Toward controlled ultra-high vacuum chemical vapor deposition processes

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Toward Controlled Ultra-High Vacuum Chemical Vapor Deposition Processes

Martijn Dresscher

Omslag: Licht passeert door een vacuüm kijkglas en kruist paden met een atoom. Een korte absorptie van het licht veroorzaakt vervolgens een richtingsverandering.

Book cover: Light passes through a vacuum window and crosses paths with an atom. A short absorption of the light subsequently causes a change in direction.

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Toward Controlled Ultra-High Vacuum Chemical Vapor Deposition Processes

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Samenvatting

Een ultra hoog vacuüm chemische damp afzetting (UHVCVD, ultra-high vacuum chemical vapor deposition) reactor wordt gebruikt om zeer dunne vaste stof lagen op gekozen oppervlakken te produceren vanuit atomaire en moleculaire precursors in de dampfase. Deze precursors kunnen met elkaar reageren nabij of op het oppervlak en nieuwe verbindingen en stoffen maken met specifieke eigenschappen. Deze processen worden onder andere gebruikt om halfgeleiders te maken voor de chip en processor industrie, maar de mogelijkheden zijn divers. Dit procestype onderscheidt zich van vergelijkbare processen doordat er zeer weinig verontreinigingen in de afgezette laag komen. Minder verontreinigingen zorgen ervoor dat de opgedampte laag beter functioneert.

Om de ultra hoge vacuüm condities te bereiken moet een reactor een thermische reiniging ondergaan. Dit houdt in dat de hele reactor voor meerdere uren op hoge temperatuur (ongeveer 330 graden Celsius) gehouden wordt, zodat achtergebleven atomen en moleculen verdampen en weggepompt kunnen worden. Een nadeel hiervan is dat veel elektronische meetinstrumenten een dergelijke behandeling niet kunnen doorstaan. Hiernaast zorgt de afwezigheid van warmtegeleidende lucht ervoor dat de temperaturen in de reactor niet snel uniform zijn en dat thermische contactmetingen onnauwkeurig worden. Deze factoren zorgen er gezamenlijk voor dat er weinig (actuele) informatie beschikbaar is over relevante toestanden in de reactor, zoals dampdrukken en temperaturen.

Om een regelsysteem te ontwerpen voor een dergelijk proces hebben we actuele (zogeheten echte-tijd) informatie nodig over de relevante toestanden. Een regelsysteem bestaat over het algemeen uit: (i) echte-tijd metingen van relevante staten voor terugkoppeling van het effect van de aansturing, (ii) een dynamisch model dat met redelijke nauwkeurigheid beschrijft hoe de aansturing de gemeten staten beïnvloed en (iii) regelalgoritmen, afgestemd op (ii), die aan de hand van (i) gepaste aansturingen bepalen.

In deze thesis leveren we bijdragen aan de ontwikkeling van regelalgoritmen voor echte-tijd deeldruk regulatie in een UHVCVD proces. We implementeren een

echte-tijd deeldruk meting die geschikt is voor gebruik door een regelalgoritme. Hiernaast hebben we een model ontwikkeld dat potentieel met hoge spatiële resolutie de gas stromen in de reactor kan beschrijven. Dit model kan daardoor de informatie over de reactoraansturing gebruiken om te voorspellen hoe de deeldruk verandert. We laten zien dat dit model accurate voorspellingen kan geven van de deeldruk wanneer we in dampdruk condities opereren. Het model, als alle componenten geïdentificeerd zijn, en de deeldruk metingen kunnen samen gebruikt worden voor het ontwerp van regelalgoritmen. We laten de toepasbaarheid van de deeldruk metingen voor het ontwerpen van regelalgoritmen zien door twee regelaars te implementeren die een minimale modelidentificatie vergen. De resultaten laten zien dat beide regelaars geschikt zijn voor deze doeleinden, voor de reactorcondities waarin ze geëvalueerd zijn.

