Model-based Control of MOCVD Rate, Uniformity and Stoichiometry

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Process Control, Diagnostics, and Modeling in Semiconductor Manufacturing III
195th Meeting of the Electrochemical Society,
Seattle, Washington, May 3, 1999

Acknowledgements

- Work funded by DARPA, administered by ONR
- Project partners: MIT, Princeton
- Several very useful discussions with Dr. L. Raja of Colorado School of Mines

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- Reactor Model
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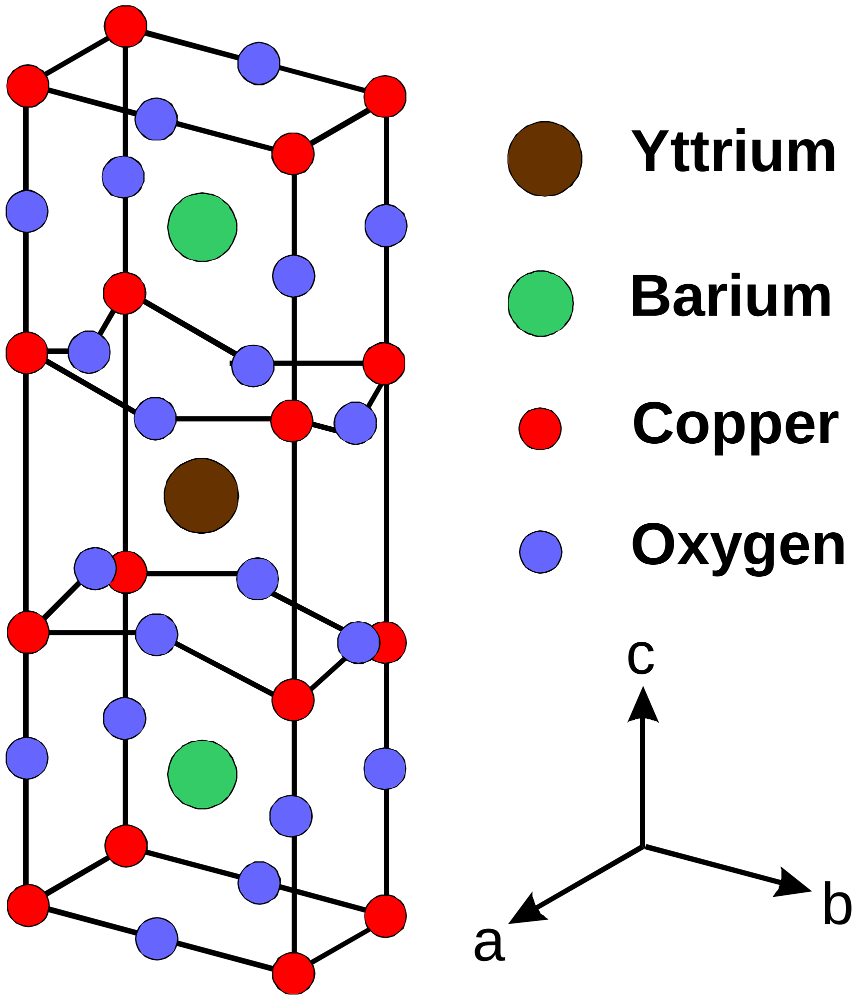
YBCO Thin Films

Fully oxygenated orthorhombic unit cell of $YBa_2Cu_3O_7$. At room temperature the lattice parameters are: a=3.819Å, b=3.883Å, c=11.687Å

Superconducting at temperatures as high as 93K

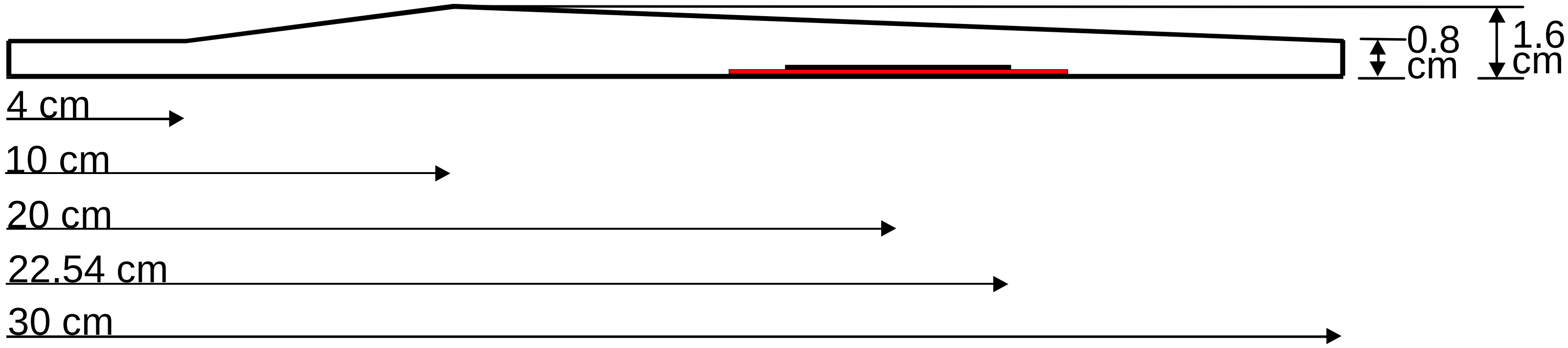


MOCVD of YBCO Thin Films

- CVD is preferable to PVD larger surfaces areas. For microwave applications (resonators, filters, antenna), a few hundred nm thick films need to be deposited on insulating substrates of diameter of at least 10 cm.

- An early challenge was finding suitable precursors for the metals. Currently, ß-diketones (general formula: $RCOCH_2COR'$) is almost exclusively used. The ones considered here is denoted by thd (or dpm) with $R=R'=C(CH_3)_3$. They are vaporized at temperatures between 100-250ºC, with vapor pressure between 0.01-1 Torr.

MOCVD of YBCO Thin Films



Schematic of a Thomas Swan MOCVD reactor

Steady-state operating conditions:

Gas mixture enters reactor at 10 Torr with mean velocity of 2 m/s and temperature of 240ºC. The inlet mole fractions are: $O_2 = 0.44$, $N_2 = 0.47$, Ar = 0.088, $Y(dpm)_3 = 2.72\times10^{-5}$, $Ba(dpm)_2 = 4.41\times10^{-5}$, $Cu(dpm)_2 = 2.35\times10^{-5}$. Walls at 800ºC.

