LOW PRESSURE CHEMICAL VAPOUR DEPOSITION OF POLYCRYSTALLINE SILICON: VALIDATION AND ASSESSMENT OF REACTOR MODELS

W.L.M. Weerts*, M.H.J.M. de Croon and G.B. Marin**

Eindhoven University of Technology, Laboratorium voor Chemische Technologie,
P.O. Box 513, 5600 MB Eindhoven, The Netherlands

Abstract - The layer thickness of polycrystalline silicon grown in a commercial LPCVD reactor at 25-50 Pa and 850-950 K is accurately simulated with a one-dimensional two-zone model without adjusting any model parameter and applying independently determined reaction kinetics. The radial non-uniformity of the layer thickness is limited to 4%. At a surface-to-volume ratio of 200 m⁻¹ up to 20% of the growth originates from silylene and disilane. This contribution is the cause of the radial non-uniformity.

INTRODUCTION

The hot-wall multiwafer low-pressure chemical vapour deposition (LPCVD) reactor, first introduced in the late seventies (Rosler, 1977), is the most common reactor for the deposition of polycrystalline silicon from silane. It realizes a large packing density of silicon wafers perpendicularly to the axis of the tube and an excellent layer thickness uniformity across each wafer as well as from wafer to wafer.

To gain quantitative insights in the interaction of the mass transport phenomena with the chemical reaction kinetics in such a reactor, several mathematical reactor models of different degrees of sophistication have been proposed up to now (Jensen and Graves, 1983; Roenigk and Jensen, 1985,1987; Middleman and Yeckel, 1986; Roenigk, 1987; Yeckel and Middleman, 1987; Yeckel et al., 1989; Joshi, 1987; Vinante et al., 1989; Duverneuil and Couderc, 1992; Badgwell et al., 1992a,b; Azzaro et al., 1992,1994). The majority of these studies is based on the pioneering work of Jensen and Graves (1983). Basically, the model developed by these authors consists of a combination of two sets of one-dimensional, i.e. axial in the annular zone between the reactor wall and the edges of the wafers and radial in the interwafer zone, continuity equations for the species considered, coupled to each other by the boundary conditions at the wafer edges. Besides such simplified one-dimensional models, more sophisticated two-dimensional models have been developed recently, treating the hydrodynamics and the mass transport with chemical reactions in a more detailed manner. In order to reduce the computational effort the modelling domain is in general confined to a single interwafer space and the corresponding annular space, situated somewhere in the middle of the wafer section to eliminate possible effects from the upstream and downstream ends of the wafer load. Most of the assumptions postulated in the simplified one-dimensional models are relaxed. The most comprehensive work in this field was reported by Azzaro et al. (1994).

The complexity and the importance of the chemical kinetics of the deposition is well recognized. Nevertheless, the kinetics applied during every single reactor modelling effort to date were either very simplified, e.g. of the Langmuir-Hinshelwood type and neglecting gas phase reactions (Jensen and Graves, 1983; Roenigk and Jensen, 1985; Wilke et al., 1986), based on theoretical calculations and thermodynamic data (Coltrin et al., 1986,1989) or obtained under conditions far removed from the
temperature and pressure ranges used in LPCVD. The present work reports on the degree of sophistication needed in the modeling to adequately describe silicon growth rate data obtained in a commercial LPCVD setup at operating conditions that are industrially relevant, i.e. total pressures around 50 Pa and temperatures around 900 K. The applied rate equations were obtained independently by regression of a set of kinetic rate data collected with a microbalance reactor at similar conditions (Weerts, 1995). Four elementary gas phase reactions between six gas phase species and ten elementary surface reactions are considered. The validity of two reactor models is tested by comparing their predictions with the experimental growth rate data obtained in the commercial LPCVD reactor. During the reactor model validation no adjustment of the kinetic parameter values is performed.

EXPERIMENTAL

The growth experiments were carried out in a conventional hot-wall multiwafer LPCVD reactor of the TEMPRESS type Junior at TNO/TPD Eindhoven. Figure 1 shows the reactor configuration. The reactor consists of four distinct sections: 1) premixing section (not shown), 2) preheating section, 3) wafer section and 4) downstream section. The premixing section is separated from the preheating section by an insulation ring which serves to maintain uniform temperature control in the wafer section and prevents heating of the gas inlets. The preheating and downstream sections have lengths similar to that of the wafer section.

