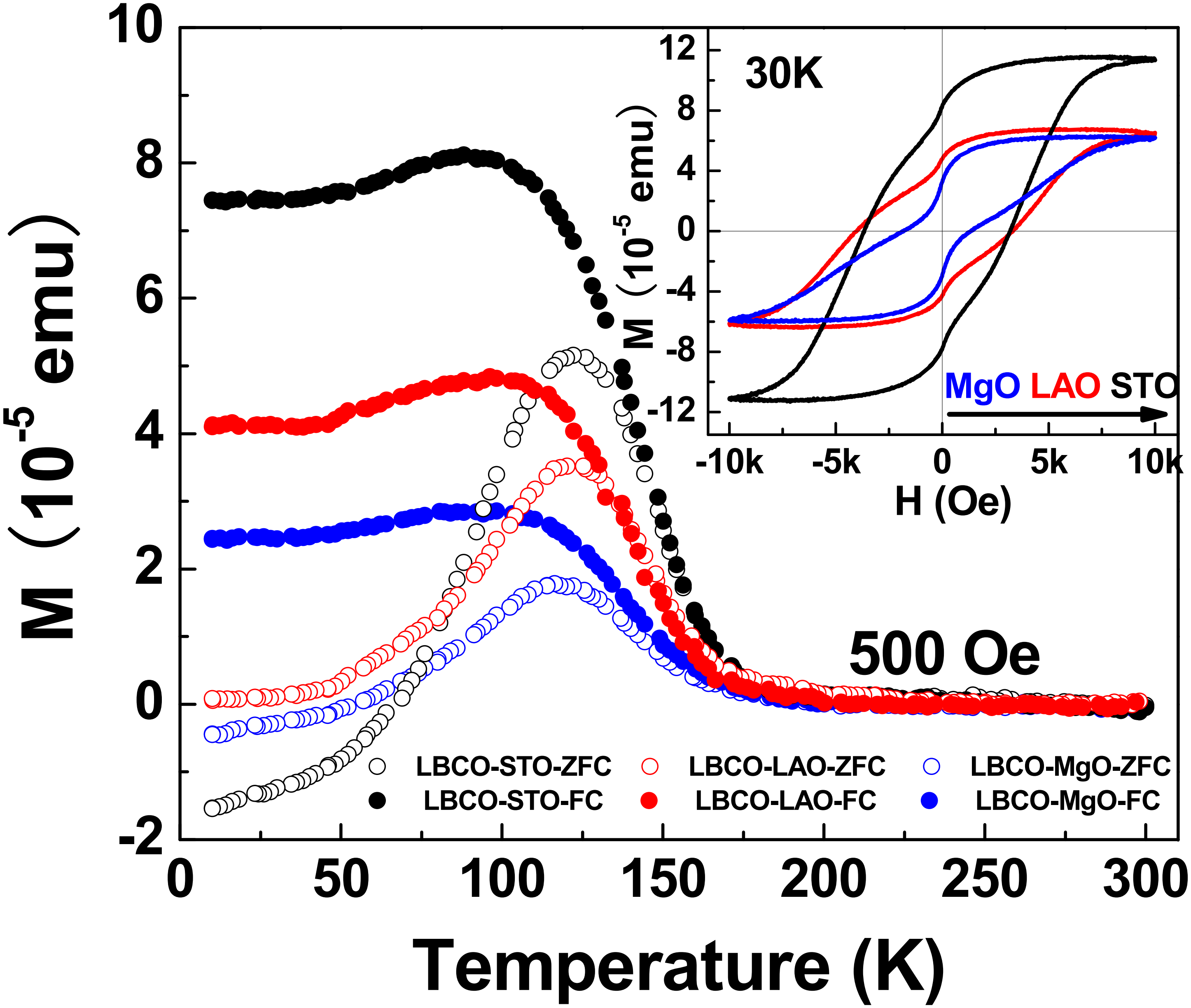


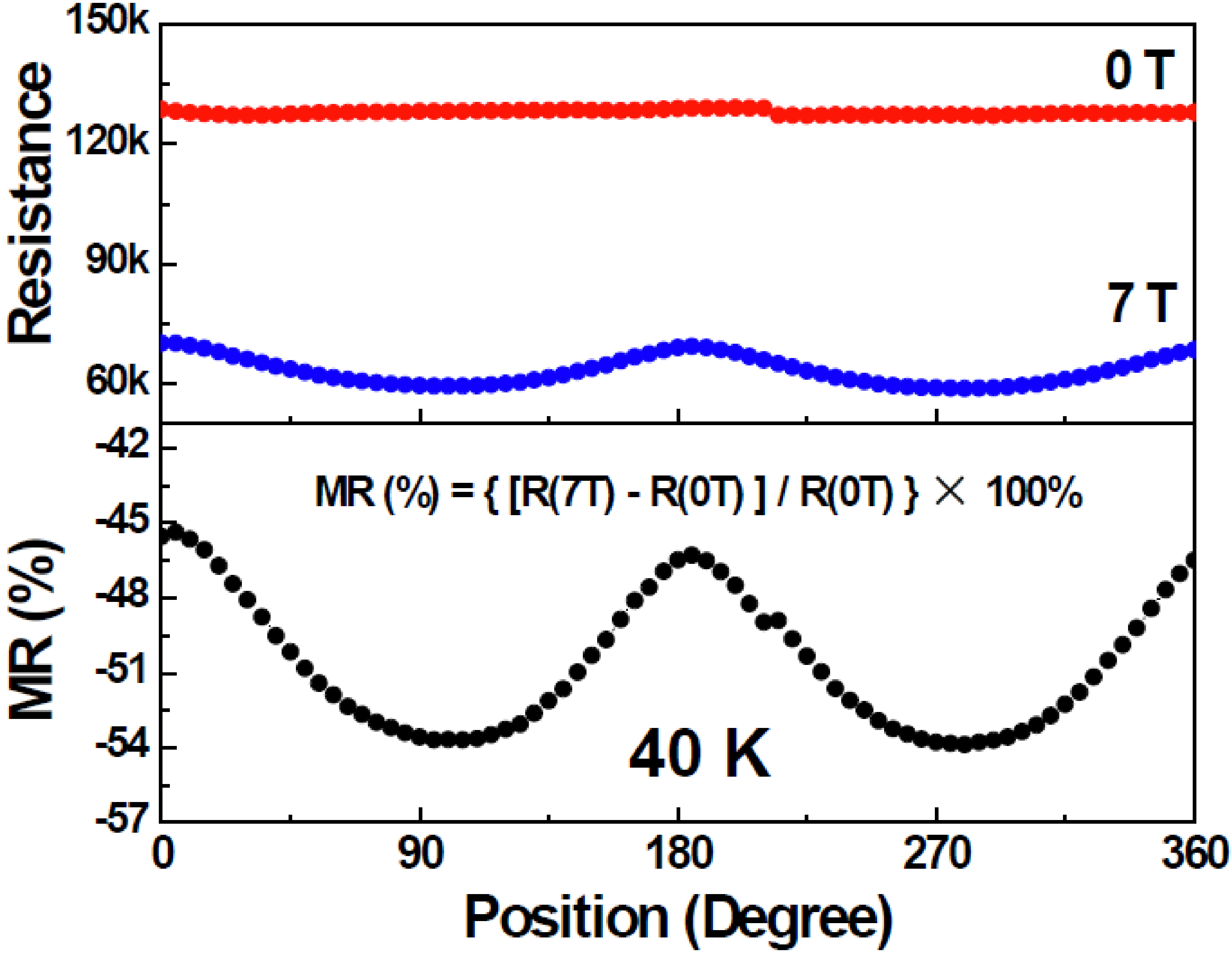
ρ_T / ρ_{295K}
Temperature (K)
NA
OA
MR (%)
H (Oe)
160K
100K
40K
10K



MgO
LAO
STO
NGO
0
-10
-20
-30
-40
-50
MR (%)
160K
100K
40K
10K
MgO
STO
NGO
LAO
Different substrates
10^4
10^3
10^2
10^1
10^0
ρ_T / ρ_{295K}
0
50
100
150
200
250
300
Temperature (K)

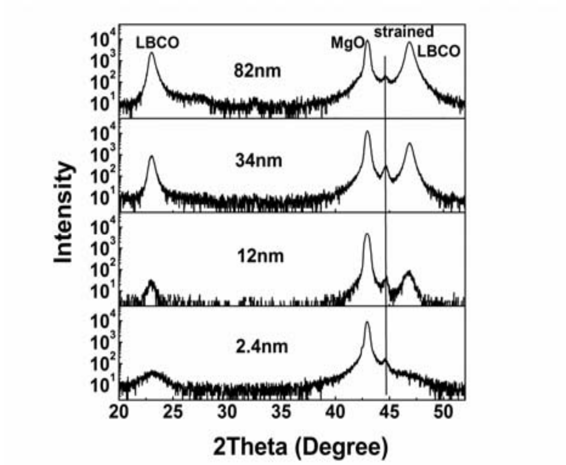
Substrate-induced Strain on Transport Behavior and Magnetic Properties of Highly Epitaxial $(LaBa)Co_2O_{5.5+\delta}$ Thin Films

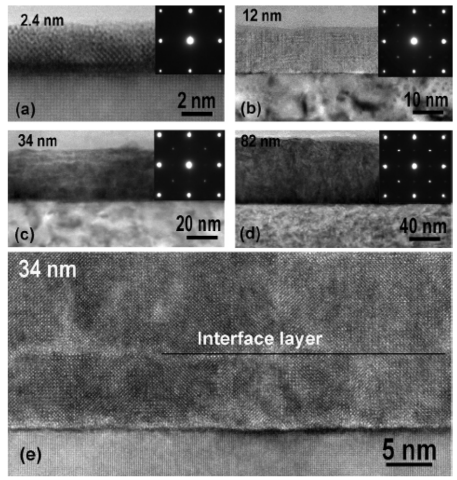


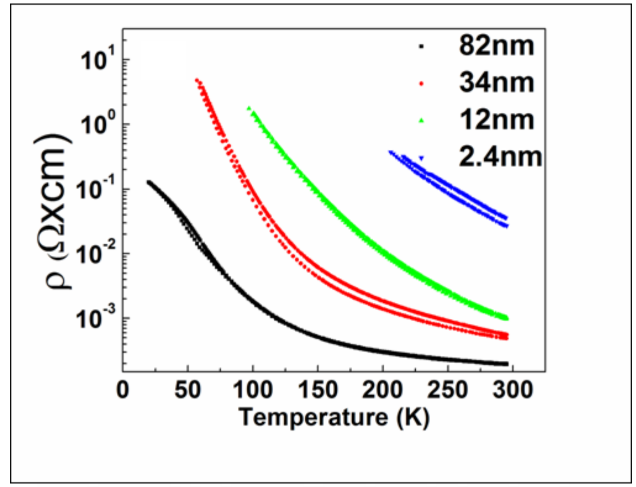


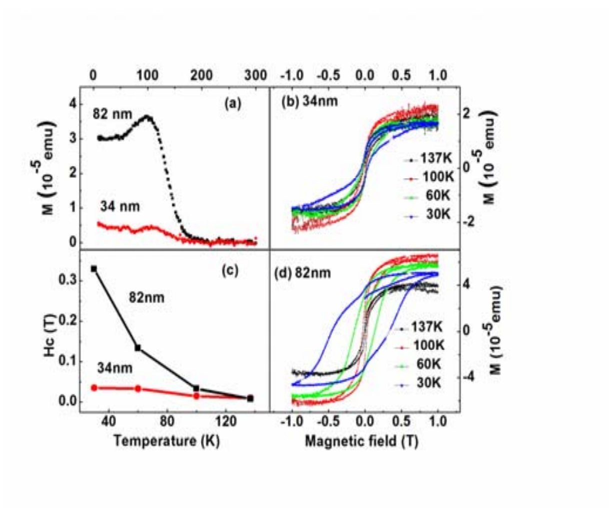
150k
120k
90k
60k
Resistance
0 T
7 T
-42
-45
-48
-51
-54
-57
MR (%)
MR (%) = { [R(7T) - R(0T)] / R(0T) } × 100%
40 K
0
90
180
270
360
Position (Degree)