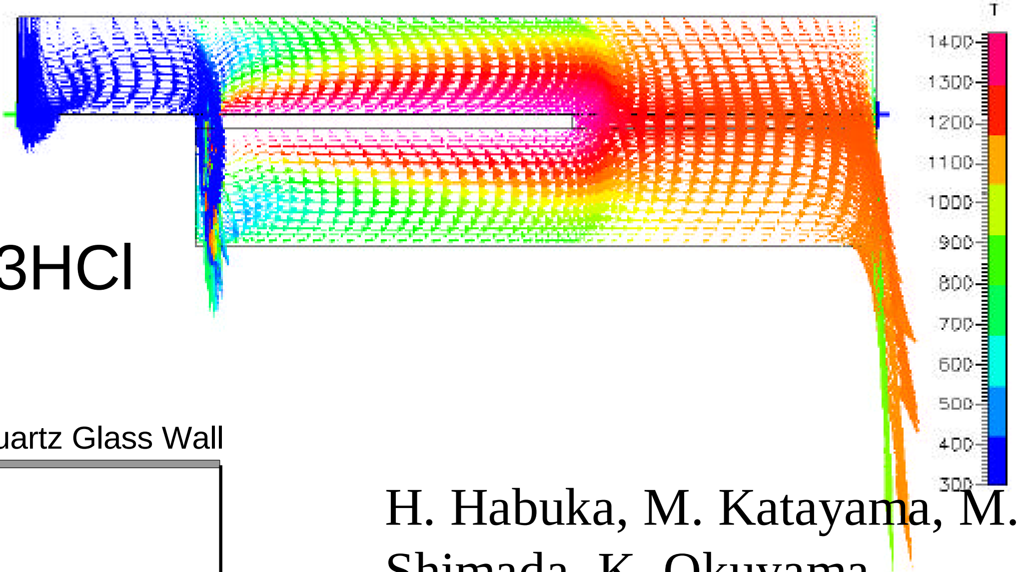
MOCVD of YBCO Thin Films

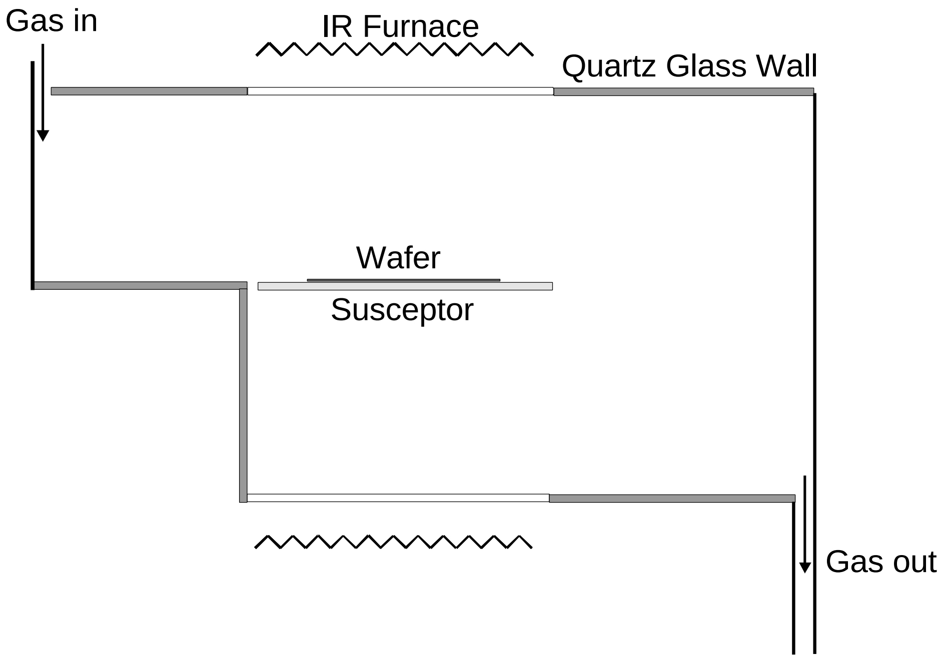
- Detailed process chemistry (gas phase and surface kinetics) is not known very well.
- Precursor decomposition, and oxide formation is currently being studied using quantum-mechanical (DFT) calculations as part of this project.
- Meantime, we are using a simplified kinetic mechanism consisting of mostly first-order finite-rate reactions for precursor decomposition, followed by very fast oxide formation. The oxides then diffuse the substrate, with surface kinetics modeled using sticking coefficients.
- CVD model developed using CFDRC's CFD-ACE® software

CVD Validation Study

Silicon Epitaxy:

$SiHCl_3 + H_2 \rightarrow Si + 3HCl$

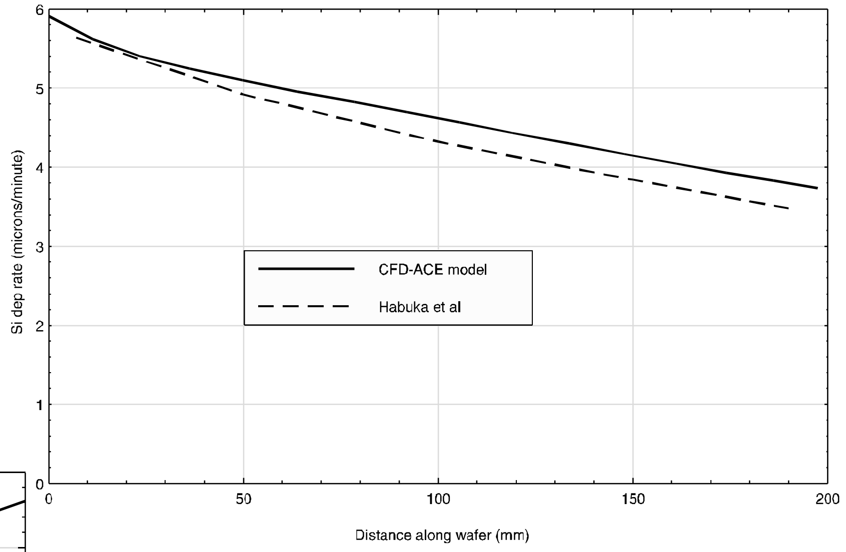


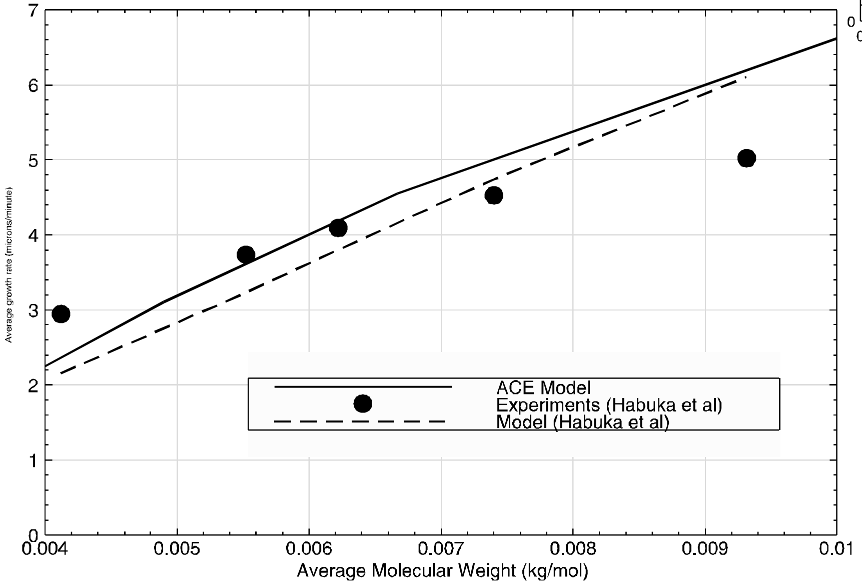


H. Habuka, M. Katayama, M. Shimada, K. Okuyama, "Numerical Evaluation of Silicon Thin Growth from $SiHCl_3$-H_2 Gas Mixture in a Horizontal Chemical Vapor Deposition Reactor," *Jpn. J. Appl. Phys*., 1994. **33**: p.1977-1985

CVD Validation Study: Si Epitaxy

Model used temperature-dependent properties, Soret diffusion, multi-component Stefan-Maxwell diffusion

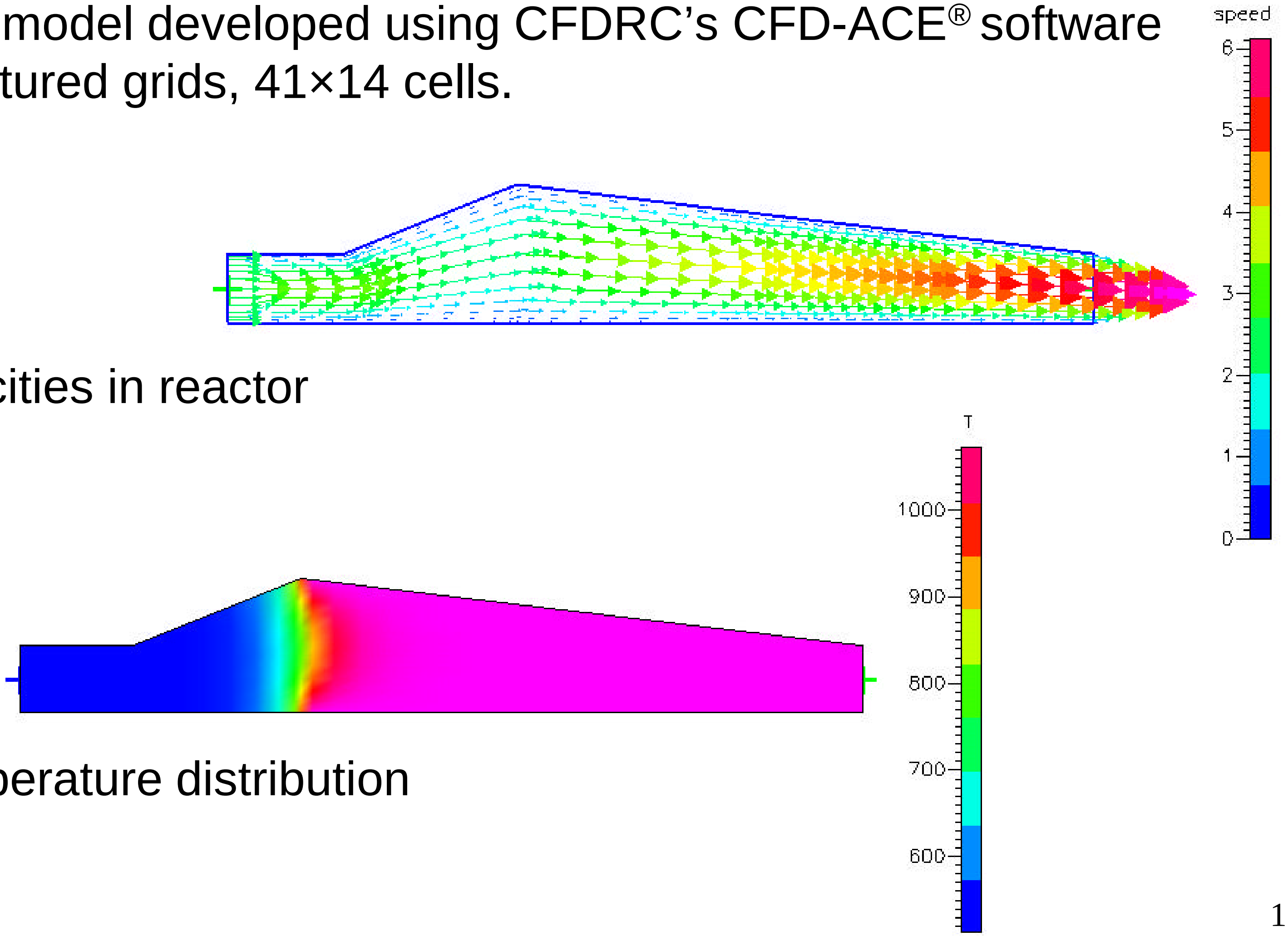




Comparison with Habuka’s results for epitaxial CVD deposition show dep rates within 6% of Habuka *et al*

