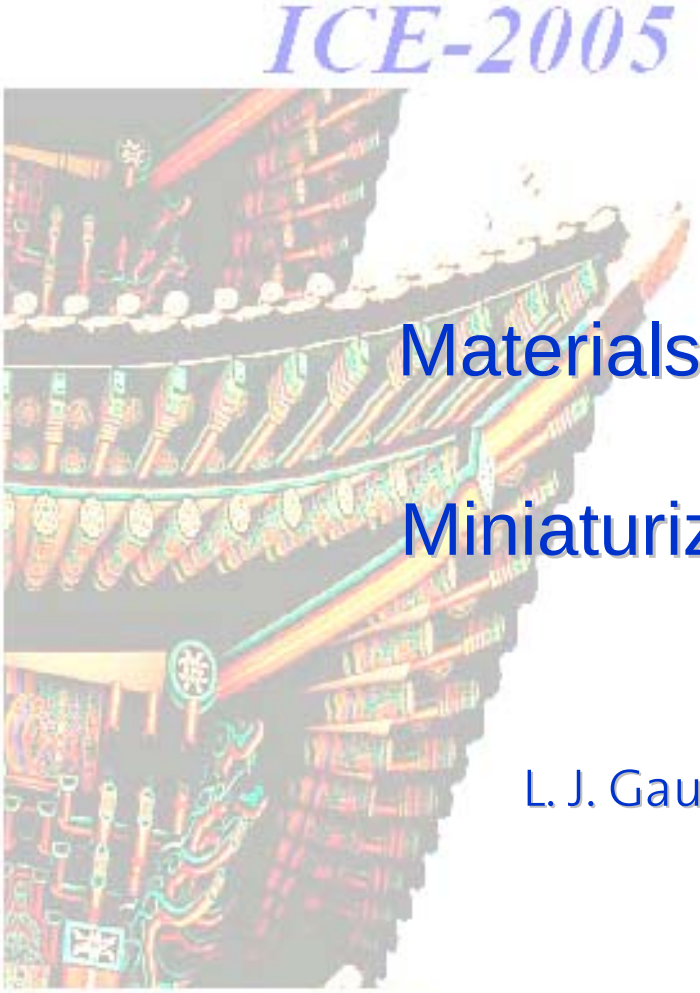
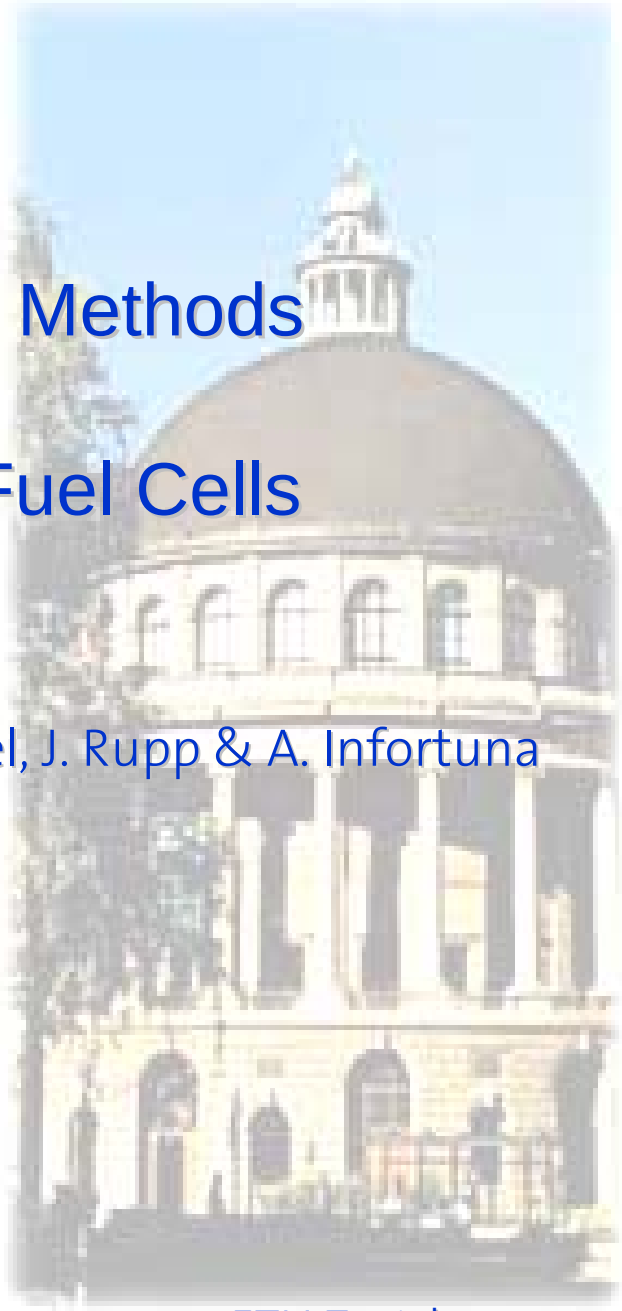


Materials and Preparation Methods for Miniaturized Solid Oxide Fuel Cells

L. J. Gauckler, U. Mücke, D. Beckel, J. Rupp & A. Infortuna

Department Materials
ETH Zurich
Switzerland



Outline

- **Motivation**
- **μ - Fuel Cell Systems**
- **μ - Solid Oxide Fuel Cell Hot Plate**
  - **Pulsed Laser Deposition**
  - **Spray Pyrolysis**
  - **Electrolyte**
  - **Cathode**
  - **Anode & Current Collector**
- **Acknowledgement**

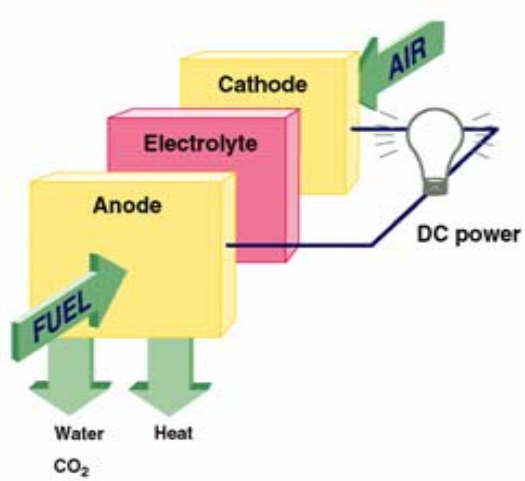
ONEBAT Project Goals

The goal of the ONEBAT project:

Miniaturized solid oxide fuel cell (SOFC) technology for small portable electronic applications such as cellular phones.

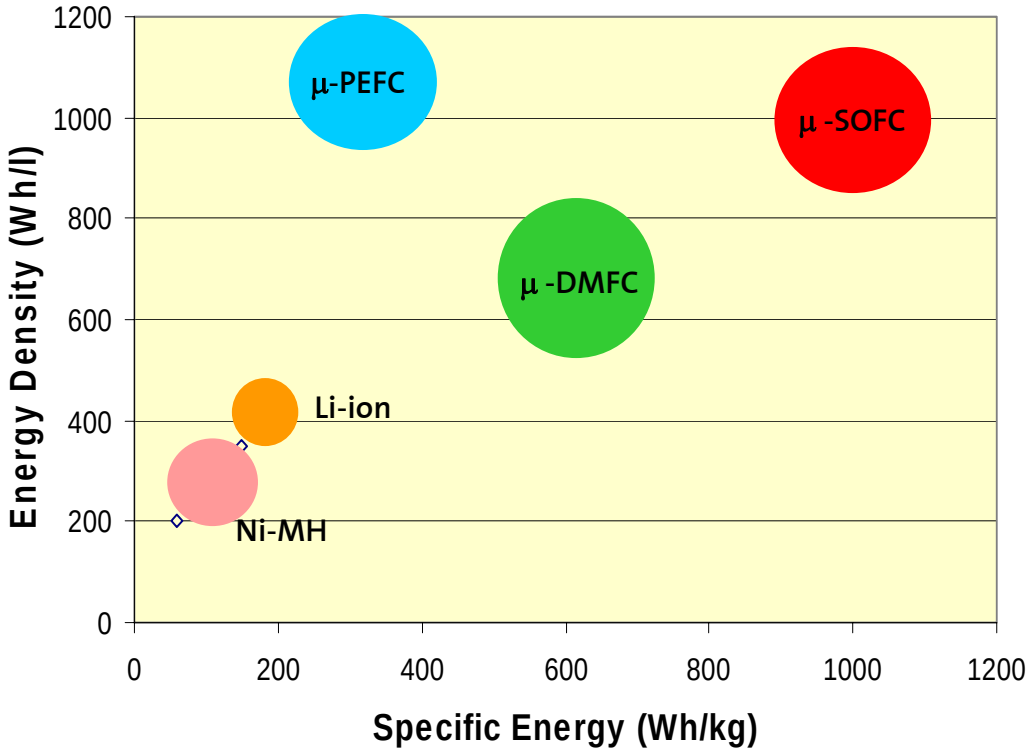
&

$$OCV = \frac{kT}{4q}\ln\left(\frac{p_{O_2}cathode}{p_{O_2}anode}\right)$$



- Very high energy capacity (at least **3 times more than current batteries**)
- **Immediate charging** (using compressed gas as a fuel)
- Power network and **geographical independence**

Systems Energy Densities



- Values as announced by system developers
- µ-PEFC lays very high but metal hydride technology is not down-scalable
- All the published energy density values for µ-DMFC are significantly lower than press releases statements (e.g. "5 times longer run-time than Li-ion"...)

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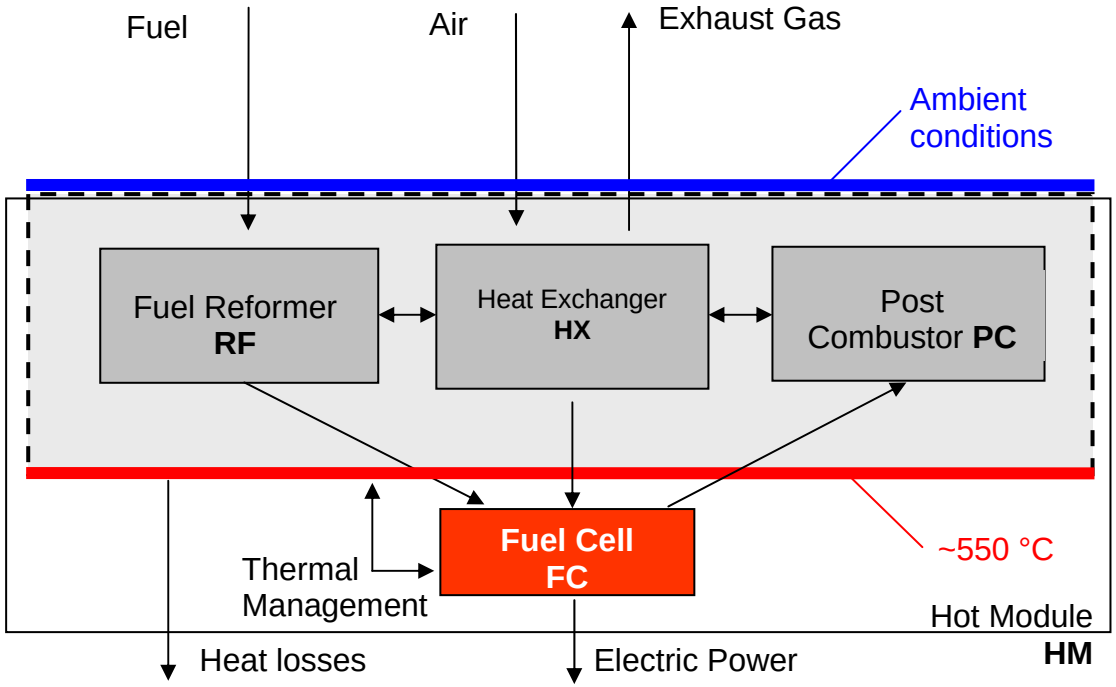
Scientific and technological objectives

SOFC pack with **μ-fuel-cell** with:

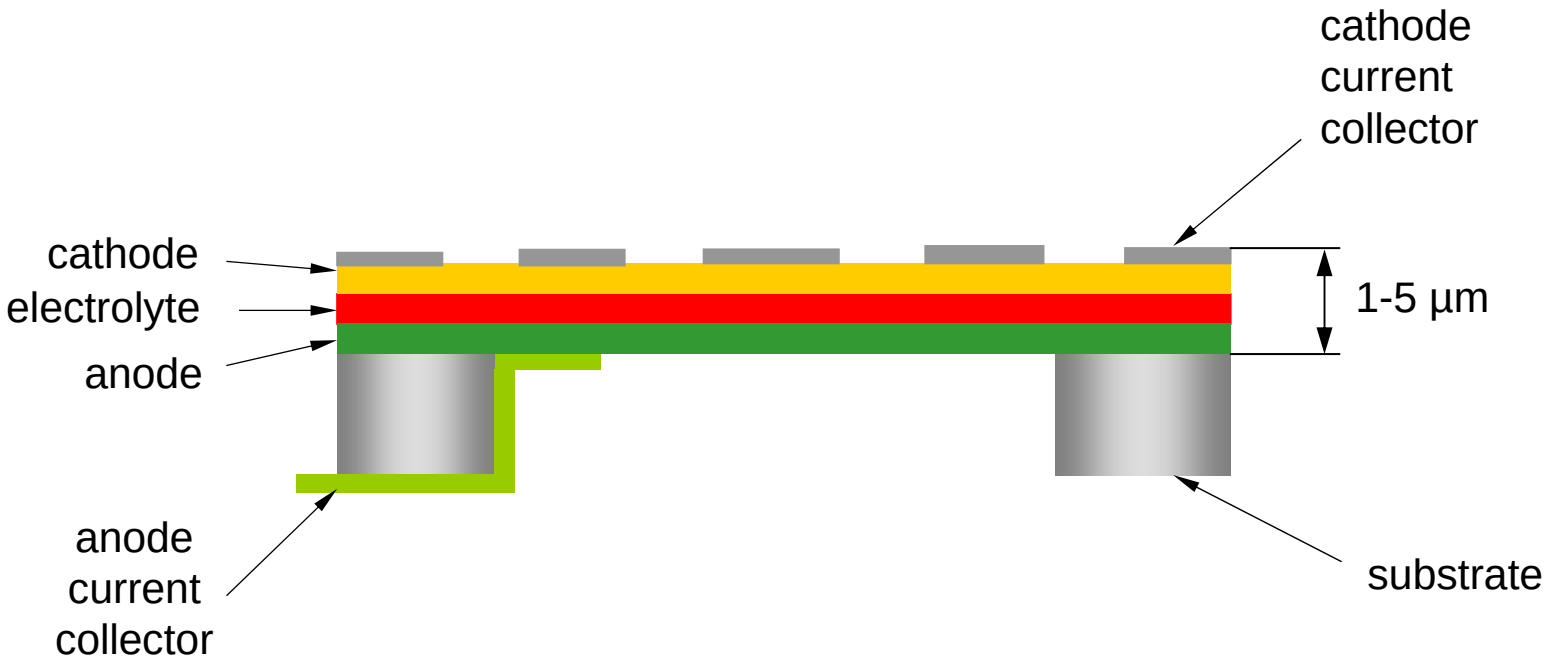
- **1 W** continuous power **(5 W peak)** within **30 cm³** (incl. 15 cm³ fuel)
- operating directly on liquid gas (e.g. **butane**)
- reaching an unprecedented battery capacity **(1000 Wh/litre & 1000 Wh/kg)**
- integrating fuel cell on the chip using **micro-fabrication technology**

μ-Solid Oxide Fuel Cell System

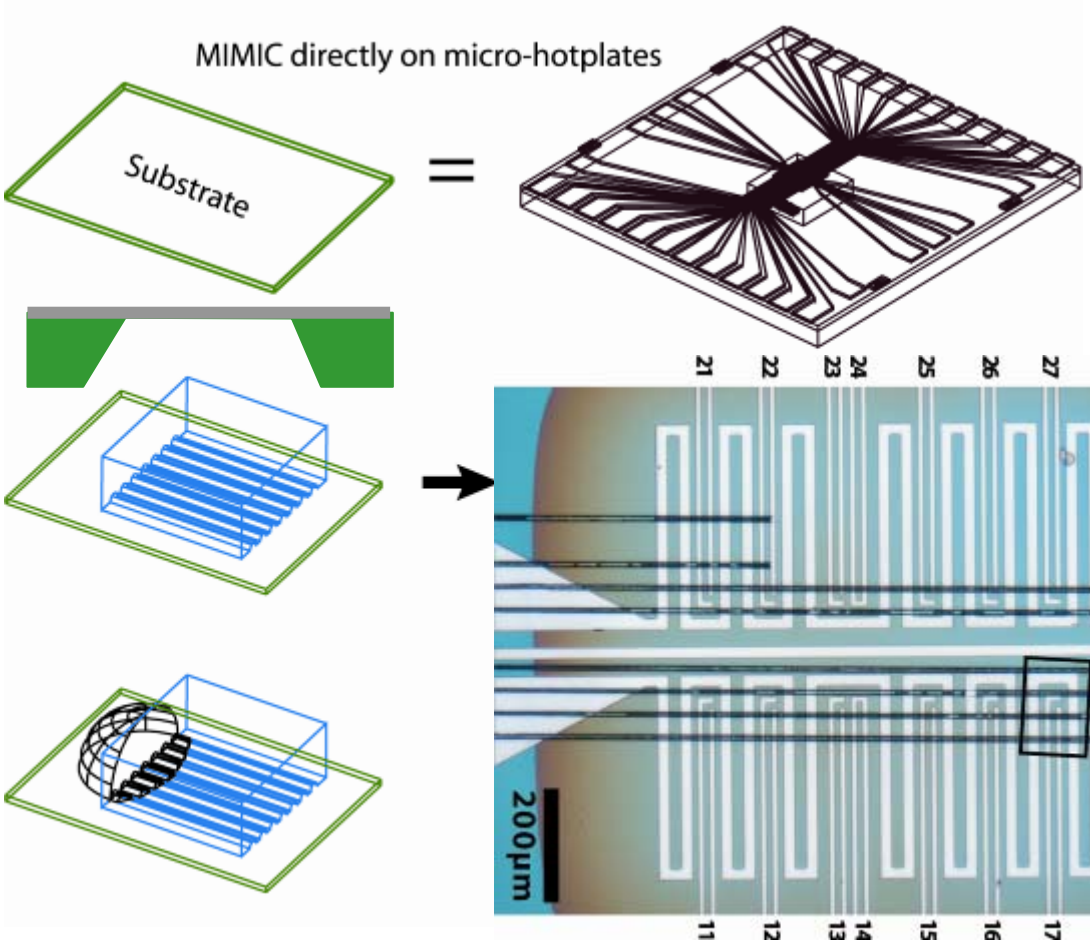


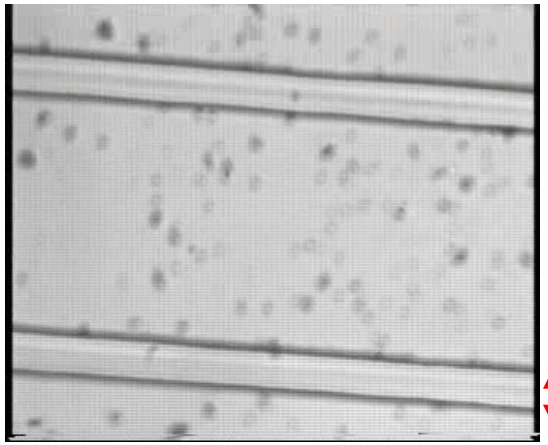
The hot module consists of four subsystems: the fuel cell (FC), the fuel reformer (RF), heat exchanger (HX), and the post combustor (PC)

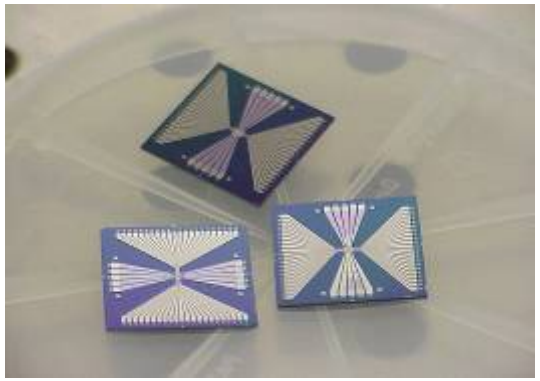
µ-SOFC test unit



SnO2 sensor array on micro-hot plate by MIMIC



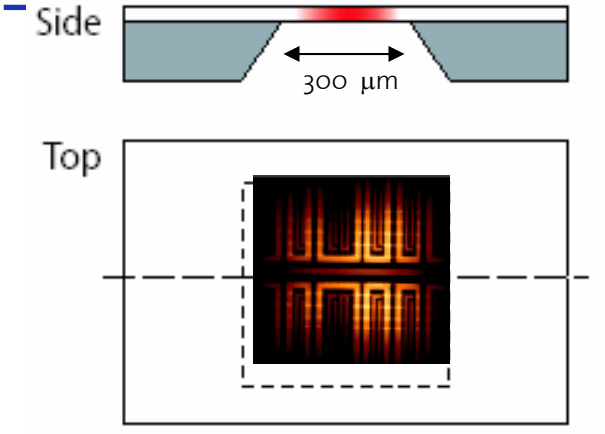


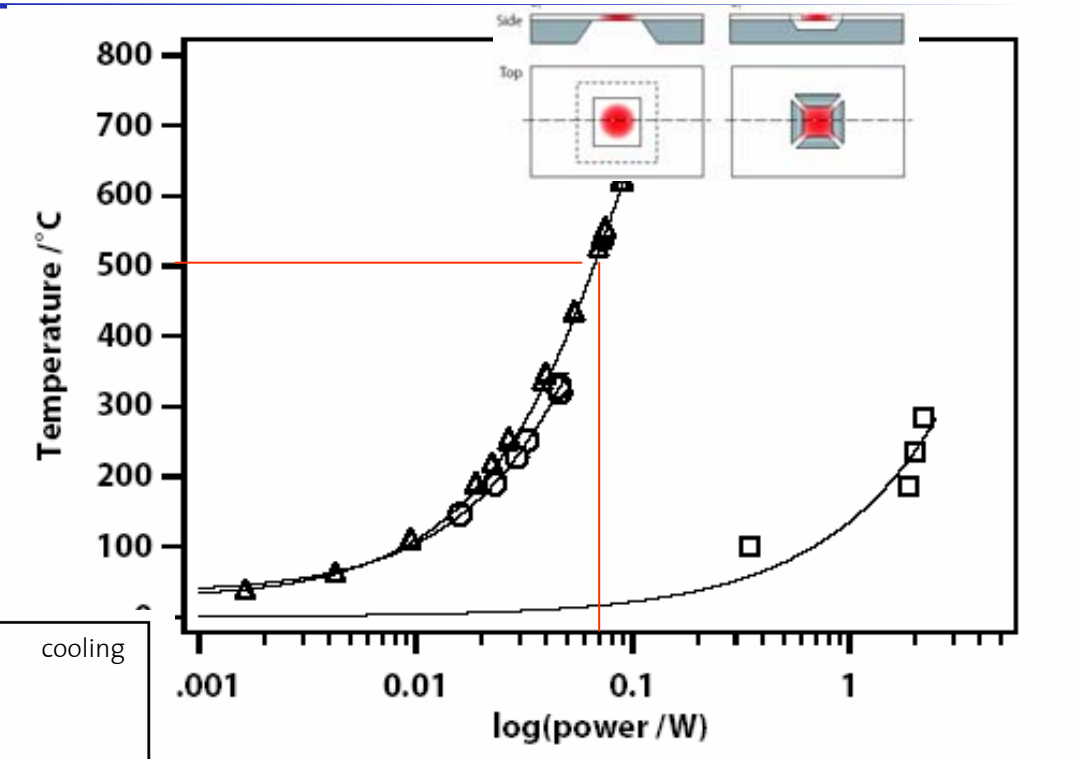


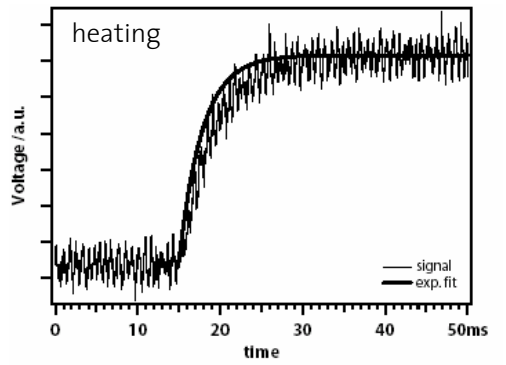
Test hotplates with 20 two-point contacts.