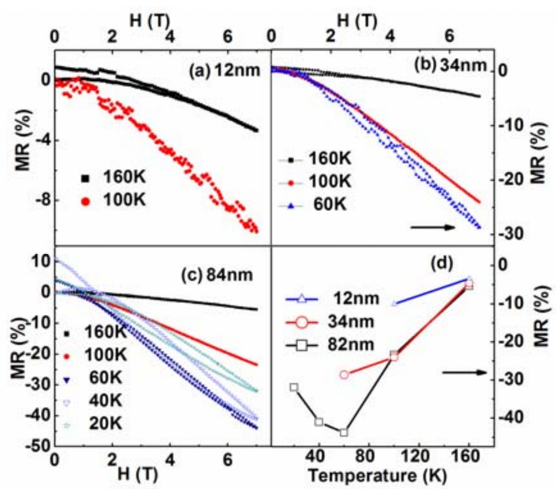
Strain from Various Film Thickness

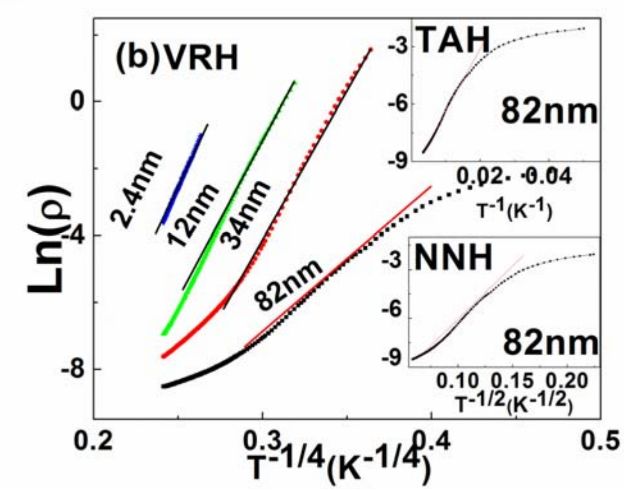




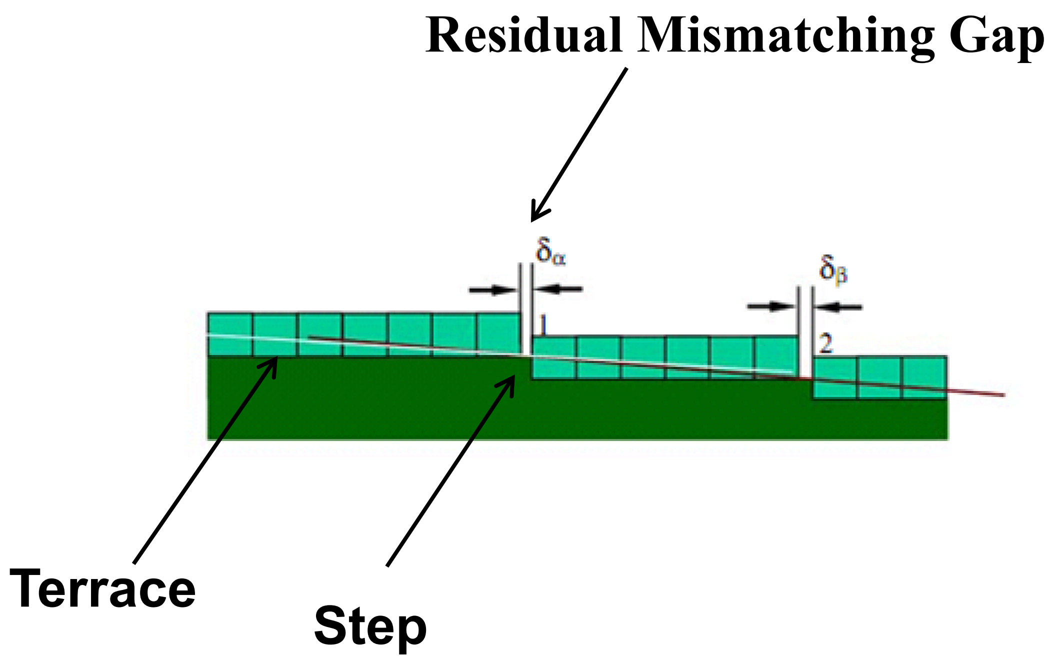




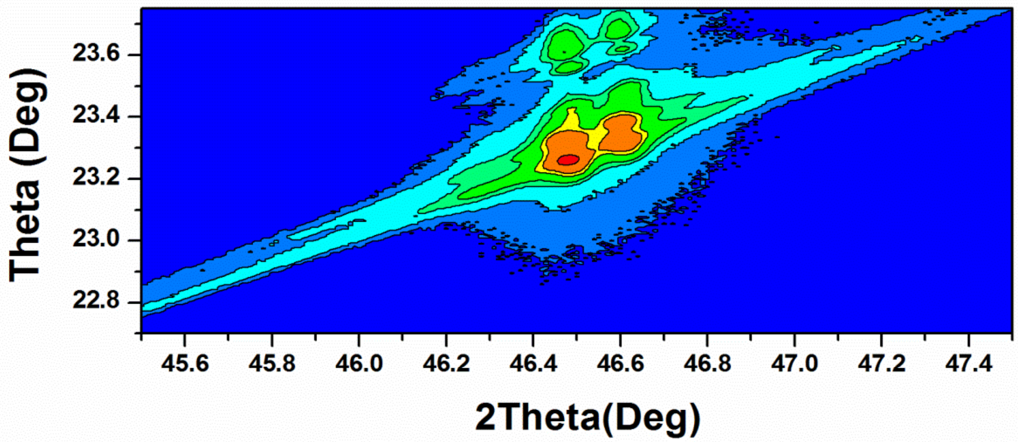




Interface Strain From Vicinal substrate



X-ray Characterization of LBCO on Vicinal Substrate



Substrate FWHM: 0.04°

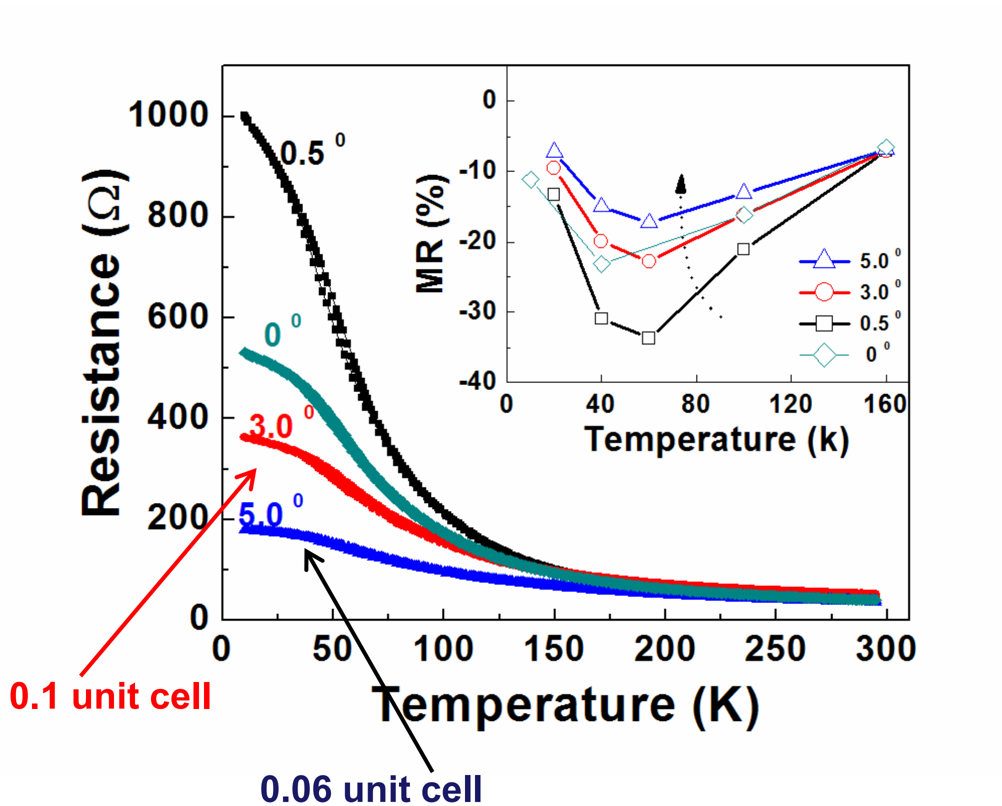
Film FWHM: 0.05°

Good Epitaxial Quality

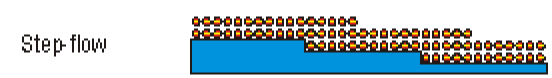
The average width of the substrate terrace in the epitaxial LBCO thin films grown on the vicinal (001) $SrTiO_3$, and how many unit cells of the substrate $SrTiO_3$ and LaBCO (n_s and n_f, respectively) that the average terrace can accommodate

Specimen	Terrace width (nm)	N_s	N_f	
0.5^0	44.748	114.6	115.16	0.5 unit cell
3^0	7.458	19.1	19.2	0.1 unit cell
5^0	4.474	11.46	11.52	0.06 unit cell

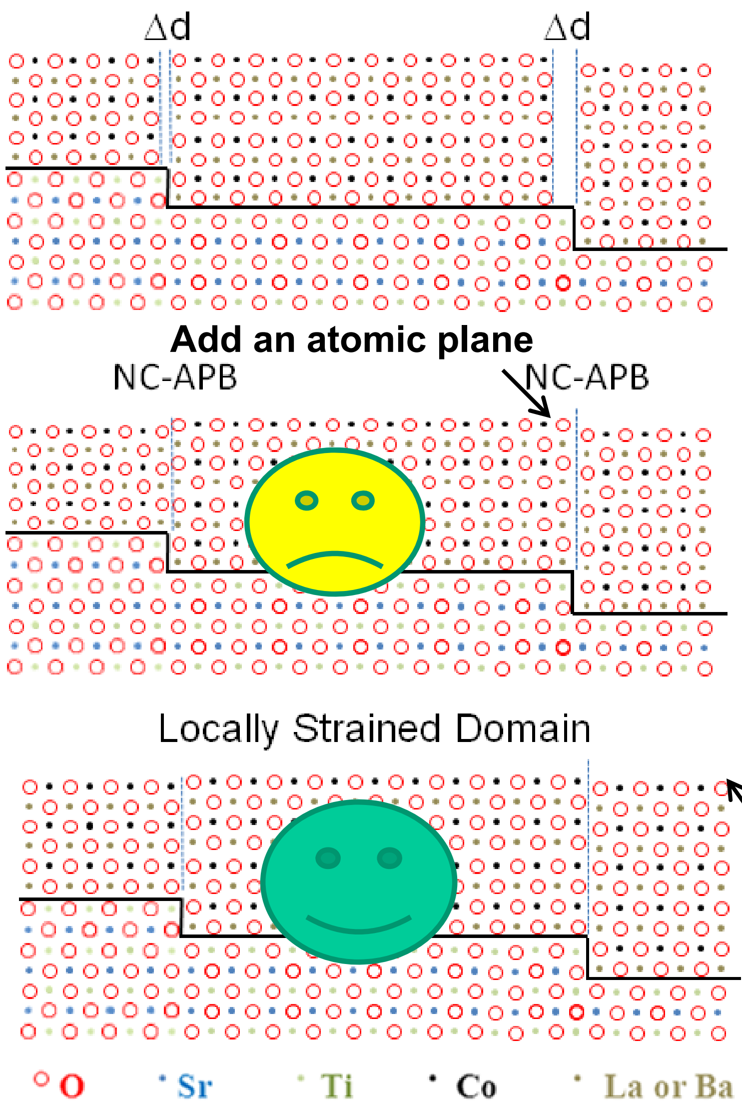
Transport Properties of LBCO Thin Film on Vicinal Substrate



The terrace width is narrower and smaller than the mean surface atom diffusion length at the growth temperature. with increasing the miscut angle, the film growth will follow the step-flow mechanism resulting in the single domain formation. The single-domain growth and small lattice-misfit strain will reduce the scattering rate of the carriers with strain lattice and domain boundaries.



Local strain from terrace is very small



~~Residual Mismatching Gap~~

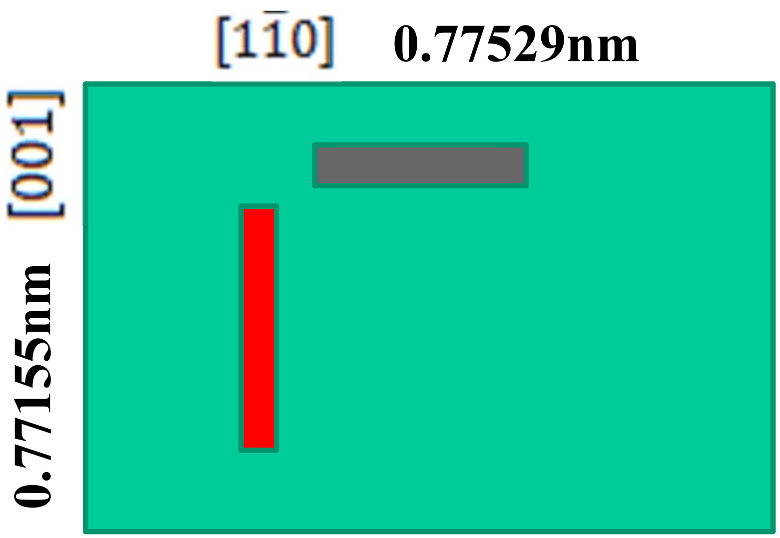
The last atomic plane of the film will always occupy the terrace end by rearranging the local atomic structure

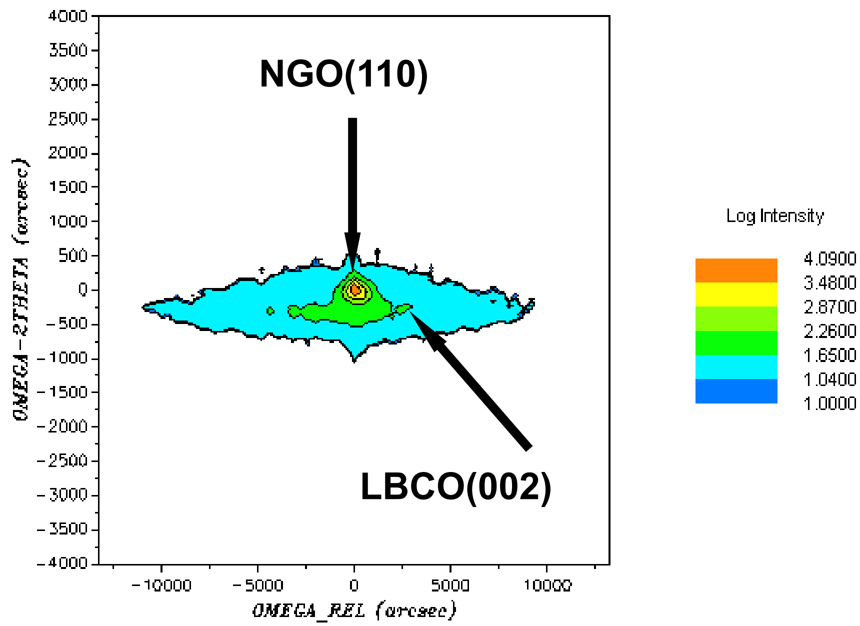
a huge amount of interface strain energy will be generated due to the same charge repulsion at the domain boundary and interface

Tensile strain in plane, compressive strain out of plane

locally-strained domain will enhance strong transport electron scattering behavior, inducing the higher resistance

Interface Strain from Different Direction of the Same Substrate





[001] 1.09% "1"

$[1\bar{1}0]$ 0.6% "2"

Compressive Strain

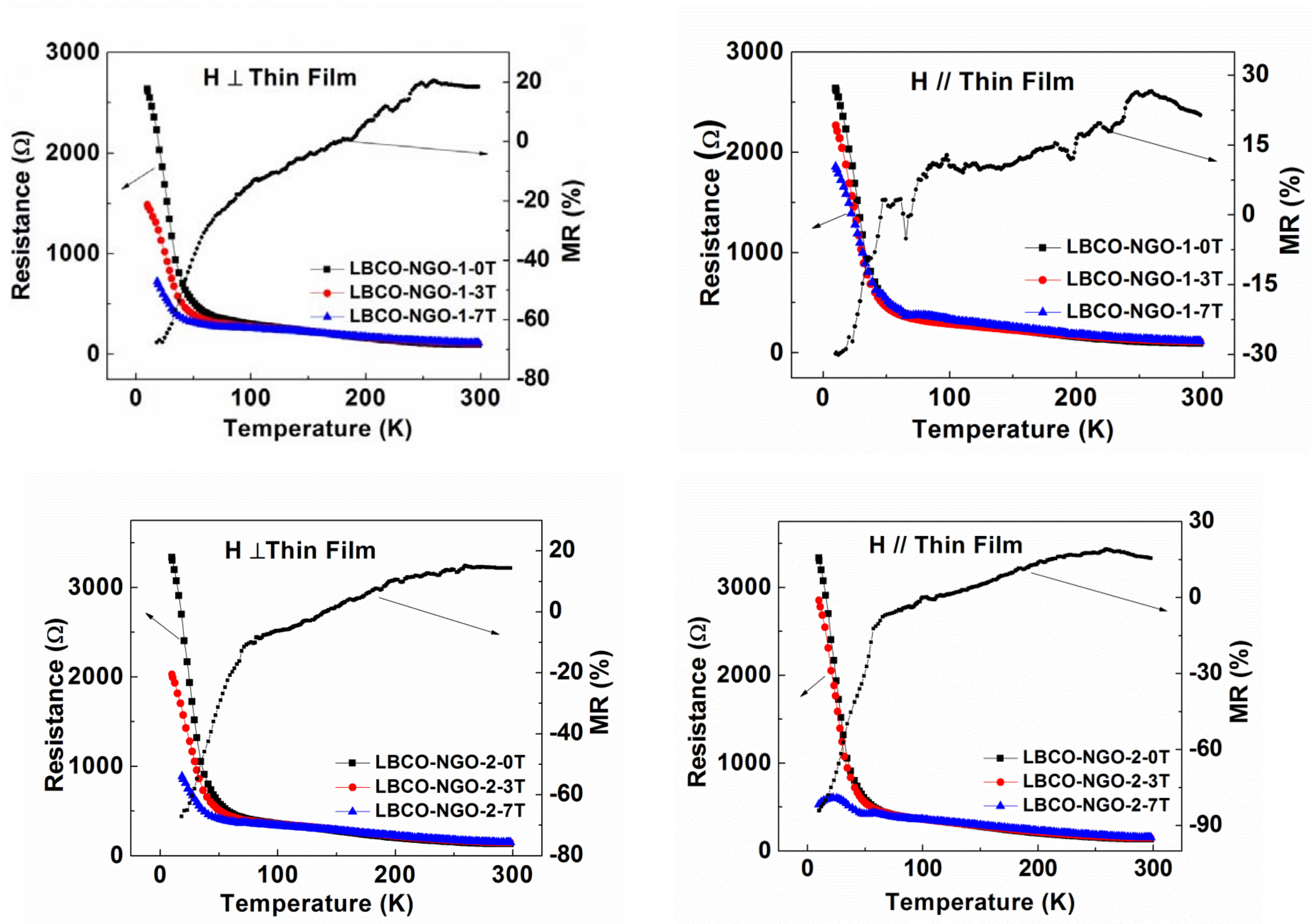
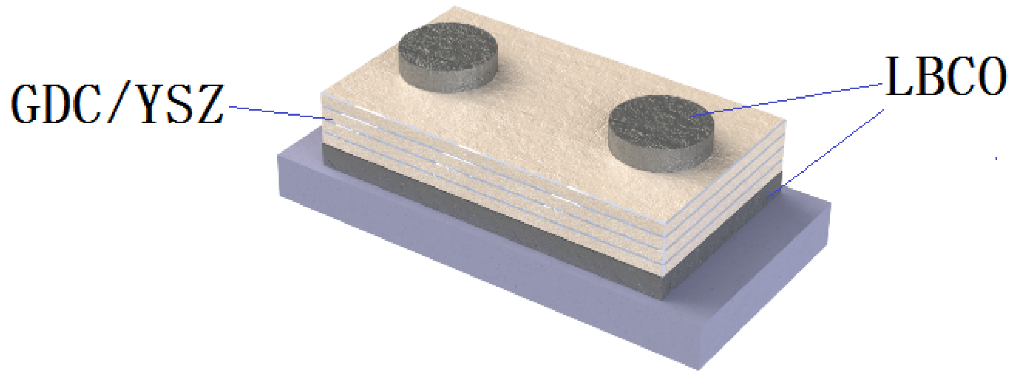
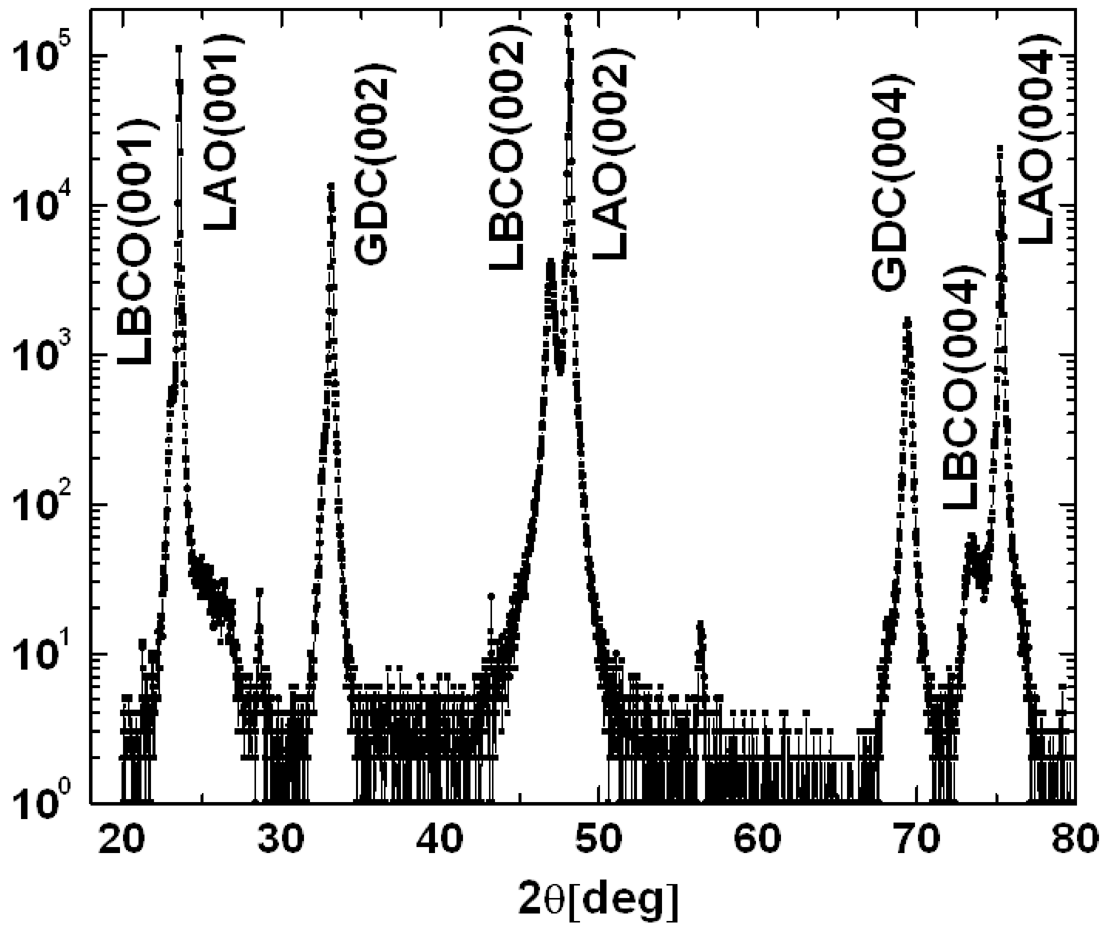