MOCVD of YBCO Thin Films

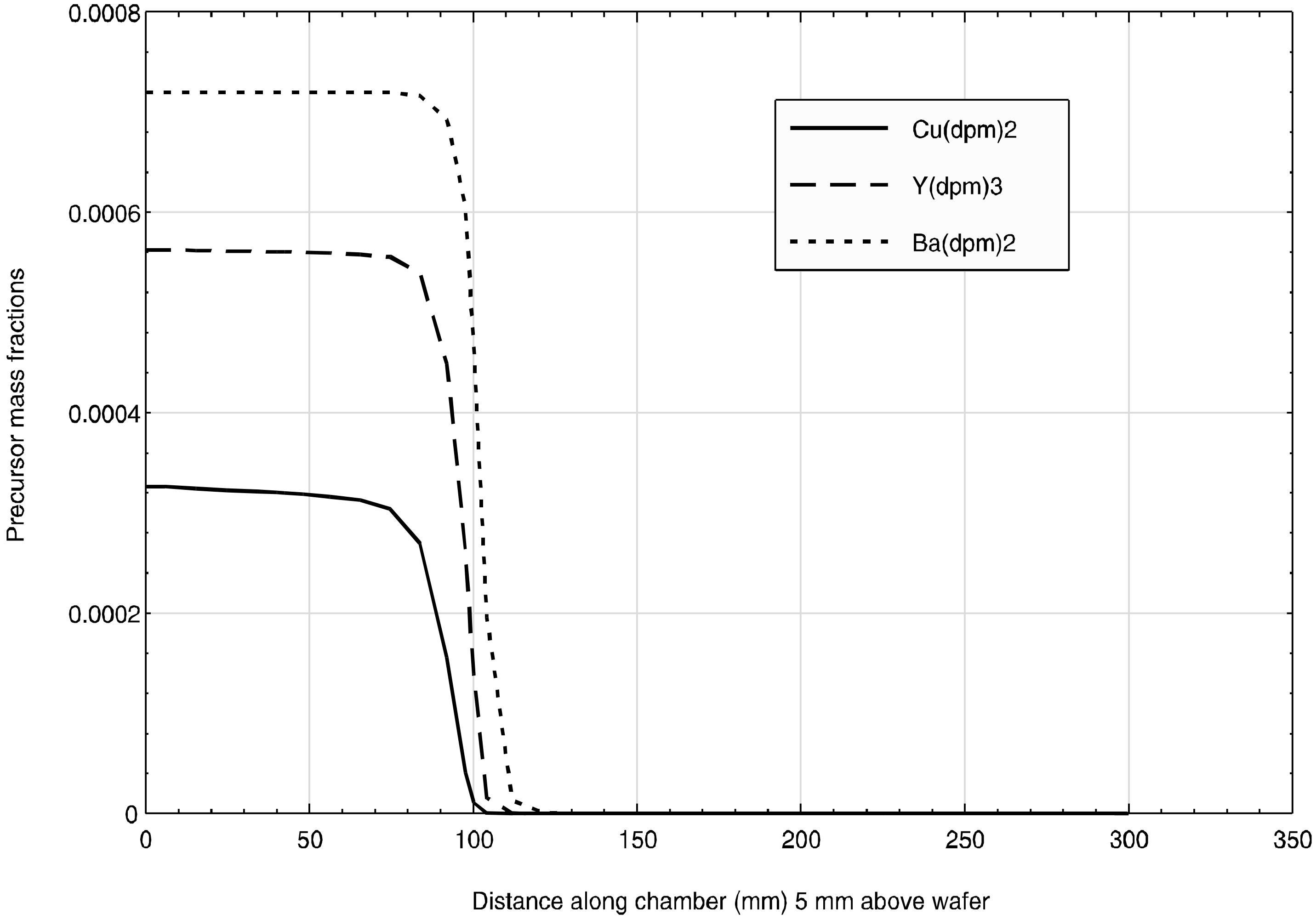
CVD model developed using CFDRC’s CFD-ACE® software
Structured grids, 41×14 cells.



