Model-based Control of MOCVD Rate, Uniformity and Stoichiometry

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YBCO Thin Films

Fully oxygenated orthorhombic unit cell of YBa$_2$Cu$_3$O$_7$. At room temperature the lattice parameters are: $a=3.819\,\text{Å}$, $b=3.883\,\text{Å}$, $c=11.687\,\text{Å}$

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 100-250\(^\circ\)C, with vapor pressure between 0.01-1 Torr.
MOCVD of YBCO Thin Films

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: \( O_2 = 0.44, N_2 = 0.47, Ar = 0.088, Y(dpm)_3 = 2.72 \times 10^{-5}, \) \( Ba(dpm)_2 = 4.41 \times 10^{-5}, Cu(dpm)_2 = 2.35 \times 10^{-5} \). 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:

\[ \text{SiHCl}_3 + \text{H}_2 \rightarrow \text{Si} + 3\text{HCl} \]

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 deposition 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

![Graph showing precursor mass fractions vs distance along chamber (mm) 5 mm above wafer.]

- **Cu(dpm)2**
- **Y(dpm)3**
- **Ba(dpm)2**
MOCVD of YBCO Thin Films

Growth occurs in a mass transport-limited regime
MOCVD of YBCO Thin Films

- Yttrium oxide dep rate
- Binary diffusivity with oxygen (m^2/s)

Distance along wafer (mm)

Temperature (K)

Sticking coefficient = 1
Sticking coefficient = 0.2

BAR
YBCO
MOCVD of YBCO Thin Films

Chemistry:

\[ \text{Y(dpm)}_3 + \text{O}_2 \rightarrow \text{Y} + 3 \text{(dpm)} + \text{O}_2 \]

\[ \text{Y(dpm)}_3 \rightarrow \text{Y} + 3 \text{(dpm)} \]

\[ \text{Cu(dpm)}_2 \rightarrow \text{Cu} + 2 \text{(dpm)} \]

\[ \text{Ba(dpm)}_2 \rightarrow \text{Ba} + 2 \text{(dpm)} \]

\[ 4 \text{Y} + 3 \text{O}_2 \rightarrow 2\text{Y}_2\text{O}_3 \]

\[ 2 \text{Ba} + \text{O}_2 \rightarrow 2\text{BaO} \]

\[ 2 \text{Cu} + \text{O}_2 \rightarrow 2\text{CuO} \]
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

Ex-situ measurements:
metrology (dep thickness, uniformity), stoichiometry

In-situ Substrate Temperature Sensor

Dynamic Inner-loop Controller
Actuator (bubbler heater)

nominal process set points

commanded set points

Process:
MOCVD Reactor

nominal process set points

commanded set points

Temperature Sensor

Dynamic Process (Temperature) Controller
Actuator (Substrate Heater)

In-situ Substrate Temperature Sensor

Run-to-run Controller

Ex-situ measurements: metrology (dep thickness, uniformity), stoichiometry

disturbances

disturbances
Introduction to Run-to-Run Control

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

\begin{quote}
Adjust recipe for next run based on results of previous runs such that product quality improves
\end{quote}
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:

\[
e_t(i) = \frac{y_t(i) - y_{\text{des}}(i)}{y_{\text{tol}}(i)}, \quad i = 1, \ldots, n
\]

- Adjust recipe according to:

\[
r_t = r_{\text{nom}} + u_t,
\]

\[
u_t = u_{t-1} - G e_{t-1}, \quad u_0 = 0
\]
Static Linear Error System

Introduction

• Assume actual process is a static linear error system:

\[ e_t = w_t + Gu_t, \quad t = 0,1,2,\ldots \]

• \( w_t \) is vector of product quality errors due to nominal control:

\[ w_t = e_t \mid r_t = r_{nom} \]

• Error and control:

\[ e_t = (I_n - GG)e_{t-1} + w_t - w_{t-1}, \quad e_0 = w_0 \]

\[ u_t = (I_m - GG)u_{t-1} - Gw_{t-1}, \quad u_0 = 0 \]
• 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.:

\[ w_t = b + ct + \nu_t \]

then

\[ w_t - w_{t-1} = c + \nu_t - \nu_{t-1} \]

with \( b \) denoting bias, and \( c \) denoting drift rate, and \( \nu_t \) a zero-mean random variable.

• However, if \( \nu_t \) has variance \( \sigma^2 \), then \( \nu_t - \nu_{t-1} \) has variance \( 2\sigma^2 \)!
• Suppose $G$ is square ($n = m$) and invertible, then the stability analysis suggests the choice:

with $\mu$ a real scalar.

• The error and control equation are now given by:

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

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

• The system is stable for:

$$0 < \mu < 2.$$
Obtaining $G$ from Response Surface calculations:

Effect of perturbations of $Y$ (mol/mol) about nominal point:

Effect of perturbations of $C_2H_5OH$ about nominal point:

Effect of perturbations of $B$ (mol/mol) about nominal point:

Effect of perturbations of $O_2$ 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.