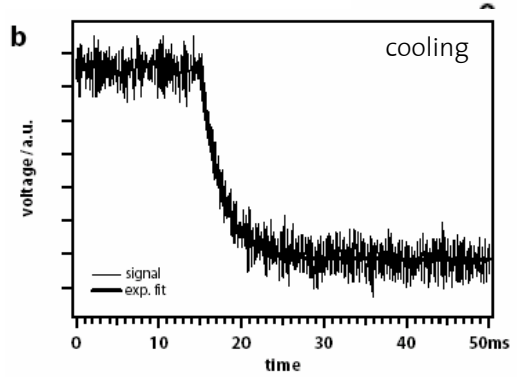
M. Heule, L. J. Gauckler
Sensors and Actuators B 93 (2003) 100–106

Heat loss from micro-hotplates.









65 mW are sufficient to heat a 300 x 300 µm² membrane to 500 °C
τ = 10-20 ms

M. Heule, L. J. Gauckler, Adv. Mater. 13, No. 23, 1790-93, 2001

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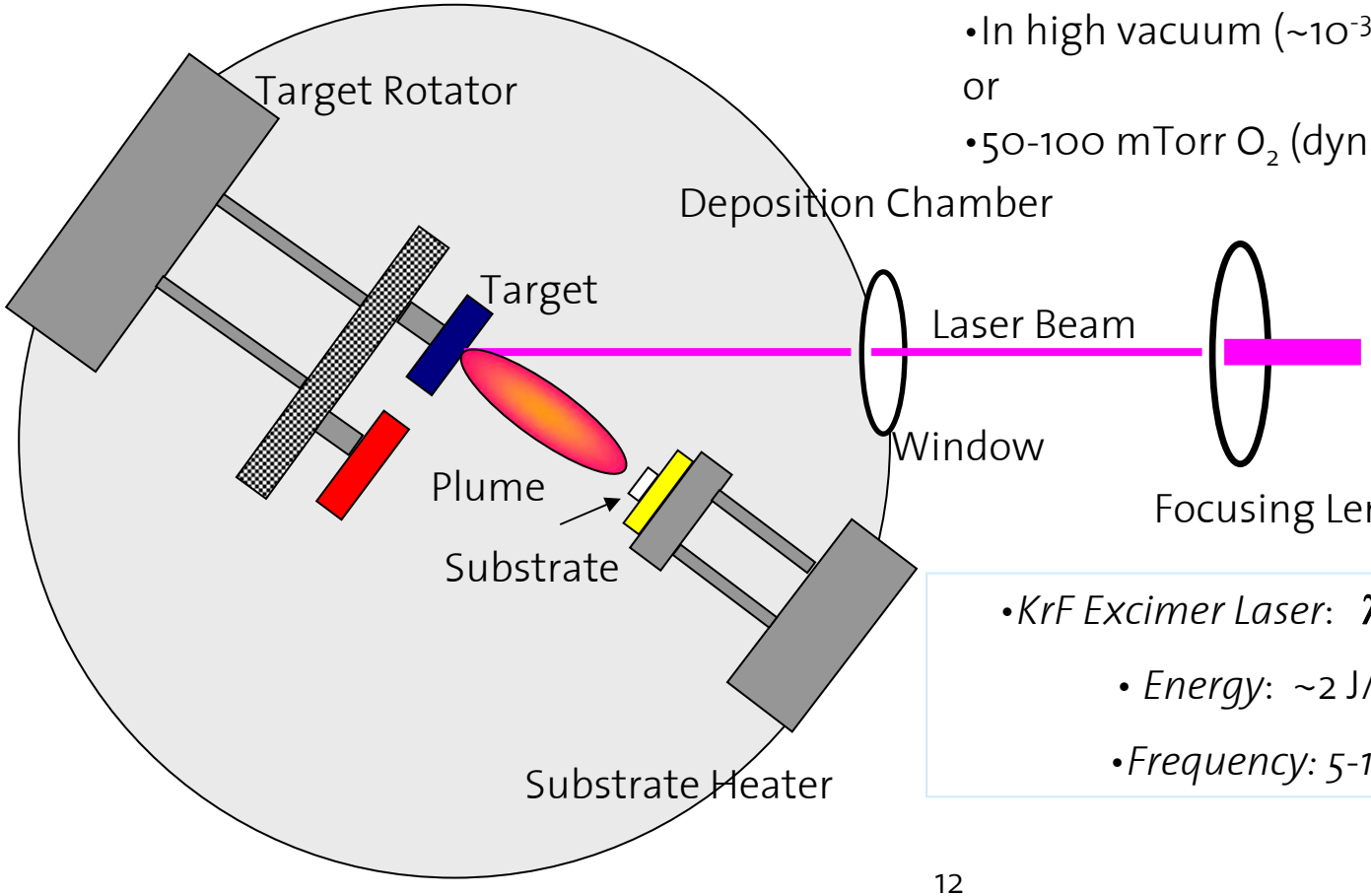
PLD-Experimental

Typical PLD Synthesis Method

- Targets synthesized by solid state techniques
- Films deposited
  - at room temperature
  - In high vacuum (~10^{-3} mTorr)

  or
  - 50-100 mTorr O_2 (dynamic)

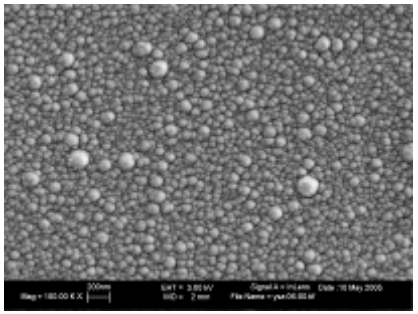
Pulsed Laser Deposition

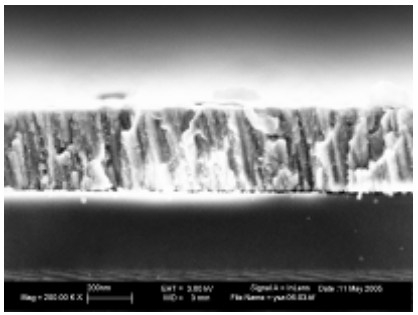


- *KrF Excimer Laser*: λ = 248 nm
- *Energy*: ~2 J/cm²
- *Frequency: 5-10 Hz*

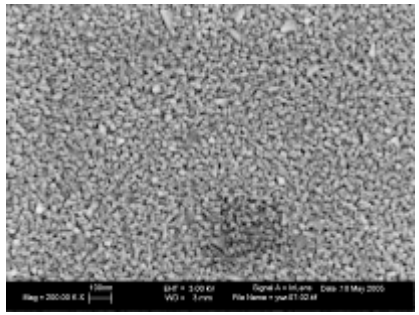
PLD of CGO and YSZ Films: Influence of pressure

RT, 20 m Torr

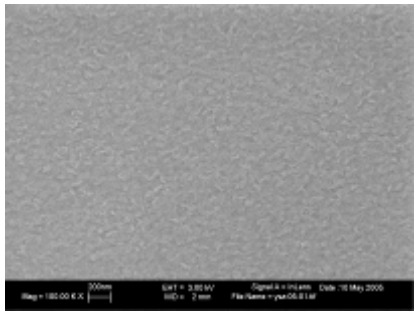


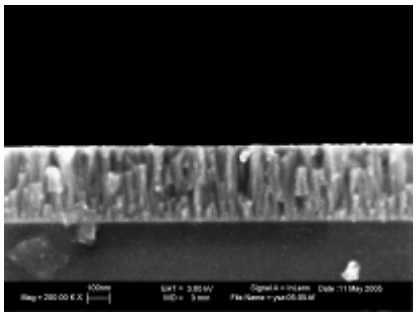


400°C, 50 m Torr

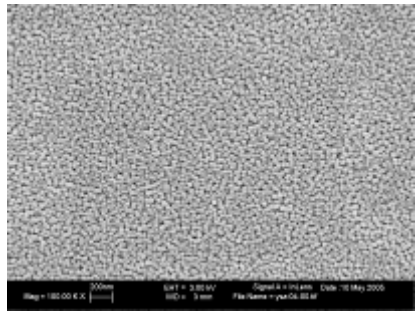


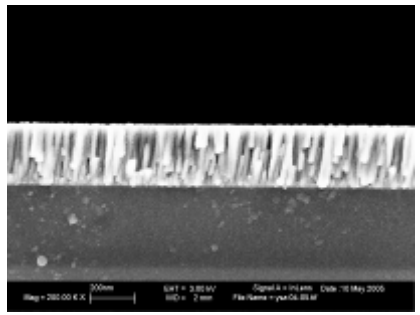
600°C, 20 m Torr





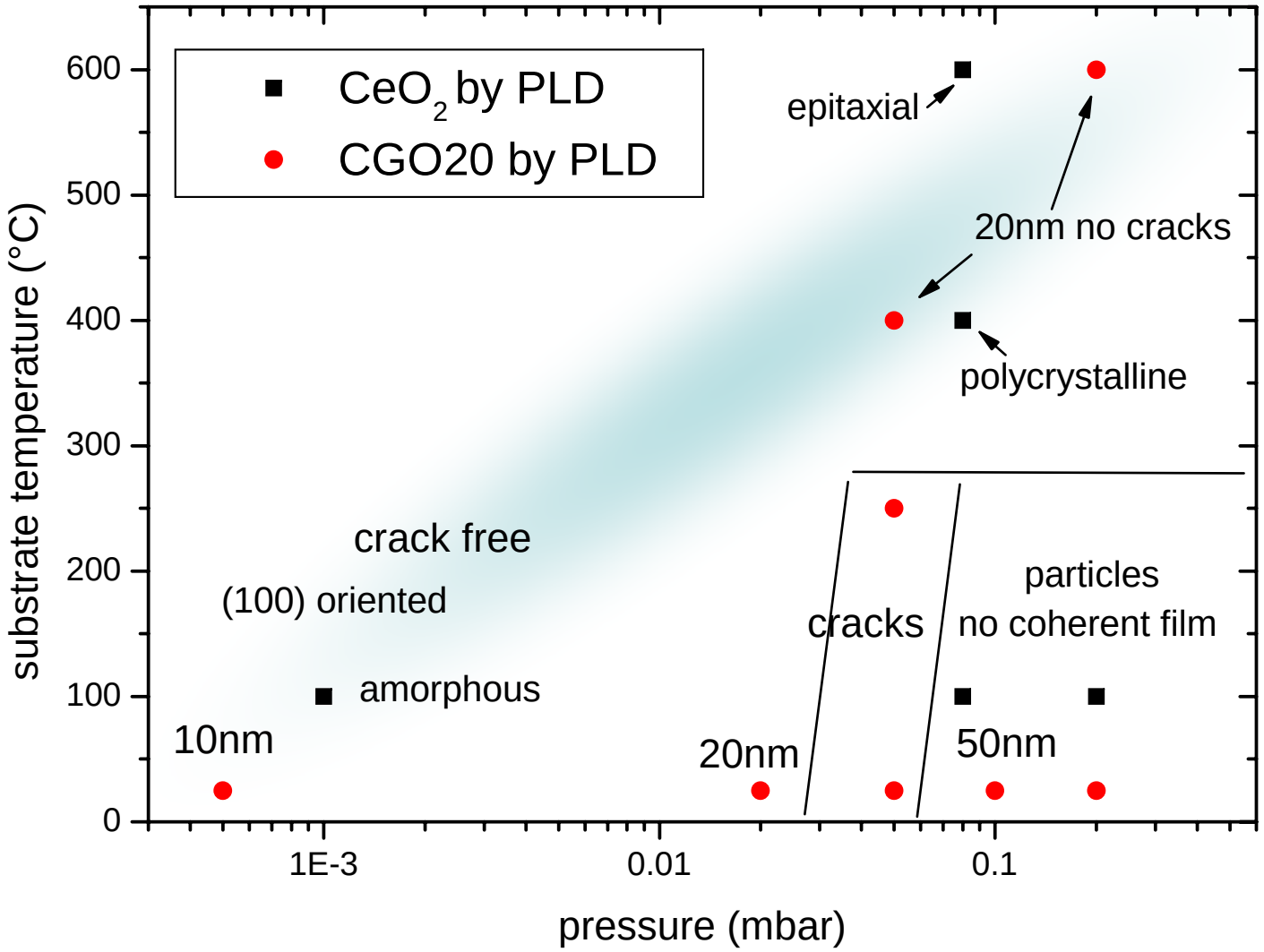
600°C, 200 m Torr



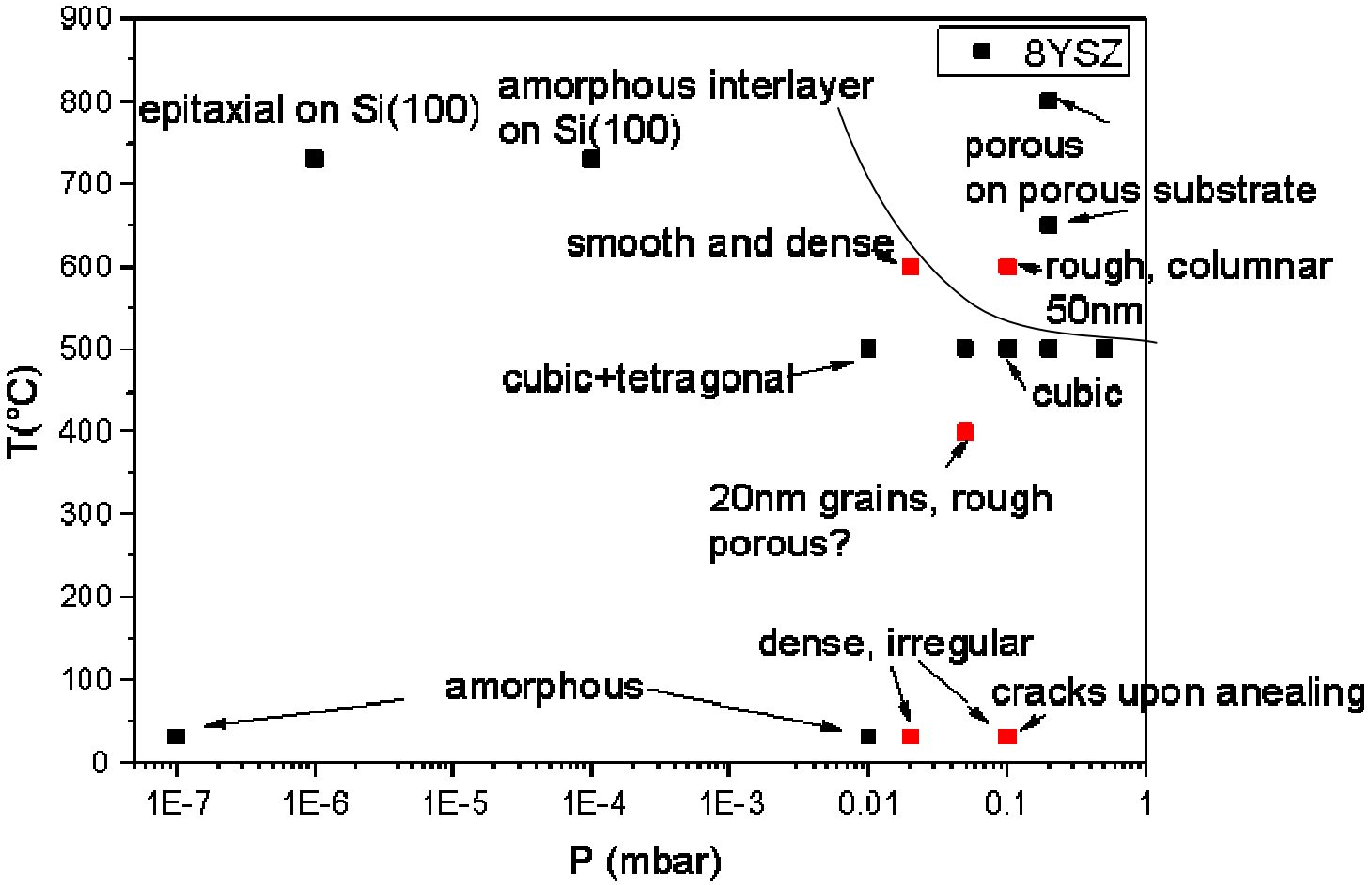


CGO prepared by PLD

A. Infortuna, L. J. Gauckler, Thin Solid Films, in press, 2005
and
Trtik, V., et al. Appl Phys A, 1999. 69: p. S815-S818.
Norton, D.P., et al. Appl, Phys Lett, 1999. 74(15): p. 2134.



PLD preparation of 8-YSZ films



A. Infortuna, L. J. Gauckler, Thin Solid Films, in press, 2005
and
Trtik, V., et al. Appl Phys A, 1999. 69: p. S815-S818.
Norton, D.P., et al. Appl, Phys Lett, 1999. 74(15): p. 2134.

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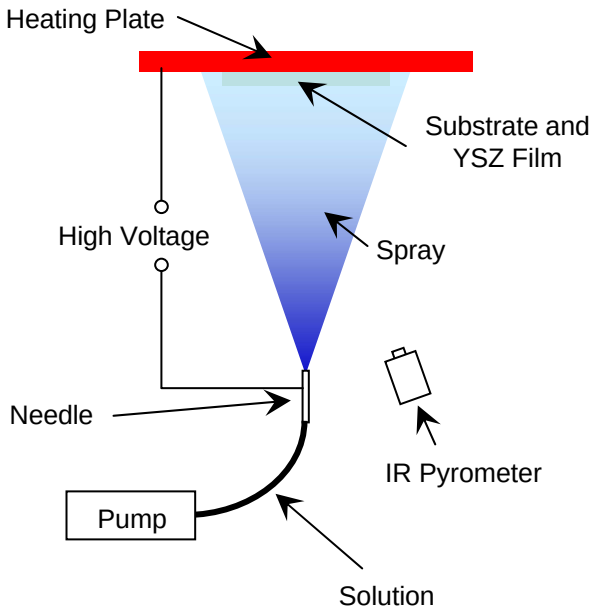
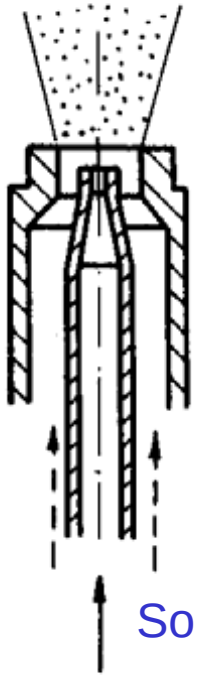
Electr spray Deposition

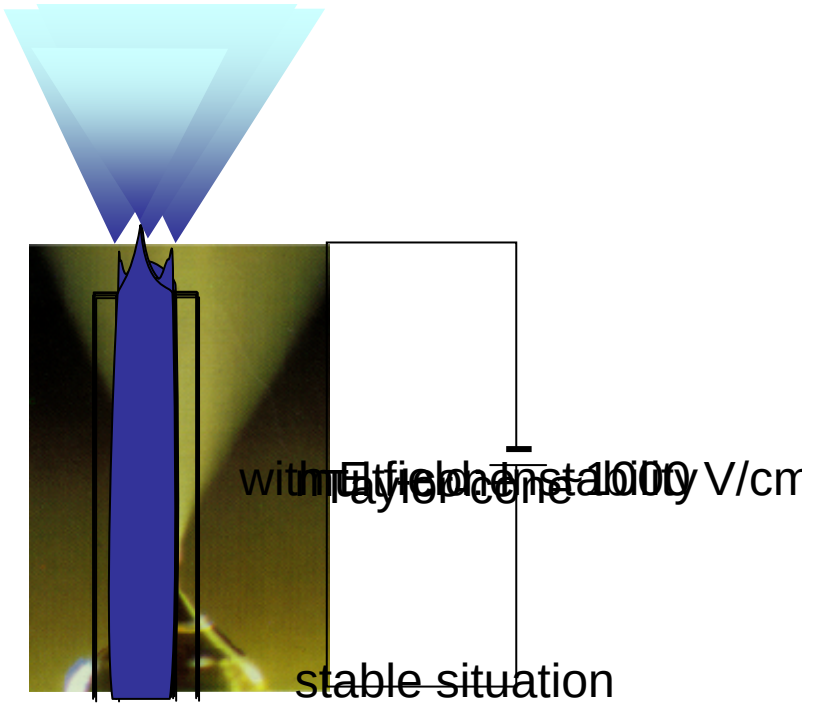




A.M. Ganan-Calvo et al., *Journal of Aerosol Science*, **28**, 249 (1997).

C.H. Chen et al., *Journal of Materials Chemistry*, **6**, 765 (1996).

D. Perednis et.al.; Thin Solid Films; 474 ; 84-95; 2005

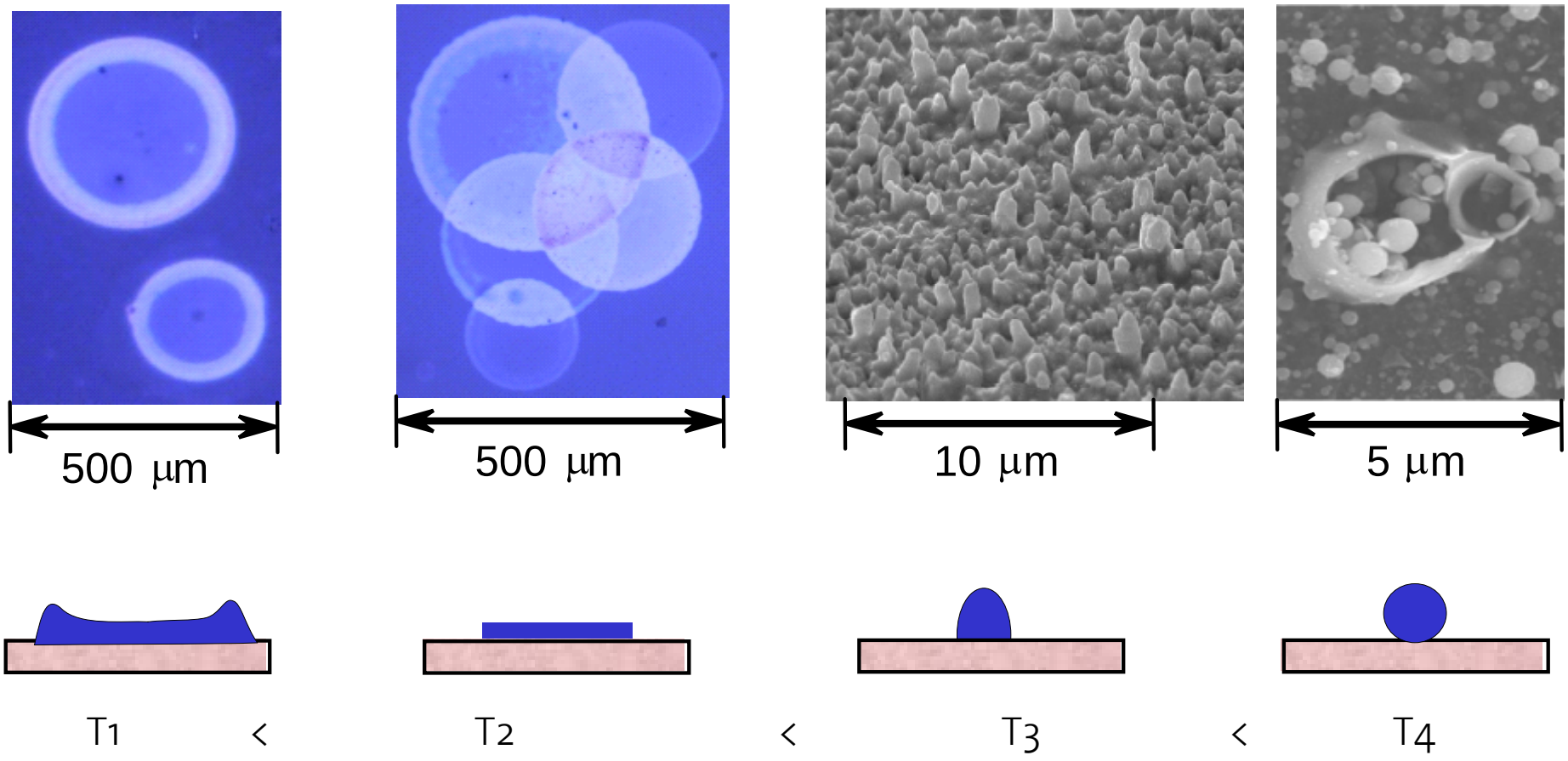


Electrostatic

Taylor cone & multi cone mode

Solvent [vol.%]	50% C_2H_5OH 50% $C_8H_{18}O_3$	50% C_2H_5OH 50% $C_8H_{18}O_3$
Salts	$Zr(C_6H_7O_2)_4$ $YCl_3 \cdot 6H_2O$	$Zr(C_6H_7O_2)_4$ $YCl_3 \cdot 6H_2O$
Concentration [mol/l]	0.1	0.1