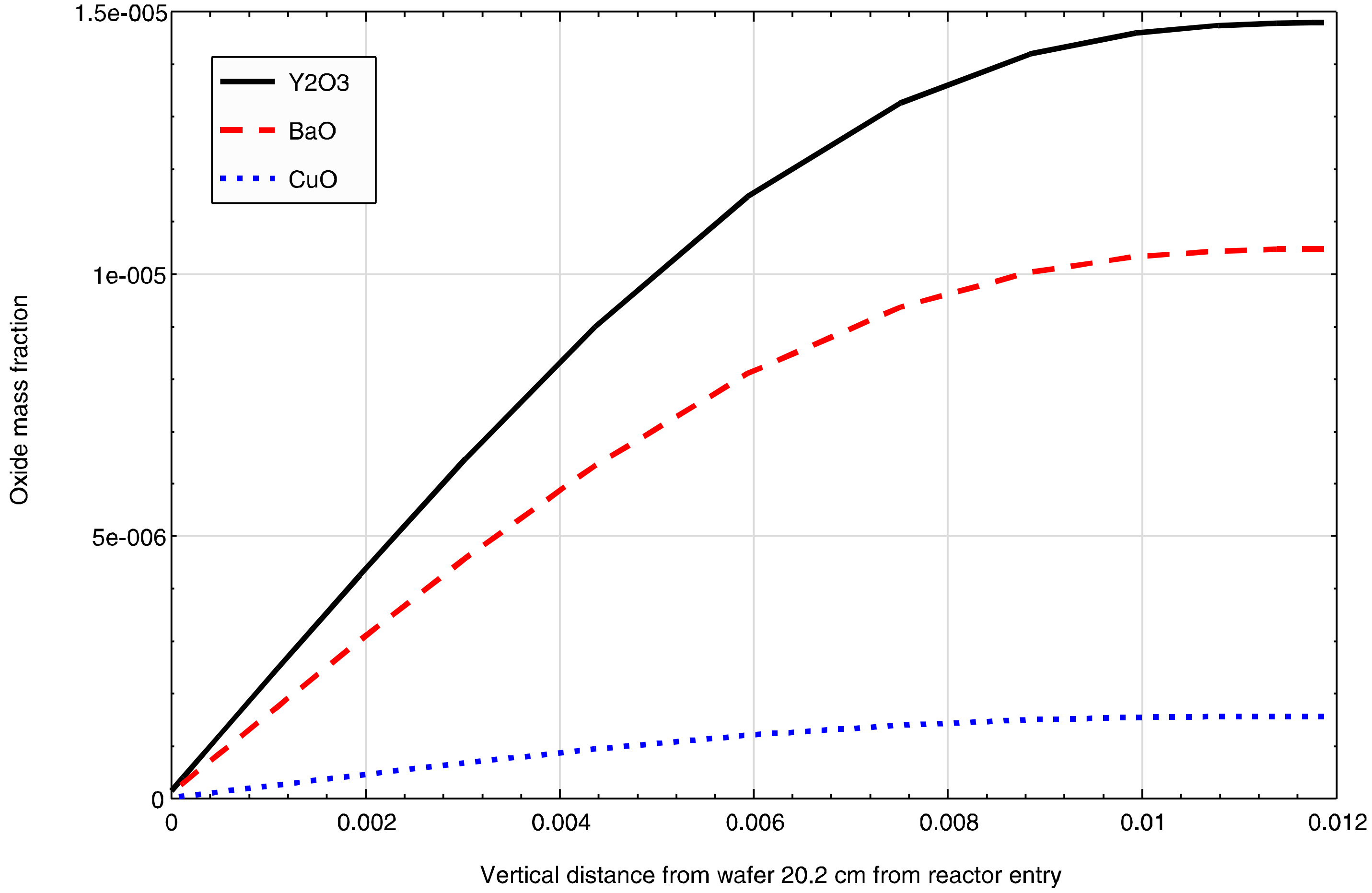
Velocities in reactor

Temperature distribution

MOCVD of YBCO Thin Films

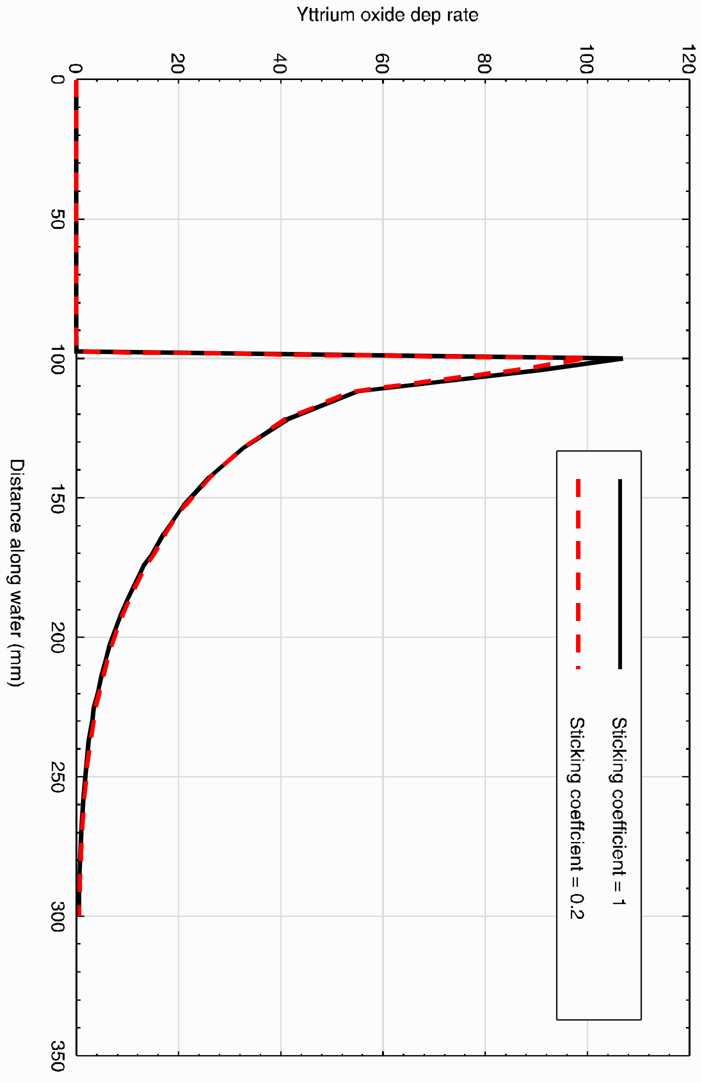


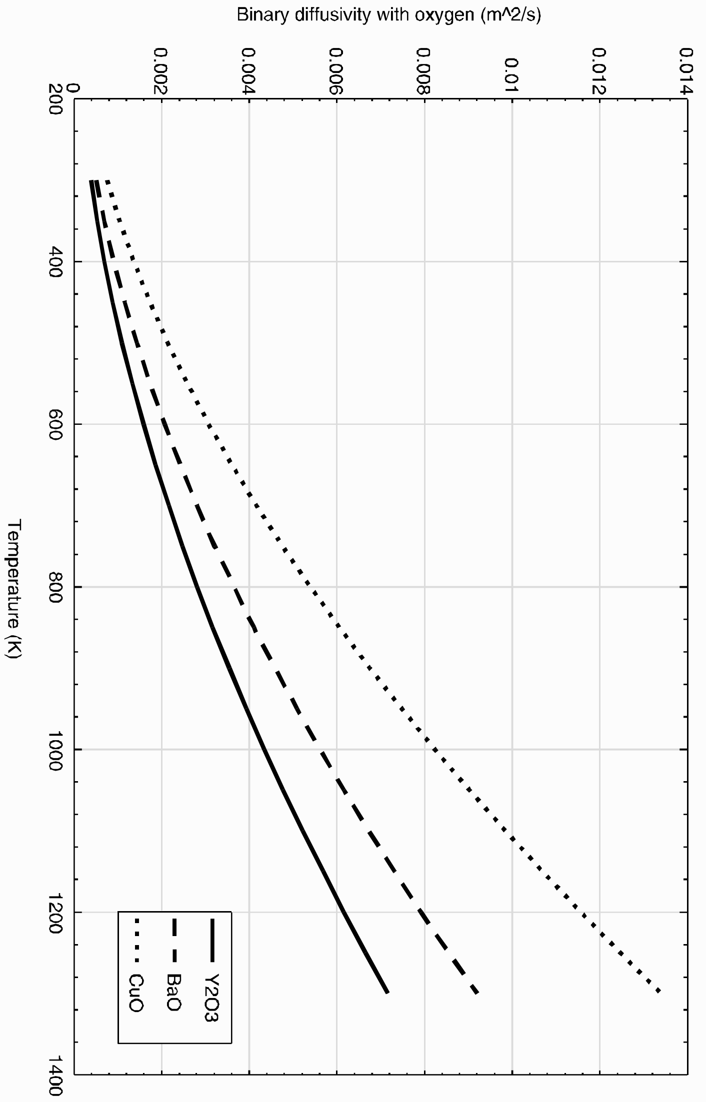
MOCVD of YBCO Thin Films



Growth occurs in a mass transport-limited regime

MOCVD of YBCO Thin Films





MOCVD of YBCO Thin Films

Chemistry:

$$Y(dpm)_3 + O_2 \rightarrow Y + 3\,(dpm) + O_2$$

$$Y(dpm)_3 \rightarrow Y + 3\,(dpm)$$

$$Cu(dpm)_2 \rightarrow Cu + 2\,(dpm)$$

$$Ba(dpm)_2 \rightarrow Ba + 2\,(dpm)$$

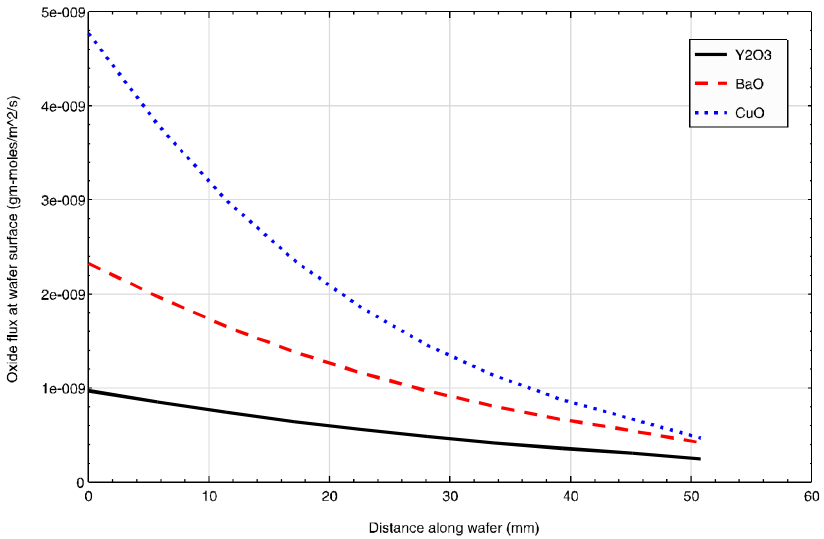
$$4\,Y + 3\,O_2 \rightarrow 2Y_2O_3$$

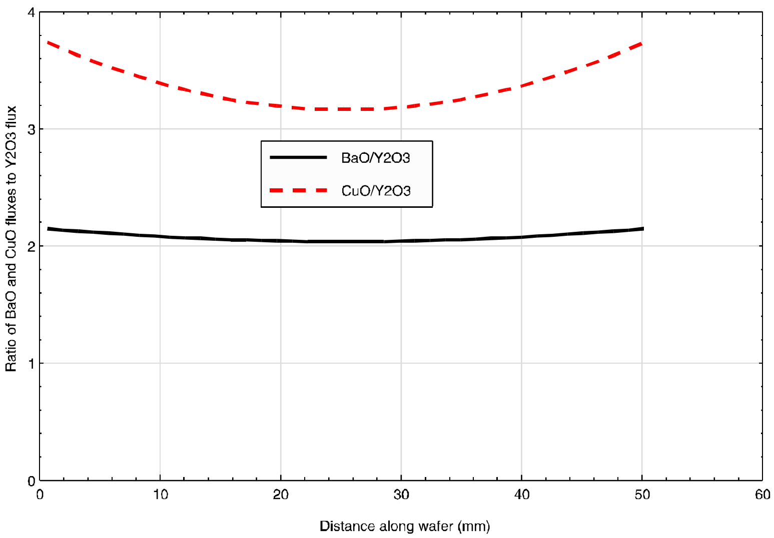
$$2\,Ba + O_2 \rightarrow 2BaO$$

$$2\,Cu + O_2 \rightarrow 2CuO$$

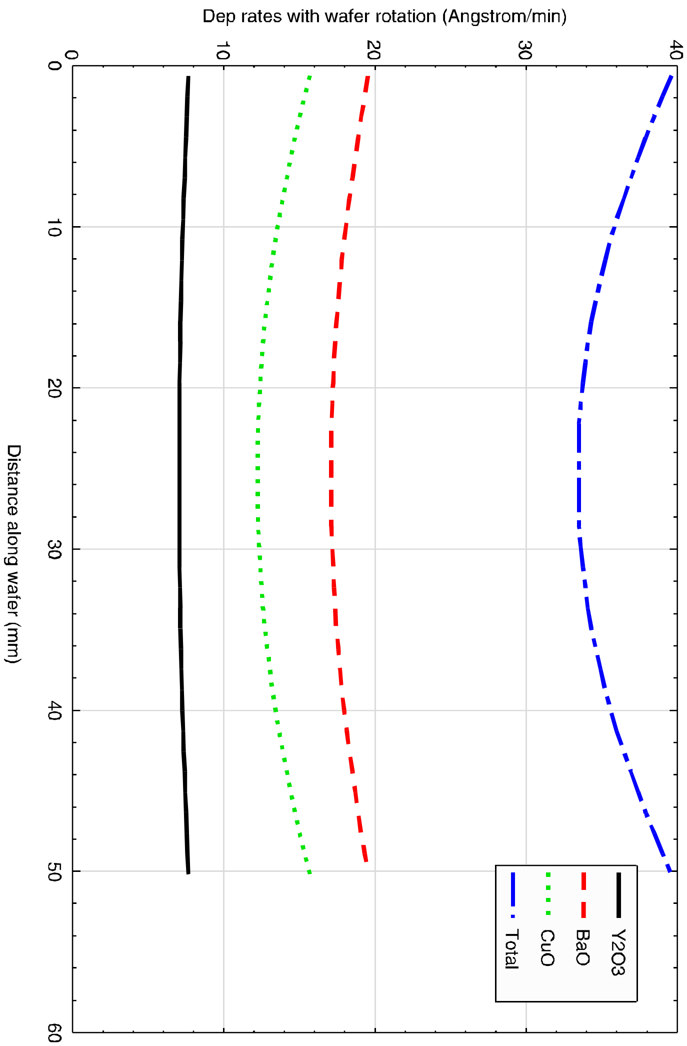
MOCVD of YBCO Thin Films

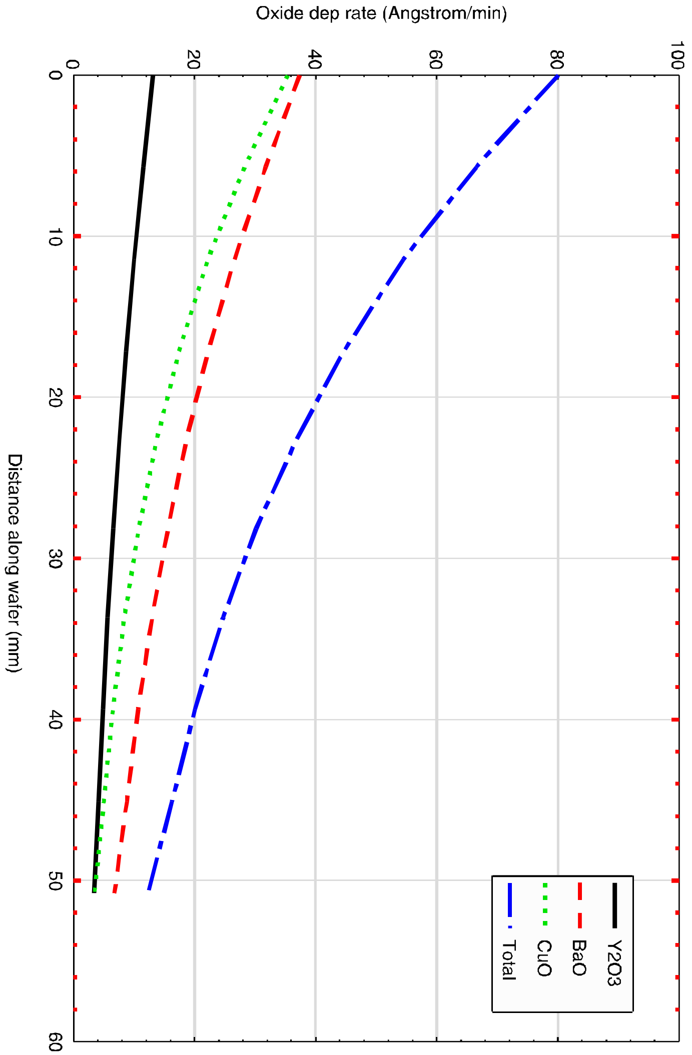
YBCO stoichiometry varies along wafer surface. Precursor concentration control can be used to restrict atom ratio within specified bounds (e.g, avoiding BaO-rich deposits)



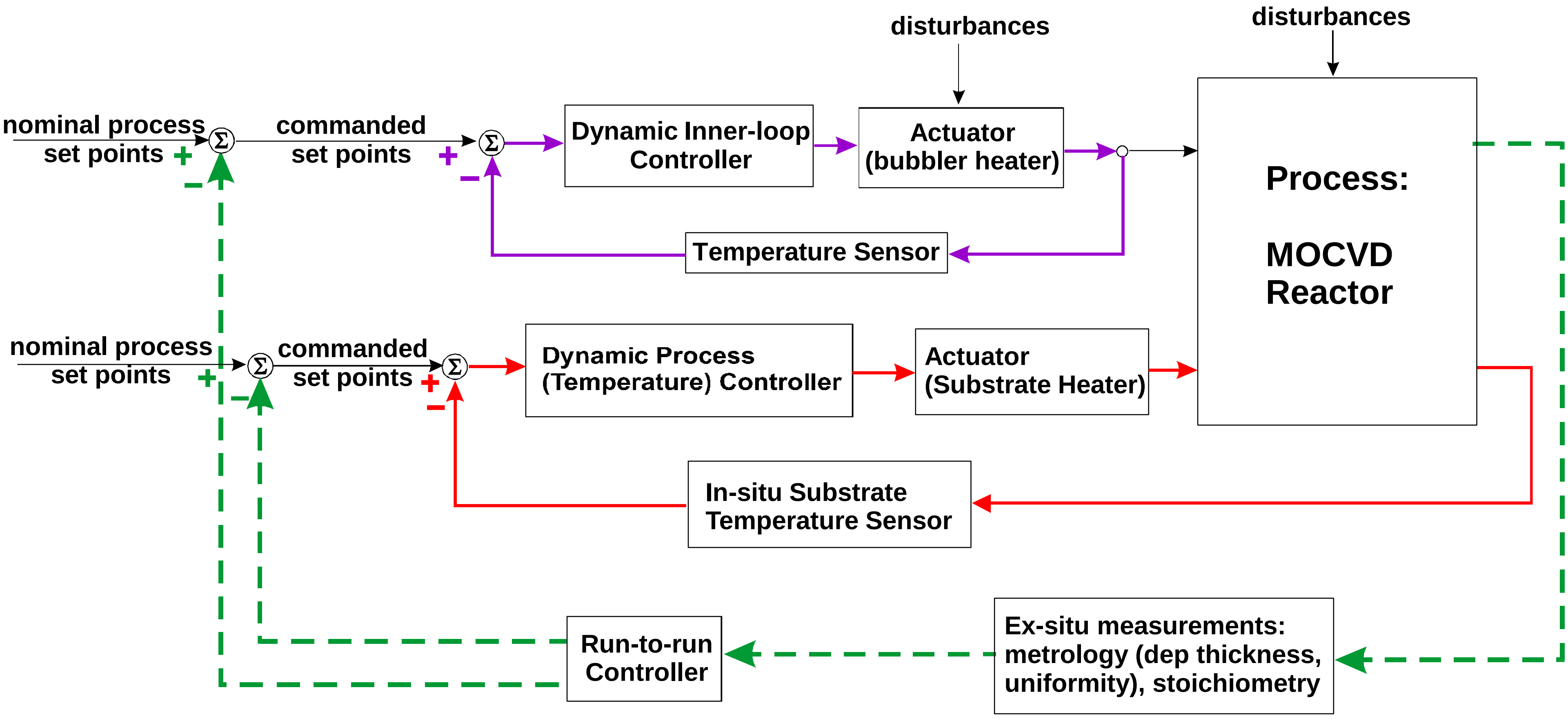


MOCVD of YBCO Thin Films





MOCVD Control Strategy



Introduction to Run-to-Run Control

- Manufacturing: multiple copies of same product
- Product quality determined *after* manufacturing (run)
- Product quality is influenced by *recipe variables*
- Recipe variables are *pre-set* and *fixed* during the run
- Run-to-Run control problem:

Adjust recipe for next run based on results of previous runs such that product quality improves

Proportional Error Control