H ⊥ Thin Film
Resistance (Ω)
MR (%)
Temperature (K)
LBCO-NGO-1-0T
LBCO-NGO-1-3T
LBCO-NGO-1-7T
H // Thin Film
Resistance (Ω)
MR (%)
Temperature (K)
LBCO-NGO-1-0T
LBCO-NGO-1-3T
LBCO-NGO-1-7T
H ⊥Thin Film
Resistance (Ω)
MR (%)
Temperature (K)
LBCO-NGO-2-0T
LBCO-NGO-2-3T
LBCO-NGO-2-7T
H // Thin Film
Resistance (Ω)
MR (%)
Temperature (K)
LBCO-NGO-2-0T
LBCO-NGO-2-3T
LBCO-NGO-2-7T

fig 1

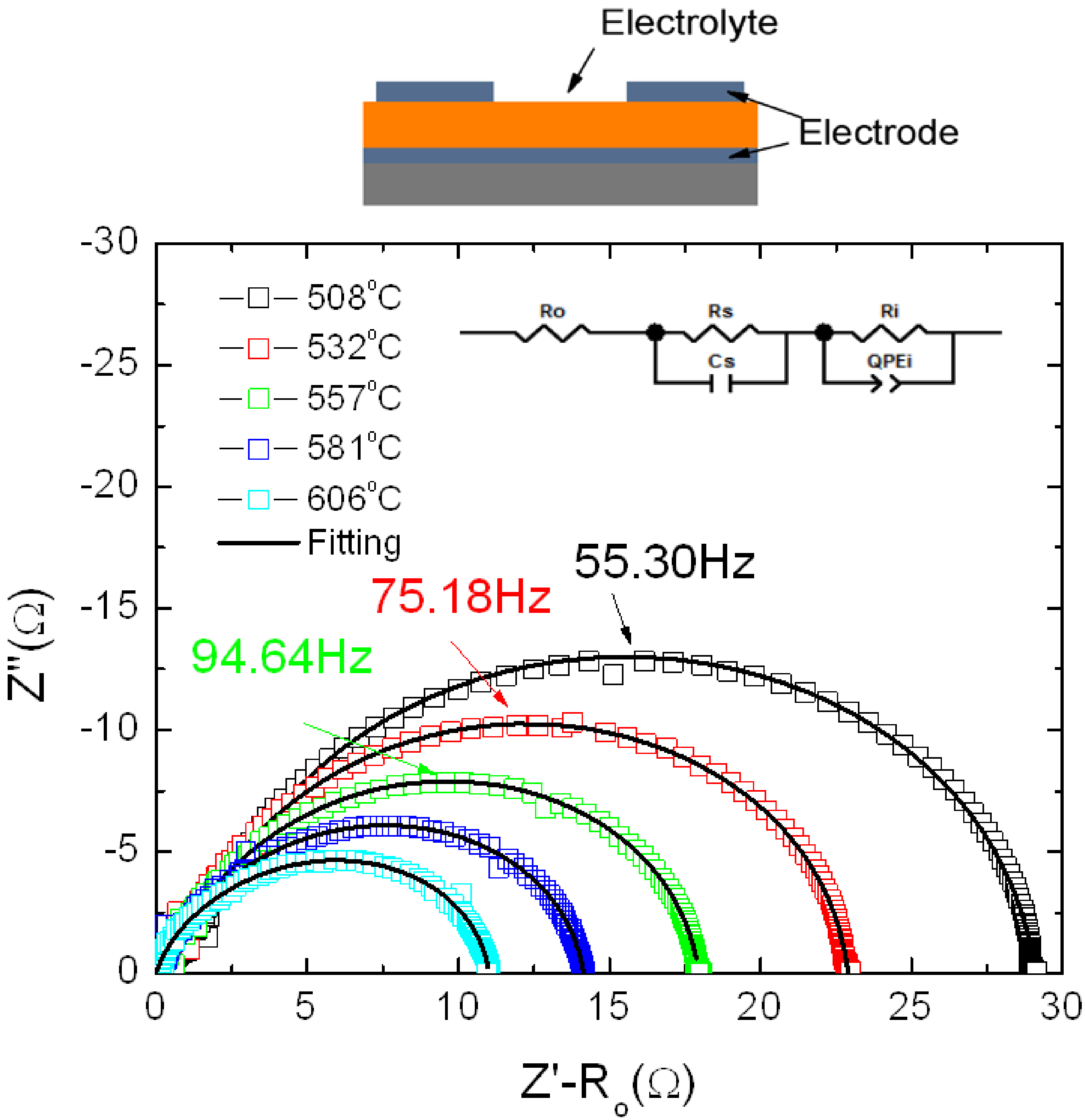


GDC/YSZ
LBCO
a



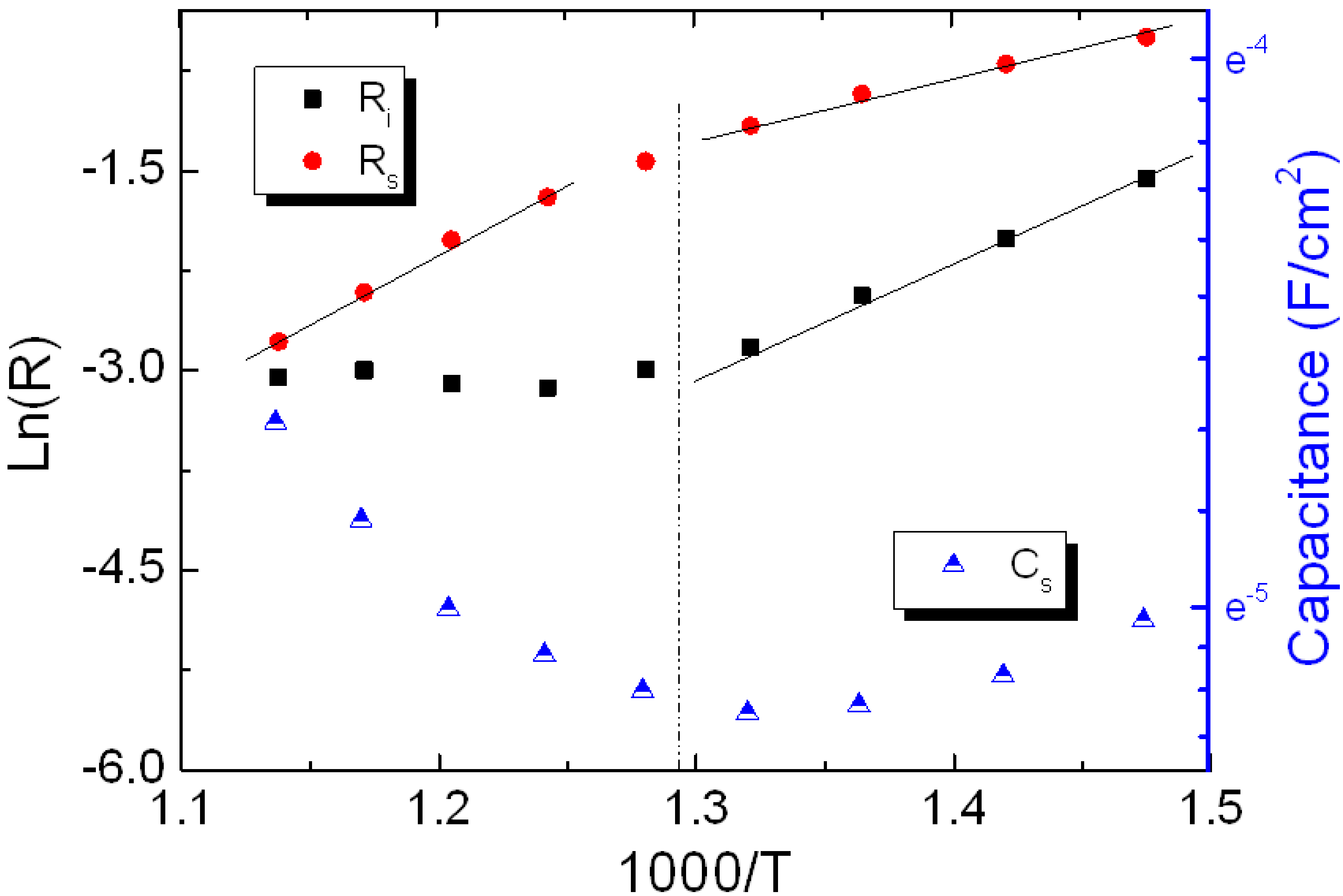
LBCO(001)
LAO(001)
GDC(002)
LBCO(002)
LAO(002)
GDC(004)
LBCO(004)
LAO(004)
10^5
10^4
10^3
10^2
10^1
10^0
20
30
40
50
60
70
80
2θ[deg]
b

fig 2 a



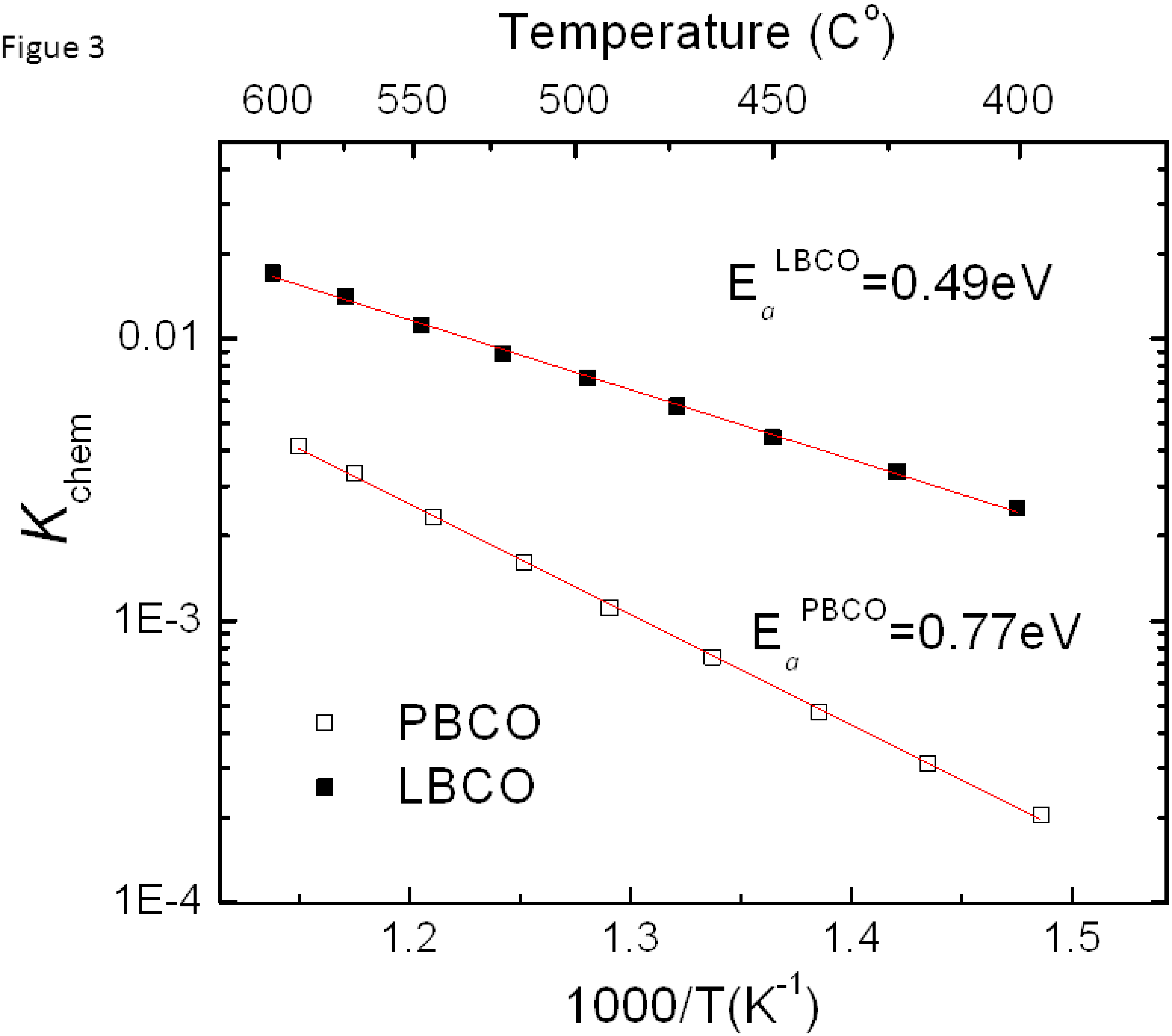
Electrolyte
Electrode
-30
-25
-20
-15
-10
-5
0
Z''(Ω)
508°C
532°C
557°C
581°C
606°C
Fitting
Ro
Rs
Ri
Cs
QPEi
55.30Hz
75.18Hz
94.64Hz
0
5
10
15
20
25
30
Z'-Ro(Ω)

Figur2 b



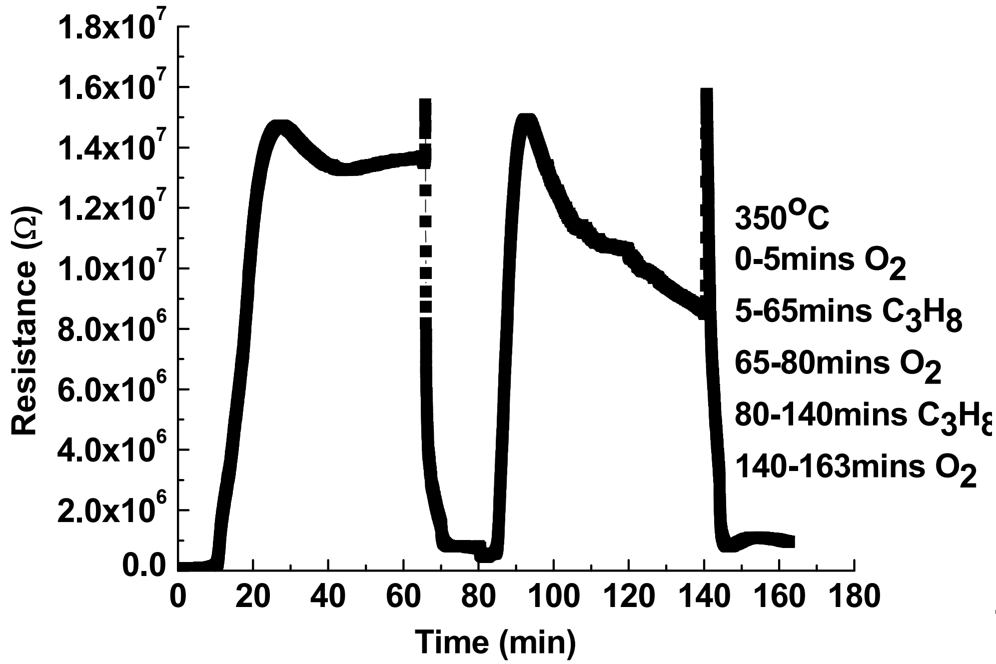
R_i
R_s
C_s
-1.5
-3.0
-4.5
-6.0
Ln(R)
1.1
1.2
1.3
1.4
1.5
1000/T
e^-4
e^-5
Capacitance (F/cm^2)