Film Formation



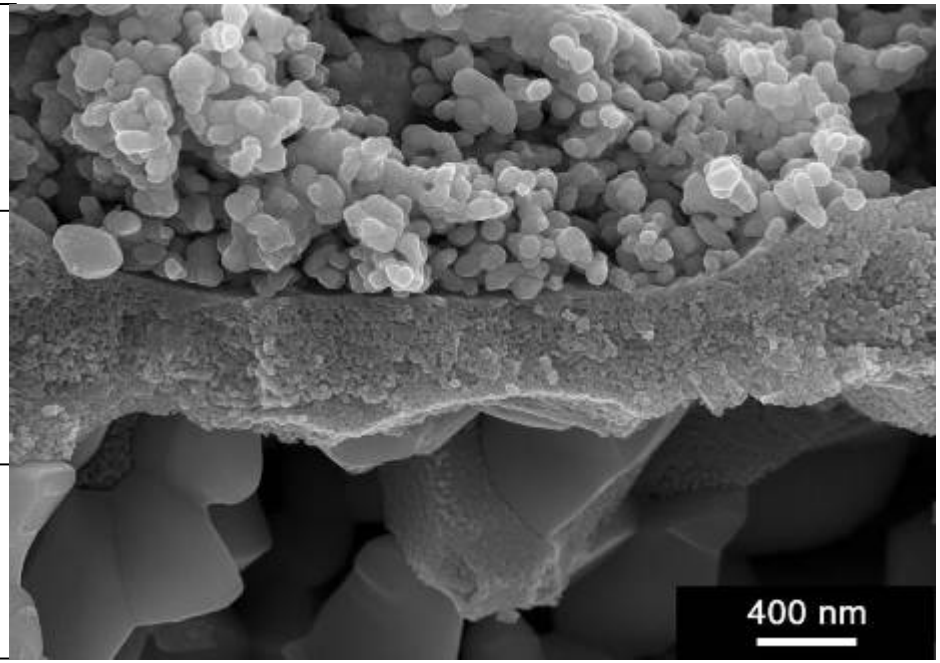
Cross-section of the fuel cell

Perednis, D.; Wilhelm, O.; Pratsinis, S.E.; Gauckler, L. J.;Thin Solid Films (474) ; 84-95 ; 2005

CATHODE
$La_{0.6}Sr_{0.4}Co_{0.2}Fe_{0.8}O_3$

ELECTROLYTE
YSZ

ANODE
Ni-YSZ



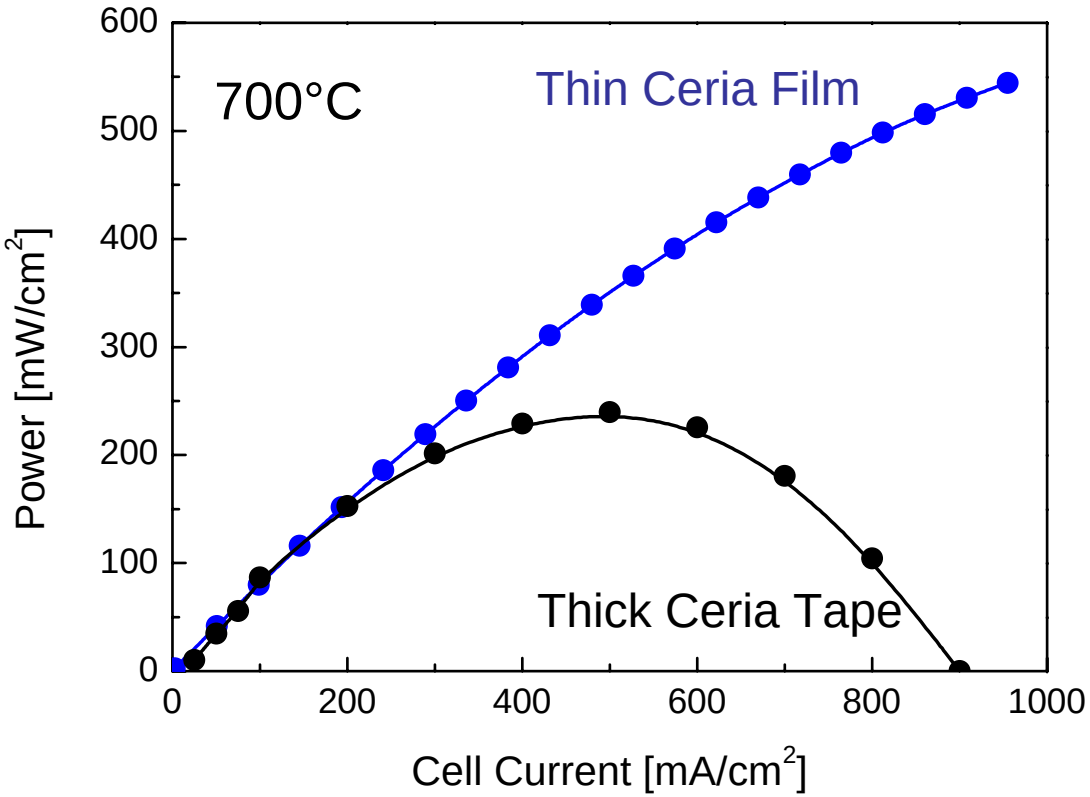
cathode screen printed
electrolyte YSZ
anode support

Dense electrolyte with thickness of 500 nm on porous anode support substrate

Grain size 30-50 nm

Cell with CeO_2ss / YSZ / CeO_2ss composite electrolyte

Perednis, D.; Wilhelm, O.; Pratsinis, S.E.; Gauckler, L. J.;Thin Solid Films (474) ; 84-95 ; 2005



Sprayed composite electrolyte:

$Ce_{0.8}Y_{0.2}O_{1.9-x}$	(0.25 μm)
YSZ	(0.25 μm)
$Ce_{0.8}Y_{0.2}O_{1.9-x}$	(0.25 μm)

Tape cast electrolyte:

$Ce_{0.8}Sm_{0.2}O_{1.9-x}$ (240 μm)

Thick Ceria Tape: M.Gödickemeier and L.J.Gauckler, *J. Electrochem. Soc.*, **145**, 414 (1998).

Improved power output due to multilayer electrolyte of

$Ce_{0.8}Y_{0.2}O_{1.9}/Zr_{0.85}Y_{0.15}O_{1.925}/Ce_{0.8}Y_{0.2}O_{1.9}$

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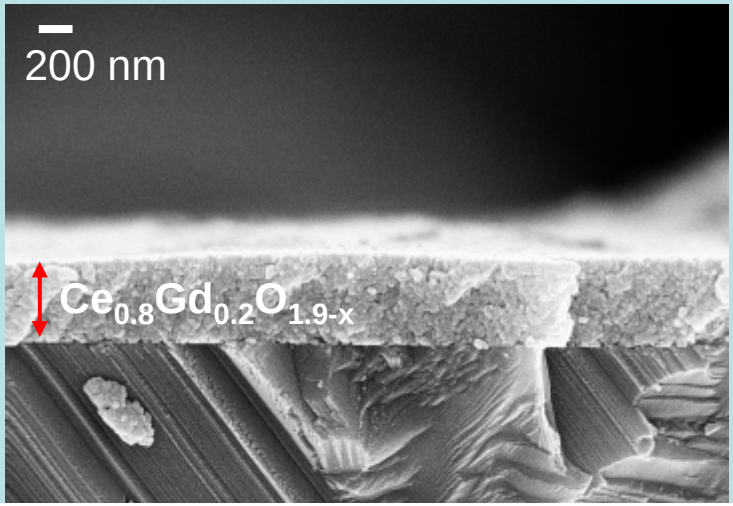
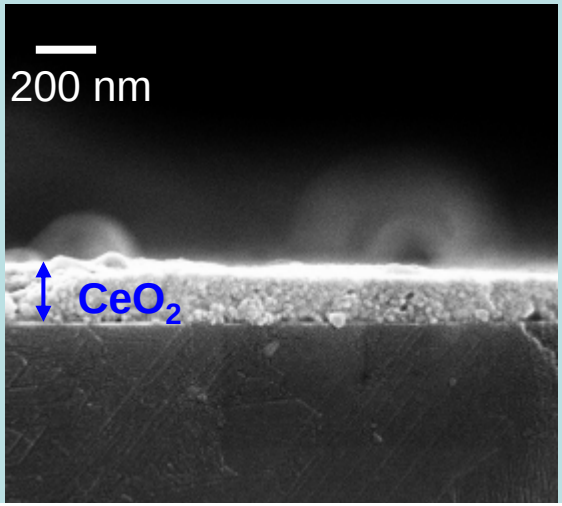
Spray pyrolysis and PLD thin films
CeO2 and CGO

Spray pyrolysis films

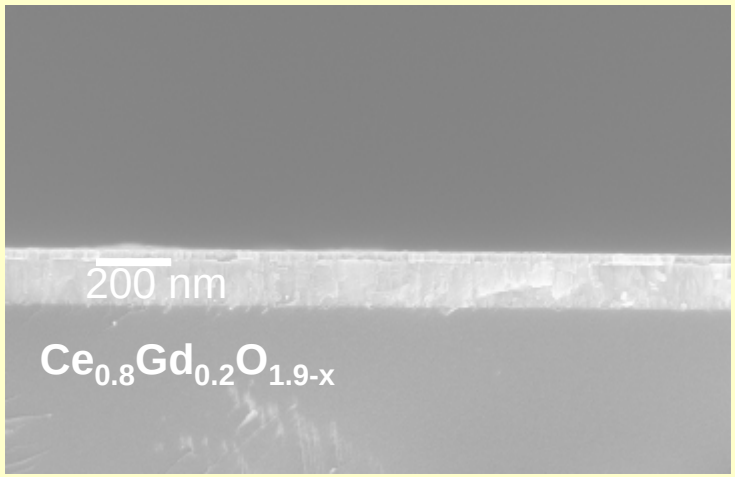
$Ce_{0.8}Gd_{0.2}O_{1.9-x}$ films



CeO_2 film



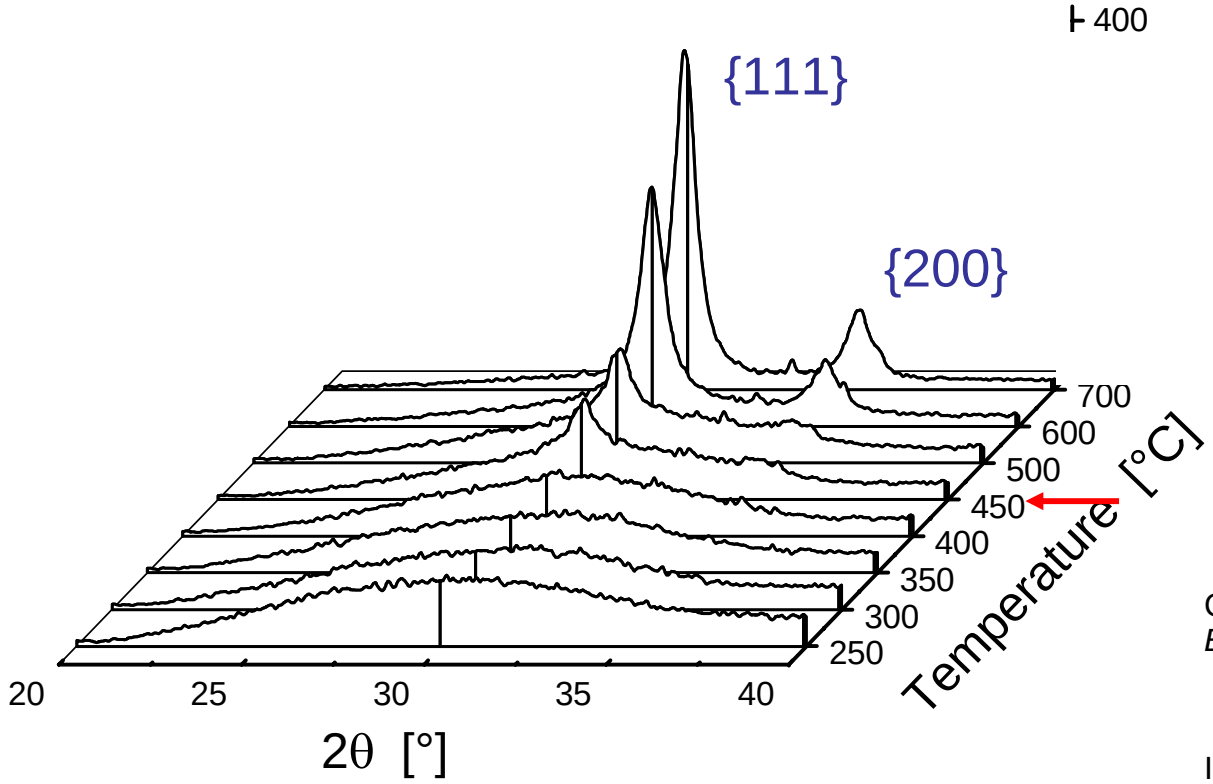
PLD film



Substrate sapphire.

Crystallization of SP films by thermal treatment

XRD patterns of YSZ film deposited on Inconel 600 at 275°C, annealing time 15 minutes



Crystalline

T. Nguyen et al, *Solid State Ionics,* **138**, 191 (2001)

325°C

C. Laberty-Robert et al, *Materials Research Bulletin,* **36**, 2083 (2001)

450°C

I. Kosacki et al, *Solid State Ionics,* **136-137**, 1225 (2000)

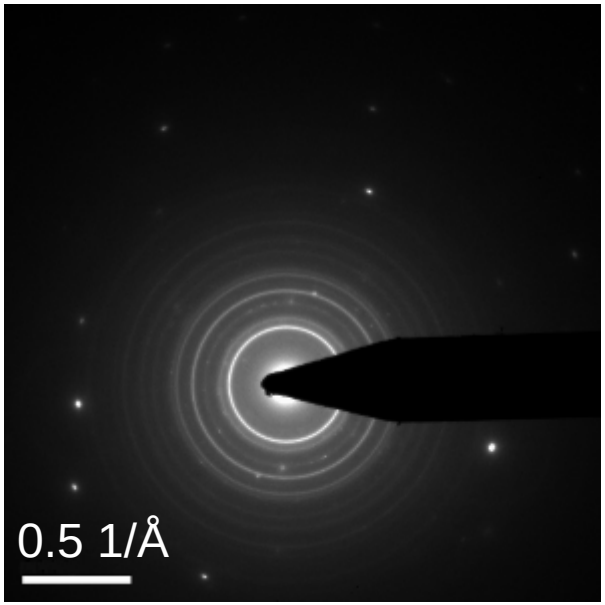
As deposited films are amorphous

Amorphous→crystalline transition onset at ~ 450°C

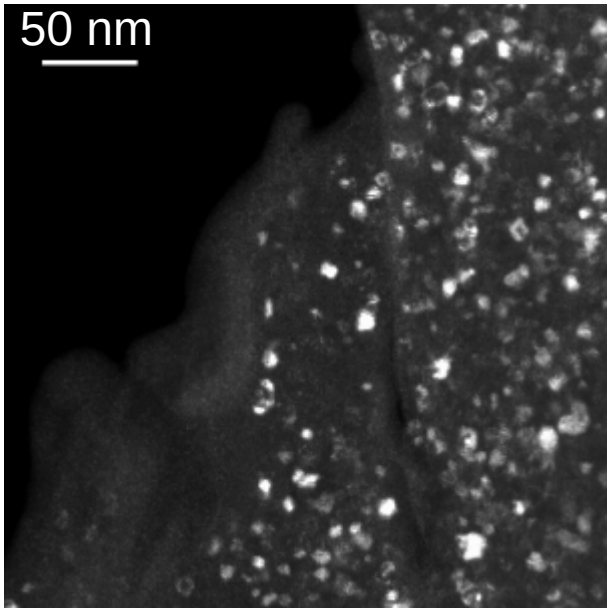
Perednis, D.; Wilhelm, O.; Pratsinis, S.E.; Gauckler, L. J.;Thin Solid Films (474) ; 84-95 ; 2005

Nano-crystalline microstructure

YSZ film on Inconel 600 after annealing at 700°C for 2 hrs



TEM Diffraction Pattern



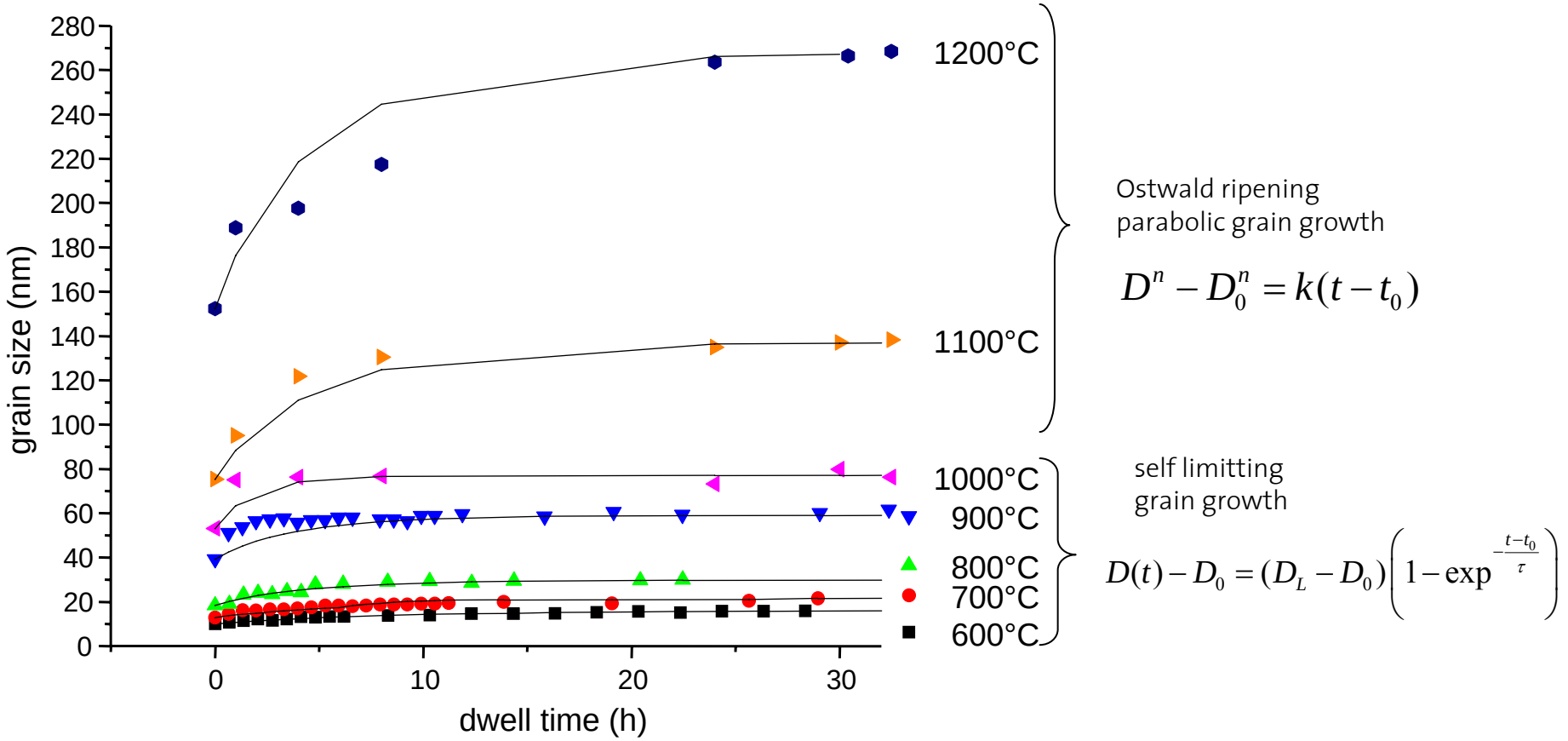
TEM Dark Field Image

⇒ Crystalline

⇒ Grain Size ~10 nm

Perednis, D.; Wilhelm, O.; Pratsinis, S.E.; Gauckler, L. J.;Thin Solid Films (474) ; 84-95 ; 2005

Thermal stability of $Ce_{0.8}Gd_{0.2}O_{1.9-x}$



Model of limiting grain size holds for grains below 120 nm

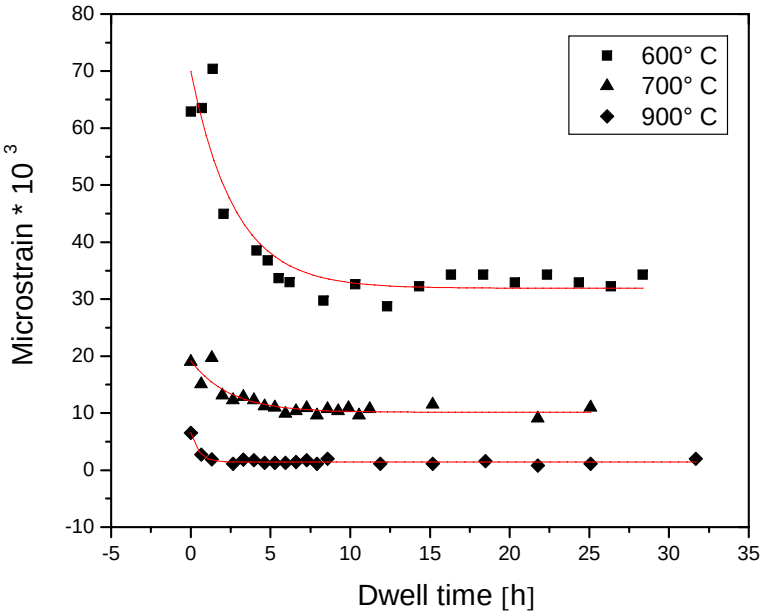
Parabolic grain growth law holds for μm grains

J. Rupp, Nonmetallic Materials ETH Zurich, Acta Mat, in press, 2005

Microstrain and Grain Growth of nano- $Ce_{0.8}Gd_{0.2}O_{1.9-x}$

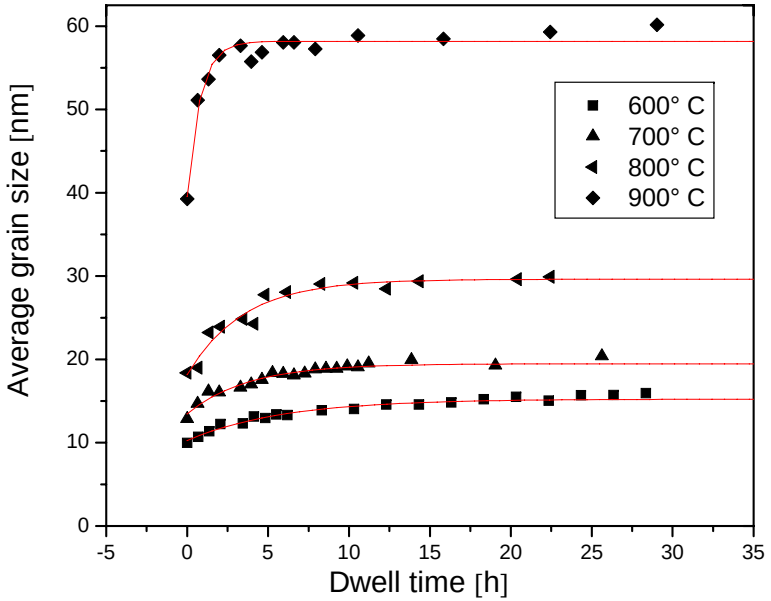
Microstrain of $Ce_{0.8}Gd_{0.2}O_{1.9-x}$

spray pyrolysis films



Grain growth of $Ce_{0.8}Gd_{0.2}O_{1.9-x}$

spray pyrolysis films



$$\varepsilon = \varepsilon_0 + D_L \exp^{-\frac{t}{\tau}}$$

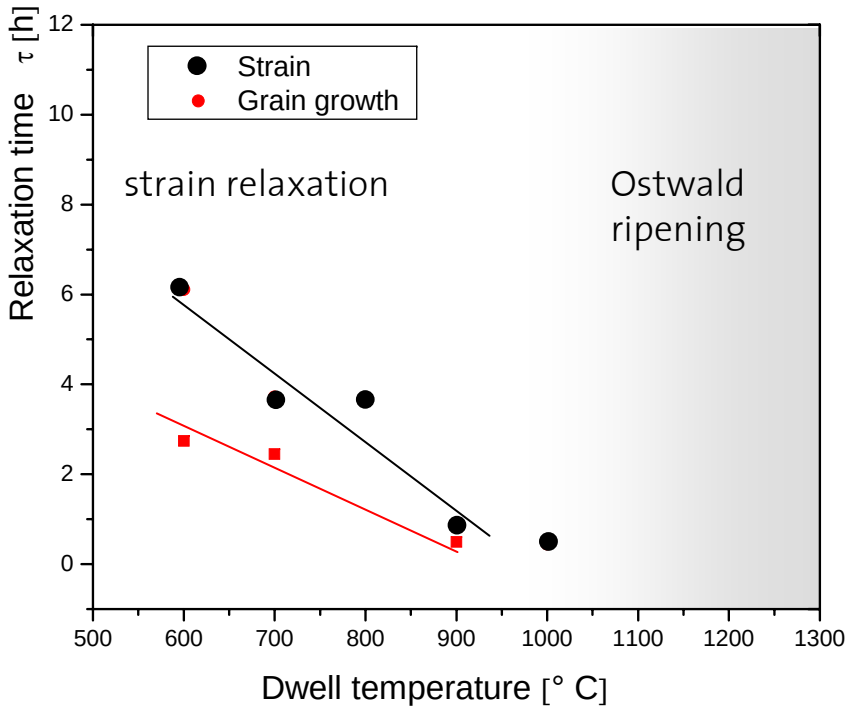
$$D(t) - D_0 = (D_L - D_0)\left(1 - \exp^{-\frac{t-t_0}{\tau}}\right)$$

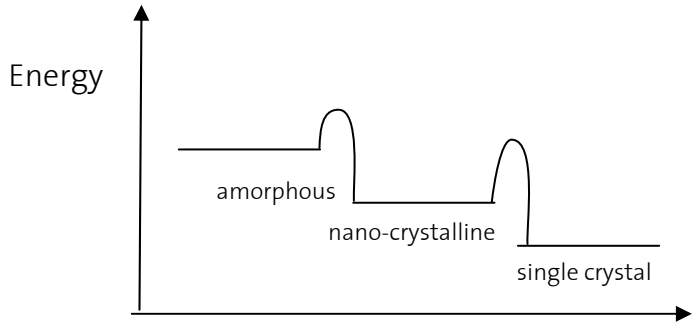
D_0 = start grain size, D = grain size, D_L = limiting grain size.