Naast de bovenstaande bijdragen, leveren we ook bijdragen aan de ontwikkeling van theoretische regelalgoritmes voor systemen die omschreven kunnen worden met deterministische dynamica en stochastische initiële staten. We laten zien dat onze regelalgoritmes theoretische garanties kunnen geven dat het effect van de variatie beperkt blijft voor een vooraf bepaald deel van de initiële staten.

Summary

An ultra-high vacuum chemical vapor deposition (UHVCVD) reactor is used to deposit thin solid layers on desired surfaces from atomic and molecular precursors in the vapour phase. These precursors can react near or on these surfaces to form new compounds with specific properties. One of the applications these processes are used for is to build semiconductors for the chips and processor industries, but the range of applications is wide. These processes are distinct from other deposition processes because they can deposit layers of extreme high purity. This high purity in turn allows the deposited layer to perform better.

A UHVCVD reactor has to undergo a thermal cleaning process in order to reach ultra-high vacuum conditions. This entails keeping the entire reactor on a high temperature (approximately 330 degrees Celsius) for multiple hours. This allows residual atoms and molecules to evaporate so that they can be pumped away from the system. A disadvantage of this procedure is that many electronic measurement devices cannot survive prolonged exposure to such temperature. The absence of heat conducting air furthermore causes reactor temperatures to be non-uniform, which in turn causes thermal measurements to be local. The availability of (real-time) information on the relevant reactor states, like pressures and temperatures, is accordingly limited.

We need real-time information on the relevant reactor states for the control system of such a process. The control system typically requires three components: (i) real-time measurements of relevant reactor states to provide feedback on the effect of the actuation, (ii) a dynamical model that can, with reasonable accuracy, describe how the actuation influences the states, and (iii) a control algorithm, that is based on (ii), that calculated in real-time the required actuation, based on (i).

In this thesis we contribute to the development of real-time partial pressure controllers for UHVCVD processes. Firstly, we develop a real-time partial pressure measurement that is suitable for the implementation of model-based controllers. Secondly, we have developed a mathematical model that can describe the evolution of particle fluxes inside the reactor with a high spatial resolution. This model uses the information on the reactor inputs for this purpose and provides estimated partial pressures inside the reactor as output. We show that this model can provide accurate predictions of the partial pressure in vapor pressure conditions. The provided model, once identified, and the partial pressure measurements can together be used for controller design. We furthermore show the efficacy of the real-time partial pressure measurement for controller design by implementing two controllers. These controllers rely on minimal modeling and identification efforts. The obtained results show that both controller designs are suitable for such purposes under the evaluated reactor conditions.

In addition to the contributions listed above, we provide contributions to the development of theoretical control algorithms for systems that can be described through deterministic dynamics and stochastic initial condition of the states. We show that our control algorithms can give theoretical guarantees that the impact of the variations remains bounded for a predetermined fraction of the initial states.

Acknowledgments

The majority of my time during the last four years has been spent obtaining knowledge and skills that were required for delivering and communicating the content of this thesis. This has been a journey that would have been significantly more difficult without the people close to me, on both a professional and a social level.

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To my friends and family, which I need not name individually. I am lucky to have so many good contacts, even if it is sometimes infrequent. Your support and friendship these years kept me going, whether we had a meal together, did sports, had drinks, simply watched a movie or went on holiday. I could not have completed this challenging project without the great interactions with you. I dearly hope that we can maintain these contacts in the future, despite any challenging circumstances caused by geographic distance or busy schedules.