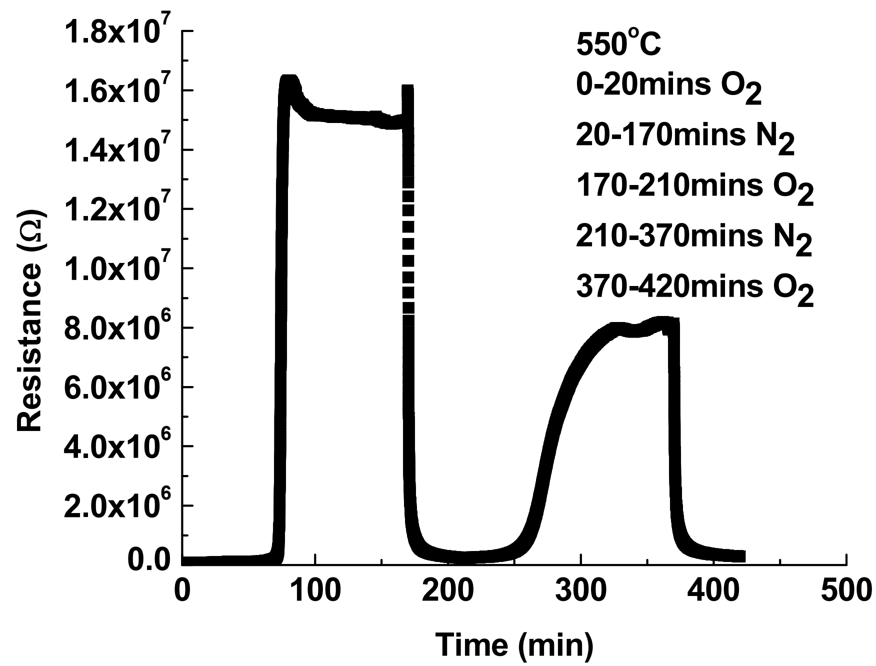
Figue 3

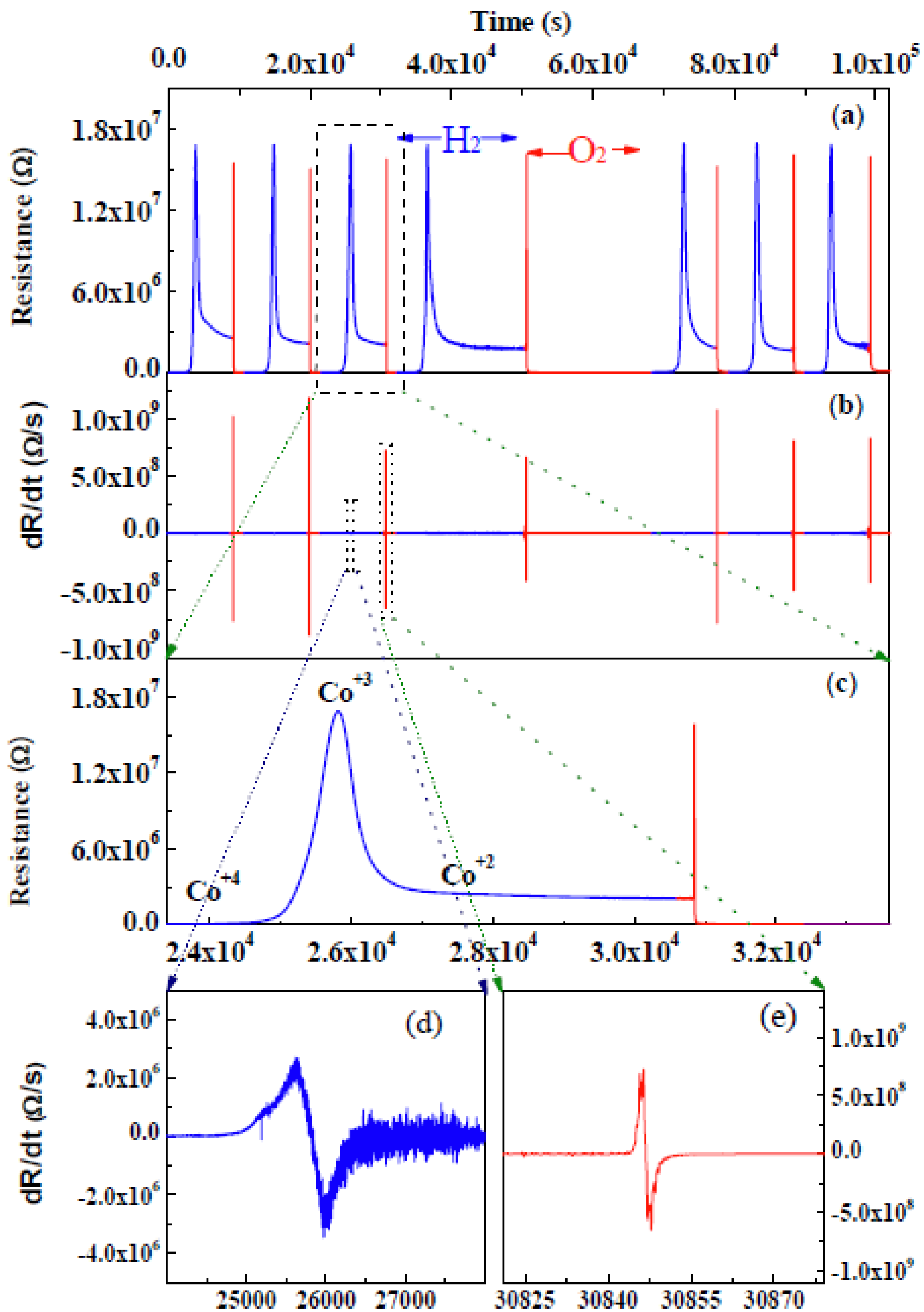


Temperature (C°)
600
550
500
450
400
0.01
1E-3
1E-4
K_{chem}
E_a^{LBCO}=0.49eV
E_a^{PBCO}=0.77eV
PBCO
LBCO
1.2
1.3
1.4
1.5
1000/T(K^{-1})

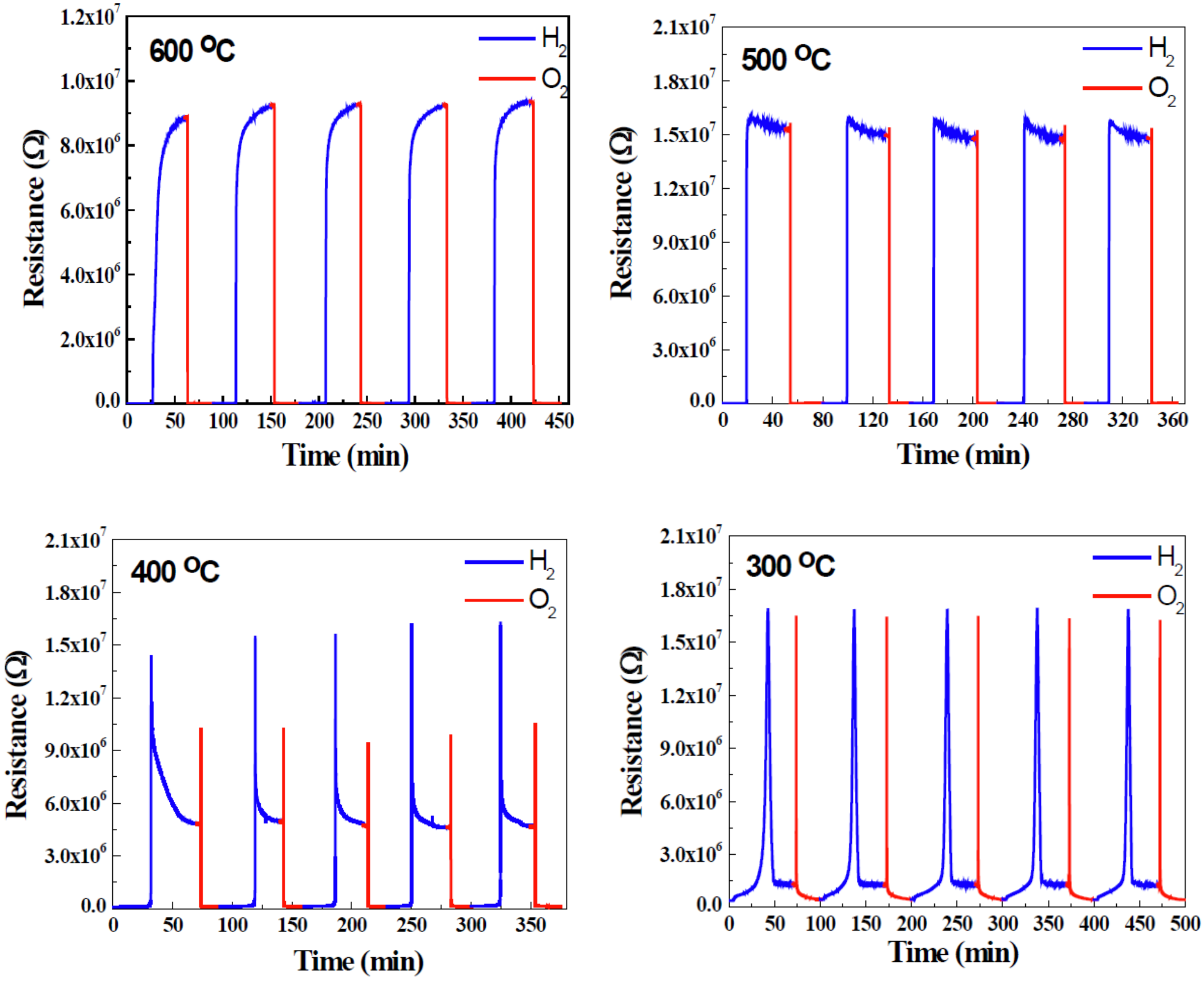
LBCO Films in Other O_2/Fuel Systems



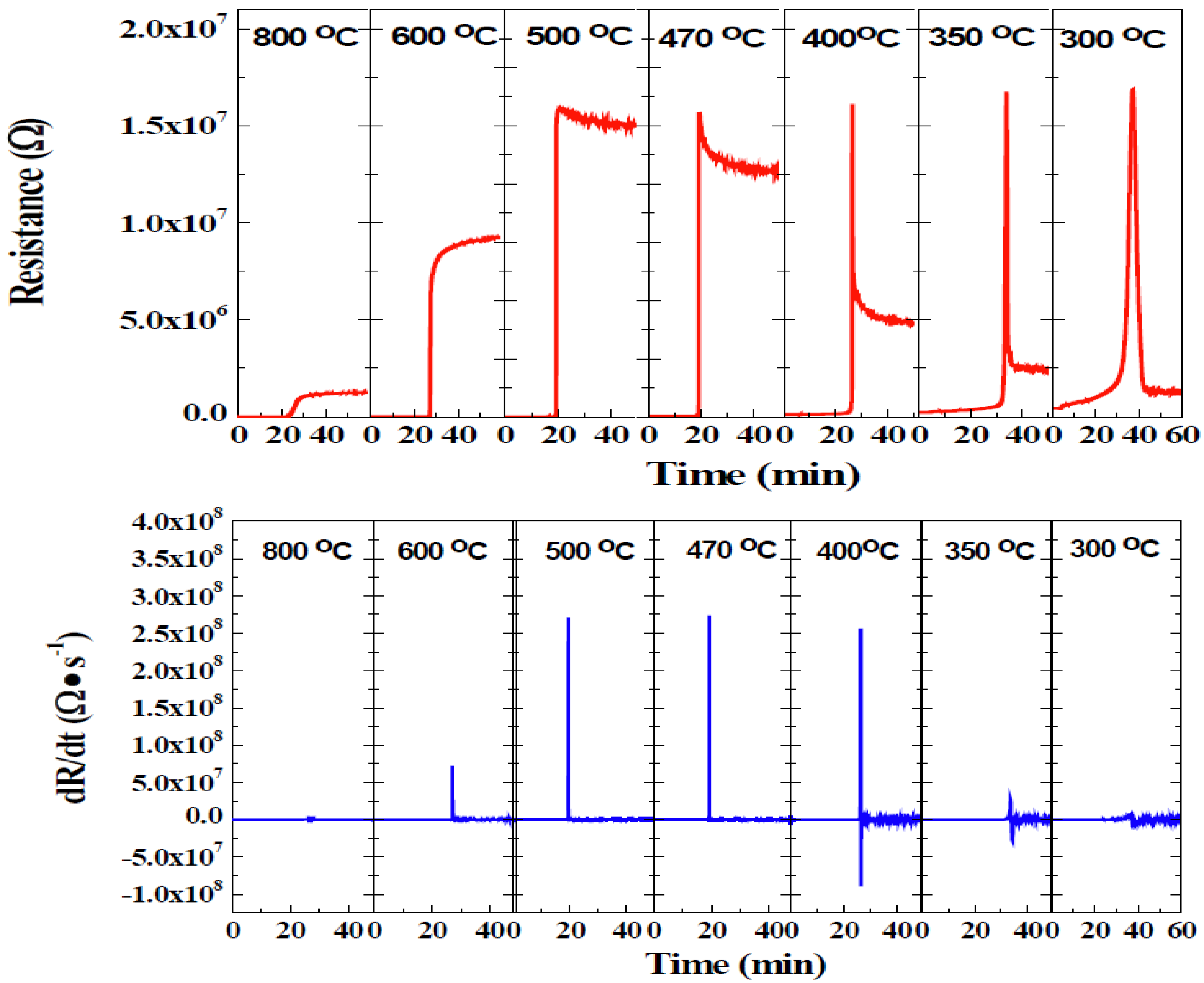




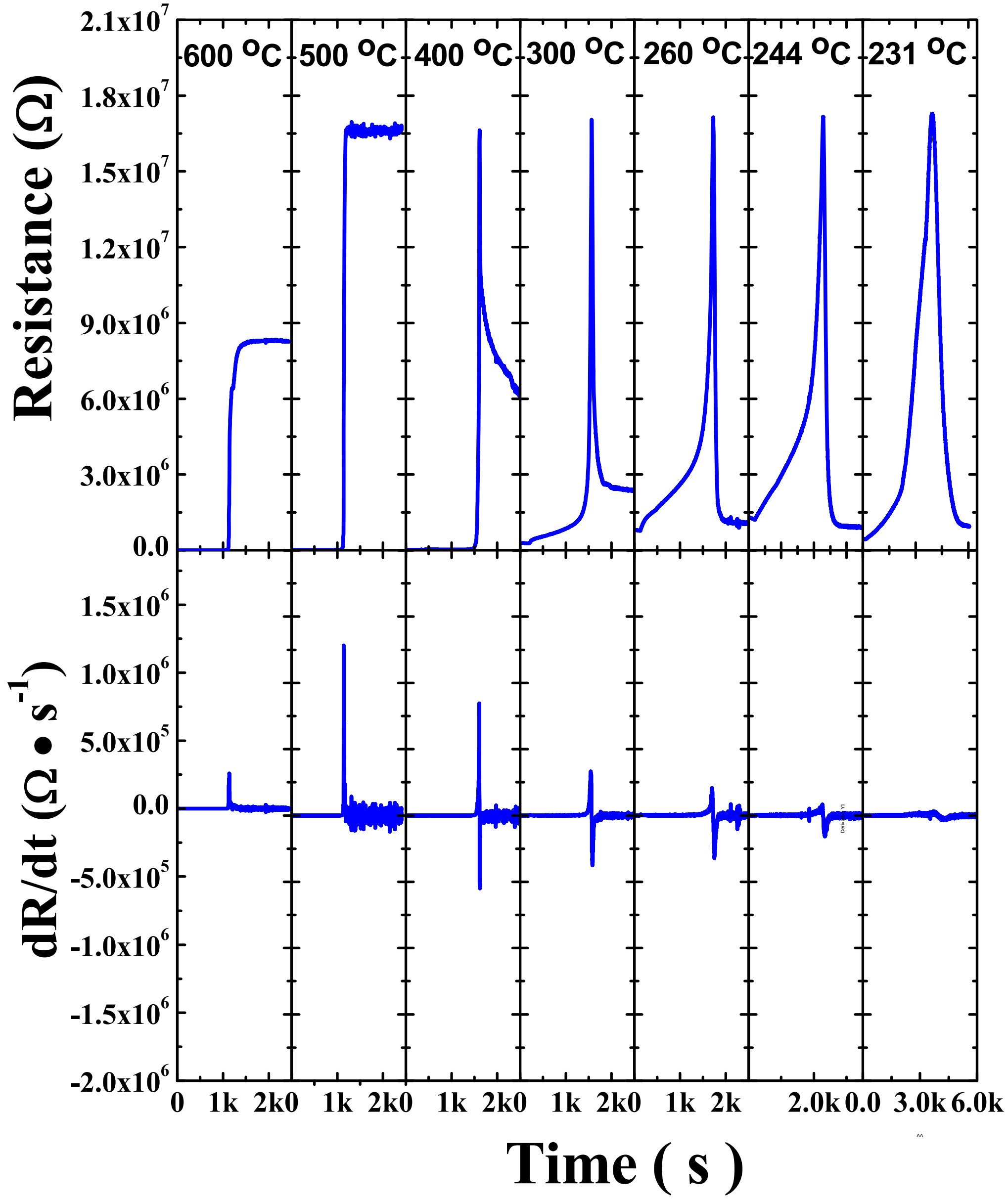
Time (s)
0.0
2.0x10^4
4.0x10^4
6.0x10^4
8.0x10^4
1.0x10^5
(a)
H2
O2
Resistance (Ω)
1.8x10^7
1.2x10^7
6.0x10^6
0.0
(b)
dR/dt (Ω/s)
1.0x10^9
5.0x10^8
0.0
-5.0x10^8
-1.0x10^9
(c)
Co+3
Co+4
Co+2
Resistance (Ω)
2.4x10^4
2.6x10^4
2.8x10^4
3.0x10^4
3.2x10^4
(d)
(e)
dR/dt (Ω/s)
4.0x10^6
2.0x10^6
0.0
-2.0x10^6
-4.0x10^6
25000
26000
27000
30825
30840
30855
30870
1.0x10^9
5.0x10^8
0.0
-5.0x10^8
-1.0x10^9