- Microstrain and grain growth cease within first 10hrs of isothermal dwell.
- Both properties follow exponential laws with a characteristic relaxation time τ

J. Rupp, Nonmetallic Materials ETH Zurich, Acta Mat, in press, 2005

Characteristic times for grain growth and strain relaxation of of nano- $Ce_{0.8}Gd_{0.2}O_{1.9-x}$ prepared by spray pyrolysis





- Increasing T (600-900°C) results in faster grain growth + faster microstrain relaxation

  →Self limitting grain growth. Metastable nanosized microstructures establishes.

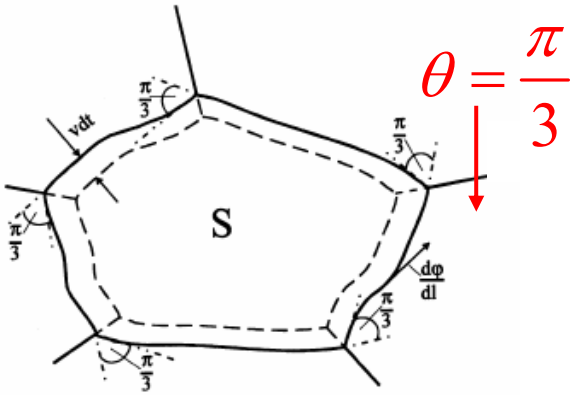
- At higher T (> 1100° C) fast microstrain relaxation and Ostwald ripening of grains.

  → Parabolic grain growth ; not limited anymore.

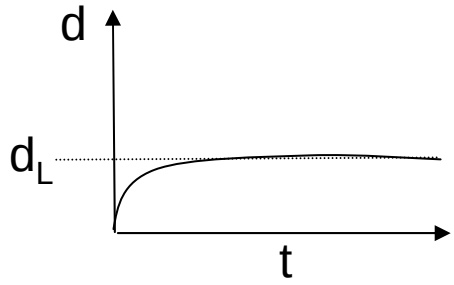
J. Rupp, Nonmetallic Materials ETH Zurich, Acta Mat, in press, 2005

Limiting Grain Growth due to Grain Size

Influence of tripple junction mobility (γ_{tj}) on GG kinetics can not be neglected for very small grains like in nano-materials.



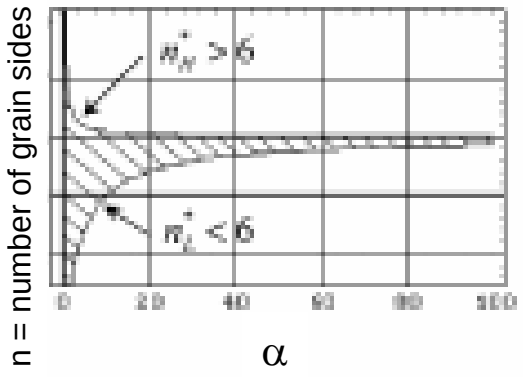
$$\alpha = \frac{\gamma_{tj} D}{\gamma_{gb}} = \frac{2\theta}{2\cos\theta - 1}$$



Small grains, small γ_{tj}:

→ Straight grain boundaries $\alpha << 1, \theta \to 0$

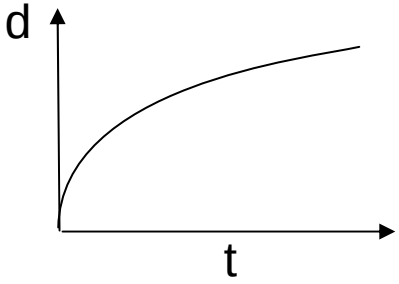
→ GG ceases !



$\alpha >> 1, \theta \to \frac{\pi}{3}$

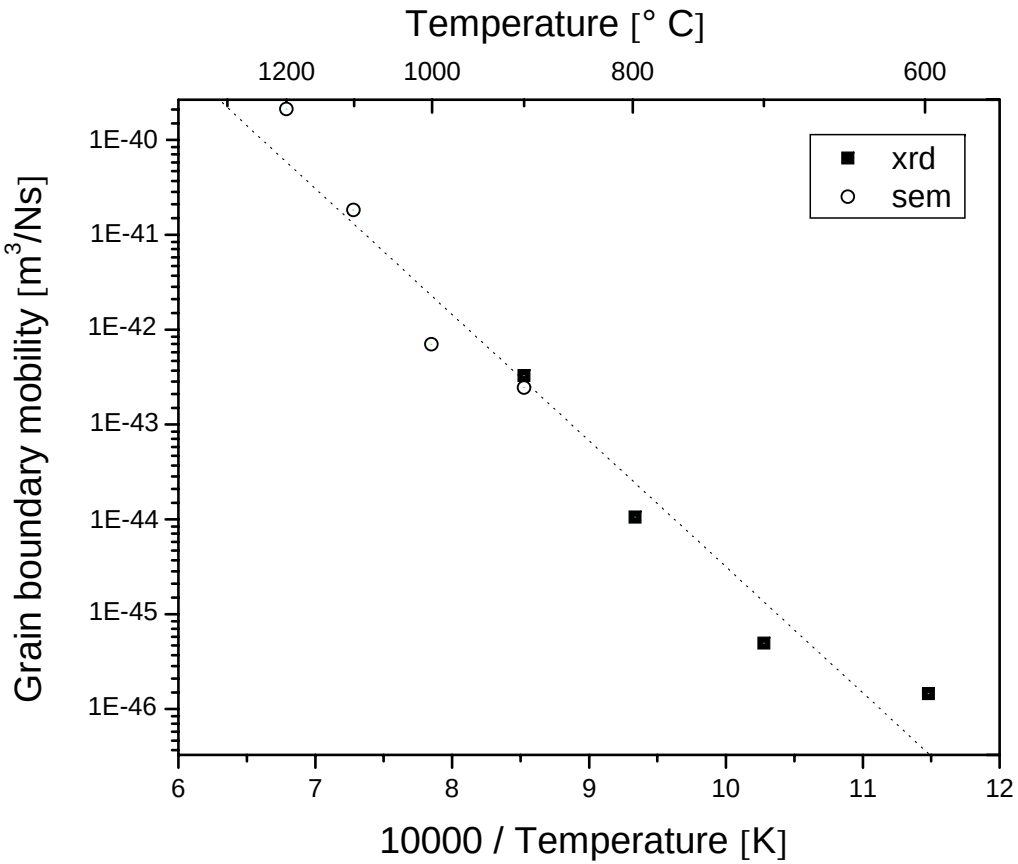
Normal parabolic gg:

→ Neumann relation holds.



Gottstein G. and Shvindlerman L.S., Acta materialia. **50, 703, 2002*
Weygand D., Brechet Y., Lepinoux J., Acta materialia. **46, 6559, 1998*
Gottstein G., Ma Y., Shvindlerman L.S., Acta materialia. **2005, in press*

Grain boundary mobility
Thermal stability of $Ce_{0.8}Gd_{0.2}O_{1.9-x}$



$$k = k_0 \exp\left(-\frac{Q}{R}\frac{1}{T}\right) \propto M$$

activation energy

d=grain size
n=grain growth exponent
t=time
R=gas constant
T=temperature
γ=grain boundary energy=0.3J/m² (*)

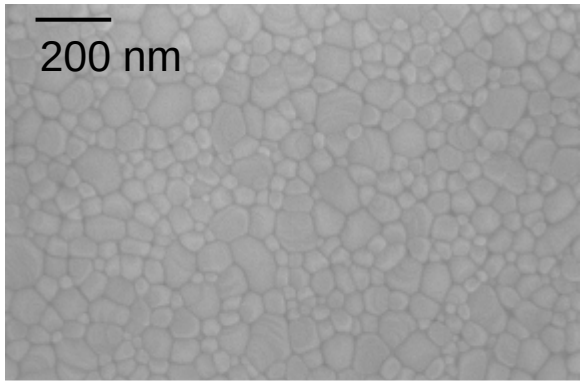
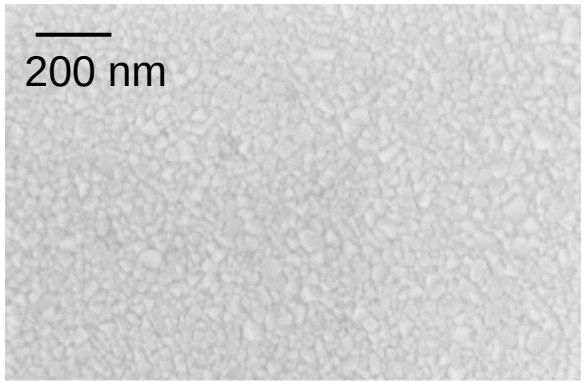
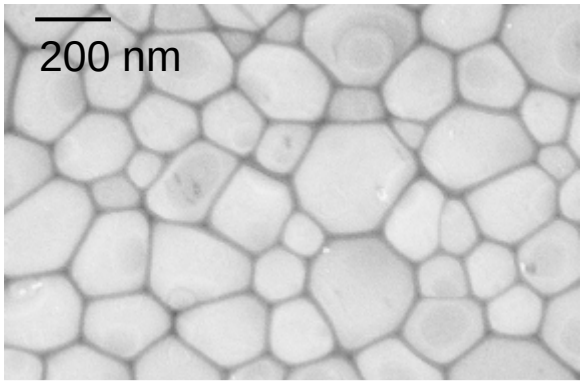
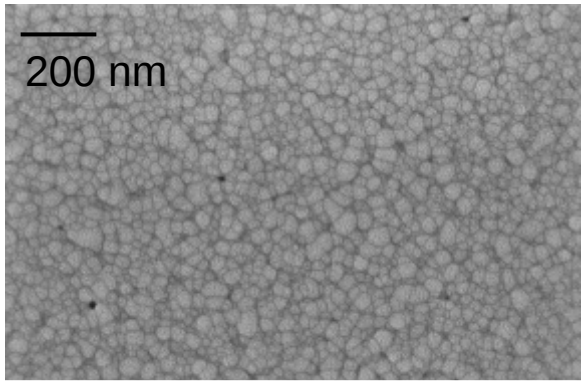
Activation energy ~1.13 eV for nm grain sized CGO
→ grain boundary diffusion

J. Rupp, Nonmetallic Materials ETH Zurich
Acta Mat, in press, 2005

Influence of doping on Grain Growth

J. Rupp, Nonmetallic Materials ETH Zurich

Spray pyrolysis thin films.

	Undoped CeO_2	**$Ce_{0.8}Gd_{0.2}O_{1.9-x}$**
1000° C		
1100° C		

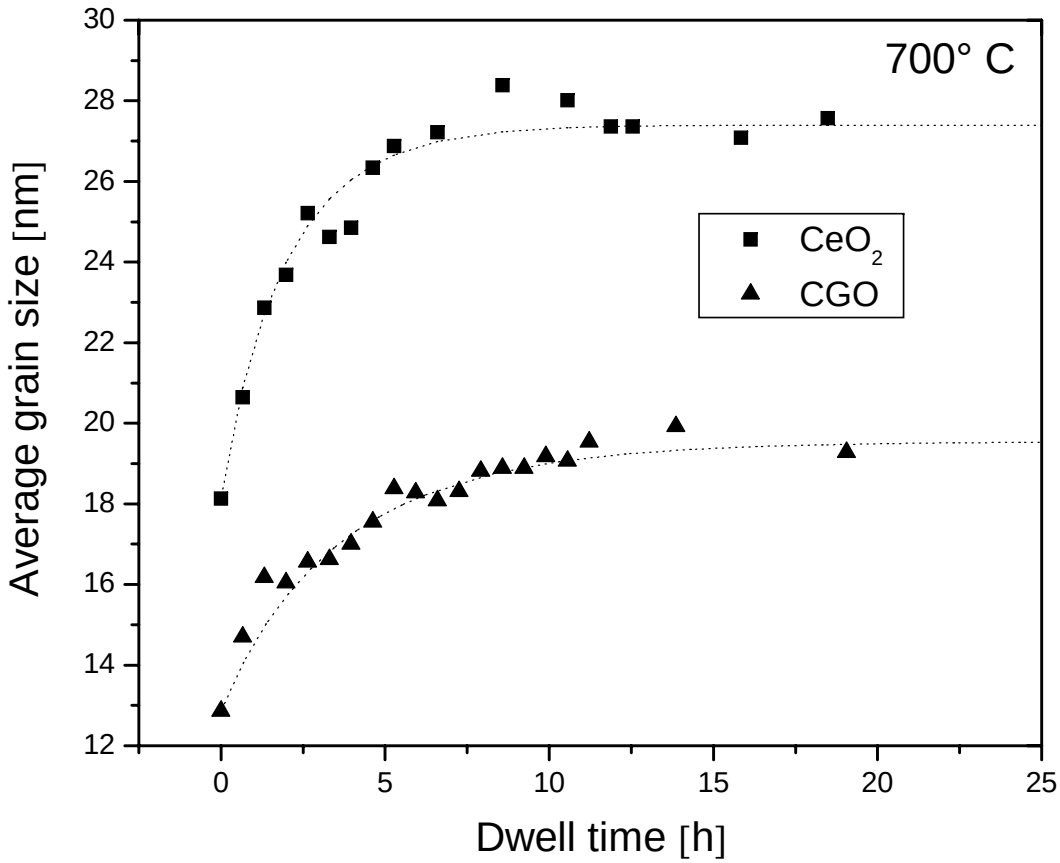
Fast grain growth in CeO_2 films

Slow grain growth in $Ce_{0.8}Gd_{0.2}O_{1.9-x}$ due to solute drag.

Influence of doping on Grain Growth

J. Rupp, Nonmetallic Materials ETH Zurich

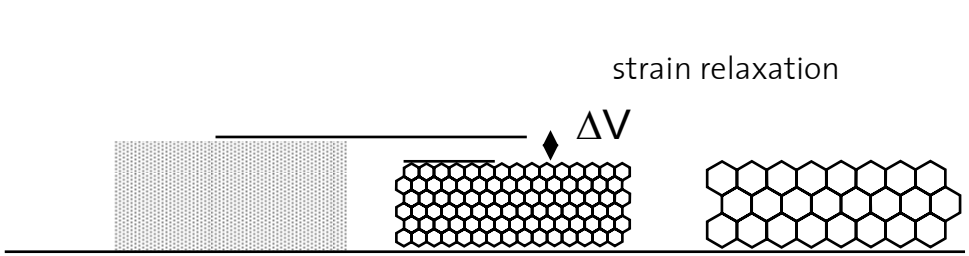
Spray pyrolysis thin films.



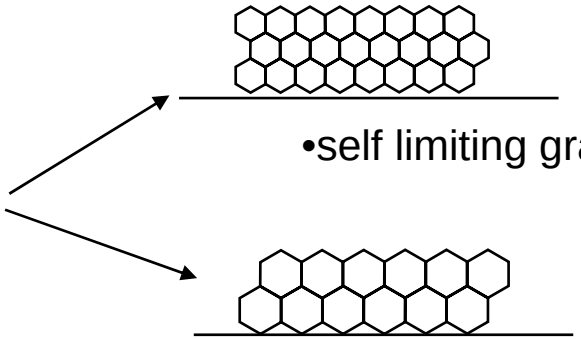
Stable microstructures after first 10 h of isothermal dwell.

Microstructural evolution: CGO thin films

J. Rupp, Nonmetallic Materials ETH Zurich
Acta Mat, 2005



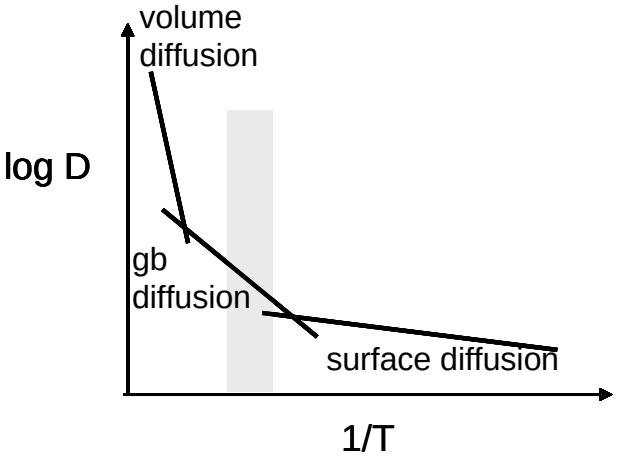
amorphous → crystalline → grain growth



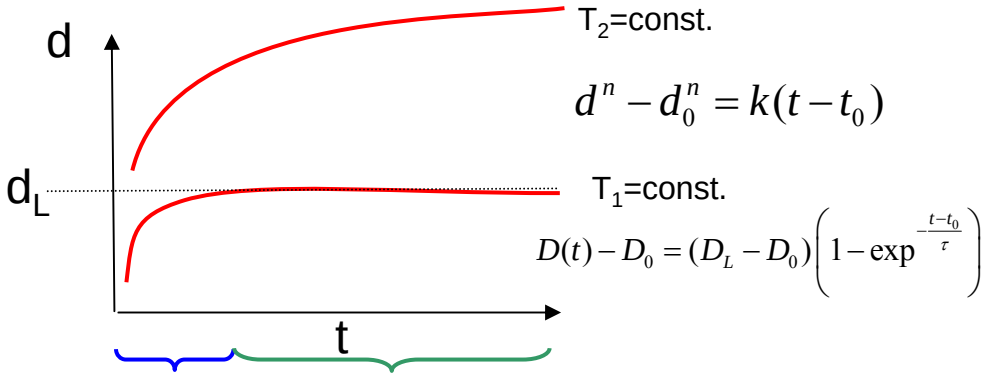
•self limiting grain growth

•parabolic grain growth

- nucleation and growth
- free volume change & shrinkage
  → dens films



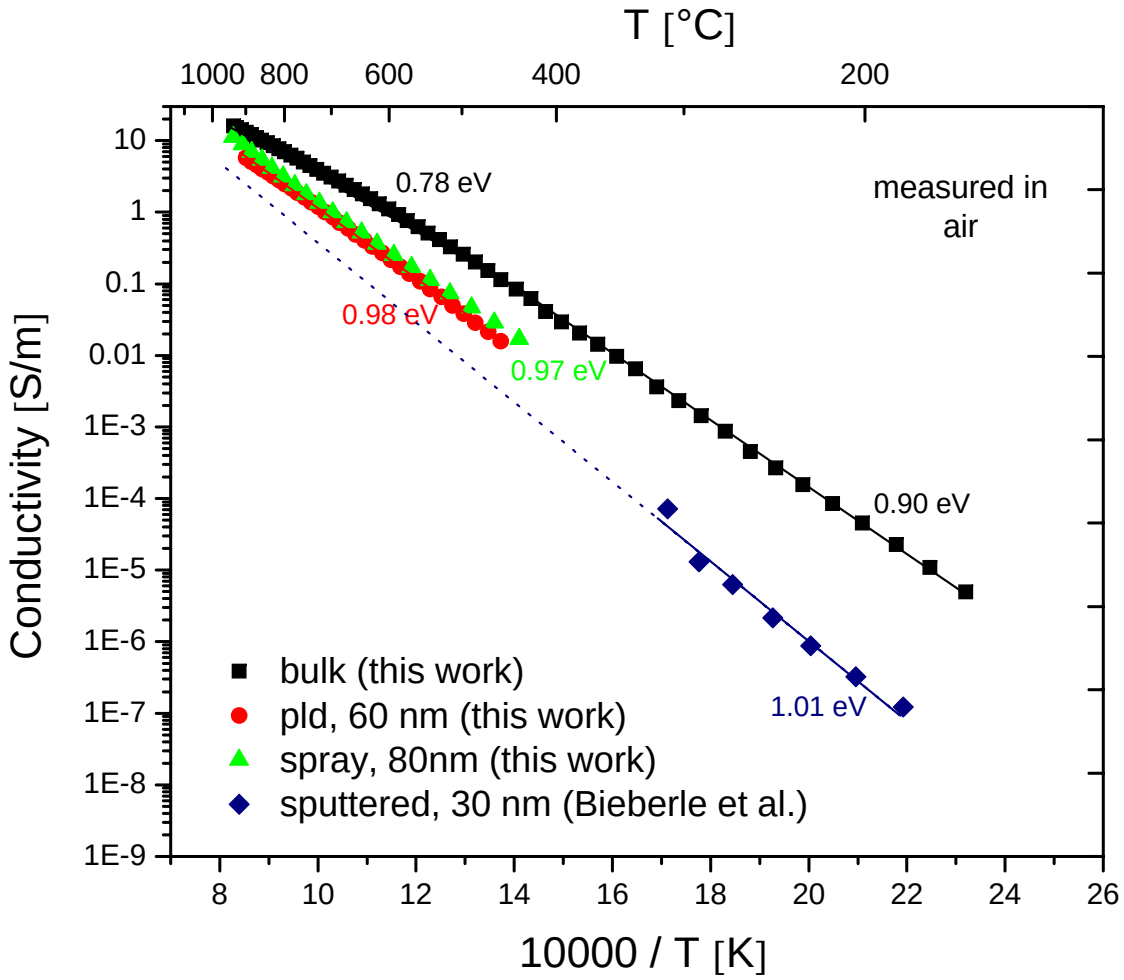
shrinkage due to
gb diffusion



nucleation and growth,
shrinkage due to
gb & volume diffusion

•normal grain growth
•self limitting grain growth

Electrical properties of $Ce_{0.8}Gd_{0.2}O_{1.9-x}$



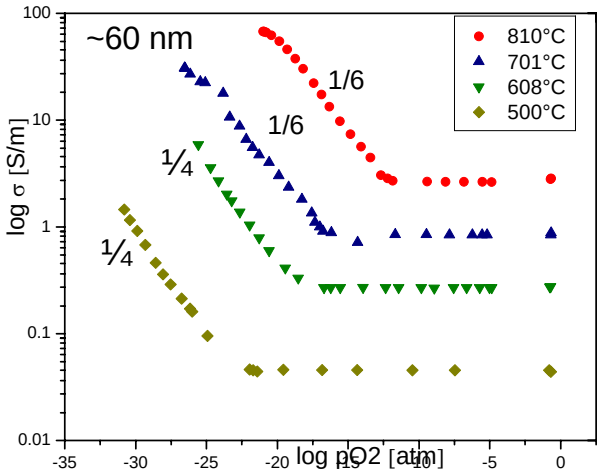
• Nanocrystalline $Ce_{0.8}Gd_{0.2}O_{1.9-x}$ thin films show lower ionic conductivity compared to microcrystalline bulk samples due to large amount of GB.

