CONF- 9605110 -- 1

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# A Reaction Mechanism for Titanium Nitride CVD from TiCl4 and NH3

(To appear in the proceedings of the Thirteenth International Conference on Chemical Vapor Deposition held May 5–10, 1996 in Los Angeles, CA)

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for the United States Department of Energy
under Contract DE-AC04-94AL85000

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# A REACTION MECHANISM FOR TITANIUM NITRIDE CVD FROM $TiCl_4$ AND $NH_3$

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## ABSTRACT

A gas-phase and surface reaction mechanism for the CVD of TiN from $TiCl_4$ and $NH_3$ is proposed. The only gas-phase process is complex formation, which can compete with deposition. The surface mechanism postulates the stepwise elimination of Cl and H atoms from $TiCl_4$ and $NH_3$, respectively, to form solid TiN and gaseous HCl. The mechanism also accounts for the change in oxidation state of Ti by allowing for liberation of $N_2$. Provided that the surface composition is at steady state, the stoichiometry of the overall reaction is reproduced exactly. In addition, the global kinetic law predicted by the mechanism is successfully fit to new deposition data from a rotating disk reactor and is shown to be consistent with literature results.

## INTRODUCTION

Thin films of titanium nitride (TiN) have several actual and potential applications in microelectronics, in particular as diffusion barriers between silicon and aluminum layers. While TiN has traditionally been deposited via physical processes such as reactive sputtering, the need for improved step coverage in structures of high aspect ratio has led to the investigation of chemical vapor deposition (CVD) as an alternative. TiN can be deposited from a number of chemical precursor systems, but each has particular disadvantages with regard to integrated circuits. For example, deposition from $TiCl_4 + N_2 + H_2$ requires temperatures that are much too high, while the use of metalorganic compounds such as tetrakis(dimethylamido)titanium (generally in conjunction with $NH_3$) leads to films that can have significant carbon, oxygen, and hydrogen contamination (1). A compromise of sorts is provided by the $TiCl_4 + NH_3$ process, which is feasible at moderate temperatures ($> 750$ K) and yields films whose main drawback is chlorine contamination (2). Experimental efforts to improve this process are underway, and the present paper is an attempt to develop a more detailed understanding.

Several quantitative studies of the deposition rate of TiN from $TiCl_4$ and $NH_3$ have been reported (3–8), and in two of these (3,4) a global rate law is inferred from the data. In addition, at least one theoretical description of some of the elementary surface processes has appeared (9). To date, however, no attempt has been made to derive an overall rate law from a complete reaction mechanism. In this paper a schematic but self-consistent mechanism is proposed, and an expression for the deposition rate is obtained in terms of the (unknown) individual rate constants. The latter are then evaluated by fitting the predictions of the model to some new experimental data. Finally, the results of this analysis are shown to be generally consistent with the observations of previous investigators.

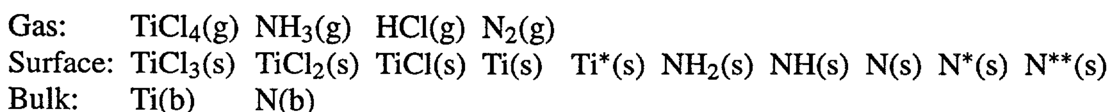
## REACTION MODEL

In constructing a mechanism for the TiN CVD process, a few general observations must be taken into account. First, the overall reaction is essentially irreversible at the temperatures normally used, since $\Delta G$ is strongly negative for $T > 600$ K. Second, there is preliminary evidence (10) that gas-phase reactions are of minor importance in this system, i.e., the process appears to be dominated by surface reactions. A complicating factor is that the titanium atoms undergo a change in oxidation state from +4 to +3. As a consequence, the products of the reaction include (presumably) gas-phase $N_2$ as well as HCl, and the overall stoichiometry is

$$6\,TiCl_4(g) + 8\,NH_3(g) \rightarrow 6\,TiN(b) + 24\,HCl(g) + N_2(g) \qquad [1]$$

The notation (b) is used here to indicate a bulk (solid) species, as distinguished from surface species that function only as reaction intermediates.