![Figure 1: Schematic representation of reactor configuration.](image)

The most important features of the geometry of the TEMPRESS Junior reactor are listed in Table 1.

<table>
<thead>
<tr>
<th>Table 1: Geometrical features and deposition conditions</th>
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<tr>
<td><strong>TEMPRESS</strong></td>
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<tr>
<td>Reactor length (L_3/\text{m} )</td>
</tr>
<tr>
<td>Wafer section length (L/\text{m} )</td>
</tr>
<tr>
<td>Reactor tube radius (R_\text{t}/\text{m} )</td>
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<tr>
<td>Wafer radius (R_\text{w}/\text{m} )</td>
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<tr>
<td>Interwafer spacing (\Delta/\text{m} )</td>
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<tr>
<td>Position of first wafer /\text{m}</td>
</tr>
<tr>
<td>Position of last wafer /\text{m}</td>
</tr>
<tr>
<td>Number of wafers in wafer section</td>
</tr>
<tr>
<td>Total pressure (p_\text{/Pa} )</td>
</tr>
<tr>
<td>Temperature (T/\text{K} )</td>
</tr>
<tr>
<td>Silane inlet flow rate (Q_{\text{SiH}_4}/\text{Nml min}^{-1} )</td>
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<tr>
<td>Deposition time / min</td>
</tr>
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</table>

*The reactor length as specified here comprises the preheating, the wafer and the downstream section. The premixing section is not included.*
A 0.38 m long fused quartz boat containing thirty-eight 0.10 m diameter silicon wafers is mounted coaxially with the furnace axis. Thirty single-side polished (100) p-type silicon wafers are positioned at equal distances of 0.01 m in the wafer section, with the polished sides directed to the downstream end of the reactor tube. These substrates were oxidized ex-situ to a thickness of 100 nm to facilitate post-deposition layer thickness measurement. At the upstream and downstream ends of the boat, just outside the wafer section, four dummy wafers are positioned at equal distances of 0.005 m. These wafers serve to smooth out axial and radial temperature variations over the deposition wafers caused by radiative heat losses to the cooled reactor inlet and outlet doors. Temperature uniformity within 1 K was established during the deposition experiments.

Layer thickness measurements were performed on the polished sides, i.e. the back sides, of at most 7 monitor wafers in the 30 wafer load, i.e. wafer numbers (1), 5, 10, 15, 20, 25, (29), by means of interferometry and ellipsometry. Deviations between these methods were within 5%. In the range of experimental conditions applied, see Table 1, the measured radial thickness variations were smaller than the experimental accuracy. Silicon growth rates were calculated by dividing the measured layer thicknesses by the corresponding deposition times. In addition, for each of the monitor wafers a radially averaged growth rate was calculated as the arithmetic mean of all growth rates measured on the wafer.

**REACTOR MODELS**

A one-dimensional two-zone and a fully two-dimensional reactor model are compared. In the former case the reactor is thought to consist of an annular zone between the wafer edges and the reactor wall and of an interwafer zone. The corresponding model, similar to that developed by Roenigk and Jensen (1985), consists of two sets of one-dimensional continuity equations for all gas phase components. One set allows to calculate the concentration profiles along the axial coordinate in the annular zone, while the other set allows to calculate the concentration profiles along the radial coordinate in the interwafer zone. Coupling between both sets is accomplished by the boundary conditions at the wafer edges. The fully two-dimensional reactor model follows from the straightforward application of the conservation laws of mass, momentum and energy. The binary diffusion coefficients as well as the transport properties of the individual gas phase species are calculated from the Chapman-Enskog kinetic theory of gases (Hirschfelder et al., 1967).