List of symbols

Part I

$\mathbb{M}$	2-dimensional set of surfaces facing the atom cloud	22
ω_1, ω_2	Surfaces, in $\mathbb{M}$	22
$\theta_{\mathrm{d}\omega_1}, \theta_{\mathrm{d}\omega_2}$	Angles with normals of $\mathrm{d}\omega_1$ and $\mathrm{d}\omega_2$	23
$d_E(\cdot)$	Euclidean distance	23
$\phi_{d\omega_1}, \phi_{d\omega_2}$	Rotations around normals of $d\omega_1$ and $d\omega_2$	23
φ	Relative rotation $d\omega_1$ and $d\omega_2$ around central axis of cylinder	23
P	Partial pressure of a precursor	23
V	Volume of considered body holding the atom cloud	23
N	Number of moles of a precursor in atom cloud	23
R	The ideal gas constant	23
T_a	Temperature of the atom cloud	23
t	Time variable	23
$\dot{N}_{in}$	Moles per second entering the volume holding the atom cloud	23
$\dot{N}_{out}$	Moles per second leaving the volume holding the atom cloud	23
$\dot{N}_s(\cdot)$	Function describing moles per second sorbed to surfaces	23
n	Number of discrete surfaces considered	24
$\bar{x}$	Fluxes in moles per second on a surface ω	24
p^A	Knudsen cosine law transfer matrix	24
s	Sorbed moles, on a surface ω	24
$G(\cdot)$	Input function	24
u	General model input	24
z	Time derivative of s	24
A	Corrected Knudsen cosine law transfer matrix, $A = \delta p^A$	24
δ	Time scale constant	24
I	Identity matrix	24
L	Leakage matrix	24
$f(\cdot)$	Sorption function	24

T	Collection of temperatures of surfaces in $\mathbb{M}$	24
$g(\cdot)$	Input function, relates u to y	24
m	Number of inputs	24
$\bar{y}$	Fluxes model output in partial pressure	24
$h(\cdot)$	Output function, relates x to y	24
M	Molar mass	25
$\mathbb{1}$	n-length column vector of ones	25
ℓ	Symmetric path length matrix	25
v	Average speed of a precursor in atom cloud	25
q	Directed flux matrix between surfaces	27
θ	Generalized coordinate, angle with normal	28
ϕ	Generalized coordinate, rotation around normal	28
$\vartheta(\cdot)$	Departing particle angle pdf	28
$\bar{r}$	Displacement along radial line from cylinder center	28
$\bar{H}$	Cylinder height	28
$\bar{R}$	Cylinder radius	28
$\bar{h}$	Displacement along height line from cylinder bottom	28
$\mathcal{P}$	Set of safe states	30
x_d	Desired trajectory of x	30
x^*	Desired operating point	30
x	General model states	30
$k(\cdot)$	Control law	30
$\mathcal{D}$	Set of unsafe states, complement of $\mathcal{P}$	30
$B(\cdot)$	Control barrier function	31
C^1	Class of functions that are once continuously differentiable	31
∂D	Boundary of the set $\mathcal{D}$	31
$\mathrm{Int}(\mathcal{D})$	Interior of the set $\mathcal{D}$	31
y	General system output	31
$\hat{f}(\cdot)$	General nonlinear vector field function	31
$\hat{g}(\cdot)$	General nonlinear input vector field function	31
$\hat{h}(\cdot)$	General nonlinear output vector field function	31
$\mathcal{L}_{\hat{f}}(\cdot)$	Lie-derivative over $\hat{f}$ at a point	31
$\mathcal{V}(\cdot)$	Control Lyapunov function	31
U	Set of admissible inputs	31
X	Set of admissible states	31
c_1, c_2, c_3	Constants > 0	31
κ_1, κ_2	Constants > 0	31
α_1, α_2	Class $\mathcal{K}$ functions	32
μ	Feasible input	32
$\mathcal{P}_{\text{clf\&cbf}}$	Set of safe state where both clf and cbf constraints are active	33
$\mathcal{P}_{\text{clf}}$	Set of safe state where only clf constraints are active	33
$\gamma_{\text{clf}}, \gamma_{\text{cbf}}$	Constant components of clf and cbf contraints	33