A surface mechanism that is consistent with the above observations can now be written in terms of the following species:



Gas: $TiCl_4(g)$ $NH_3(g)$ $HCl(g)$ $N_2(g)$
Surface: $TiCl_3(s)$ $TiCl_2(s)$ $TiCl(s)$ $Ti(s)$ $Ti^*(s)$ $NH_2(s)$ $NH(s)$ $N(s)$ $N^*(s)$ $N^{**}(s)$
Bulk: $Ti(b)$ $N(b)$

The notation for the surface and bulk species requires some elaboration. First of all, a surface species is defined to be one that resides in the topmost layer of the solid, while a bulk species resides in

any layer below this. Furthermore, Ti–N bonds are not explicitly noted here. Thus, for example, $TiCl_3(s)$ is a surface species in which one of the original Ti–Cl bonds has been replaced by a Ti–N bond. Similarly, in Ti(s) all four of the Ti–Cl bonds have been replaced. In $Ti^*(s)$, however, one of the new Ti–N bonds has been severed. The nitrogen-containing species are named analogously, so that in $N^{**}(s)$ two of the N–Ti bonds have been broken. Keeping in mind the oxidation states in TiN, it is clear that $Ti^*(s)$ is the immediate precursor to a bulk titanium, Ti(b), while N(s) is the species that becomes N(b). Furthermore, an $N_2$ molecule is formed from two $N^{**}(s)$ species by breaking the last N–Ti bond to each. Needless to say, whenever a nitrogen surface species loses a bond, a titanium species must do the same.

In proposing a surface reaction mechanism, we note that the likely interactions among the species defined above fall naturally into five groups:

Titanium deposition:

1. $TiCl_4(g) + NH_2(s) + Ti^*(s) \rightarrow TiCl_3(s) + NH(s) + HCl(g) + Ti(b)$
2. $TiCl_4(g) + NH(s) + Ti^*(s) \rightarrow TiCl_3(s) + N(s) + HCl(g) + Ti(b)$

Nitrogen deposition:

3. $TiCl_3(s) + NH_3(g) + N(s) \rightarrow TiCl_2(s) + NH_2(s) + HCl(g) + N(b)$
4. $TiCl_2(s) + NH_3(g) + N(s) \rightarrow TiCl(s) + NH_2(s) + HCl(g) + N(b)$
5. $TiCl(s) + NH_3(g) + N(s) \rightarrow Ti(s) + NH_2(s) + HCl(g) + N(b)$

Surface condensation:

6. $TiCl_3(s) + NH_2(s) \rightarrow TiCl_2(s) + NH(s) + HCl(g)$
7. $TiCl_3(s) + NH(s) \rightarrow TiCl_2(s) + N(s) + HCl(g)$
8. $TiCl_2(s) + NH_2(s) \rightarrow TiCl(s) + NH(s) + HCl(g)$
9. $TiCl_2(s) + NH(s) \rightarrow TiCl(s) + N(s) + HCl(g)$
10. $TiCl(s) + NH_2(s) \rightarrow Ti(s) + NH(s) + HCl(g)$
11. $TiCl(s) + NH(s) \rightarrow Ti(s) + N(s) + HCl(g)$

Bond breaking:

12. $Ti(s) + N(s) \rightarrow Ti^*(s) + N^*(s)$
13. $Ti(s) + N^*(s) \rightarrow Ti^*(s) + N^{**}(s)$

$N_2$ liberation:

14. $2\,Ti(s) + 2\,N^{**}(s) + 2\,N(b) \rightarrow 2\,Ti^*(s) + N_2(g) + 2\,N(s)$

It should be noted that this mechanism (and each step individually) automatically conserves the total number of surface species, or sites. In reaction #1, for example, when a gas-phase $TiCl_4$ attaches to the surface, a $Ti^*(s)$ becomes buried and is thus now a bulk species instead. As a result, one bulk-phase atom is added to the solid for each one that arrives from the gas. A reverse process occurs in reaction #14.

Because the surface species are merely reaction intermediates, it is natural to assume that their concentrations will be at steady state. A conservation equation can then be written for each; however, since the total concentrations of titanium and nitrogen species are both conserved, only eight of the ten equations are independent. Denoting by $R_i$ the rate (in mol/cm$^2$s) of reaction $i$, the equations can be written concisely as

$$R_1 + R_2 = R_3 + R_6 + R_7 = R_4 + R_8 + R_9 = R_5 + R_{10} + R_{11} = 3\,R_{12} = 3\,R_{13} = 6\,R_{14} \equiv r \quad [2]$$

$$R_3 + R_4 + R_5 = R_1 + R_6 + R_8 + R_{10} = R_2 + R_7 + R_9 + R_{11} \equiv s \quad [3]$$

The net production rates of the gaseous and bulk species are then

$$
\begin{aligned}
\text{HCl(g):}\; & R_1 + R_2 + R_3 + \cdots + R_{11} = 3s && = 4r \\
\text{TiCl}_4\text{(g):}\; & -R_1 - R_2 && = -r \\
\text{NH}_3\text{(g):}\; & -R_3 - R_4 - R_5 = -s && = -\tfrac{4}{3}r \\
\text{N}_2\text{(g):}\; & R_{14} && = \tfrac{1}{6}r \\
\text{Ti(d):}\; & R_1 + R_2 && = r \\
\text{N(d):}\; & R_3 + R_4 + R_5 - 2\,R_{14} = s - \tfrac{1}{3}r = r
\end{aligned}
$$

Comparing with Eq. [1], it can be seen that the surface mechanism reproduces exactly the stoichiometry of the overall reaction.