**One-dimensional two-zone reactor model**

The continuity equation for component $i$ in the interwafer zone is given by:

$$\frac{1}{r} \frac{d}{dr} \left( r n_i^w \right) = \sum_{m=1}^{NG-1} v_{m,1} y_{a,m} + \sum_{k=1}^{NG} v_{k,1} y_{v,k}$$

(1)

There are for an ideal gas mixture in this zone containing NG components (NG-1) independent Stefan-Maxwell equations:

$$\frac{dy_i^w}{dr} = \sum_{j\neq i} \frac{y_j^w N_j^w - y_i^w N_i^w}{CD_{i,j}}$$

(2)

and:

$$\sum_{i=1}^{NG} y_i^w = 1$$

(3)
Symmetry around the reactor axis and continuity of the molar fractions from the wafer edge to the annular zone are expressed by the boundary conditions:

\[ r = 0 \quad N_i^w = 0 \] (4)

\[ r = R_w \quad y_i^w = y_i^a \] (5)

The continuity equation for component i in the annular zone is given by:

\[ \frac{dN_i^a}{dz} = \frac{2}{R_e^2 - R_w^2} \left[ R_b (1 + \alpha) \sum_{m=1}^{n_a} \nu_{m,i} r_{a,m} + R_w N_i^w \bigg|_{r=R_w} \right] + \sum_{k=1}^{n_g} \nu_{k,i} \Gamma_{v,k} \] (6)

Multicomponent diffusion effects are again accounted for by (NG-1) independent Stefan-Maxwell equations:

\[ \frac{dY_i}{dr} = \sum_{j \neq i} y_i^a N_j^a - y_j^a N_i^a \] (7)

and

\[ \sum_{i=1}^{NG} y_i^a = 1 \] (8)

Continuity of the molar fluxes across the inlet to the wafer section is expressed by:

\[ z = 0 \quad N_i^a = u_0 C y_i^a,0 \] (9)

Furthermore, it is assumed that no diffusion takes place across the plane at the end of the wafer section:

\[ z = L \quad \frac{dy_i^a}{dz} = 0 \] (10)

which implicitly means that negligible reaction occurs downstream of the wafer section.

To integrate the complete set of differential equations with corresponding boundary conditions, 6th order orthogonal collocation (Finlayson, 1972) was applied in both the r-direction of the interwafer zone and the z-direction of the annular zone. The resulting set of algebraic equations is solved using a modified Newton-Raphson method in the standard NAG-library routine C05NBF (NAG, 1991). A typical reactor simulation requires a CPU-time of 30 minutes on a Silicon Graphics Power Challenge Computer.

**Fully two-dimensional model**

The reactor configuration considered in the fully two-dimensional model is shown in Figure 1. The effects of the premixing section are lumped into the treatment of the preheating section. The model
development thus involves the description of the performance and coupling of the preheating, the wafer and the downstream sections. Compared to the one-dimensional two-zone reactor model the modelling domain is expanded so that the inlet and outlet boundaries are removed from the wafer section. In this way possible effects associated with the idealised inlet and outlet of the simplified one-dimensional model, see equations (9) and (10), are minimized.

A general derivation and the form of the two-dimensional model equations representing the conservation of mass, momentum and energy is given in standard references on transport phenomena (Bird et al., 1960; see also Kleijn, 1991). Heat effects associated with gas phase reactions and the Dufour energy flux are neglected in the energy conservation equation. Also the so-called Soret effect, causing diffusion fluxes as a result of temperature gradients has been neglected in the continuity equations for gas phase components. Diffusive fluxes are calculated from the Stefan-Maxwell equations.

The usual no-slip boundary conditions are assumed to hold on all solid surfaces. The wall and wafer temperatures inside the wafer section are supposed to be fixed by the furnace setting, whereas the walls outside the wafer section are assumed to be adiabatic. Heat effects associated with surface reactions are neglected.


KINETIC MODEL

The kinetic model used has been developed by modelling of a large set of kinetic experiments performed in the range of industrially relevant operation conditions for the deposition of polycrystalline silicon (Weerts, 1995). It consists of four elementary gas phase reactions coupled to ten elementary surface reactions. The dehydrogenation of surface species formed during adsorption reactions is assumed to proceed instantaneously towards solid silicon. Hence, besides the gas phase species hydrogen, silane, silylene (SiH2), disilane (Si2H6), disilene (H2SiSiH2) and silylsilylene (H3SiSiH3), hydrogen adatoms form the only kinetically significant surface species. The homogeneous net production rates of the above gas phase components follow from the law of mass action for the rates of forward and reverse steps of the elementary gas phase reactions. Rate coefficients of unimolecular steps are calculated using an empirical relation accounting for the pressure fall-off behaviour of these steps (Robinson and Holbrook, 1972; Weerts, 1995). The rate coefficients of the reverse steps are calculated from thermodynamics (Coltrin et al., 1986, 1989).