- Let $t = 1,2,\ldots$ denote run number, r_t the vector of recipe variables during run t, y_t the vector of product quality attributes at end of run t, and e_t the normalized product quality error with *i-th element:*

$$e_t(i) = \frac{y_t(i) - y_{des}(i)}{y_{tol}(i)}, \qquad i = 1,\ldots,n$$

- Adjust recipe according to:

$$r_t = r_{nom} + u_t,$$

$$u_t = u_{t-1} - G e_{t-1}, \qquad u_0 = 0$$

Static Linear Error System

Introduction

- Assume actual process is a *static linear error system*:

$$e_t \;=\; w_t + G u_t, \qquad\qquad t = 0,1,2,\ldots$$

- w_t is vector of product quality errors due to nominal control:

$$w_t \;=\; e_t \,\big|_{r_t = r_{nom}}$$

- Error and control:

$$e_t \;=\; (I_n - GG)e_{t-1} + w_t - w_{t-1}, \qquad e_0 = w_0$$

$$u_t \;=\; (I_m - GG)u_{t-1} - Gw_{t-1}, \qquad u_0 = 0$$

Static Linear Error System (cont'd)

Introduction

- Note that error system is driven by *variation* in nominal error. Hence effect of biases can be eliminated, and slow drifts greatly reduced, e.g.:

  $$w_t = b + ct + v_t$$

  then

  $$w_t - w_{t-1} = c + v_t - v_{t-1}$$

  with *b* denoting bias, and *c* denoting drift rate, and v_t a zero-mean random variable.