J. Rupp, Nonmetallic Materials ETH Zurich; Acta Mat,in press, 2005

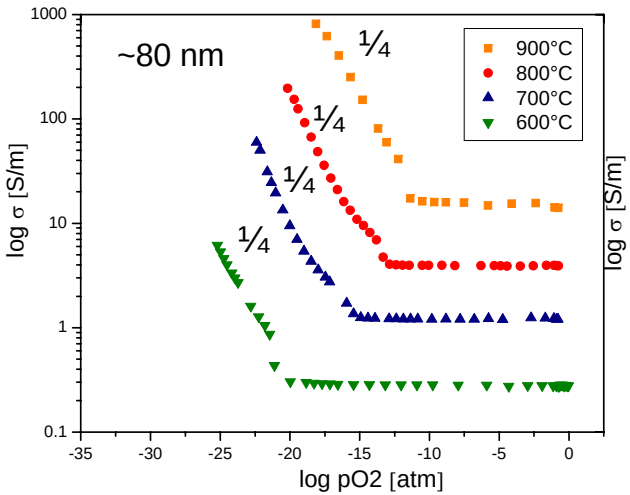
Electrical properties of $Ce_{0.8}Gd_{0.2}O_{1.9-x}$ Films & Bulk

J. Rupp, Nonmetallic Materials ETH Zurich
Acta Mat, 2005

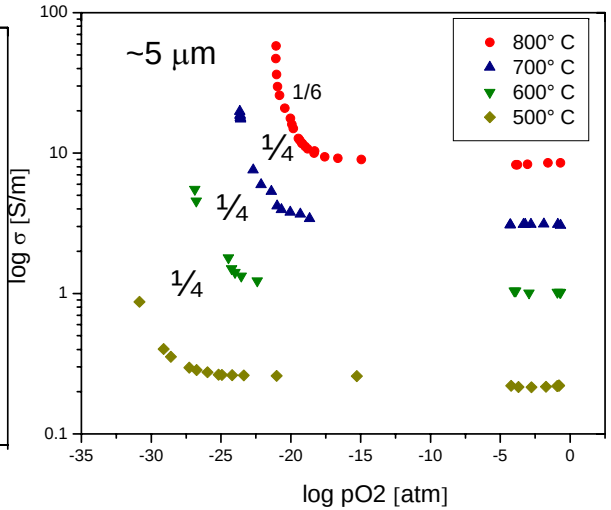
PLD



Spray pyrolysis



Bulk



- Thin film and bulk $Ce_{0.8}Gd_{0.2}O_{1.9-x}$ are predominantly ionic conductors for T < 600°C, with high enough ionic conductivity to operate as electrolytes in a SOFC system.

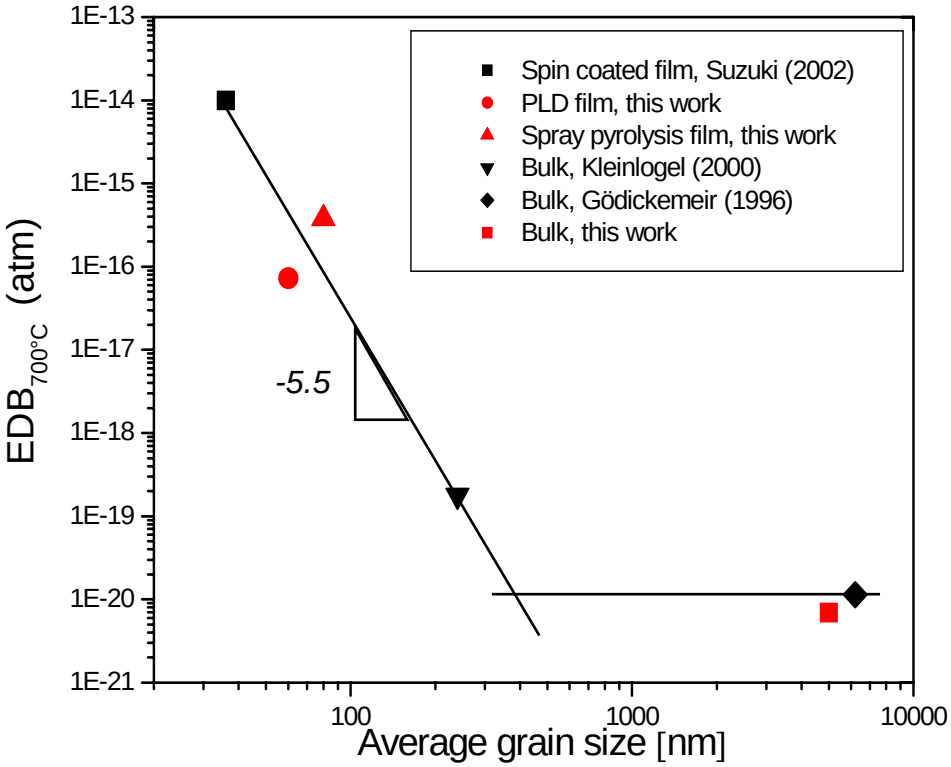
- Thin film microstructures are very stable after pre-annealing

→ low electrical conductivity degradation.

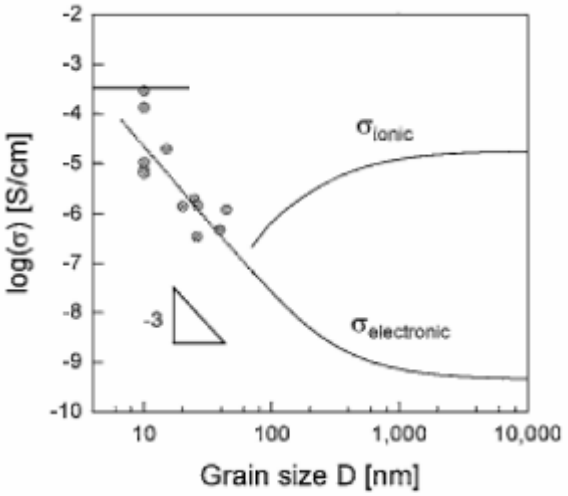
J. Rupp, Nonmetallic Materials ETH Zurich; Acta Mat, in press, 2005

Electrolytic Domain Boundary of $Ce_{0.8}Gd_{0.2}O_{1.9-x}$ and CeO_2 Thin Films

$Ce_{0.8}Gd_{0.2}O_{1.9-x}$



CeO_2



A. Tschöpe, R.Birringer,
J. Electroceramics 7, 169 (2001)

- $Ce_{0.8}Gd_{0.2}O_{1.9-x}$ and CeO_2 gets more easily reduced with decreasing grain size below ~400 nm

J. Rupp, Nonmetallic Materials ETH Zurich
Acta Mat, in press, 2005

Outline

Motivation: μ - Solid Oxide Fuel Cell & One-Bat Project

μ - Solid Oxide Fuel Cell System

μ - Solid Oxide Fuel Cell Hot Plate

Pulsed Laser Deposition
Spray Pyrolysis

Electrolyte
Cathode
Anode & current collector

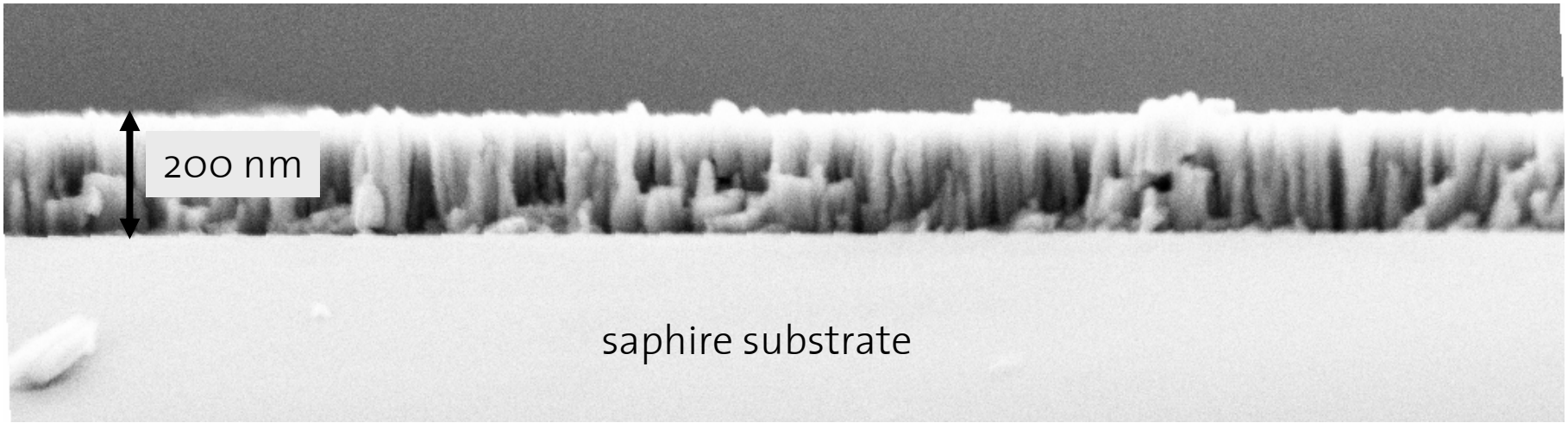
Acknowledgement

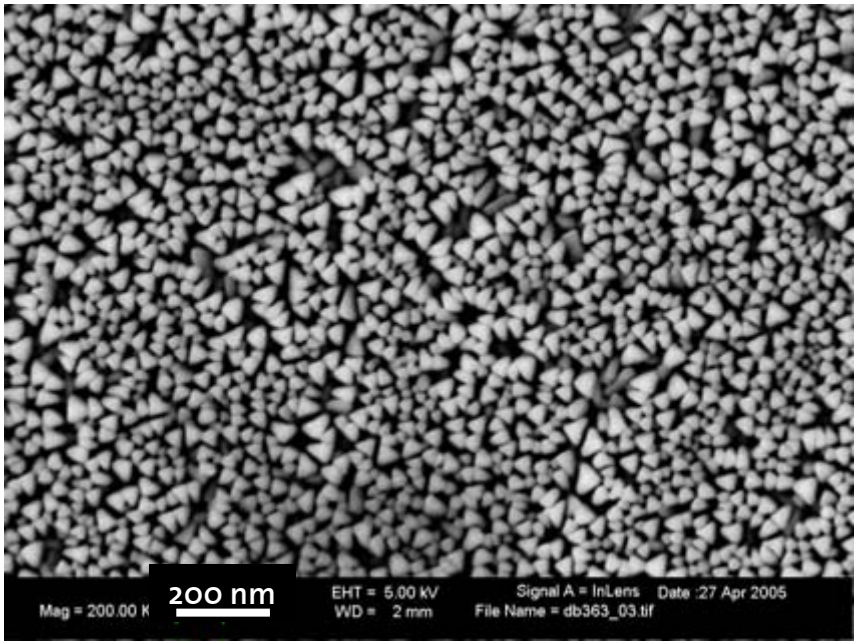
Cathode Materials

- **PLD $La_{0.6}Sr_{0.4}Co_{0.2}Fe_{0.8}O_3$ Thin Films**
- **Spray Pyrolysis $La_{0.6}Sr_{0.4}Co_{0.2}Fe_{0.8}O_3$ Thin Films**

$La_{0.6}Sr_{0.4}Co_{0.2}Fe_{0.8}O_3$ by PLD

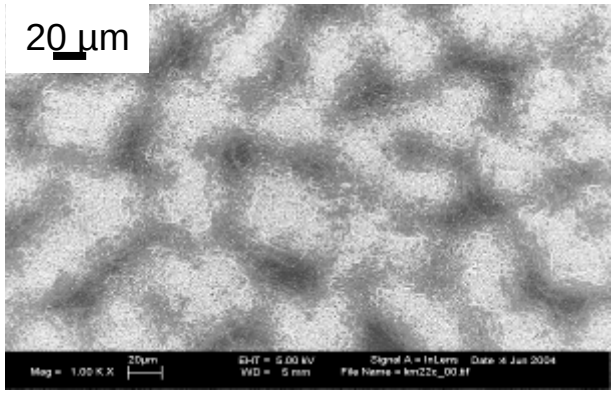
A. Infortuna; Nonmetallic Materials ETH Zurich





Ratio of Substrate Temperature to Solvent Boiling Point for SP $La_{0.6}Sr_{0.4}Co_{0.2}Fe_{0.8}O_3$

D. Beckel; Nonmetallic Materials ETH Zurich

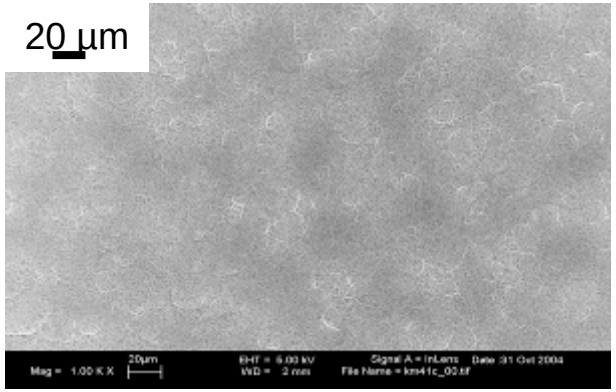


T_{Dep} = 255 °C, T_{SBP} = 180 °C

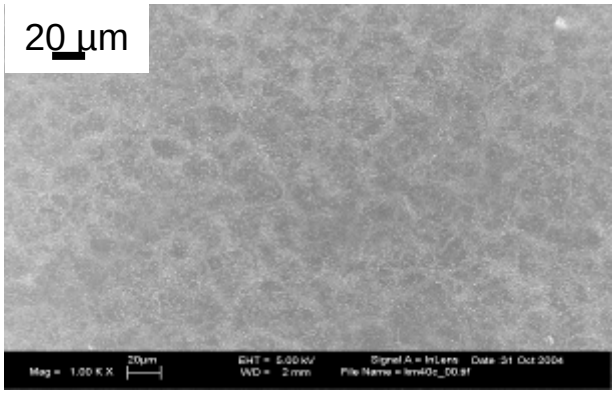
LSCF deposited at

$$\frac{T_{Deposition}}{T_{SolventBoilingPoint}} = 1.15 \ \text{(in K)}$$

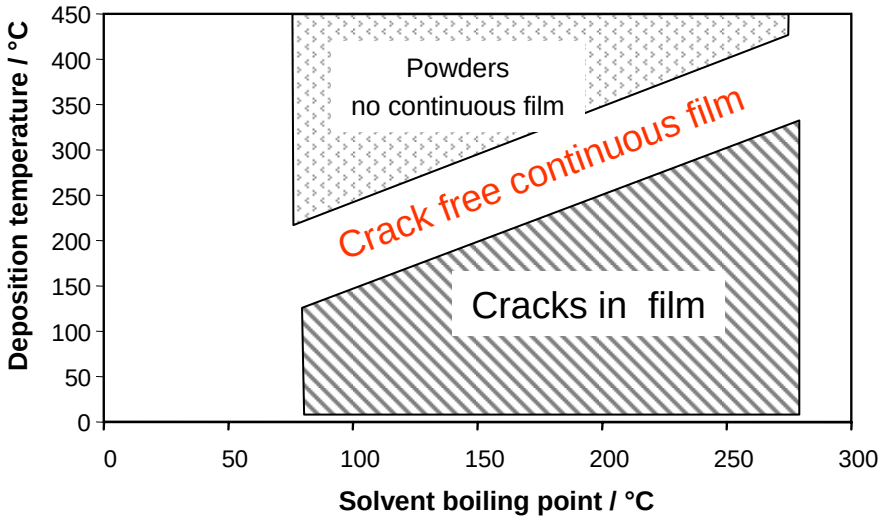
Deposition parameters:
30 ml / h, 0.02 mol / l,
60 min, 1 bar.
Films annealed



T_{Dep} = 225 °C, T_{SBP} = 155 °C



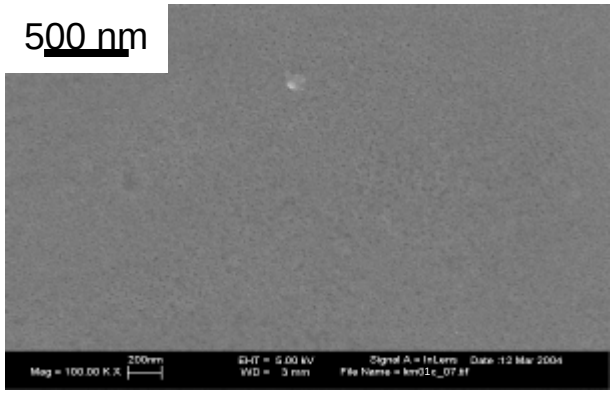
T_{Dep} = 190 °C, T_{SBP} = 130 °C



Ratio of deposition temperature to solvent boiling point most important.

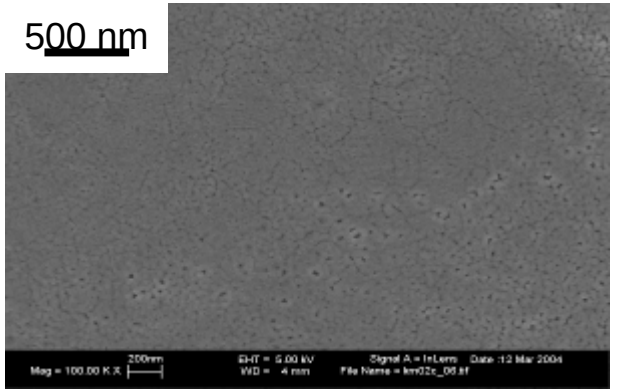
SP $La_{0.6}Sr_{0.4}Co_{0.2}Fe_{0.8}O_3$ films: microstructures after annealing

D. Beckel; Nonmetallic Materials ETH Zurich



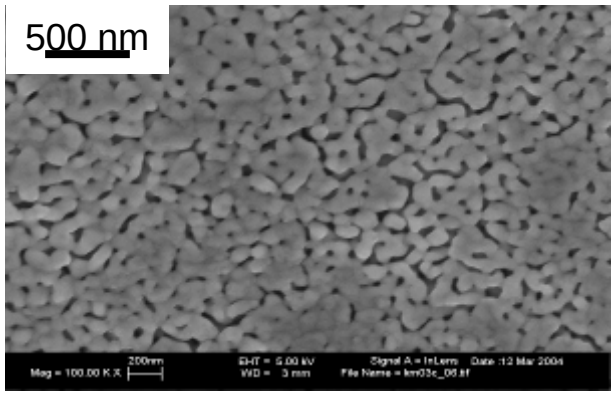
600 °C

LSCF after 3h annealing with varying annealing temperature on Si.



6 %

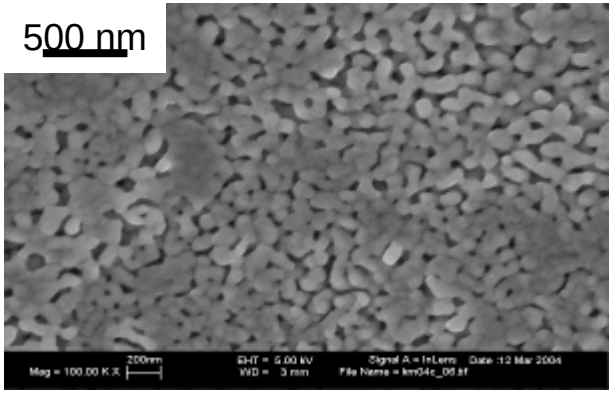
700 °C



20%

800 °C

Deposition parameters:
255 °C, 1 bar, 30 ml / h,
0.04 mol / l, 30 min.
Heating rate 2 °C / min.



30%

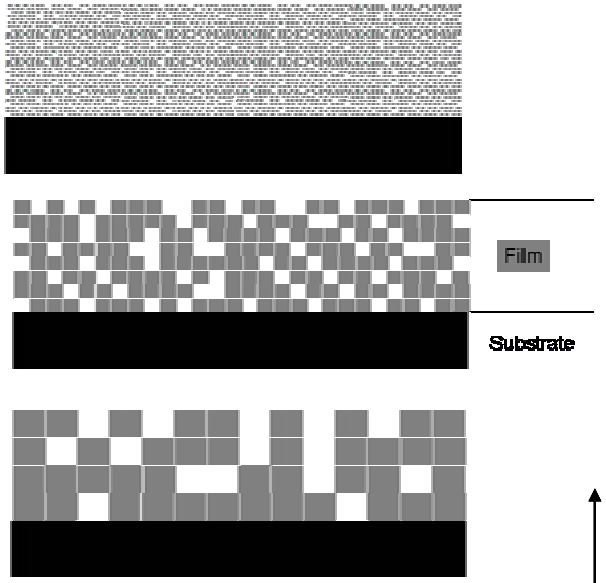
900 °C

Pores form and coalesce depending on annealing temperature.