The calculation of the heterogeneous net production rates of the above gas phase species is less straightforward. The complexity of the surface reaction network does not allow a straightforward derivation of closed kinetic expressions relating the silicon deposition rates from the individual silicon containing gas phase species to gas phase concentrations only. Writing the mass balance for hydrogen adatoms in terms of the concentration of vacant surface sites, yields a third order equation in L, the concentration of vacant surface sites, which is solved numerically. L is subsequently used to calculate the heterogeneous net production rates of the silicon containing gas phase species following the law of mass action for the rates of adsorption reactions.

After solving the complete set of governing equations of either the one-dimensional two-zone or the fully two-dimensional reactor model, the silicon growth rate is calculated from:

\[
R_{Si} = \frac{M_{Si}}{\rho_{Si}} \sum_{n=1}^{ns} \nu_{n, Si} x_{n,m}
\]

VALIDATION AND ASSESSMENT OF REACTOR MODELS

The validity of the one-dimensional two-zone and the fully two-dimensional reactor models is tested by comparing their predictions with the experimental growth rates obtained in the range of operating
conditions as listed in Table 1. Figure 2a shows that both reactor models adequately describe the radially averaged axial growth rate profiles over a broad range of temperatures at 50 Pa. The effect of total pressure is simulated adequately as well. This is shown in Figure 2b where the silicon growth rate is plotted versus the axial reactor coordinate at 900 K and pressures of 25 and 50 Pa. Within the complete range of experimental conditions, the calculated radial non-uniformities are smaller than the experimental accuracy of the layer thickness measurements.

Clearly, both the one-dimensional two-zone model and the fully two-dimensional model adequately describe the experimental growth rate data along the axial coordinate. The much higher degree of sophistication applied in the fully two-dimensional model thus seems to be redundant. In the following part the additional simplifications made in the development of the one-dimensional two-zone model will be discussed.

The calculations with the fully two-dimensional model revealed temperature uniformity within 0.2 K in the interwafer zone and a negligible total pressure drop across the reactor. The largest pressure drop relative to the reference pressure prescribed in the outlet of the reactor was calculated for 25 Pa and 900 K and amounted to 1.3 Pa. In general, a small pressure drop takes place across the wafer section, determined largely by the size of the annular zone cross-sectional area (Coronell and Jensen, 1992). The above considerations clearly justify the assumptions concerning isothermal and isobaric operation used in the development of the one-dimensional two-zone model.

Figure 3a shows a typical profile of the axial mass average velocity component versus the radial reactor coordinate at 50 Pa and 900 K midway between two successive wafers, i.e. at $z = \Delta z/2$. Clearly, the gas mixture is motionless in axial direction between successive wafers. The axial velocity components associated with the so-called Stefan flow, caused by the non-equimolar counter-diffusion, are too small in absolute sense to be captured in this figure.

In Figure 3b the radial mass average velocity component is plotted versus the axial position between two successive wafers at 50 Pa and 900 K for different radial positions. These profiles show a radial gas entrance immediately downstream of the first wafer and the corresponding radial gas exit just upstream of the second wafer. However, the small value of the radial velocity component at 4.83 $10^{-2}$ indicates that this gas circulation is confined to the outer 2 mm of the interwafer zone only. The above considerations validate the hypothesis of a purely axial gas movement in the annular zone and of a motionless gas in the interwafer zone as postulated in the development of the one-dimensional two-zone model.

Figure 4 shows the calculated silane molar fraction profiles versus the axial reactor coordinate at 50 Pa and the three considered temperatures, i.e. 850, 900 and 950 K. The dotted vertical lines reflect the boundaries of the wafer section.