600 °C
500 °C
400 °C
300 °C
H2
O2
Resistance (Ω)
Time (min)
0.0
2.0x10^6
4.0x10^6
6.0x10^6
8.0x10^6
1.0x10^7
1.2x10^7
3.0x10^6
9.0x10^6
1.5x10^7
1.8x10^7
2.1x10^7
0
50
100
150
200
250
300
350
400
450
500
40
80
120
160
240
280
320
360

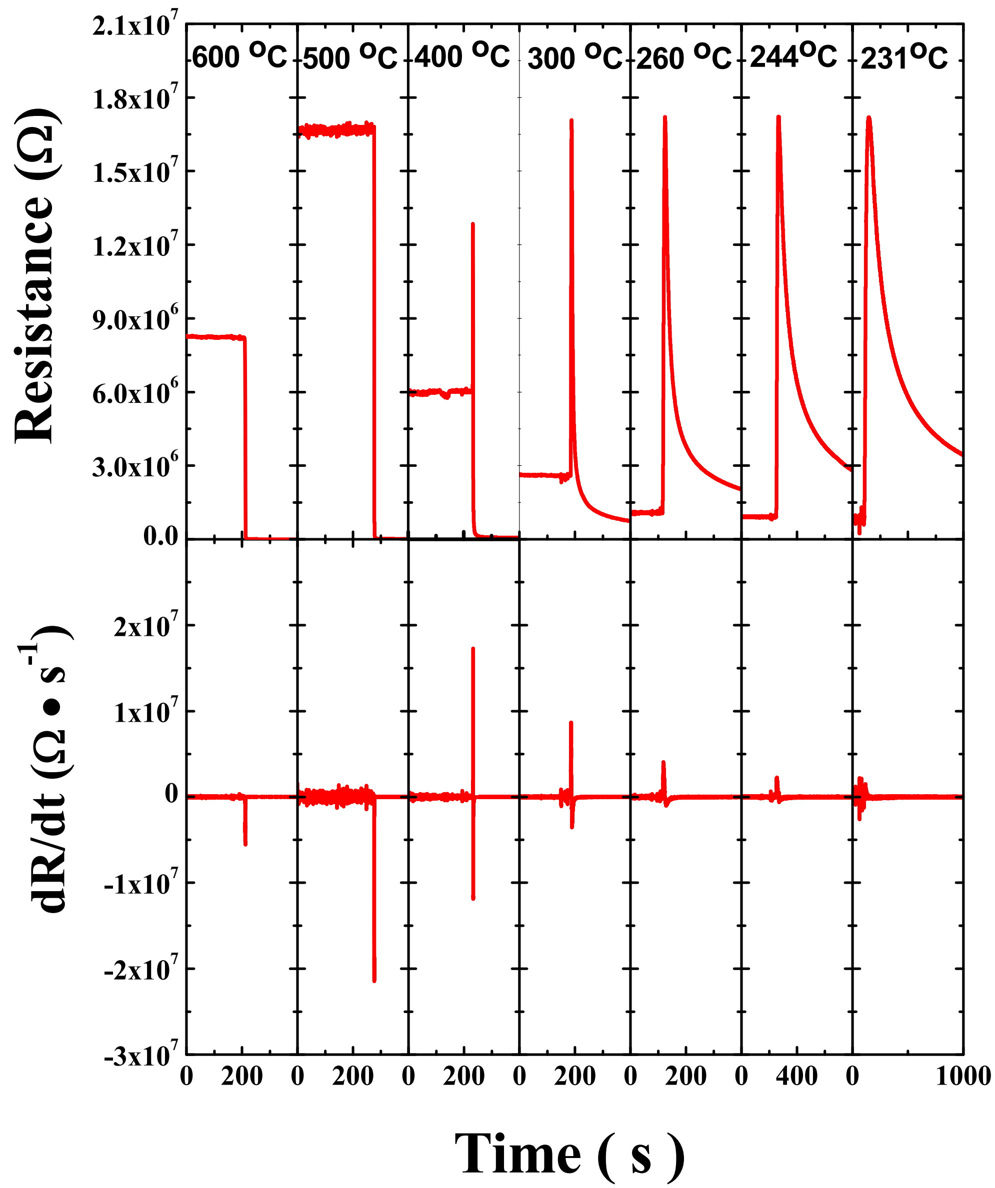
LBCO Film Reduction Property：



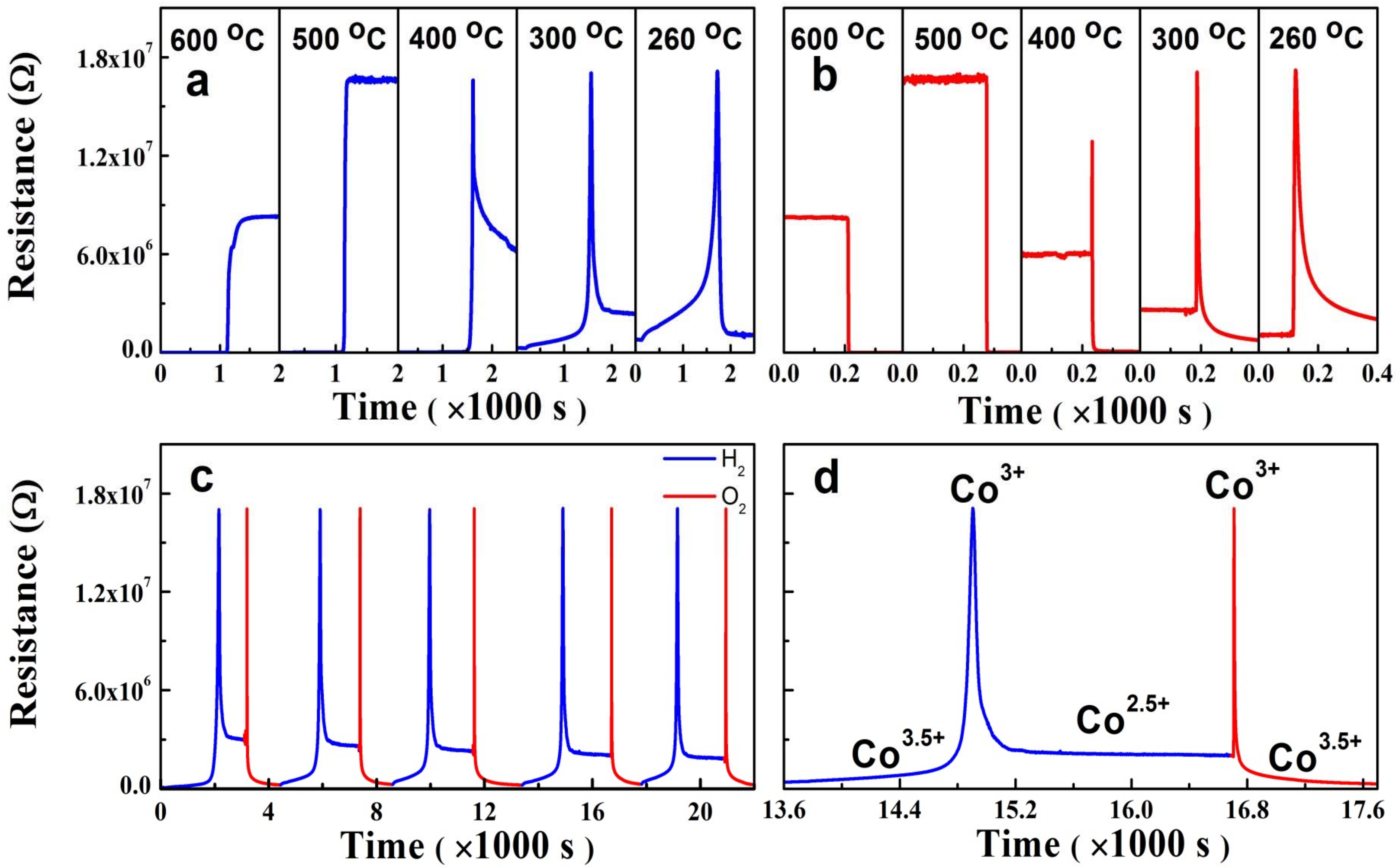
EBCO Film Reduction Property ：



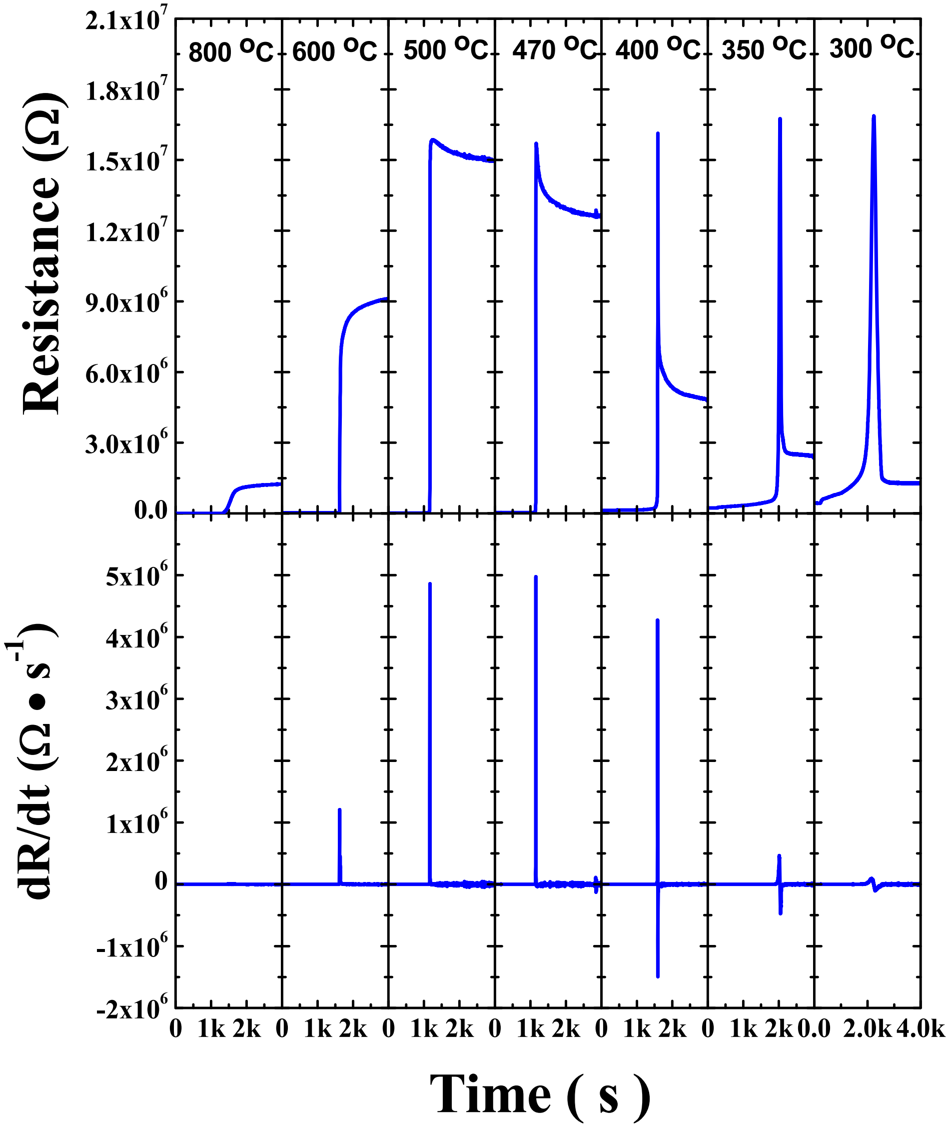
EBCO Film Oxidation Property ：



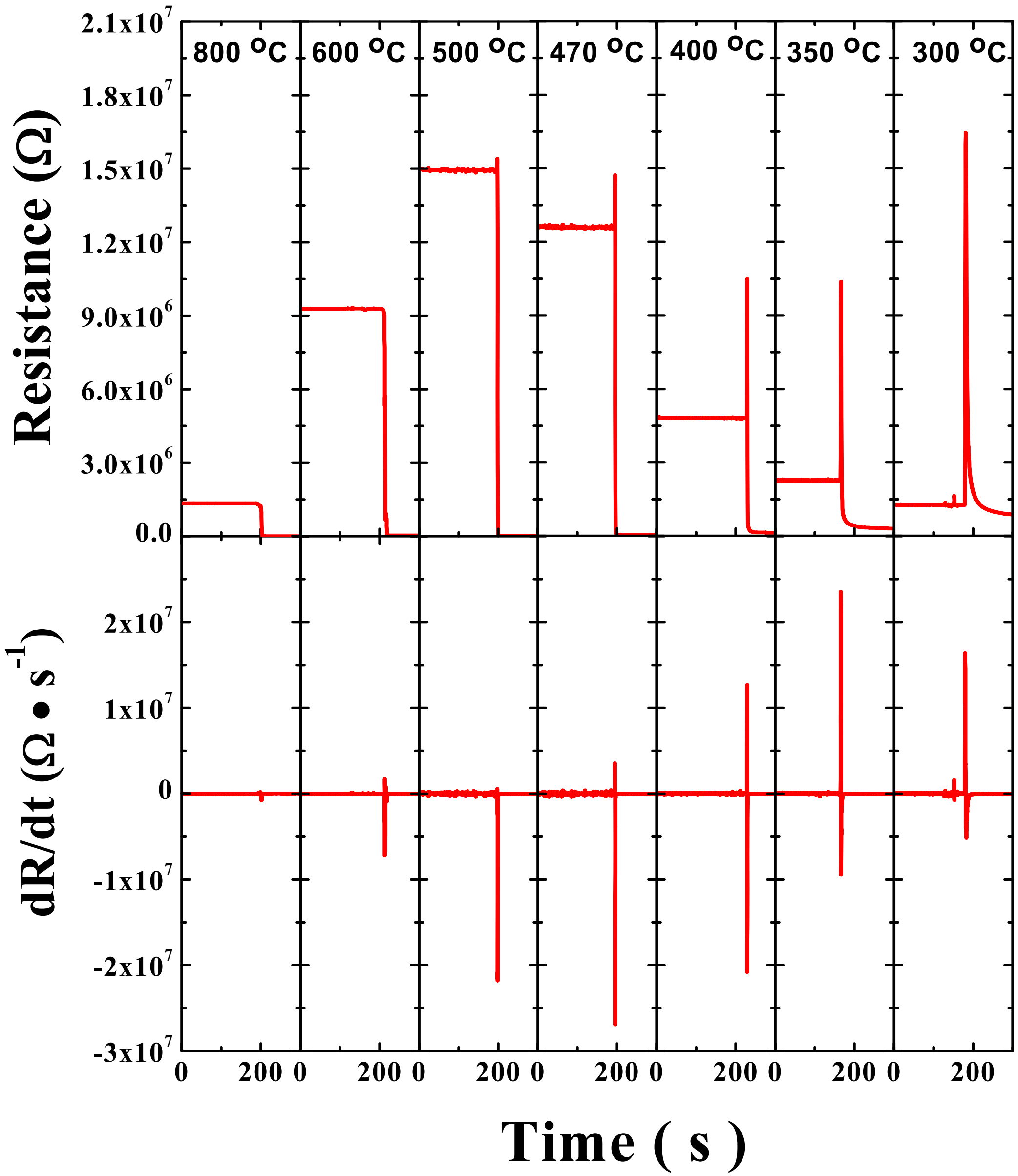
EBCO Film Oxidation Property ：

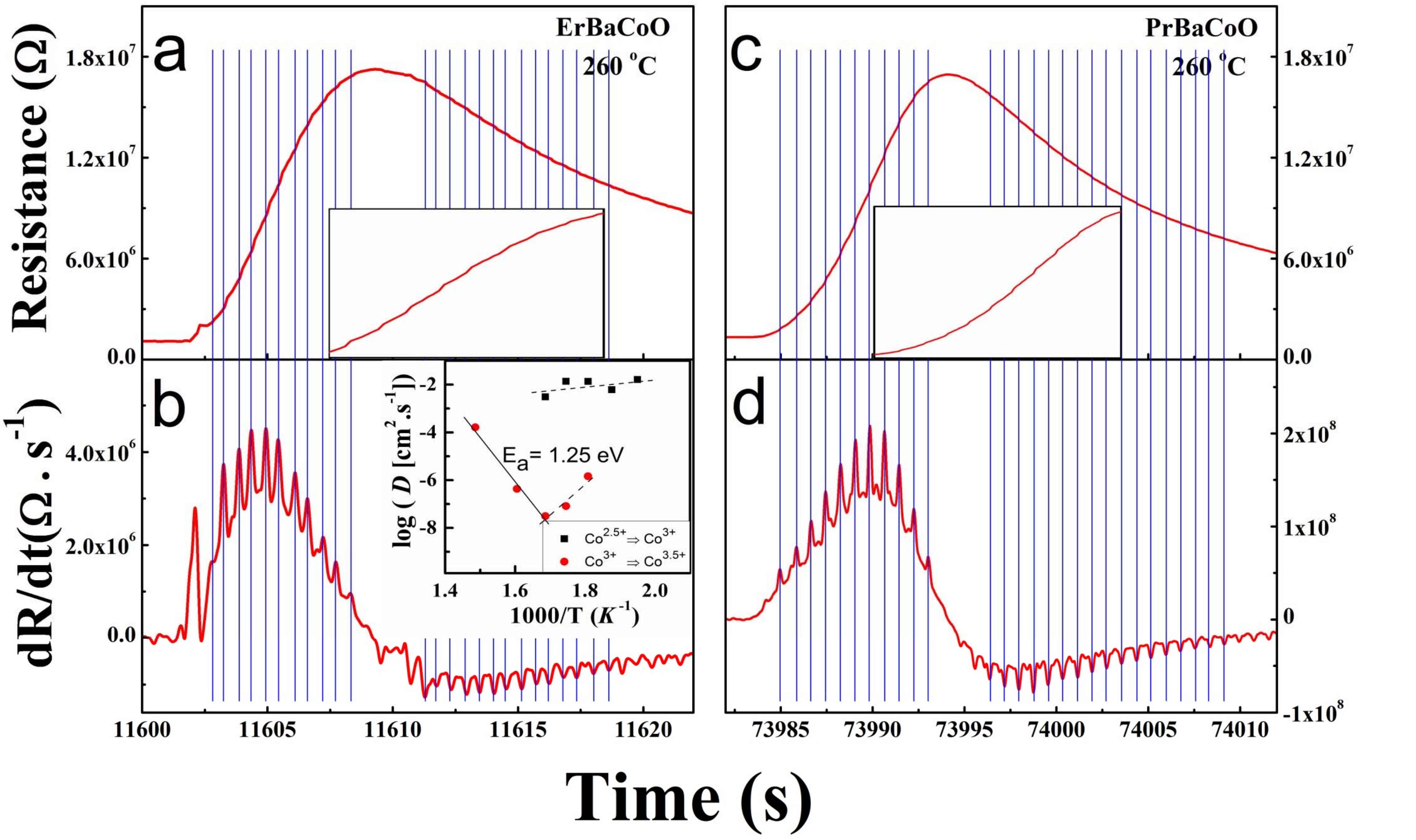


PBCO Film Reduction Property ：

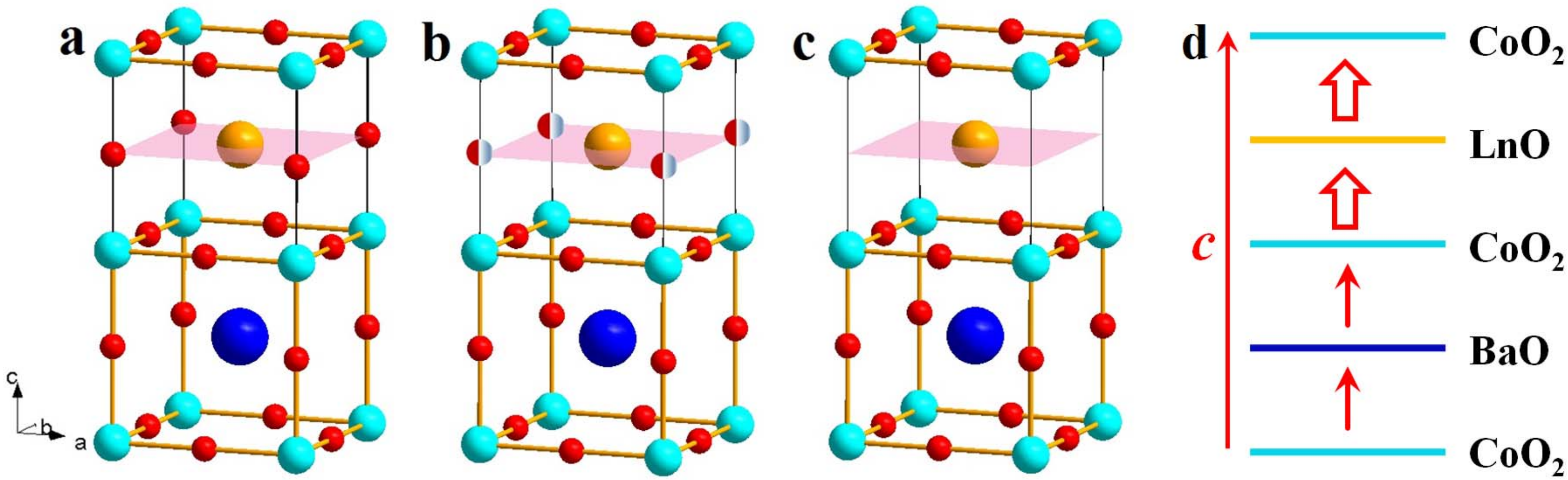


PBCO Film Oxidation Property：





ErBaCoO
260 °C
PrBaCoO
260 °C
a
b
c
d