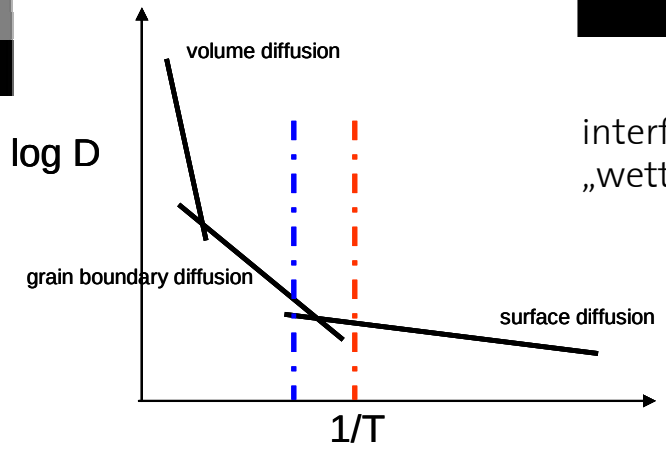
Sintering and pore coalescence during annealing of SP deposited films

grain growth and pore formation & growth
no shrinkage , no densification

$La_{0.6}Sr_{0.4}Co_{0.2}Fe_{0.8}O_3$ films

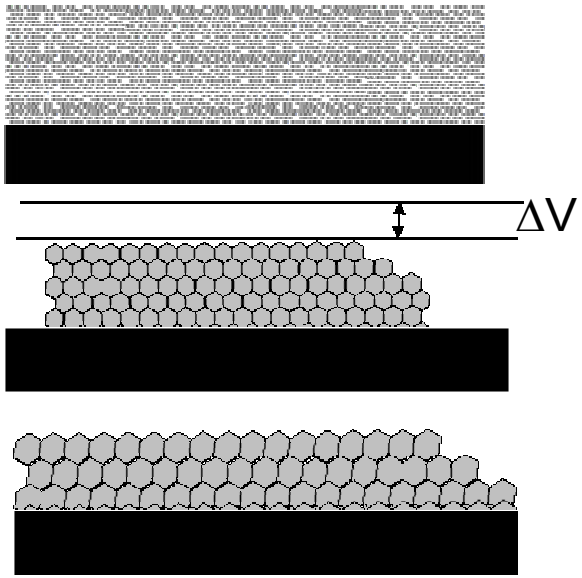


interfacial energy $\gamma_{LSCF/sobstrate}$ =large
„de-wetting“



grain growth and shrinkage

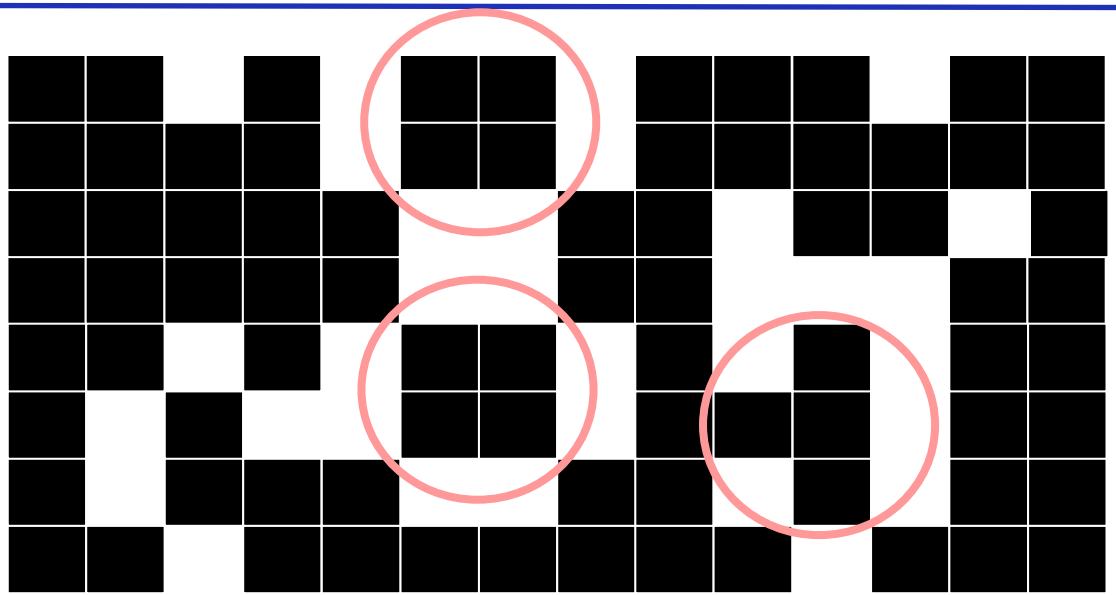
CGO films



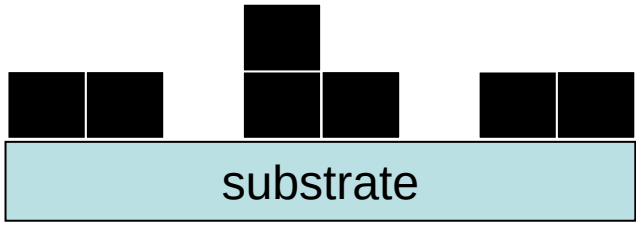
interfacial energy $\gamma_{CGO/substrate}$ =small
„wetting“

Consequences of Pore Coalescence I (grain size ~ film thickness)

$La_{0.6}Sr_{0.4}Co_{0.2}Fe_{0.8}O_3$



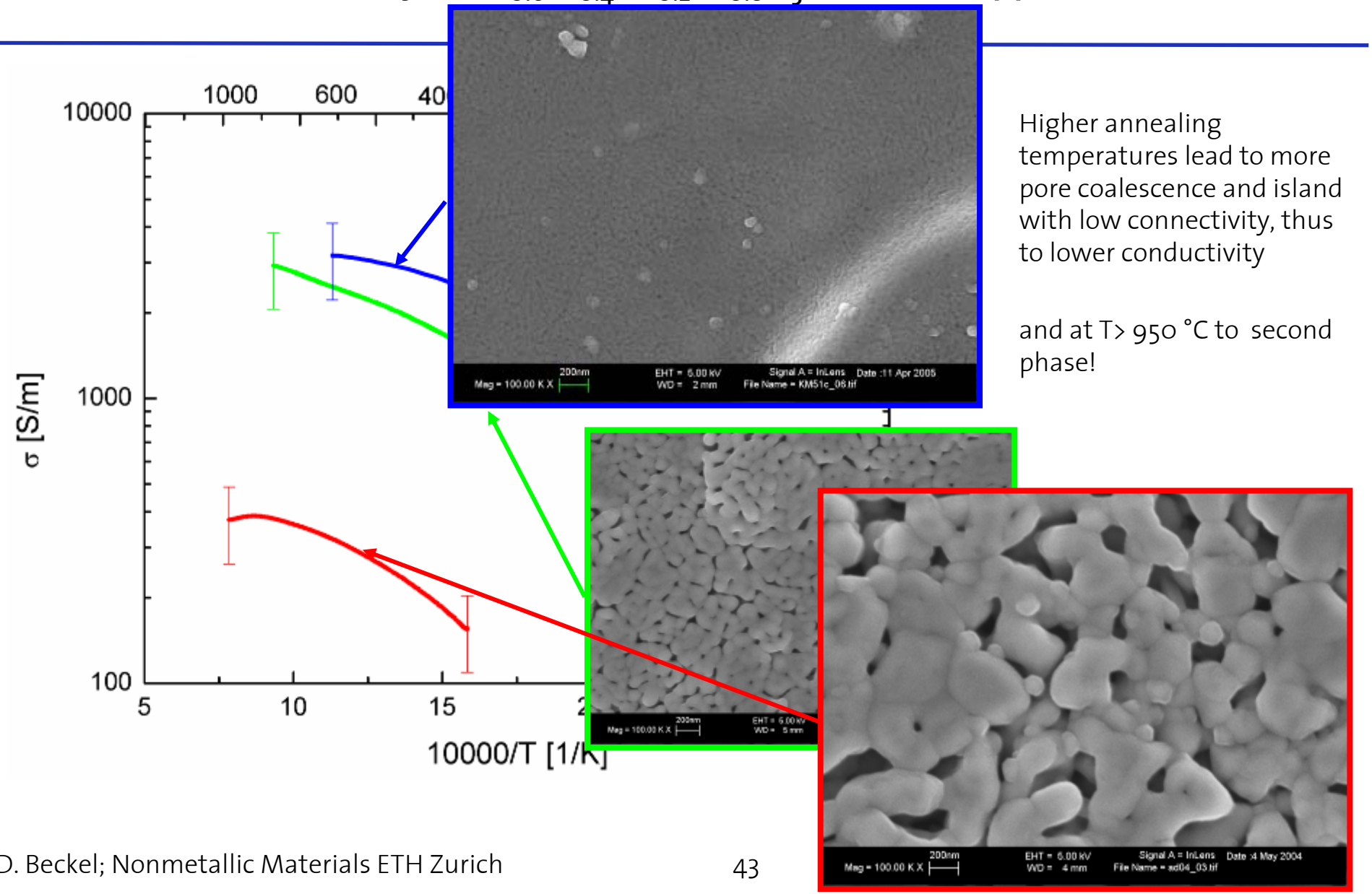
top view



side view

- Pore coalescence $\rightarrow$ islands with low connectivity.
- Isolated islands do not contribute to conductivity.
- Electric conductivity should decrease upon pore coalescence for larger grain sizes.

Electric conductivity of $La_{0.6}Sr_{0.4}Co_{0.2}Fe_{0.8}O_3$ films on sapphire annealed



Higher annealing temperatures lead to more pore coalescence and island with low connectivity, thus to lower conductivity

and at T> 950 °C to second phase!

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μ - Solid Oxide Fuel Cell Hot Plate

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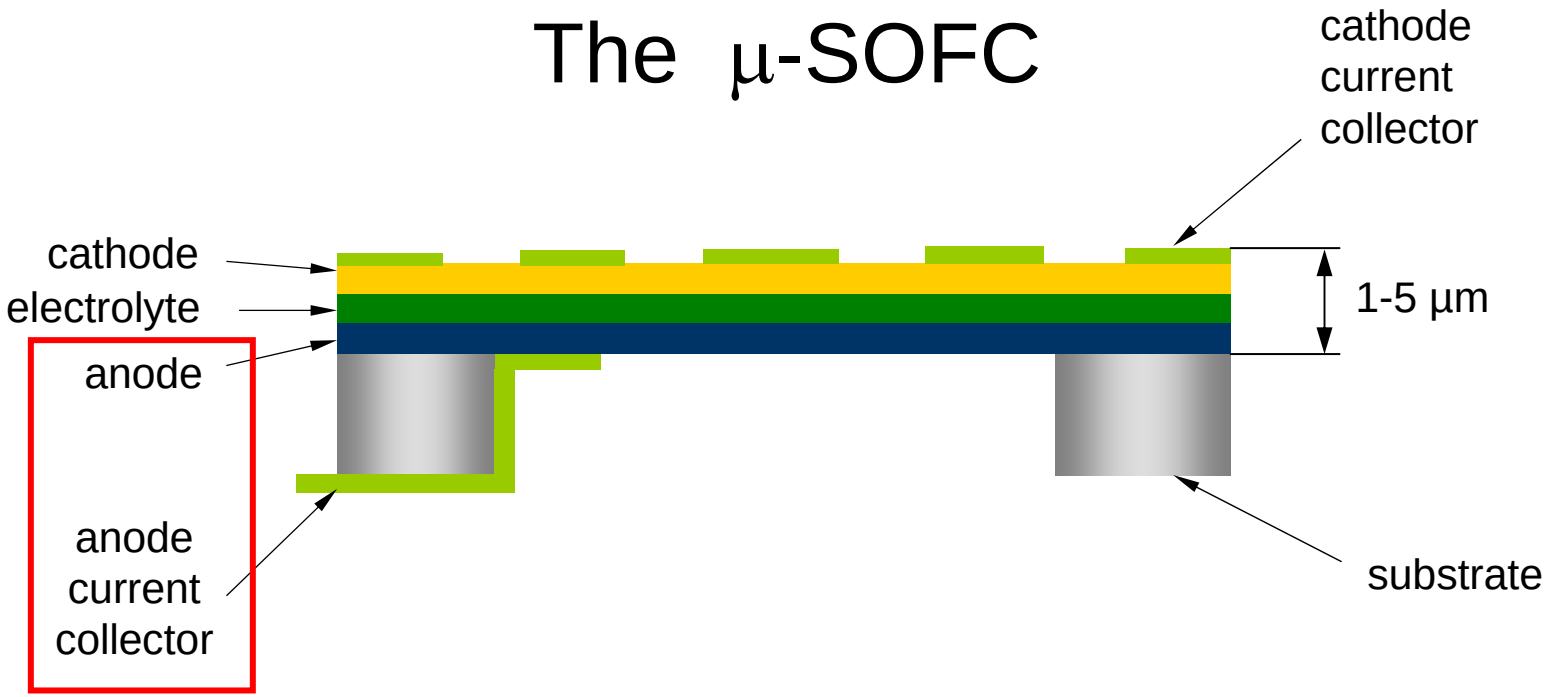
Electrolyte
Cathode
Anode & current collector

Acknowledgement

The μ-SOFC



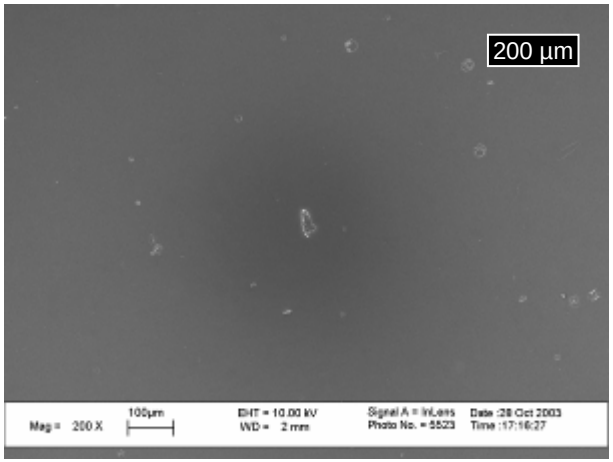
Ni-CGO anode thin film and a Ni current collector operating at 550°C.

Deposition of pure NiO Films

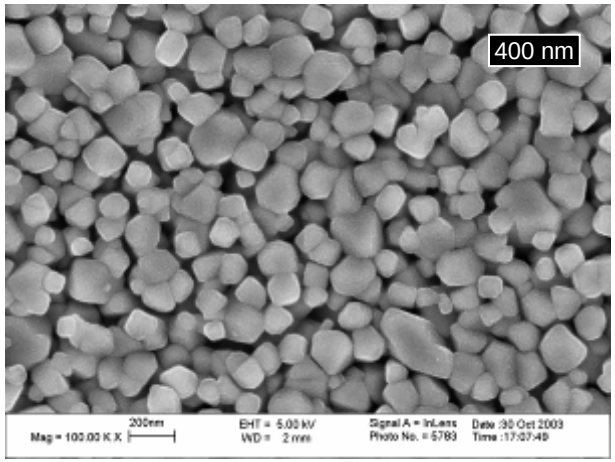
U. Mücke; Nonmetallic Materials ETH Zurich

substrate
500 µm tape casted YSZ

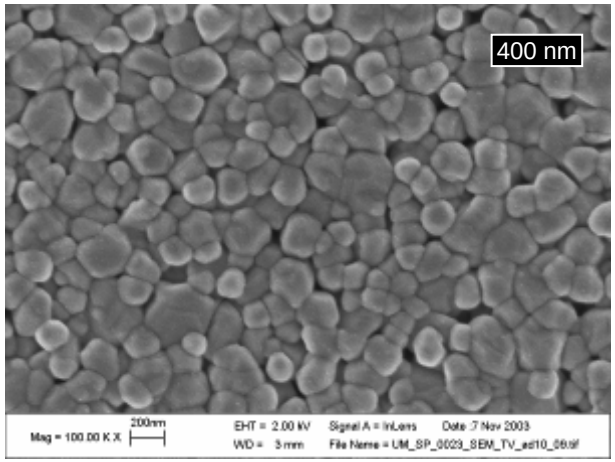
precursor
0.1 mol/l $NiNO_3 \times 6H_2O$



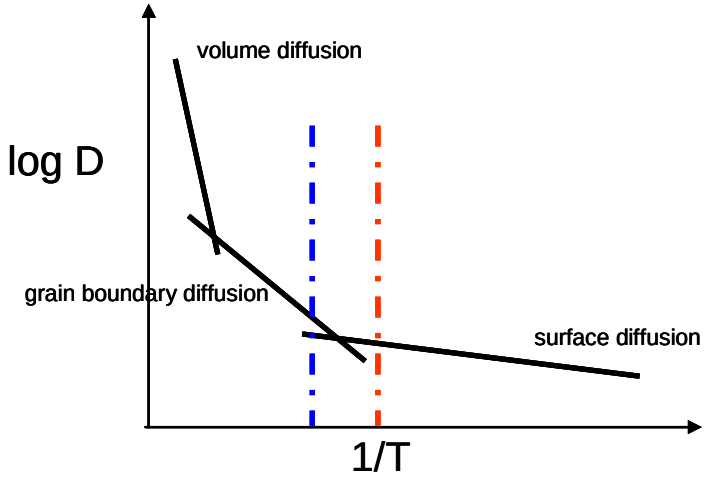
as deposited



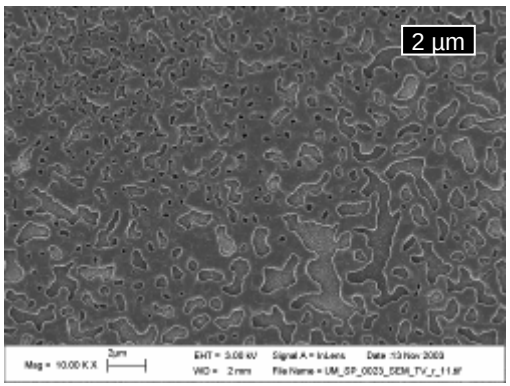
annealed at 800 °C for 10 hrs

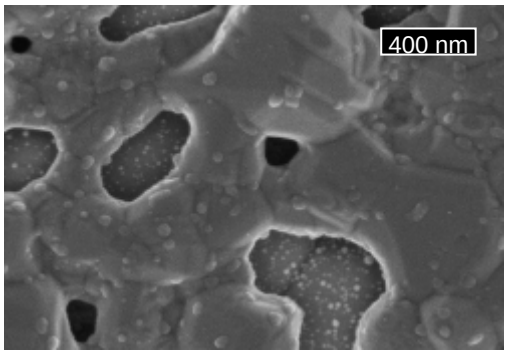


annealed at 1000 °C for 10 hrs



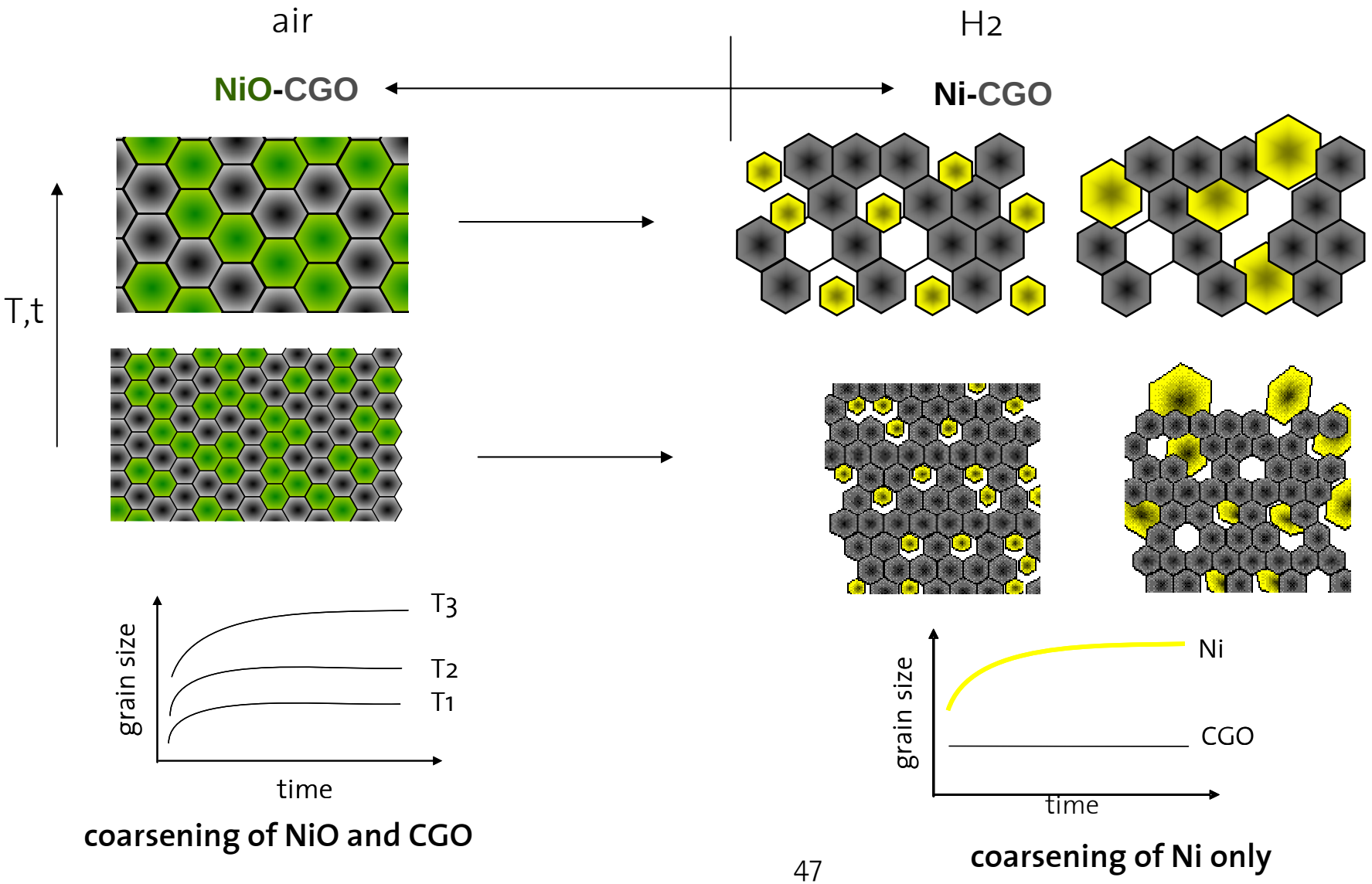
reduced @ 700°C for 1 hr





CGO and Ni growth model: Two step annealing

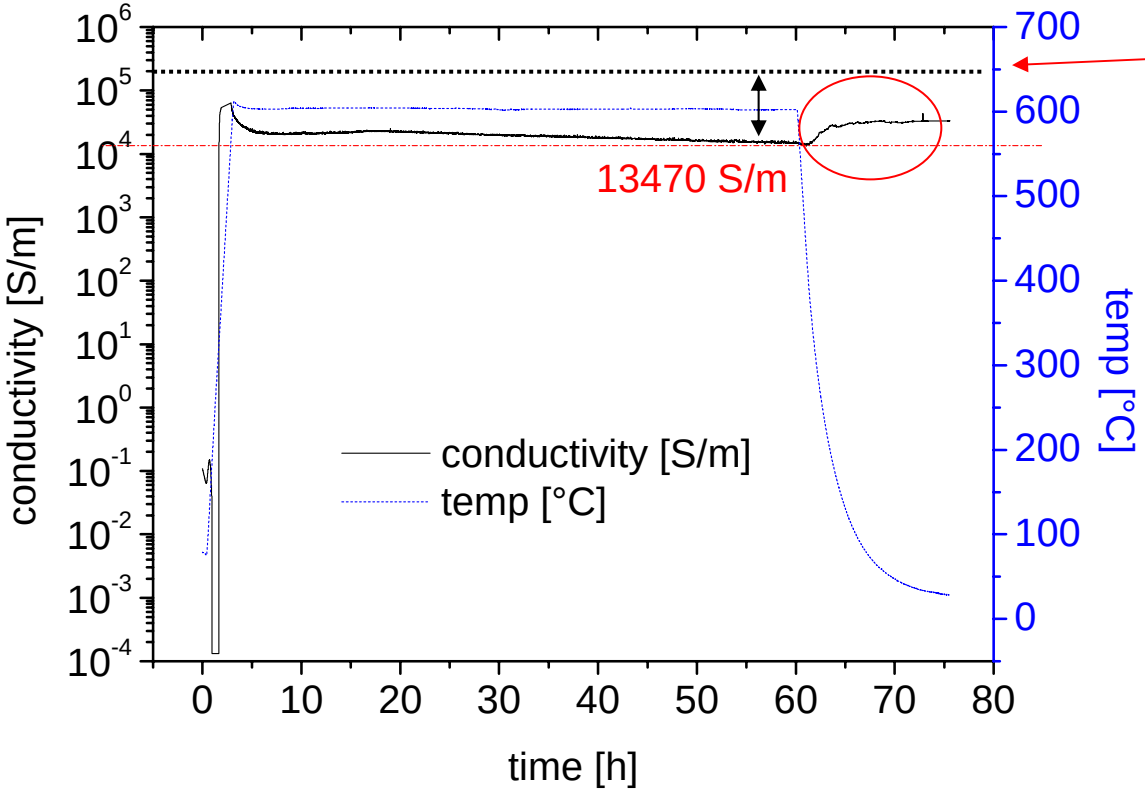
U. Mücke; Nonmetallic Materials ETH Zurich



Conductivity Data of 60/40 Ni/CGO Anode Layers

U. Mücke; Nonmetallic Materials ETH Zurich

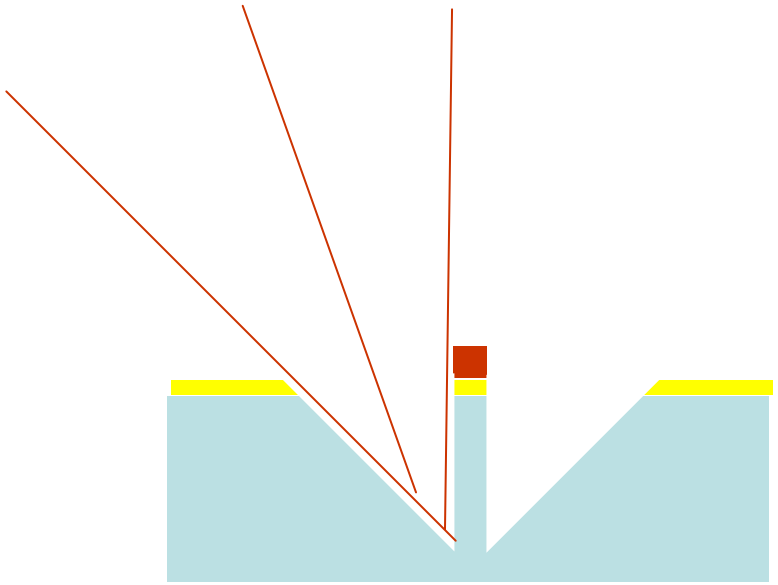
- σ=f(t) in a mixture of dry H_2:N_2
- sample remains conductive after 1 cycle => metallic conductivity