**Figure 2a&b:** TEMPRESS LPCVD reactor. Radially averaged silicon growth rate versus the axial reactor coordinate. Dashed lines: fully two-dimensional model. Full lines: one-dimensional two-zone model. Points: experiments. Conditions: $Q_{SiH_4} = 90 \text{ Nml min}^{-1}, 100\% \text{ SiH}_4$; a) $p_i = 50 \text{ Pa, } + T = 850 \text{ K, } o T = 900 \text{ K, } \Box T = 950 \text{ K}$. b) $T = 900 \text{ K, } + p_i = 25 \text{ Pa, } o p_i = 50 \text{ Pa}$. 
Figure 3a&b: TEMPRESS LPCVD reactor. Mass average velocity component versus reactor coordinate as calculated with fully two-dimensional reactor model. Conditions: $Q_{SiH_4} = 90 \text{ Nml min}^{-1}$, 100% SiH$_4$, $p_r = 50 \text{ Pa}$, $T = 900 \text{ K}$. a) axial component versus radial coordinate at $z = A/2$; b) radial component versus axial coordinate between two successive wafers at $r = 4.83 \times 10^{-2} \text{ m}$, $r = 5.00 \times 10^{-2} \text{ m}$, $r = 5.15 \times 10^{-2} \text{ m}$.

Figure 4: TEMPRESS LPCVD reactor. Silane molar fraction versus axial reactor coordinate at different temperatures. Dashed lines: fully two-dimensional model. Full lines: one-dimensional two-zone model. Conditions: $Q_{SiH_4} = 90 \text{ Nml min}^{-1}$, 100% SiH$_4$, $p_r = 50 \text{ Pa}$, $T = 850 \text{ K}$, $T = 900 \text{ K}$, $T = 950 \text{ K}$.

Clearly, the agreement between the silane molar fraction profiles calculated using the one-dimensional two-zone model and those obtained with the fully two-dimensional model is satisfactory. This underlines the correctness of omitting the preheating and downstream sections and of using the Danckwerts boundary conditions in the one-dimensional two-zone model. The Danckwerts entrance boundary condition leads to a significant step change in composition at the wafer section entrance. This concentration discontinuity results from the allowance of diffusion on the downstream side of the boundary but not on the upstream side of the boundary and, hence, confirms the relative importance of axial diffusion in the annular zone.

In case convection was the only mode of transport, a silane molar fraction equal to one would have been calculated at the same point. The axial diffusion thus reduces the variation in reactant concentration and, hence, improves the layer thickness uniformity along the wafer section. Comparison of the results shown in Figures 2 and 4 reveals that changes in the molar fraction of silane have no large impact on the silicon growth rate. This is consistent with the rather low partial reaction order of silane (Weerts, 1995). Depending on silane space time and feed composition, the partial reaction order varies between 0.28 and 0.44 at 900 K.

The full lines in Figure 5 represent typical molar fraction profiles of silylene and disilane calculated at 50 Pa and 900 K using the fully two-dimensional model. The molar fractions are plotted as a function of the radial reactor coordinate for different axial positions between two successive wafers located in the middle of the wafer section. These results clearly demonstrate the existence of significant variations in the silylene and disilane molar fractions, from high values in the annular zone to much lower values in the
interwafer zone. These differences can be linked to differences in the surface-to-volume ratios of the annular and the interwafer zone. Mass transport effects are of minor importance in this context. Note that silylene and to a lesser extent disilane show significant concentration gradients in both the axial direction of the interwafer zone and the radial direction of the annular zone.

The profiles of disilene and silylsilylene, not shown, exhibit similar gradients as silylene in both directions. Silane on the other hand, being much less reactive than the above mentioned gas phase intermediates, has completely flat profiles. Although the one-dimensional two-zone model does not account for these concentration gradients of the gas phase intermediates, silicon growth rates calculated using this strongly simplified reactor model do not significantly differ from those calculated using the fully two-dimensional model, see Figure 2. This is caused by the relatively small contribution of the reactive intermediates to the silicon growth rate. At 50 Pa and 900 K the average contribution of the most important gas phase intermediates, i.e. silylene and disilane, amounts to roughly 20%. Furthermore, the dashed lines in Figure 5 show that the radial molar fraction profiles of silylene and disilane calculated with the one-dimensional two-zone model roughly fall in between those calculated with the fully two-dimensional model at the different axial positions. Hence, the use of the one-dimensional reactor model results in molar fractions at the surface that are only slightly different from those calculated with the two-dimensional reactor model. Consequently, the errors in the calculated wafer growth rates introduced by neglecting the concentration profiles in both the axial direction of the interwafer zone and the radial direction of the annular zone are typically less than one percent only. The difference between the radially averaged growth rates predicted by these models will thus be less than one percent as well. Based on these considerations, it can be concluded that even under conditions where 20% of the silicon growth originates from gas phase intermediates a one-dimensional two-zone model is sufficient.