$k_{\text{clf\&cbf}}$	Control law in set of states where both clf and cbf are active	33
k_{clf}	Control law in set of states where only clf is active	33
$F(\cdot), H(\cdot)$	Cost functions components for quadratic program	33
$K_{\text{clf}}(\cdot)$	CLF admissible input set for a point in state space	33
$K_{\text{cbf}}(\cdot)$	CBF admissible input set for a point in state space	34
$\epsilon_a, \epsilon_r, \epsilon_h$	Cylinder discretization parameters	35
i_1, i_2, i_3	Integers used for cylinder discretization	35
$\mathcal{B}$	Matrix allocating input flux to surfaces	36
$\mathcal{C}$	Matrix relating states to output	36
$\mathcal{H}_\infty$	H infinity norm	37
$\hat{u}$	Derivative of flux input	37
$\hat{A}, \hat{B}, \hat{C}$	System matrices for reduced order model	37
$\hat{a}_1, \hat{a}_2$	Constant components of $\hat{A}$	37
$\hat{b}_1, \hat{b}_2$	Constant components of $\hat{B}$	37
y^*	Desired output	40
$\vartheta_\theta(\cdot)$	Departing particle angle pdf for given $\phi_{d\omega_1}$	43
$\vartheta_\phi(\cdot)$	Departing particle angle pdf for given $\theta_{d\omega_1}$	43
$\Theta_\theta(\cdot)$	Departing particle angle cdf for given $\phi_{d\omega_1}$	43
$\Theta_\phi(\cdot)$	Departing particle angle cdf for given $\theta_{d\omega_1}$	43
E	Photon energy	47
l_w	Wavelength of a photon	47
h_p	Planck's constant	47
c	Speed of light in a vacuum	47
T_r	Cold spot temperature reference signal	52
ν	Temperature input for experiment	52
T_c	Cold spot temperature	52
λ	Absorbance	52
Q_{in}	Light intensity of bundle entering the atom cloud	52
Q_{out}	Light intensity of bundle exiting the atom cloud	52
l	Length of absorbing path	53
k_a	absorbing coefficient	53
Q_s	Measured light intensity with shutter open	53
Q_b	Measured light intensity with shutter closed	53
Q_{ref}	Measured baseline light intensity	53
Λ	Measured sodium pressure	53
$\Psi(\cdot)$	Mapping from λ to Λ	53
P_{Na}	Theoretical sodium vapor pressure	53
$\xi(\cdot)$	Sodium vapor pressure function	53
b_1, b_2, b_3	Parameters in sorption function $f(\cdot)$	54
β_1, β_2	Parameters in sorption function $\dot{N}_s(\cdot)$	54
$\hat{y}$	Moles model output	54
e	Error signal, $\bar{y} - P_{Na}$	55

e_Λ	Absolute and normalized error of Λ w.r.t. P_{Na}	58
$e_{\hat{y}}$	Absolute and normalized error of $\hat{y}$ w.r.t. P_{Na}	58
$e_{\bar{y}}$	Absolute and normalized error of $\bar{y}$ w.r.t. P_{Na}	58
ζ	Constant component of output function $h(\cdot)$	61
a_1, a_2	Constant components of $\xi(\cdot)$	61
ρ	Measure of evaporation history	65
ς	Measure of temperature	65
$\Upsilon(\cdot)$	Pdf describing input-output probabilities	65
$\Upsilon_{u,\lambda}(\cdot)$	Marginal pdf of input current u and absorbance λ	66
$\mathcal{R}$	Domain of ρ	66
$\mathcal{T}$	Domain of ς	66
Γ	Set of input-output pairs (u, λ) for which $\Upsilon_{u,\lambda} > 0$	66
u_d	Input required to realize λ_d	67
λ_d	Desired absorbance	67
$\Phi(\cdot)$	mapping of u to λ for feedforward component of controller	68
$\mathcal{A}$	Considered domain for controller design of λ	69
k	Discrete time step counter	69
K_p	Proportional control gain	69
u_i	Integral control input component	69
e_{fb}	Error between λ_d and λ	69
K_i	Integral control gain	69
Δt_k	Discrete time step length between timestep k and $k-1$	69
u_{ff}	Feedforward control component	69
u_{fb}	Feedback control component	69
t_k	Time associated with timestep k	70
η	Online identified component in input-output relation	71
ϱ	Constant relating magnitude of input and output	71
u_{mf}	Model-free control component	71
Δ	Update parameter	72