Pratihar et al. 1993
for μ-sized Ni

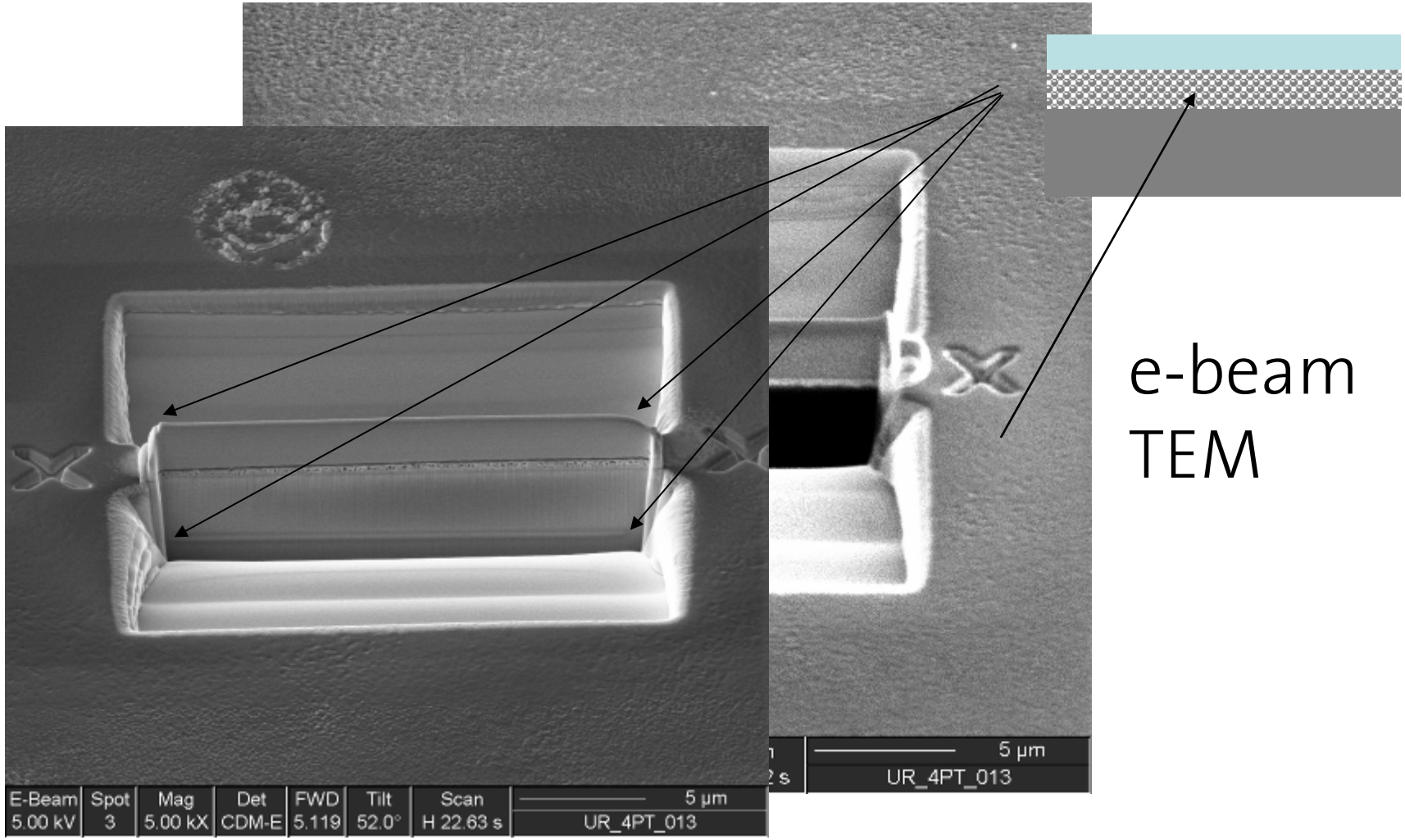
FIB preparation thin films for TEM

M. Holzer, EMPA and U. Mücke; Nonmetallic Materials ETH Zurich



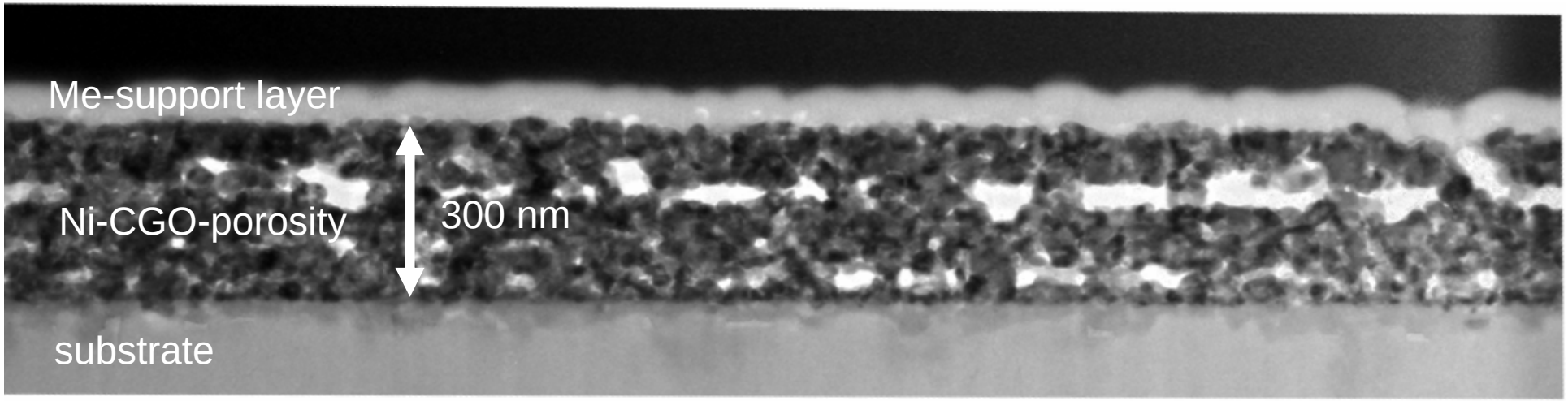
FIB preparation of TEM specimens

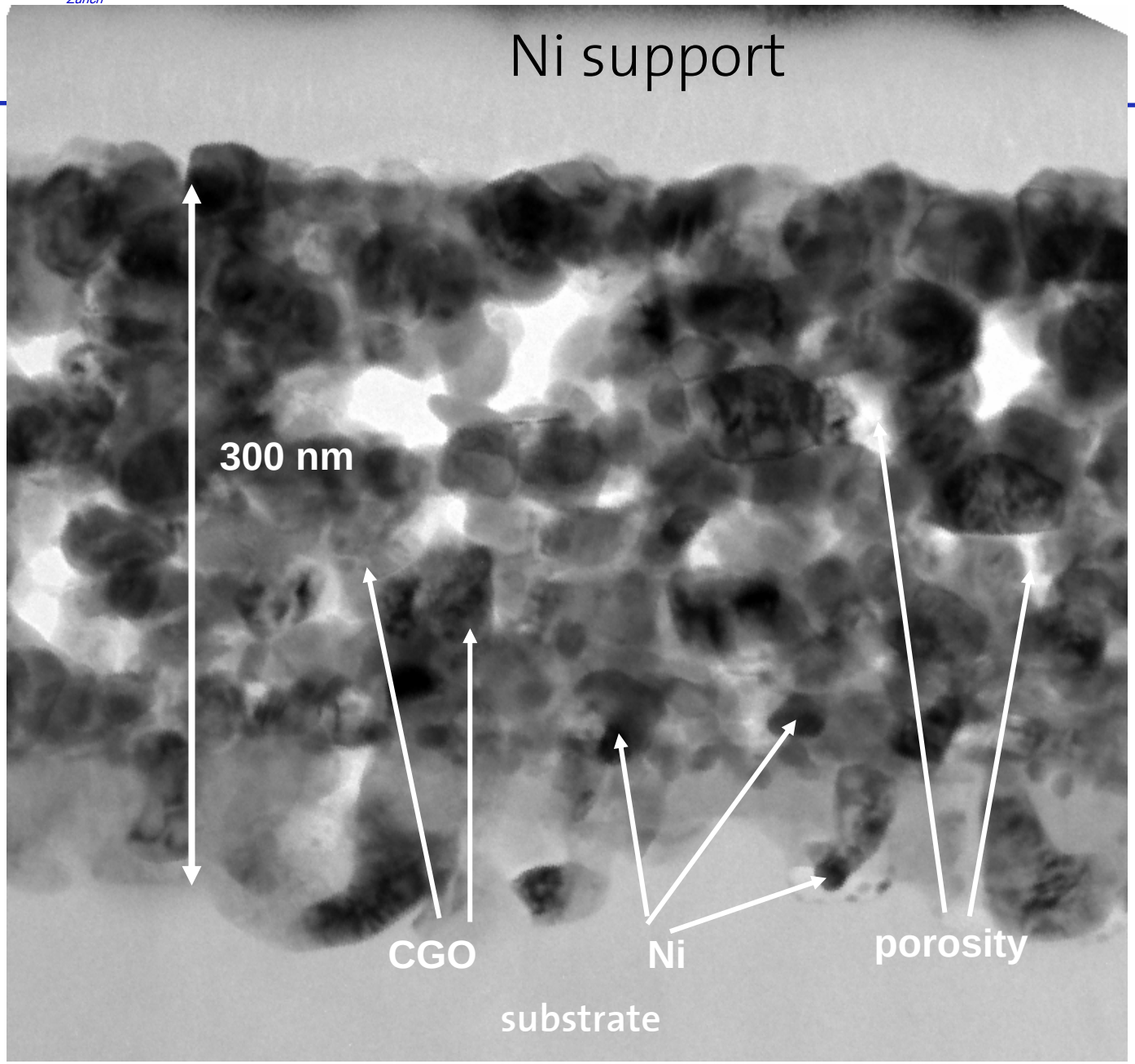
M. Holzer, EMPA and U. Mücke; Nonmetallic Materials ETH Zurich



e-beam
TEM

Ni-CGO anode




Ni support
300 nm
CGO
Ni
porosity
substrate

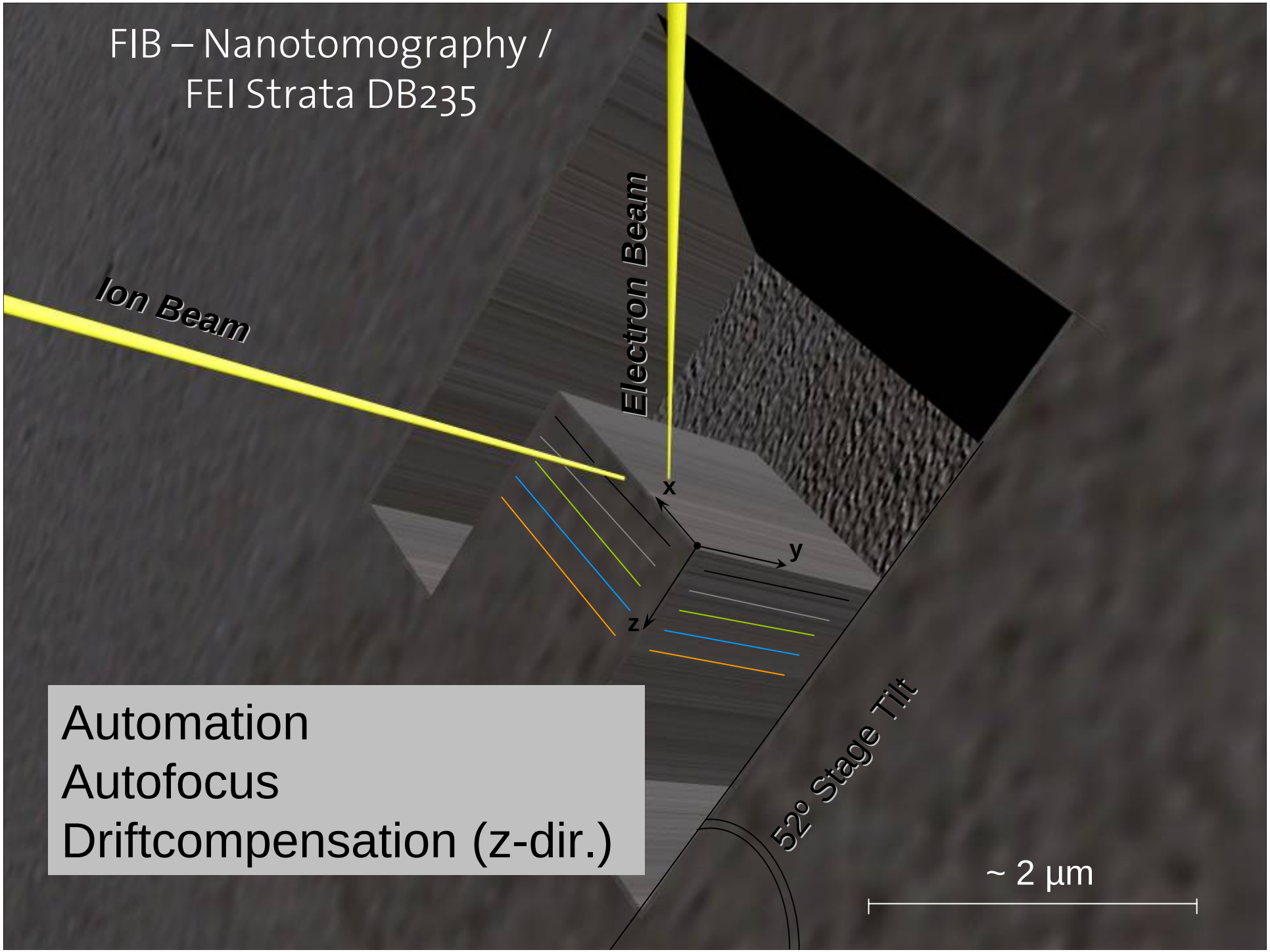
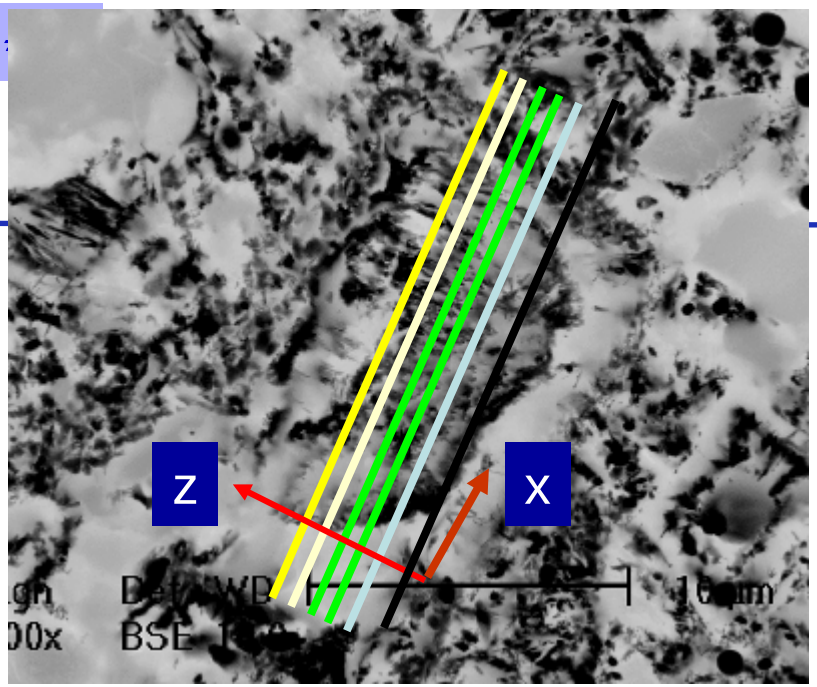
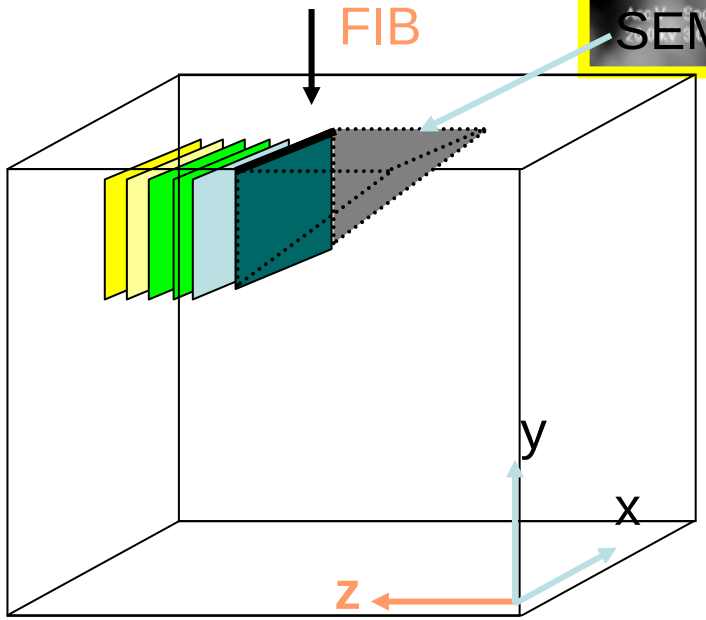
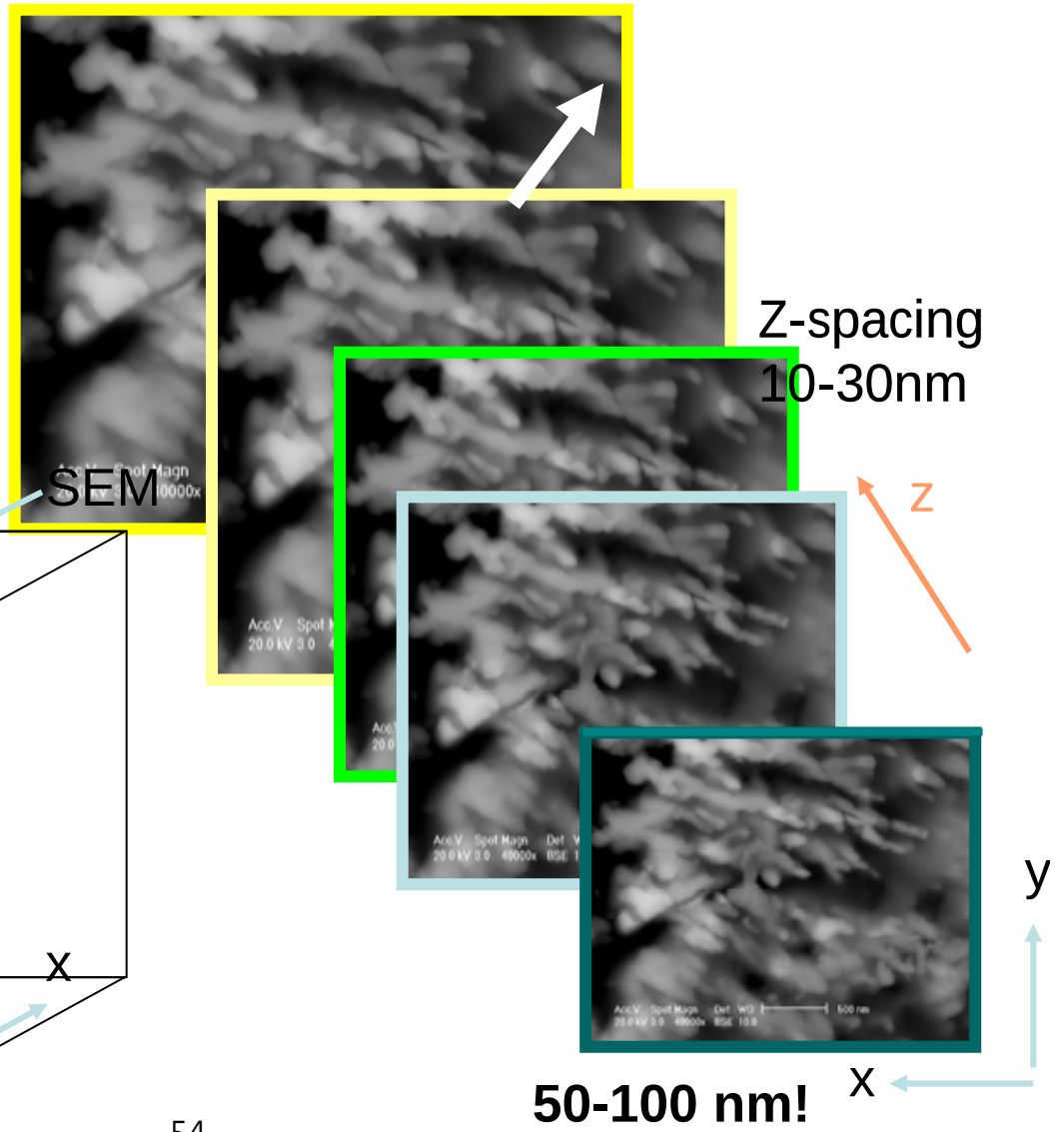

FIB – Nanotomography /
FEI Strata DB235
Ion Beam
Electron Beam
x
y
z
52° Stage Tilt
~ 2 µm
Automation
Autofocus
Driftcompensation (z-dir.)

Image processing for 3D reconstruction

M. Holzer, EMPA Zurich





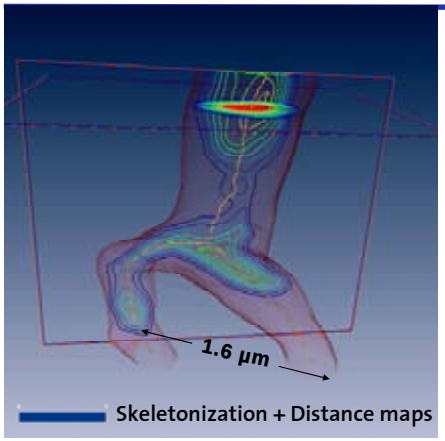


Quantitative Analysis: Skeletonization → Network analysis → Topology

M. Holzer, EMPA Zurich

Ni grain size distribution from 3D-data

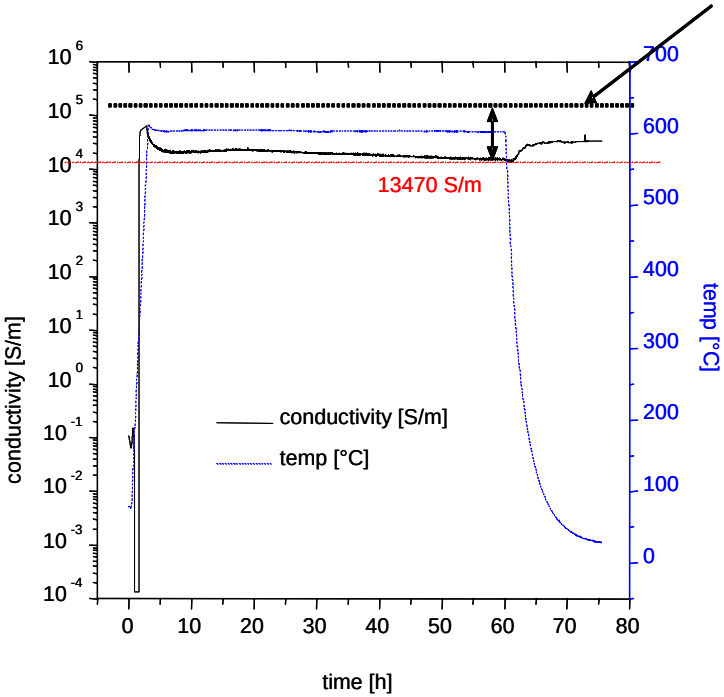
M. Holzer, EMPA Zurich



Conductivity Data of 60/40 Ni/CGO Anode Layers

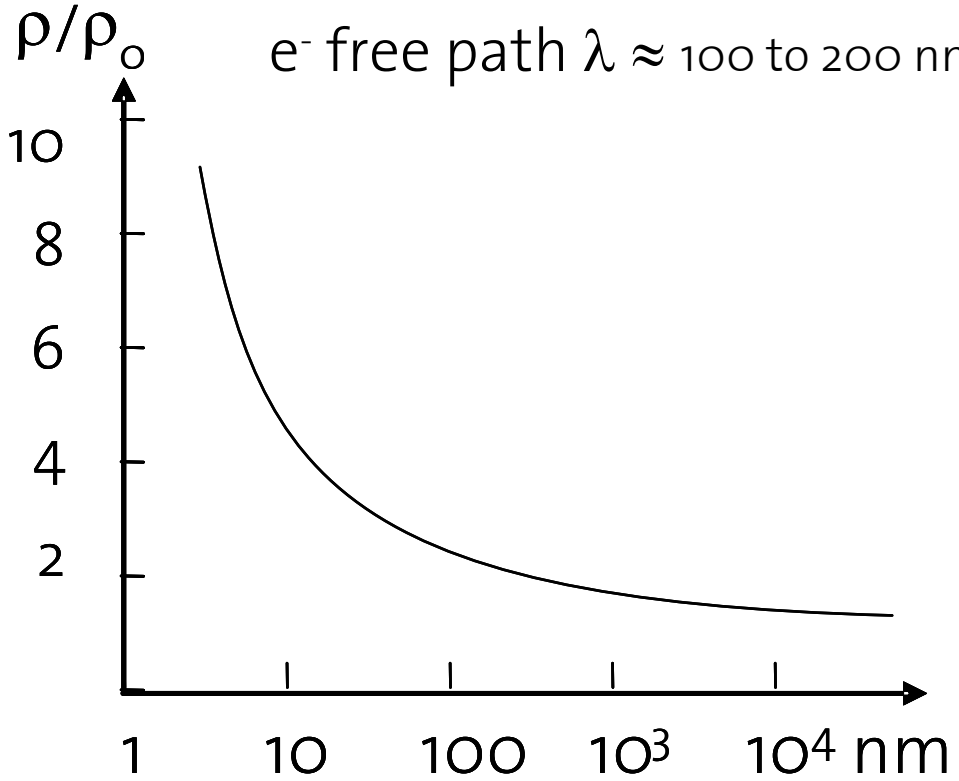
U. Mücke; Nonmetallic Materials ETH Zurich