ORIGIN OF RADIAL NON-UNIFORMITY IN AN INDUSTRIAL-SCALE REACTOR

It was shown in the previous section that, except for the layers grown at 950 K (Figure 2a), a satisfactory layer uniformity could be obtained for the batch of wafers in the TEMPRESS reactor. The axial non-uniformity at 950 K is caused by the silane conversion and, hence, can be reduced by imposing an adequate temperature profile or by feeding silane at intermediate positions as well. Radial non-uniformity can become important with increasing wafer diameter. Badgwell et al. (1992b) attributed the latter to radial temperature gradients. Figure 6 shows growth rates calculated by the isothermal one-dimensional two-zone model for an industrial-scale LPCVD reactor with wafers of a diameter of 200 mm. The operating conditions are listed in Table 1. The total growth rate is almost completely
determined by the heterogeneous decomposition of silane. The silane conversion increases from 47.8% at the entrance to 66.3% at the exit of the wafer section. The contribution from all gas phase intermediates to the growth rate is approximately constant over the complete length of the wafer section. It corresponds to the growth rate contributions from SiH₂ and SiH₆ and amounts to roughly 3%. The contribution from H₂SiSiH₂ and H₂SiSiI-I to the total growth rate due to gas phase intermediates is typically less than 1%. Figure 6 shows simulation results versus the radial coordinate in the interwafer zone at an axial position in the reactor corresponding to a silane conversion of 53.5%.

The growth rate non-uniformity sharply increases on the periphery of the wafer and shows a maximum of 6.3% at the edge of the wafer. The wafer edge non-uniformity, which decreases from 7.5 to 5.7% along the length of the wafer section, is caused by radial variations in the growth rates from both silylene and disilane. Apparently, there is no need to involve radial temperature gradients to explain radial growth non-uniformities.

**CONCLUSIONS**

A one-dimensional two-zone model can sufficiently describe the operation of a LPCVD reactor for the deposition of polycrystalline silicon at typical industrial conditions without adjustment of any model parameter. The radial non-uniformity of the layer thickness results from the contribution of silylene and disilane to the growth.

**NOTATION**

<table>
<thead>
<tr>
<th>Symbol</th>
<th>Description</th>
<th>Unit</th>
</tr>
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<tbody>
<tr>
<td>C</td>
<td>concentration</td>
<td>mol m⁻³</td>
</tr>
<tr>
<td>Dᵣ</td>
<td>binary molecular diffusion coefficient</td>
<td>m² s⁻¹</td>
</tr>
<tr>
<td>k</td>
<td>reaction rate coefficient</td>
<td>reaction dept.</td>
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<td>L</td>
<td>surface concentration</td>
<td>mol m⁻²</td>
</tr>
<tr>
<td>N</td>
<td>molar flux w.r.t. fixed coordinates</td>
<td>mol m⁻² s⁻¹</td>
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<tr>
<td>p</td>
<td>pressure</td>
<td>Pa</td>
</tr>
<tr>
<td>Rₛᵢ</td>
<td>silicon growth rate</td>
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<tr>
<td>r</td>
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<td>r</td>
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<td>u</td>
<td>molar/mass averaged velocity</td>
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<td>y</td>
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<tr>
<td>z</td>
<td>axial coordinate</td>
<td>m</td>
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<tr>
<td>Δ</td>
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<td>m</td>
</tr>
<tr>
<td>v</td>
<td>stoichiometric coefficient</td>
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</table>

**Superscripts:**

- a: annular zone
- w: interwafer zone

**Subscripts:**

- a: axial
- i: gas phase component
- k: gas phase reaction
- m: surface reaction
- t: reactor tube
- R: radius
- v: volumetric
- w: wafer
- O: inlet
- *: vacant site

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REFERENCES