- However, if v_t has variance σ^2, then $v_t - v_{t-1}$ has variance $2\sigma^2$!

Static Linear Error System

Inverse Control

- Suppose G is square (n = m) and invertible, then the stability analysis suggests the choice:

  with *m* a real scalar.
- The error and control equation are now given by:

$$e_t \;=\; (1 - \mu)e_{t-1} + w_t - w_{t-1}, \qquad e_0 = w_0$$

$$u_t \;=\; (1 - \mu)u_{t-1} - \mu G^{-1} w_{t-1}, \qquad u_0 = 0$$

- The system is stable for:

$$0 < \mu < 2.$$

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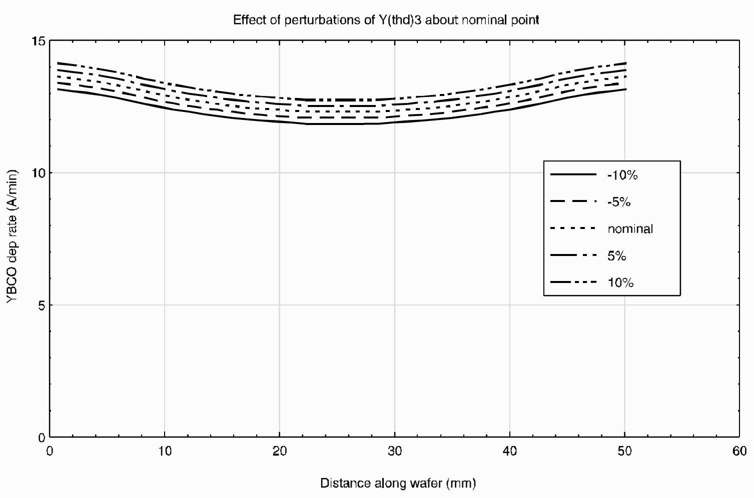
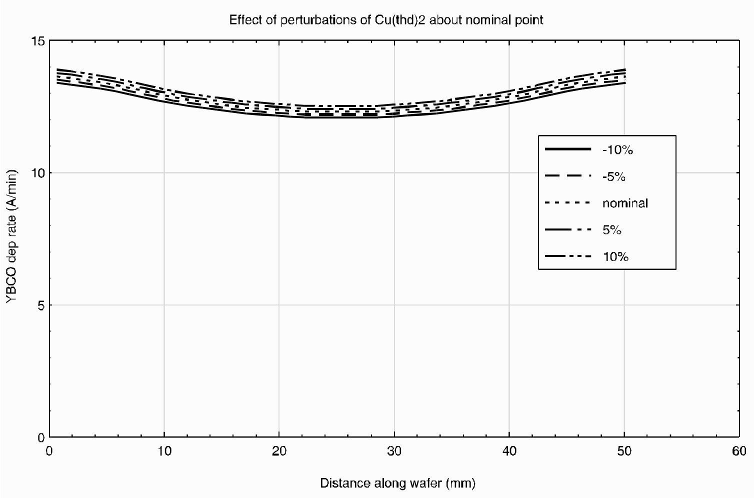
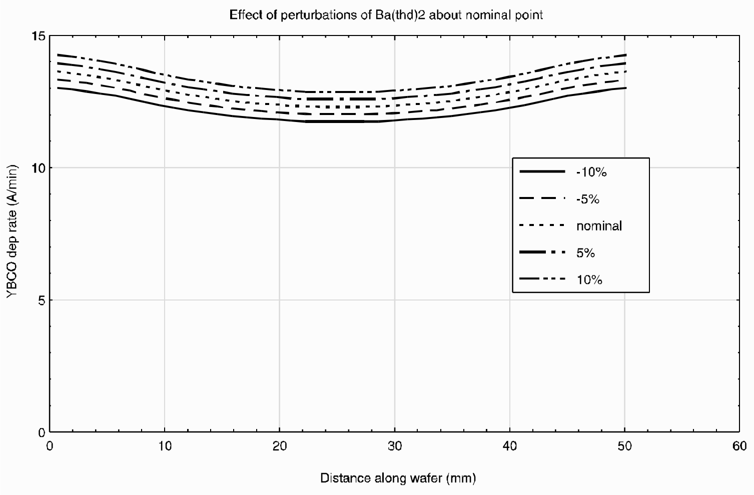
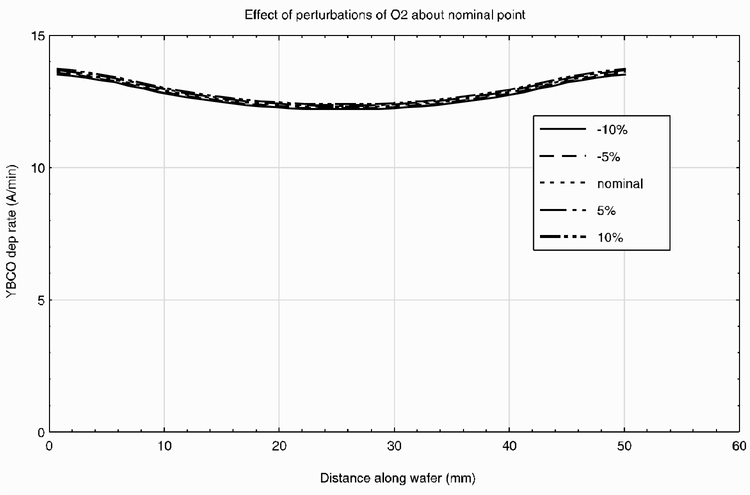
MOCVD Control

Obtaining *G* from Response Surface calculations:


Effect of perturbations of Y(thd)3 about nominal point


Effect of perturbations of Cu(thd)2 about nominal point


Effect of perturbations of Ba(thd)2 about nominal point


Effect of perturbations of O2 about nominal point

Summary

- A 2D model of MOCVD reactor has been developed for deposition of YBCO thin films.

- System characterization showed the need for control of growth rate, deposition uniformity, and oxide stoichiometry at the surface.

- A run-to-run control architecture was developed, and is currently being implemented.