Part II

x	Stochastic state variable	82
X	Domain of x	82
u	System input, generalized force	82
U	Domain of u	82
t	Time indicator	82
$f(\cdot)$	System vector field	82
x_0	Stochastic initial condition	82
X_0	Domain of x_0	82
$\phi_{x_0}(\cdot)$	Probability density function of x_0	82
$\phi_{x_0,t}(\cdot)$	Probability density function of $x(t)$	82
T	Transient time for transient performance specification	82
Ξ	Subset of X, desired containment set	82
$\Phi_{\Xi,T}(\cdot)$	Cumulative density of $\phi_{x_0,T}$ over the set Ξ	82
ξ	Integrator variable	82
σ	Second moment of a probability density function	82
μ	Mean value of probability density function	82
p^*	Desired containment level	83
$d(\cdot)$	Unspecified distance	83
x_d	Desired asymptotic trajectory of x	83
$k(\cdot)$	Control law	83
A, B	System matrices for linear system	84
x^*	Constant desired asymptotic convergence value	84
K	Control gain matrix	84
$\tilde{x}$	Stochastic state variable after coordinate change	84
$\tilde{x}_0$	Stochastic initial condition after coordinate change	84
$\mathcal{N}(\cdot)$	Normal distribution	84
$\tilde{\Xi}$	Desired containment set after coordinate change	84
$\tilde{x}_{T,low}, \tilde{x}_{T,up}$	Bounds on $\tilde{\Xi}$	84
$\tilde{\Xi}_0$	Initial time containment set after coordinate change	84
$\tilde{x}_{0,low}, \tilde{x}_{0,up}$	Bounds on $\tilde{\Xi}_0$	84
p_{max}	Maximum containment level	84
$\mathrm{erf}(\cdot)$	The error function	84
τ	Time at which desired asymptotic behavior can be realized	85
u_d	Desired input signal associated with desired trajectory x_d	85
$d_E(\cdot)$	Euclidean distance between two points	85
ϵ_1, ϵ_2	Center of ball or distance set	86
κ_1, κ_2	Radius of ball or distance set	86
$\mathbb{B}_\kappa(\cdot)$	Ball set defined with respect to Euclidean distance	86
x_r	Tracking reference signal	86
u^*	Feedforward control input	86

z	State of reference system	86
ζ	State of error system	86
ζ_0	Stochastic initial condition of error system	86
λ	Contraction exponential rate constant	86
$d_F(\cdot)$	Finsler distance	87
$f_c(\cdot)$	Closed-loop system vector field	88
C	Contraction region	88
$\mathbb{D}_\kappa(\cdot)$	Distance set defined with respect to Finsler distance	88
$\mathcal{X}$	Domain of robot manipulator states	90
$\mathcal{Q}$	Domain of q	90
q	Generalized position vector	90
$\mathcal{S}$	Unitary circumference	90
p	Generalized momentum vector	90
θ_1, θ_2	Rotational positions robot joints	90
s	Translational position of robot joint	90
$p_{\theta_1}, p_{\theta_2}, p_s$	Momentum of robot joints	90
$M(\cdot)$	Inertia matrix	90
F_1, F_2, F_3	Robot manipulator input components	90
$\mathbf{0}$	Zero matrix	90
I	Identity matrix	90
$H(\cdot)$	Hamiltonian function	90
$D(\cdot)$	Damping matrix	90
$G(\cdot)$	Input matrix	90
$V(\cdot)$	Potential energy	90
m_1, m_2, m_3	Robot manipulator link masses	90
M_1, M_2	Inertia matrix components	90
l_1, l_2	Robot manipulator link lengths	90
q_0	Stochastic initial condition of robot manipulator position	90
μ_q	Mean of q	90
Σ_q	Covariance of q	90
$\mu_{q,1}, \mu_{q,2}, \mu_{q,3}$	Mean values of q components	91
Σ	Covariance matrix	91
$\sigma_{q,1}, \sigma_{q,2}$	Variance of q components	91
p_0	Initial condition of robot manipulator momentum	91
g_c	Gravitational constant	91
q_d	Desired asymptotic trajectory for generalized position	91
p_d	Desired asymptotic trajectory for generalized momentum	91
q_r	Reference trajectory for generalized position	91
ϵ_q	Point associated to $q_r(T)$	91
$\tilde{q}$	Error in q, defined as $q - q_d$	91
ω	Error in p, defined as $p - p_r$	91
p_r	Reference trajectory for generalized momentum	91