Resistance (Ω)
dR/dt(Ω · s⁻¹)
log (D [cm².s⁻¹])
E_a = 1.25 eV
Co2.5+ ⇒ Co3+
Co3+ ⇒ Co3.5+
1000/T (K⁻¹)
Time (s)



Comparison of the diffusion rates: D(H) > D(a-Ag_{2+d}S) or D(a-Ag_2Te)!

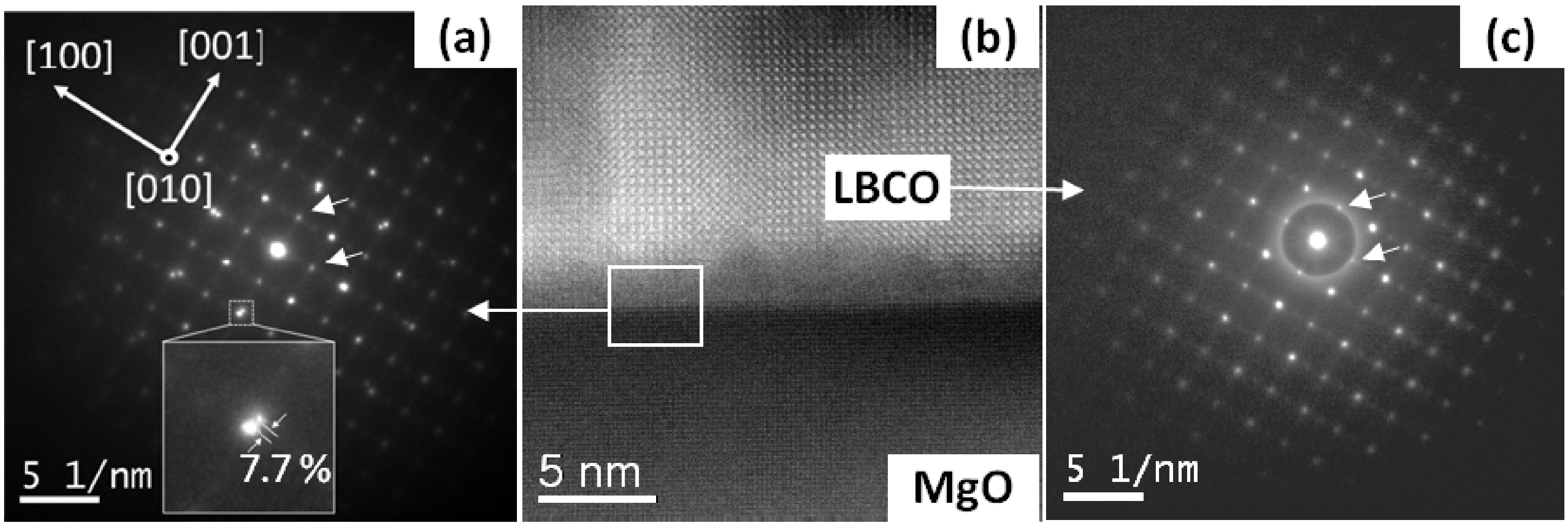
Model for diffusion of carriers

$$\frac{c(x,t)-c_1}{c_2-c_1}=1-\sum_{i=0}^{\infty}\frac{4(-1)^i}{\pi(2i+1)}\cos[\pi(2i+1)\frac{x}{L}]\exp[-\pi^2(2i+1)^2\frac{Dt}{L^2}$$

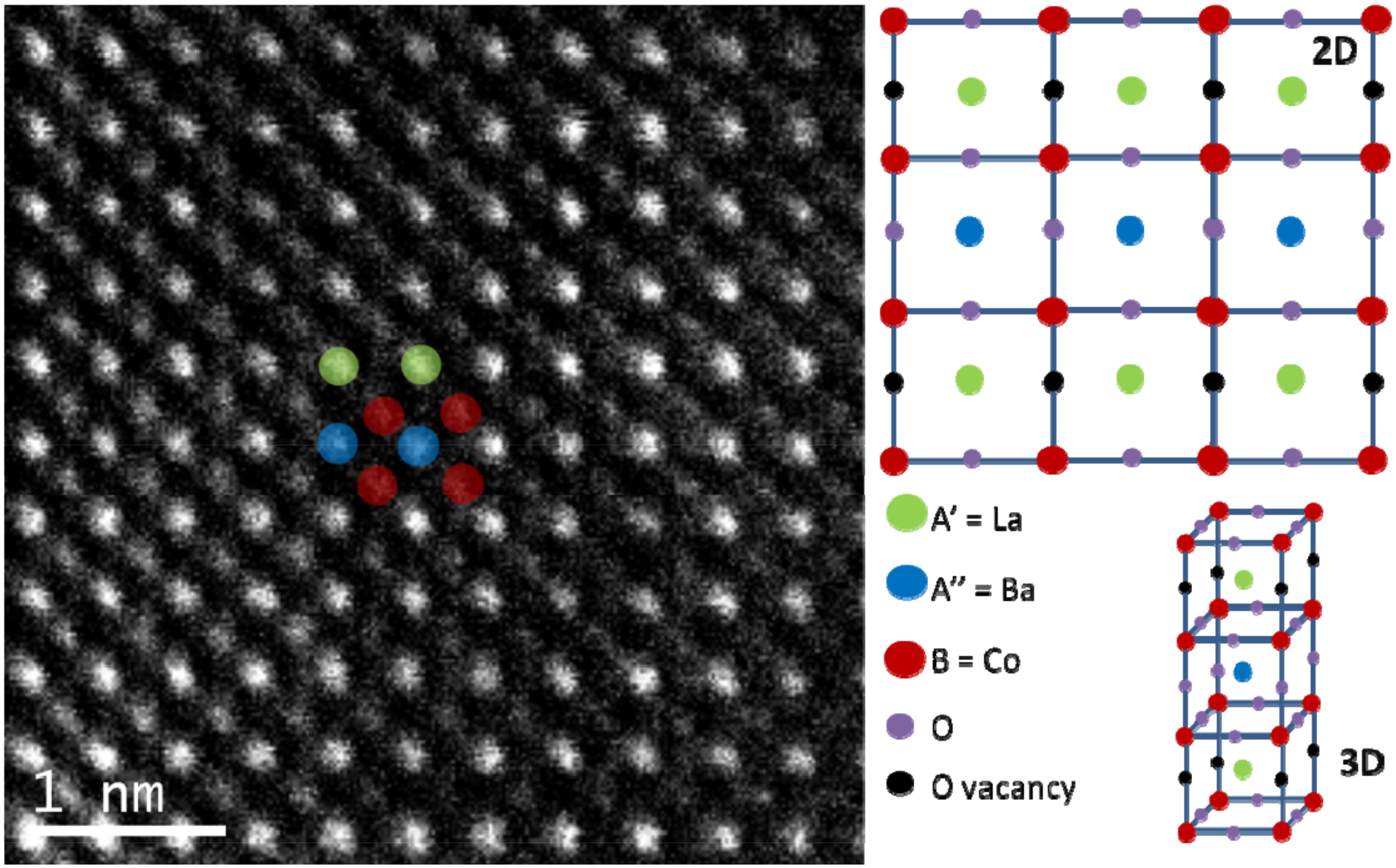
$$\frac{\sigma_m-\sigma_1}{\sigma_2-\sigma_1}=1-\frac{8}{\pi^2}\sum_{i=0}^{\infty}\frac{1}{(2i+1)^2}\exp[-(2i+1)^2\frac{t}{\tau}]$$

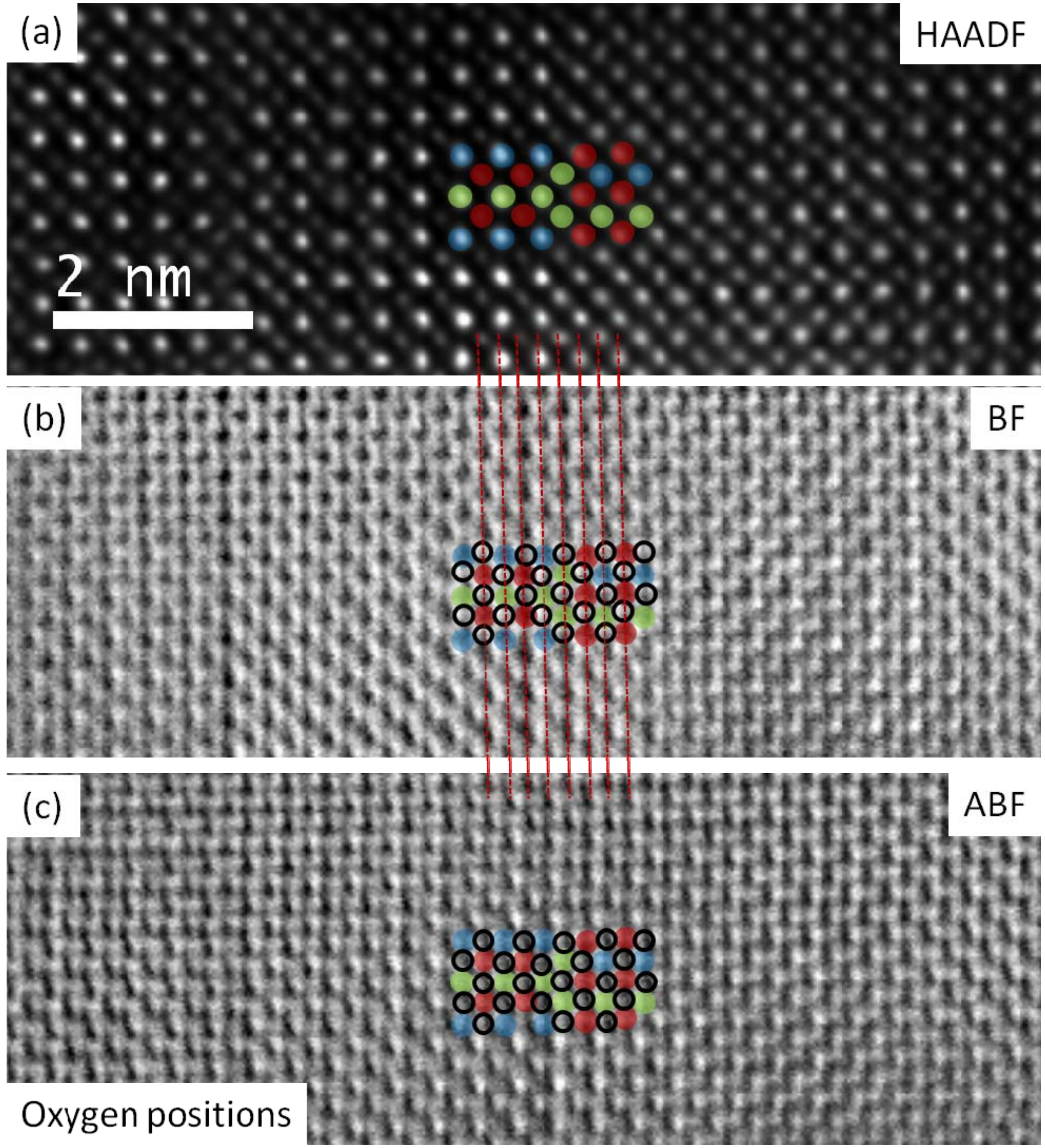
$$\tau=\frac{L^2}{\pi^2 D}$$

$$\frac{\sigma_m-\sigma_1}{\sigma_2-\sigma_1}\approx 4\pi^{-3/2}\sqrt{\frac{t}{\tau}}$$