$\sigma = f(T, X_i)$ and $\sigma = f$ (Ni-grain size& distribution)



Pratihar et al. 1993

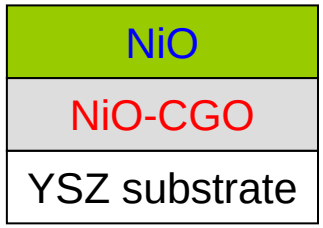
for μ-sized Ni



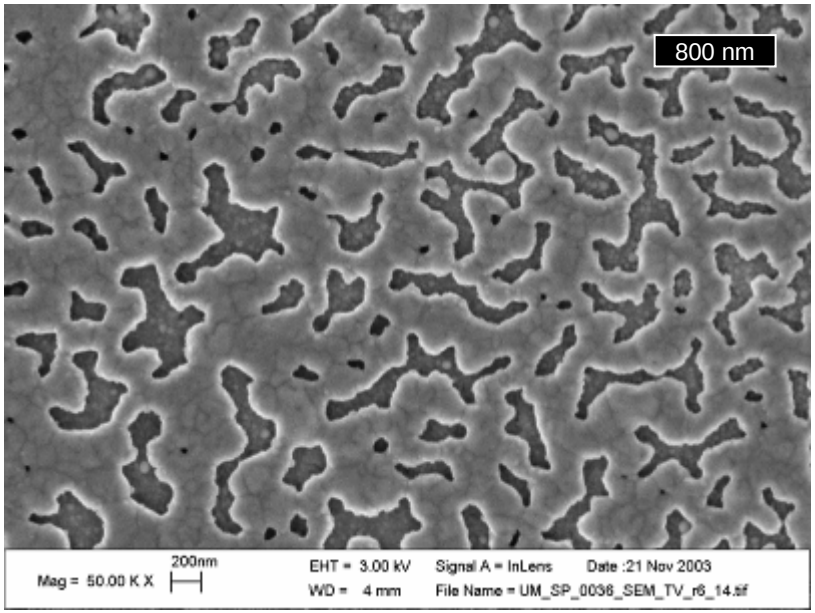
Ni + Ni/CGO Composite Layer

U. Mücke; Nonmetallic Materials ETH Zurich

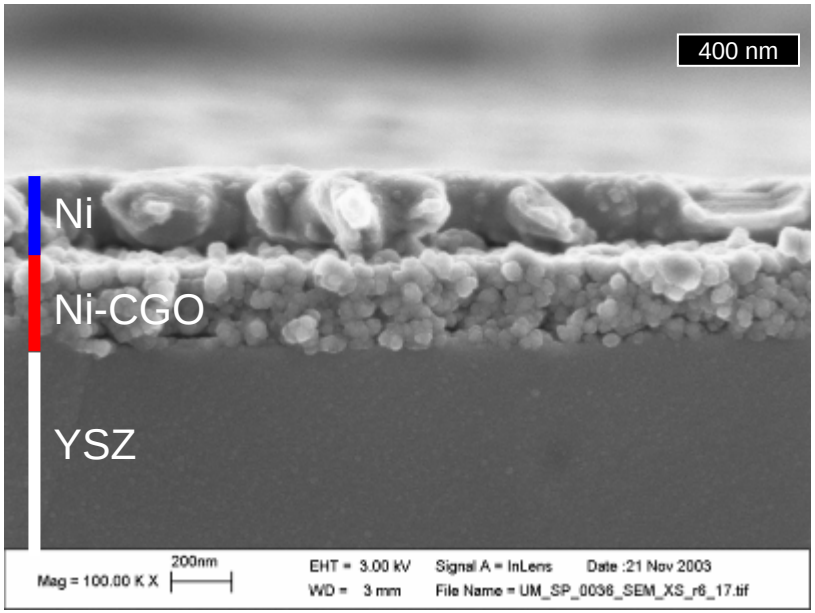
- porous Ni as current collector on Ni-CGO layer?
- annealed @ 600 °C for 1 hrs with 1°C up, then 1 hr in 5% H_2 in N_2 and 2°C down



top view



cross section



Electrochemical Characterization: Cathode/Electrolyte/Anode Tri-Layer

Measurement Techniques:

- 4 point conductivity
- impedance spectroscopy

@ T = 550°C:

$R_{p\ electrolyte}$ = 0.05 Ωcm²

$R_{p\ anode}$ = 1.5 Ωcm²

$R_{p\ cathode}$ = 8.4 Ωcm²

predicted performance

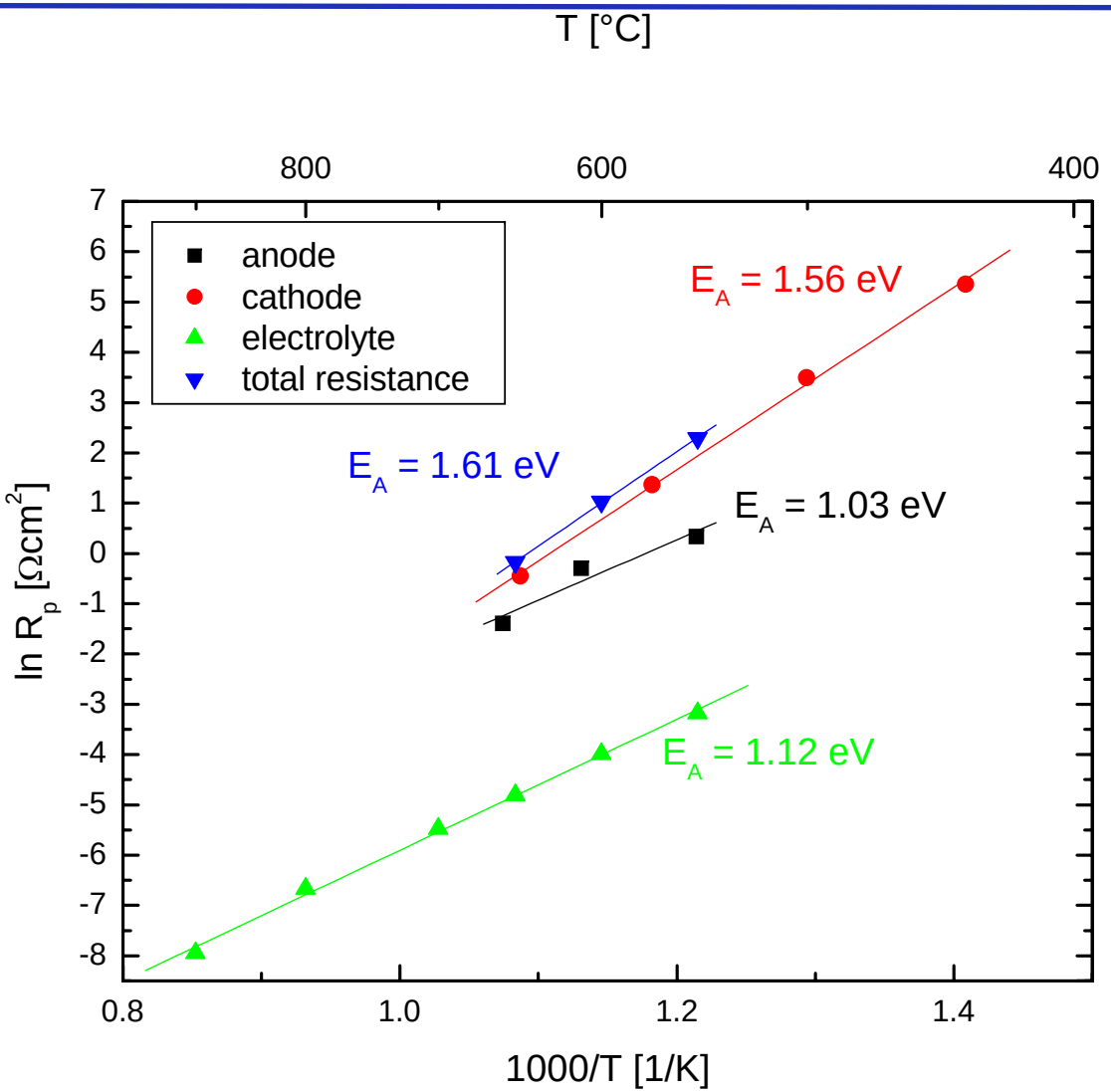
(E_{cell} = 0.7 V, H_2/air):

P = 50 mW/cm² @ T = 550°C

P = 600 mW/cm² @ T = 650°C

measured performance

P = 600 mW/cm² @ T = 720°C



Bieberle, Rupp, Beckel, Mücke, Gauckler, mstnews, in print, June, 2005

Outline

Motivation: μ - Solid Oxide Fuel Cell & One-Bat Project

μ - Solid Oxide Fuel Cell System

μ - Solid Oxide Fuel Cell Hot Plate

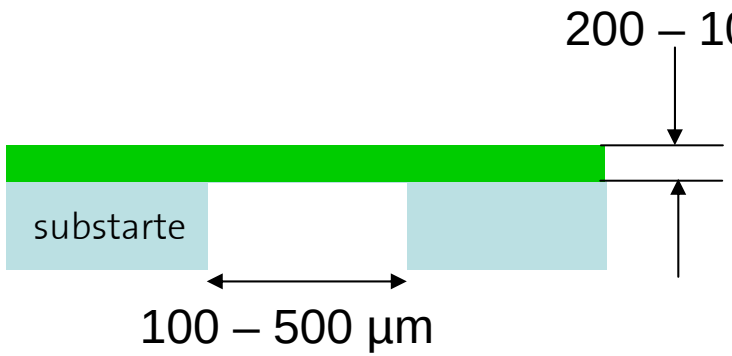
Pulsed Laser Deposition
Spray Pyrolysis

Electrolyte
Cathode
Anode & current collector

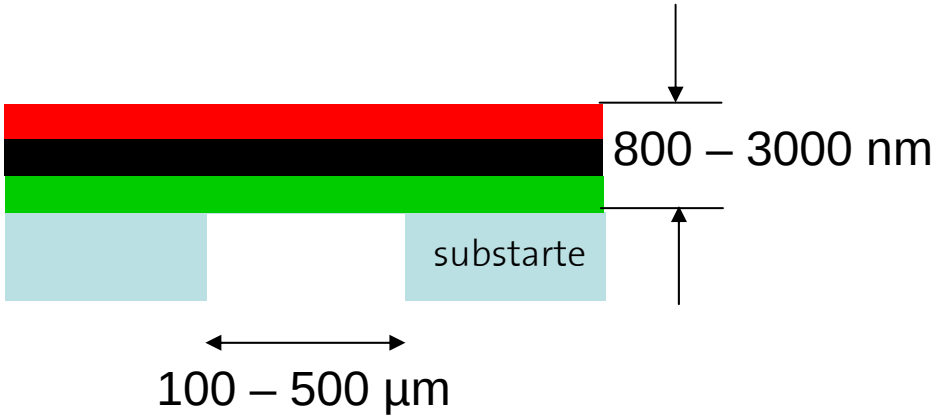
Outlook

Acknowledgement

Substrates for free standing ceramic membranes



First tests with NiO-CGO anodes



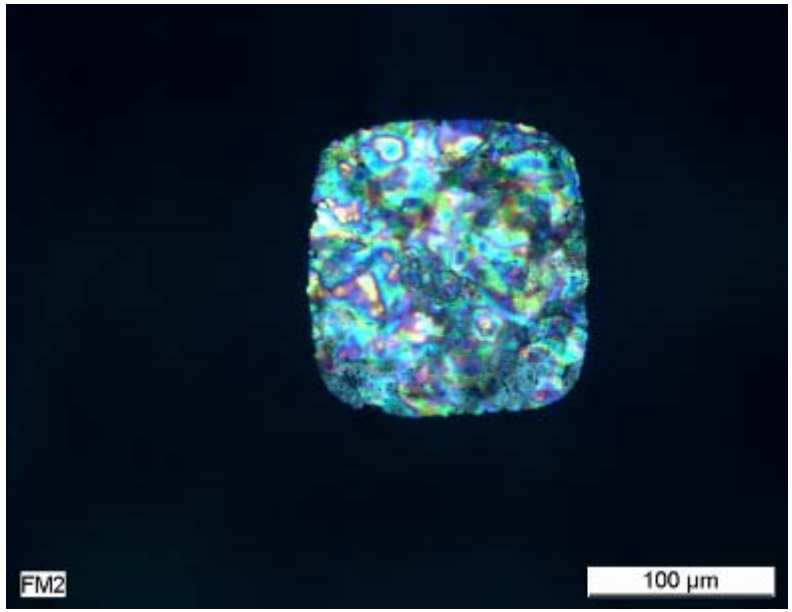
Free standing triple layer
(anode, electrolyte, cathode)

Free Standing 60/40 NiO/CGO Membranes

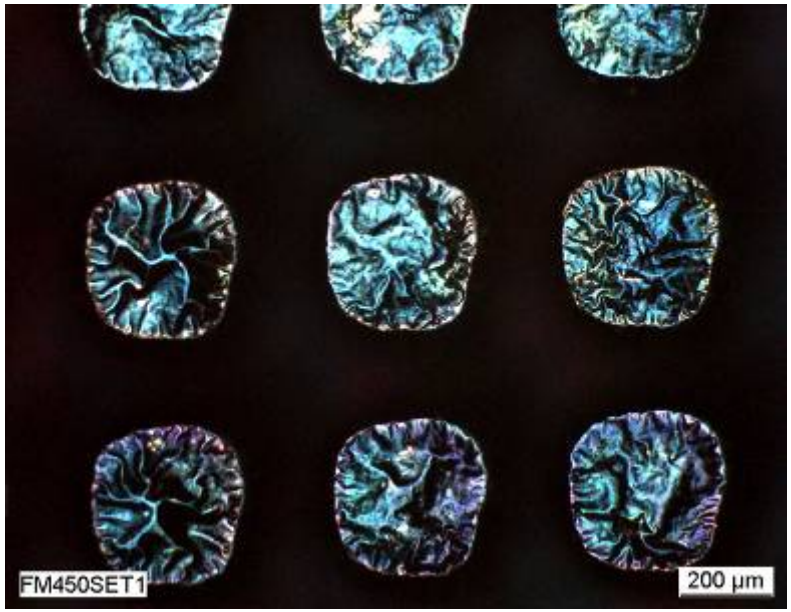
U. Mücke; Nonmetallic Materials ETH Zurich

- membranes can up to now withstand
  - sudden heating up to 600 °C without rupturing
  - normal handling in lab
- size can be up to 0.5 x 0.5 mm²

membrane after spraying and etching

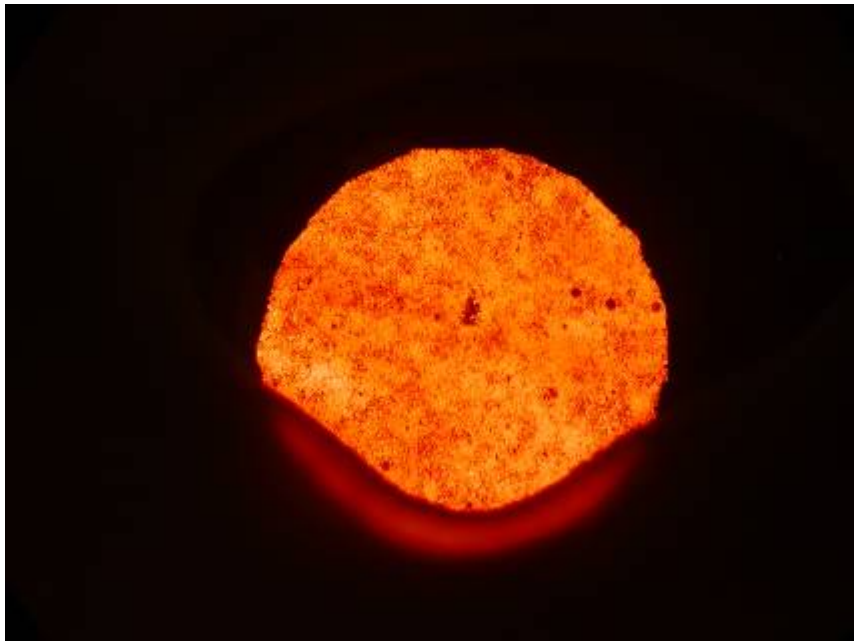


membrane after heat treatment at 450 °C

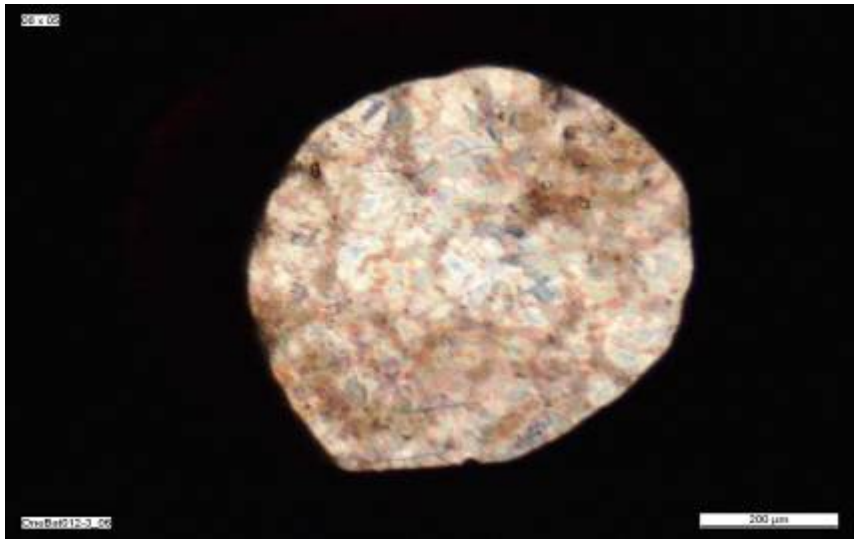


Free standing triple layer

After etching

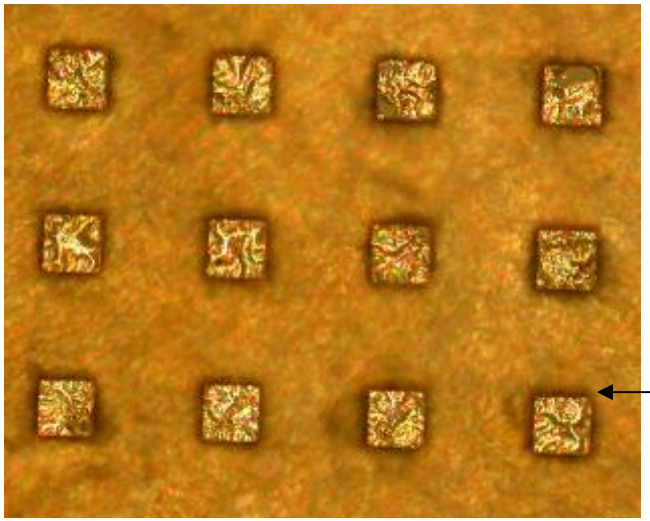


After annealing at 600 °C

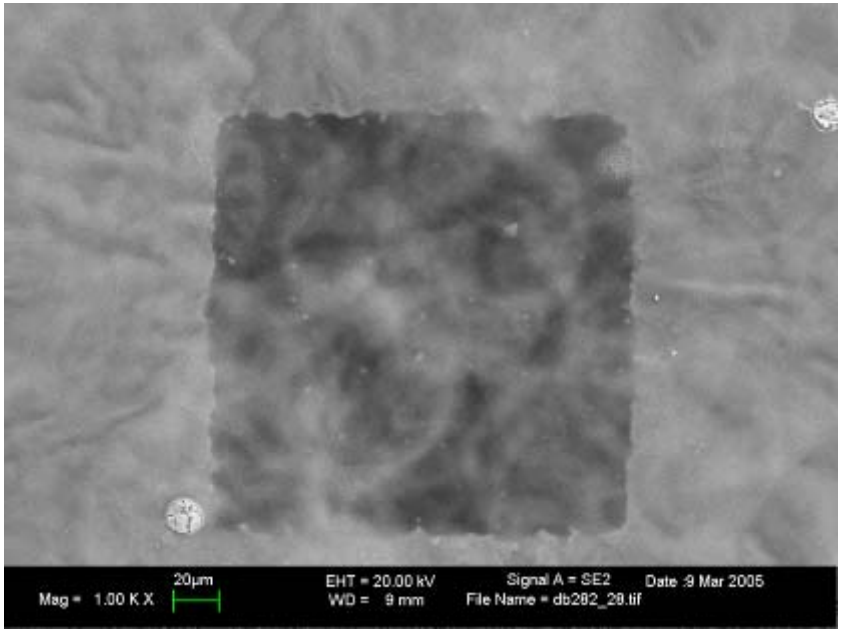


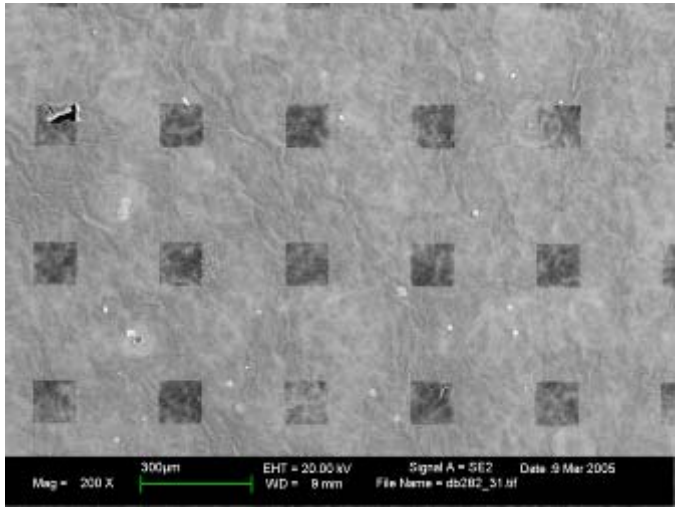
Light microscope view from backside on anode (light shining through)

Free standing NiO/CGO anodes



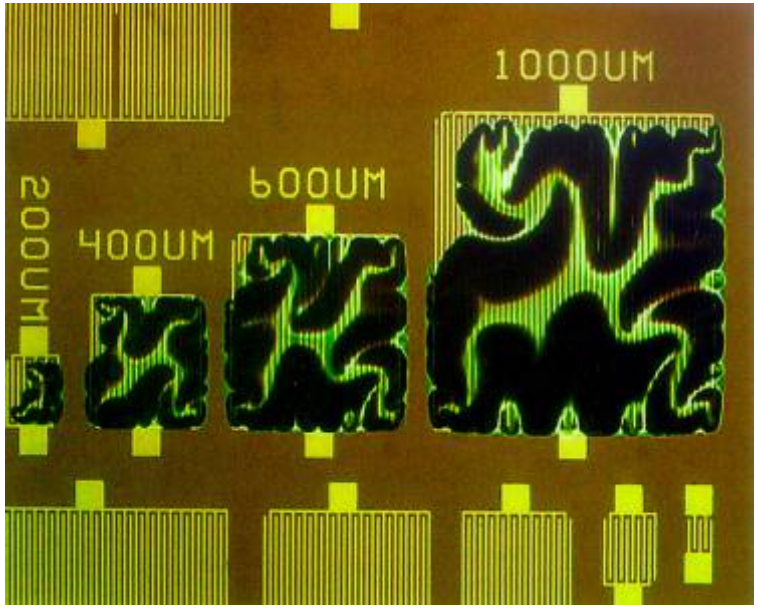
After etching
(light microscope,
view from backside)





After annealing at 600 °C
(SEM top view, high voltage to make membranes transparent)

CGO Membranes with electrodes on Si



$Ce_{0.8}Gd_{0.2}O_2$ electrolyte
Membrane thickness: 400 nm
Largest Membrane: 1 mm
Stable up to 350 °C.

600 µm version

Paul Muralt,
N. Setter; EPFL

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Anode & current collector

Acknowledgement


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Anja Bieberle


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Ulrich Mücke

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- KTI (CH)


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