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Toward controlled ultra-high vacuum chemical vapor deposition processes

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Document Version

Publisher's PDF, also known as Version of record

Publication date:

2019

[Link to publication in University of Groningen/UMCG research database](#)

Citation for published version (APA):

Dresscher, M. (2019). *Toward controlled ultra-high vacuum chemical vapor deposition processes*. Rijksuniversiteit Groningen.

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“To know that we know what we know, and to know that we do not know what we do not know, that is true knowledge.”

– Nicolaus Copernicus.

The work presented in this thesis has been done to improve the performance of in particular ultra-high vacuum chemical vapor deposition (UHVCVD) production processes, but can have wider implications for vacuum based deposition systems. The conclusions have been divided into three sections. In Section 7.1 we present the conclusions related to Part I of the thesis, which considers accordingly the design, modeling and control of UHVCVD. In Section 7.2 we present the conclusions related to Part II of the thesis and hence considers the control design for a more general class of systems, namely deterministic systems with stochastic initial conditions, which can be of use for UHVCVD process improvement in the future. In Section 7.3 we lastly present our recommendations for future research.

7.1 Conclusions Part I

Part I of this thesis starts with contributions to modeling and associated controller design in Chapter 2. We have presented a flux-based model that can connect reactor inputs to partial pressure measurements, suitable for model-based controller design. A crucial part of the flux dynamics, namely the transfer matrix, has been validated with results from literature. We have furthermore shown the suitability of our flux model for model-based control design through a numerical simulation, using a Lyapunov-based controller design with guaranteed safety, for a high-resolution model realization without nonlinear components. Following this, we have numerically evaluated the suitability of such a Lyapunov-based controller for some of the more realistic dynamics that we have observed experimentally, again without some of the nonlinear components. We found a good performance for such dynamics. The results listed above provide some theoretical foundations that are required for model-based controller design. They are however not validated experimentally and therefore lack a solid connection with reality. The proposed controller designs furthermore consider only a part of the real dynamics that can be relevant in such a

system. Experimental realization is expected to require explicit consideration of (currently unidentified) nonlinear components.

The results from Chapter 2 provide a starting point for model identification and subsequent controller design. To proceed in this direction, we have designed an experimental reactor which allows for suitable actuation and measurements. We have presented and implemented this reactor design in Chapter 3. Here, we have firstly provided an exposition on possible measurement techniques that can facilitate a real-time partial pressure measurement inside a vacuum. This exposition has led to selection of the atomic absorption spectroscopy (AAS) based sensor. The presented experimental reactor design accordingly incorporates this sensor. We have subsequently used the experimental reactor design to validate the AAS measurements and our flux-based model. We have shown that both the measurements and the flux-based model are suitable for real-time characterization of partial pressure measurements in the vapor pressure regime, for a single element. We have furthermore compared the flux-based model performance with that of a more simple and lower resolution mole-based model, which can serve as a benchmark. We found that the quality of the predictions made by the two models is comparable. The results from Chapter 3 accordingly provide the connection between the flux model and reality. We have provided and validated two components of the controlled reactor, namely the AAS-based partial pressure measurement and the flux model suitable for model-based control design. The flux model has been identified for sodium vapor pressure estimation with controlled cold spot temperature, using the structure of semi-empirical vapor pressure functions used in the literature. However, this causes the structure to be suitable for the vapor pressure operation region only. The flux model accordingly still requires more intensive identification in order to be suitable for design of a model-based controller that can also operate outside this vapor pressure region.

We estimated that further identification of the flux model was too time consuming for us to pursue. We have accordingly moved forward with controller design that requires a very minimum of modeling and that is tested experimentally. The controller designs that we have presented aim to make the AAS measurement track a specified reference, for a single element in the process. These controller designs are presented and implemented in Chapter 4. We have shown good control performances for a proportional integral controller with a feedforward component (PIFF) and for a model-free proportional integral (MFPI) controller. Both controllers can effectively deal with the large degree of variations that exists in the relation between the applied input current to the evaporation source and the AAS measurement. We consider such controller flexibility to be important, as the variations can get exacerbated when, for example, multiple elements are present in the process that interact with each other. The controllers have not been optimized and it is therefore difficult to describe relative performances. We furthermore cannot provide analytic guarantees of performance, since we lack the knowledge on the system dynamics required

for such analysis. Our results do indicate that both controllers are stable, where the PIFF seems relatively robust when compared to the MFPI. On the other hand, we found that the error of the MFPI is less sensitive to changes in reactor conditions than the error for the PIFF, for constant controller gains. This suggests that the MFPI is less sensitive to parameter tuning than the PIFF controller.