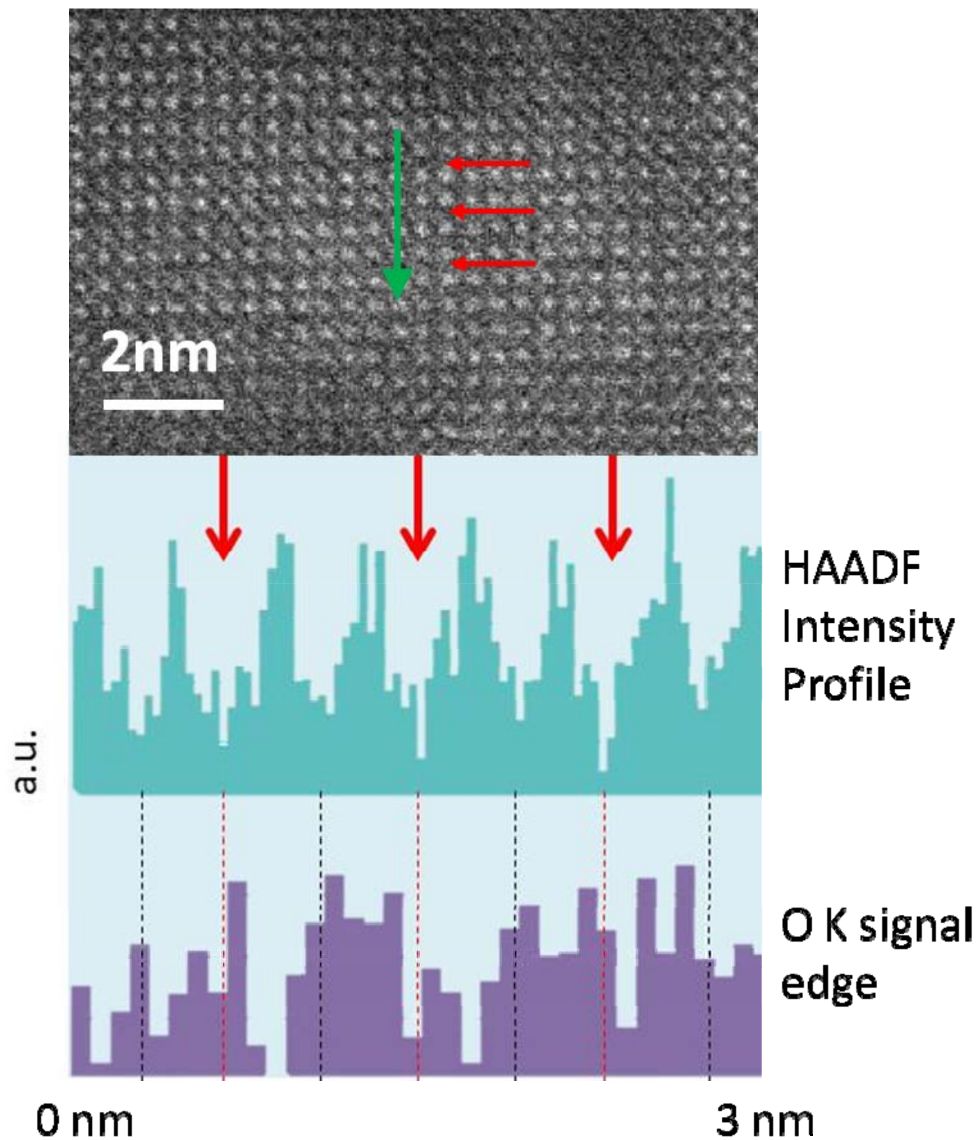


(a)
[100]
[001]
[010]
7.7 %
5 1/nm
(b)
LBCO
5 nm
MgO
(c)
5 1/nm

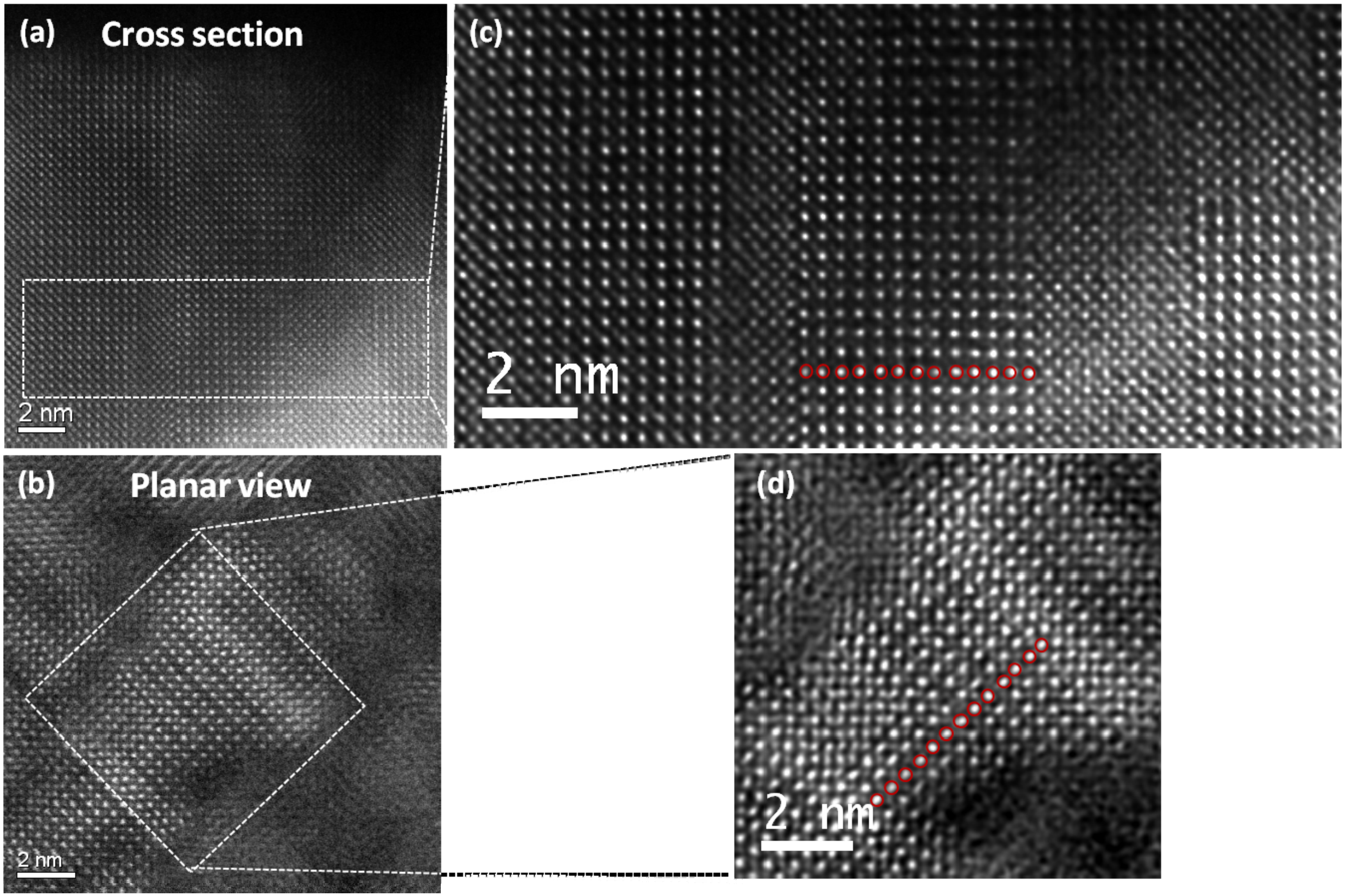

1 nm
2D
A' = La
A'' = Ba
B = Co
O
O vacancy
3D



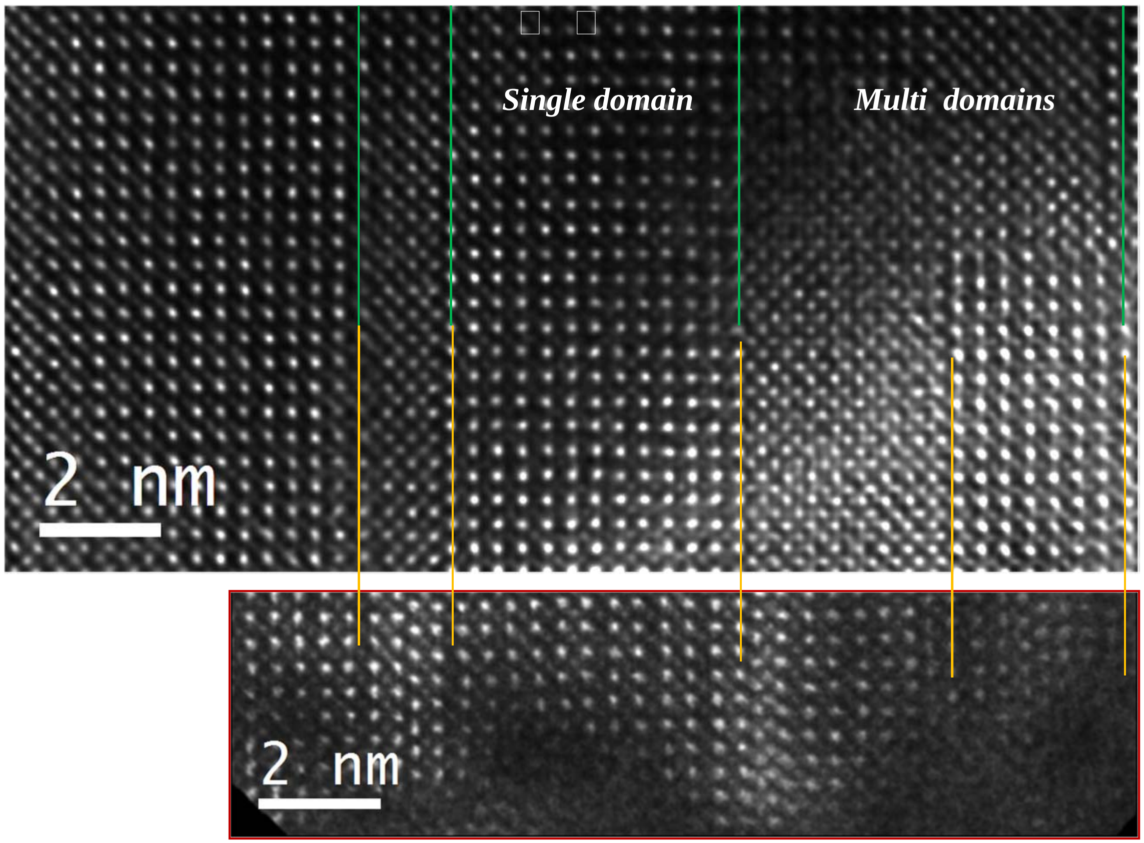
(a)
HAADF
2 nm
(b)
BF
(c)
ABF
Oxygen positions

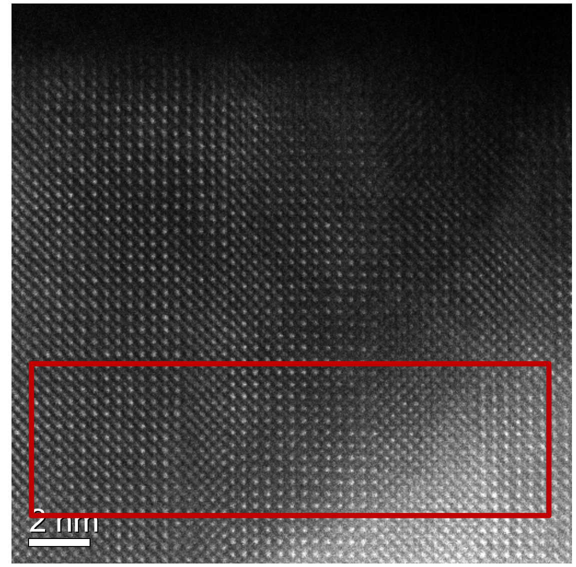


2nm
HAADF Intensity Profile
a.u.
O K signal edge
0 nm
3 nm

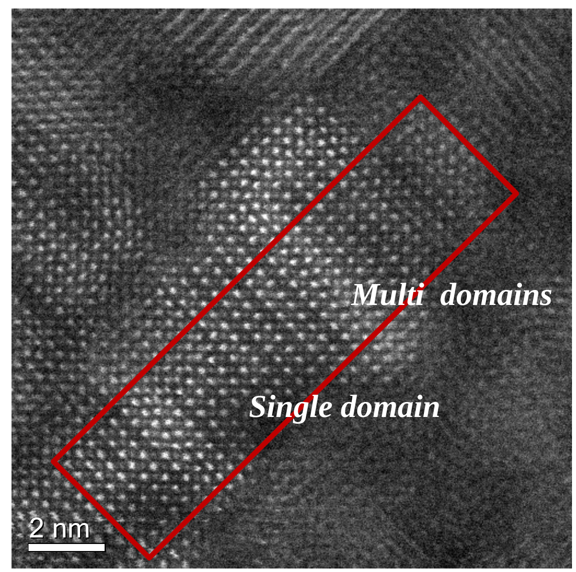

(a) Cross section
2 nm
(b) Planar view
2 nm
(c)
2 nm
(d)
2 nm

***d*-spacing 0.39 nm**





Cross section



Planar view

Future Research

- Continually study physical properties of LnBCO thin films and related materials at various chemical environments (gases, pressures, temperatures, etc.)

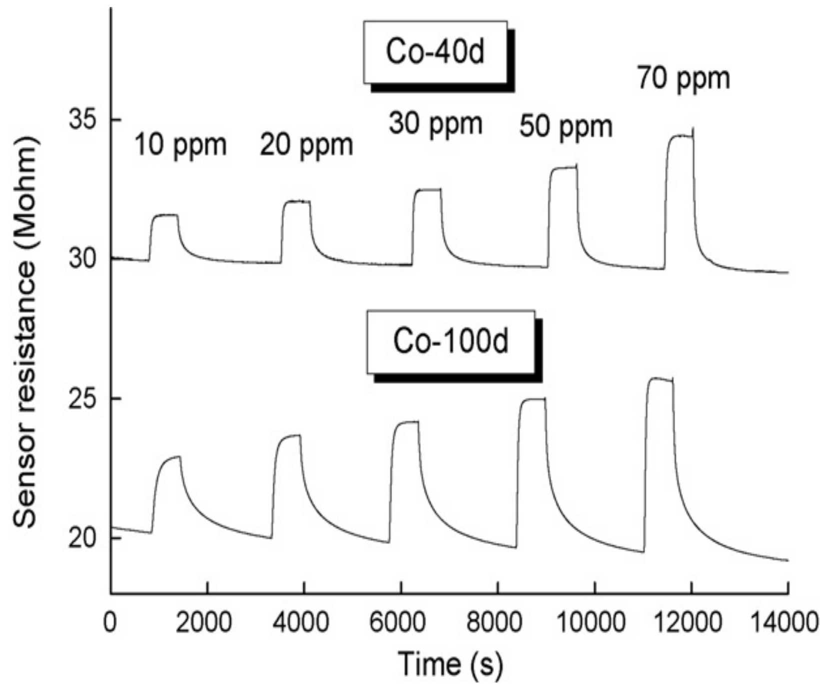
- Design and characterize the full scale (low & high temperature) chemical sensors, not only qualitatively but also quantitatively

- Explore novel materials for the development of new sensors and transducers (temperature, pressure, infra, etc.)