$p_{d\omega}$	Reference component for p	91
Λ	Controller component	91
ϵ_p	Point associated to $p_r(T)$	91
$u_{\text{eq}}, u_{\text{at}}$	Input components for robot manipulator controller	92
K_d	Controller gain matrix	92
$V_F(\cdot)$	Finsler-Lyapunov function	92
x_v	Virtual system state	92
δx_v	Virtual system tangent vector	92
$\delta q_v, \delta p_v$	Virtual system tangent vector components	92
Θ	Constant component of V_F for robot manipulator example	92
$P(\cdot)$	Functional component of V_F for robot manipulator example	92
$\Gamma(\cdot)$	Collection of γ curves between two points	92
$\gamma(\cdot)$	Differentiable curve between two points	92
$\Upsilon(\cdot)$	Functional component of contraction rate λ	92
r	Polar coordinate	93
$\rho(\cdot)$	Angle used in figures	94
$\mathbf{1}(\cdot)$	Step function	94
T_xX	Tangent space of point x	97
TX	Tangent bundle of all points in X	97
c_1, c_2, c_3	Constants used for defining Finslet-Lyapunov function	97
F	Finsler structure	97
$\mathcal{I}$	Domain existing between 0 and 1	98
$\alpha(\cdot)$	Function defining contraction properties	98
$\mathcal{B}(\cdot)$	The Bhattacharyya coefficient	100
$d_h(\cdot)$	The Hellinger distance	100
$d_b(\cdot)$	The Bhattacharyya distance	100
ℓ	Desired Hellinger distance	100
Y	Domain of a pdf, specified to define matching property	101
$\varphi(\cdot)$	Arbitrary pdf	101
η, β	Matrices describing linear matching of pdf	101
ε	Scaling correction for linear matching of pdf	101
$\tilde{\varepsilon}$	Scaling of pdf caused by system dynamics	102
$\tilde{\eta}, \tilde{\beta}$	Matrices describing pdf change through system dynamics	102
$\tilde{y}, y$	Points after two different coordinate changes from x	103
Z	Domain of a pdf, specified to define matching property	104
$\vartheta(\cdot)$	Correction for elongation of pdf for nonlinear matching map	104
$\Psi(\cdot)$	Function relating two pdfs in nonlinear matching map	104
μ_d	Desired mean point	106
Σ_d	Desired covariance matrix	106
μ_T	Realized mean	107
Σ_T	Realized covariance matrix	107

List of Figures

List of abbreviations

AAS	Atomic absorption spectrometry
CBF	Control barrier function
CCP	Containment control problem
CLF	Control Lyapunov function
CVD	Chemical vapor deposition
DOF	Degree of freedom
EIES	Electron impact emission spectrometry
ES-CBF	Exponentially safe control barrier function
ES-CLBF	Exponentially stable control Lyapunov-barrier function
ES-CLF	Exponentially stable control Lyapunov function
FMF	Free molecular flow
HCL	Hollow-cathode lamp
KKT	Karush-Kuhn-Tucker
LPCVD	Low pressure chemical vapor deposition
MBE	Molecular beam epitaxy
MC	Monte-carlo
MFPI	Model-free proportional integral controller
MS	Mass spectrometry
pdf	Probability density function
PI	Proportional integral
PID	Proportional integral derivative
PIFF	Proportional integral controller with feedforward component
PVD	Physical vapor deposition
QP	Quadratic program
SCARA	Selective compliance assembly robot arm
SCP	Shape control problem
UHV	Ultra-high vacuum
UHVCVD	Ultra-high vacuum chemical vapor deposition