7.2 Conclusions Part II

In Part II we started by considering a containment control problem (CCP) in Chapter 5. We have formulated the CCP so that it requires the probability density function (pdf) of the state to satisfy a prespecified cumulative density over a set, during a specific transient time, in addition to the standard asymptotic convergence criterion. This is a very general control problem formulation as it includes both the deterministic case and the case where the initial condition is unbounded. The transient criterion is furthermore comparable to traditional performance criteria like rise-time and settling-time. We have presented sufficient conditions, that are very general, to solve the CCP for both linear and nonlinear systems. We have furthermore shown the efficacy of our controller design for nonlinear systems through numerical simulation of a robot manipulator. The linear results are relatively simple to implement, as we rely on a simple control law to realize our results. The results for nonlinear systems are less straightforward, as they require design of a nonlinear control law so that desired contraction properties are obtained.

In Chapter 6, we have considered a shape control problem (SCP). The SCP requires the pdf of the state to satisfy a prespecified distance criterion with a desired pdf, in addition to the standard asymptotic convergence criterion. This control problem formulation is considerably more specific than the CCP, as it requires the pdf to maintain a closeness to a desired shape instead of a cumulative density over a set, which can be attained with (almost) arbitrary shape. We have provided sufficient conditions to solve the SCP for linear systems only, where the initial and the desired pdf are furthermore required to have a known relation that can be expressed through a linear or nonlinear matching formalism. We have furthermore performed additional numerical evaluation of our controller design for linear matching pdfs. The obtained results are simple to implement, given the knowledge on the matching relation between the initial and desired pdfs.

The nonlinear CCP result, presented in Theorem 3 in Chapter 5, can be applied to UHVCVD process once a model, such as presented in Chapter 2, has been identified. Application of such a controller will be sensible if the variations can be described through the initial conditions. If this is not the case, e.g. when variations have to be described through the vector field, then the nonlinear CCP result is unsuitable in its current form. Describing the variations through the initial conditions is generally difficult for such processes, because the dynamics of such a state with variations

have to be well understood. This required in-depth knowledge on states that are difficult to measure. We therefore expect that further development of such controllers presented in Part II of this thesis is required for the UHVCVD implementation. The linear results CCP and SCP that we have presented are expected to be unsuitable for most nonlinear systems, since the linearization procedure is expected to cause inaccuracies in the vector field that can be relatively large and influential compared to the variations in the initial conditions.

7.3 Recommendations for future research

The work in this thesis has produced candidate partial pressure controllers that rely on a minimum of modeling. The controllers work well in the considered setting, but cannot anticipate on fast changes in reference or system dynamics. Our results furthermore indicate that the controllers are not performing well when exposed to such changes. We hence recommend to put future effort in producing a model-based controller design, so that the controller can anticipate on such abrupt changes using model predictions.

The flux model that we have presented in this thesis is a suitable model to base such a controller design on, but still requires identification. The model can be complemented by a thermodynamic model to cover the full reactor dynamics. The identification process is expected to yield interesting mathematical and physical insights in the evaporation and sorption phenomena that are being identified. It involves creating a physical understanding of the UHVCVD process (with components such as shown in Fig. 1.4) and determining suitable mathematical descriptions. Examples of these descriptions are then the input and sorption function components of the flux model. The AAS-based measurement that we have implemented and validated can be used for such identification procedures.

One cannot perform the identification procedure of the process without explicitly considering the run-to-run variations. Such an identification will accordingly yield a model that has dynamics, states or both which are subject to these variations. However, as most of the sources of these variations will in turn be subject to dynamics, it should be possible to describe such variations through stochastic states having deterministic dynamics. This requires significant modeling effort, but allows for deterministic control design with performance guarantees such as we have presented in Part II of this thesis. If it is not possible to describe these variations in this way, then the vector field of the model can instead contain these run-to-run variations. For such a model, the controller design then needs to be such that the controllers guarantees a good performance for all realizations of the vector field, in addition to the states. This can be seen as an extension of the controller designs for deterministic systems with stochastic initial conditions.