- Fundamentally understand the sensing mechanisms

Tunable interconnectivity of mesostructured cobalt oxide materials for sensing applications

- The sensor resistance change of the mesostructured cobalt oxide at different template temperatures.



Publications – published/revised

US Patent: C. L. Chen and J. Liu, "**US61/351,576**: Highly Epitaxail Thin Films for High Temperature/Highly Sensitive Chemical Sensors for Critical and Reducing Environment".

1. W. Donner, C. L. Chen, M. Liu, A. J. Jacobson, Y.-L. Lee, M. Gadre, and D. Morgan, "Epitaxial Strain-Induced Chemical Ordering in $La_{0.5}Sr_{0.5}CoO_{3-d}$ Films on $SrTiO_3$", ***Chem. Mat*****,** **23** (2011) 984
2. Jian Liu, Gregory Collins, Ming Liu, Chonglin Chen,* Jie He, Jiechao Jiang, and Efstathios I. Meletis, **"**Ultrafast Oxygen Exchange Kinetics on Highly Epitaxial $PrBaCo_2O_{5+d}$ Thin Films", ***Appl. Phys. Lett***., **100** (2012) 193903.
3. Chunrui Ma, Ming Liu, Gregory Collins, Jian Liu, Chonglin Chen,* Jie He, Jiechao Jiang, E. I. Meletis : "Thickness Effects on Magnetic and Electrical Transport Properties of Highly Epitaxial $LaBaCo_2O_{5.5+\delta}$ Thin Films on MgO Substrates", ***Appl. Phys. Lett***. **101** (2012) 021601
4. M. Liu, C. R. Ma, J. Liu, G. Collins, Y. M. Zhang, C. L. Chen,* J. He, J. C. Jiang, E.I. Meletis, Y. Lin, Li Sun, A. J. Jacobson, and Q. Y. Zhang, "Magnetic properties and anomalous transport phenomena in highly epitaxial double perovskite nano-ordering $(LaBa)Co_2O_{5.5+d}$ thin films on (001) MgO", ***Applied Materials & Interface***, **4** (2012) 5524-5528
5. Jian Liu, Gregory Collins, Ming Liu, and Chonglin Chen,* "Superfast Oxygen Exchange Kinetics on Highly Epitaxial $LaBaCo_2O_{5+d}$ Thin Films for Intermediate Temperature Solid Oxide Fuel Cells", ***APL Mat*** (in press)
6. Chunrui Ma, Ming Liu, Gregory Collins, Haibing Wang, Shanyong Bao, Xing Xu, , Erik Enriquez, **Chonglin Chen,*** Yuan Lin, and Myung-Hwan Whangbo, "Magnetic and Electrical Transport Properties of Highly Epitaxial $LaBaCo_2O_{5.5+\delta}$ Thin Films on Vicinal (001) $SrTiO_3$ Surfaces", ***Applied Materials & Interface*** **5** (2013) 451

Publications – papers submitted

1. Shanyong Bao, Chunrui Ma, Garry Chen, Xing Xu, Erik Enriquez, Chonglin Chen,* Yamei Zhang, Jerry L. Bettis, Jr., Myung-Hwan Whangbo, Chuang Dong, and Qingyu Zhang, "Ultrafast Chemical Exchange Dynamics with Layer-by-Layer Oxygen Vacancy-Exchange Diffusion in Cobalt Double-Perovskite Thin Films", ***Angew. Chem. Int. Ed***., (under review).
2. H. B. Wang, S. Y. Bao, J. Liu, G. Collins, C. R. Ma, M. Liu, C. L. Chen,* C. Dong, and M.-H. Whangbo, "Superfast Oxidation/Redox Chemical Dynamics on Highly Epitaxial $LaBaCo_2O_{5+\delta}$ Thin Films", ***Scientific Reports*** (under review).
3. Q. Zou, G. Q. Wang, H. L. Lu, T. Z. Yang, H. M. Guo, H. J. Gao, M. Liu, C. R. Ma, H. B. Wang, X. Xu, and C. L. Chen, "Tunable Anisotropic Transport Properties of Highly Epitaxial $LaBaCo_2O_6$ thin film on Stepped $SrTiO_3$ Substrates", ***APL Mat.***
4. G. Collins, J. Liu, C. R. Ma, H. B. Wang, C. L. Chen,* "Interface Engineered Heterostructures Electrolyte for Novel Low Temperature Thin Film Solid Oxide Fuel Cells", ***Adv. Energy. Mat.***
5. C. R. Ma, M. Liu, J. Liu, G. Collins, Y. M. Zhang, H. B. Wang, C. L. Chen,* Y. Lin, J. He, J. C. Jiang, E. I. Meletis, "Interface Strain Induced Anomalous Electronic Transport Behavior in Highly Epitaxial $LaBaCo_2O_{5.5+d}$ Films", ***Chem Mat***.
6. M. Liu, C. R. Ma, E. Enriquez, H. B. Wang, C. L. Chen, Y. Lin, "Physical Properties of Highly Mixed Conductive $LaBaCo_2O_{5.5+d}$ Thin Films directly Integrated on Si (100)", ***Materials Letters***
7. Chunrui Ma, Ming Liu, Gregory Collins, Haibin Wang, Shanyong Bao, Xing Xu, Erik Enriquez, Chonglin Chen*, and Y. Lin, "Anisotropic Strain Induced Anomalous Metal-Insulation Transition in on Highly Epitaxial $LaBaCo_2O_{5.5+\delta}$ Thin Films on (110) $NdGaO_3$", **Nature Physics** (to be submitted)
8. Fransisco Ruiz-Zepeda, Chunrui Ma, Daniel B. Uribe, J. Cantu-Valle, Haibing Wang, Xing Xu, Arturo Ponce, Miguel Yacaman* and C. L. Chen*, "Nanodomain Induced Anomalous Magnetic Electronic Transport Properties of $LaBaCo_2O_{5.5+\delta}$ Epitxial Films", **Chem Mat** (to be submitted)

Several other manuscripts are preparing for publication

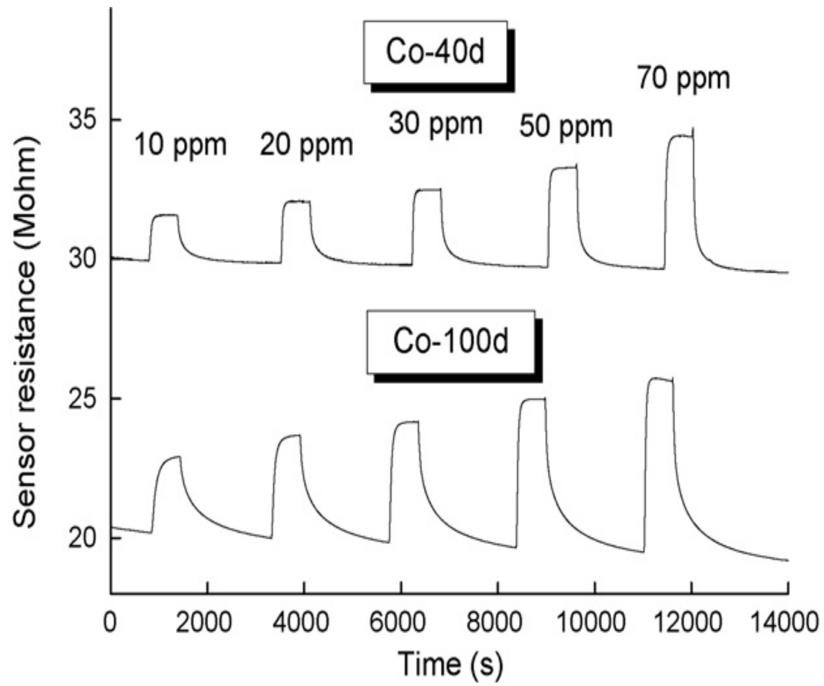
Summary

- Mixed ionic/electronic conductive double perovskite $LaBaCo_2O_{5.5}$ thin films have been successfully grown on various substrates for full scale chemical sensors.
- Superfast chemical dynamic behavior was found from the symmetric cell structures.
- A new oxygen vacancy exchange diffusion mechanism was discovered from the colbaltate systems.
- Various new/interesting physical phenomena have been found and achieved in the LBCO materials.
- More experimental and theoretical works are needed to understand the superfast chemical oxidation/redox dynamics and to explore the interface physics.

Thank you very much for your attention!

Tunable interconnectivity of mesostructured cobalt oxide materials for sensing applications

- The sensor resistance change of the mesostructured cobalt oxide at different template temperatures.



Optical CO Gas Sensor Using a Cobalt Oxide Thin Film Prepared by Pulsed Laser Deposition under Various Argon Pressures

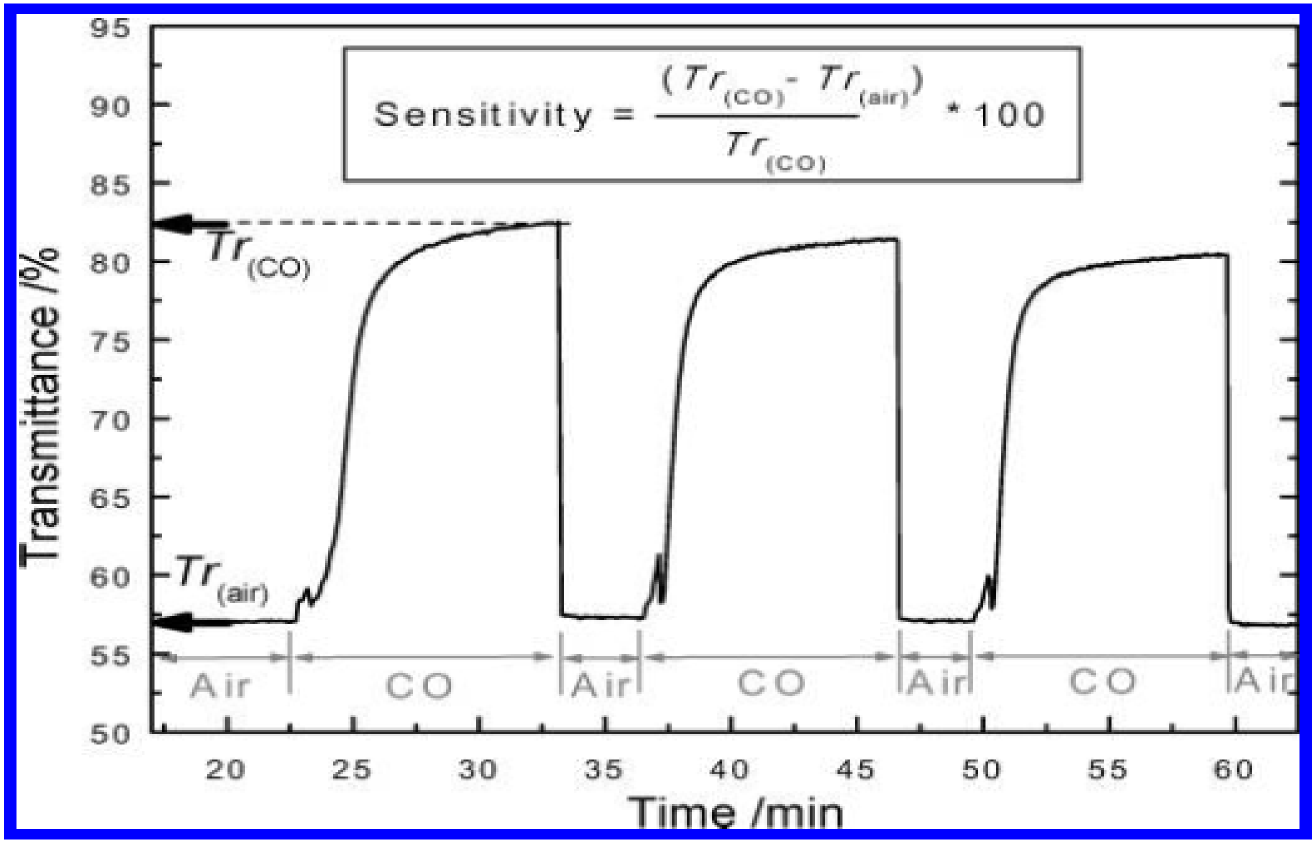


Figure 3. Transmittance change of a cobalt oxide film prepared at 133 Pa of Ar. The transmittance of the film at 625 nm was monitored in dry air and 200 ppm of CO gas at 350 °C. Sensitivity is defined as $(Tr_{(CO)} - Tr_{(air)})/Tr_{(CO)} \times 100$, where $Tr_{(CO)}$ and $Tr_{(air)}$ are transmittance in CO gas and dry air, respectively, at 350 °C.

Hierarchically Structured Cobalt Oxide (Co3O4): The Morphology Control and Its Potential in Sensors

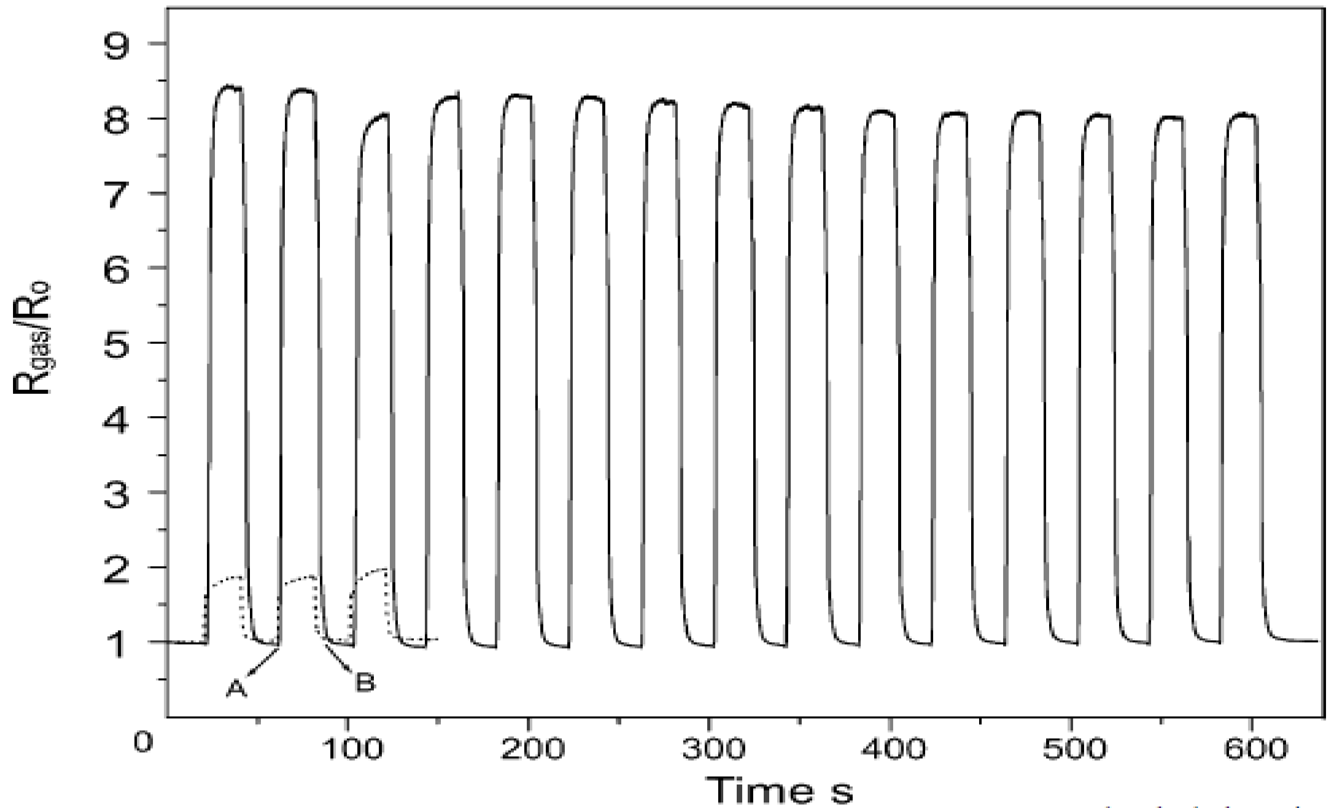


Figure 10. The alcohol sensing curve in a Co_3O_4-based sensor at 300 °C. The solid line is the response to 50 ppm alcohol and the dash line is to 1000 ppm CO, showing the selectivity of the sensor. The sensor is exposed to alcohol vapor or CO at A and for 20 s, then it is switched to dry air at B. The gas sensitivity is defined as the resistance ratio of R_{gas} to R_o, where R_{gas} and R_o are the electrical resistance for the sensor in alcohol or CO and in air, respectively.