Next, the kinetic law for the process can be derived by inserting expressions for the rates of the individual steps into Eqs. [2] and [3], assuming mass-action kinetics in each case. For simplicity, the rate constants will be taken to be equal for all reactions within a given group; the values for the five groups will be denoted by $\alpha$, $\beta$, $\gamma$, $\lambda$, and $\delta$, respectively. The goal is to write the overall deposition rate $r$ in terms of these rate constants and the concentrations $C_{\mathrm{TiCl_4}}$ and $C_{\mathrm{NH_3}}$ of the gas-phase reactants. Since the concentration of N(b) is a constant, it can be incorporated into the rate constant $\delta$ with no loss of generality. In order to eliminate the concentrations of the ten surface species, one can use the eight conservation equations together with the condition that the total concentrations of Ti- and N-containing species are each equal to 1/2 of the overall surface site density $\rho$. After a fair amount of algebra, the following expression for the deposition rate is obtained:

$$
r = \frac{3\lambda}{4(\phi_n + 2)^2}\left\{\left[\frac{\lambda}{2\delta} + \rho^2\frac{(\phi_n+2)\theta\phi_t\phi_n}{1+\phi_t+\theta\phi_t\phi_n}\right]^{1/2} - \left(\frac{\lambda}{2\delta}\right)^{1/2}\right\}^2 \qquad [4]
$$

where $\phi_t = (5\alpha/3\gamma)\,C_{\mathrm{TiCl_4}}$, $\phi_n = (5\beta/4\gamma)\,C_{\mathrm{NH_3}}$, and $\theta = \gamma/5\lambda$. Needless to say, the way in which the deposition rate varies with the reactant concentrations is, according to Eq. [4], rather complicated. However, the behavior in various limiting cases is simple and physically realistic. For example, if $\phi_t$ and $\phi_n$ are both arbitrarily small, then

$$
r \doteq \frac{25}{384}\,\delta\left(\frac{\alpha\beta\rho^2}{\gamma\lambda}\cdot C_{\mathrm{TiCl_4}}\cdot C_{\mathrm{NH_3}}\right)^2 \qquad [5]
$$

and the overall reaction is second-order in each reactant. On the other hand, if $\phi_n$ is arbitrarily small but $\phi_t \gg 1$, then

$$
r \doteq \frac{3}{128}\,\delta\left(\frac{\beta\rho^2}{\lambda}\cdot C_{\mathrm{NH_3}}\right)^2 \qquad [6]
$$

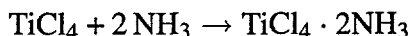
so that the reaction is still second-order in $NH_3$ but now zeroth-order in $TiCl_4$. This accords reasonably well with the results that have been reported in the literature (3,4); a small negative order in $TiCl_4$ is actually observed, but this has been attributed to competing gas-phase complex formation. In any case, it should be noted that Eq. [6] does not involve the rate constants $\alpha$ and $\gamma$, which may therefore be difficult to determine.

## EVALUATION OF RATE CONSTANTS

Since there are currently no methods available for calculating $\alpha$, $\beta$, $\gamma$, $\lambda$, and $\delta$ from first principles, the only option is to infer them from experimental data for the overall deposition rate. Of course, this rate is invariably reported as a function of the inlet gas composition, while the surface

mechanism is written in terms of local concentrations. In order to account for such effects as mass transport and reactant depletion, one must use the mechanism in conjunction with a computational reactor model. Unfortunately, literature studies generally fail to specify some key parameters, such as the surface/volume ratio and the residence time, that would be necessary input to a reactor simulation. The only data set for which such information is available is that generated recently at Sandia/New Mexico in an MRC rotating disk reactor (11). Therefore, this data will be used to determine the rate constants in the analysis to follow.

As noted above, the expression in Eq. [4] is at worst of order 0 in $TiCl_4$, whereas most experimental studies show a small negative order. This has been attributed to a competing gas-phase reaction that produces the (possibly) nonreactive complex $TiCl_4 \cdot 2NH_3$ (3). Therefore, the surface mechanism will be augmented here with the homogeneous reaction



$$TiCl_4 + 2\,NH_3 \rightarrow TiCl_4 \cdot 2NH_3$$

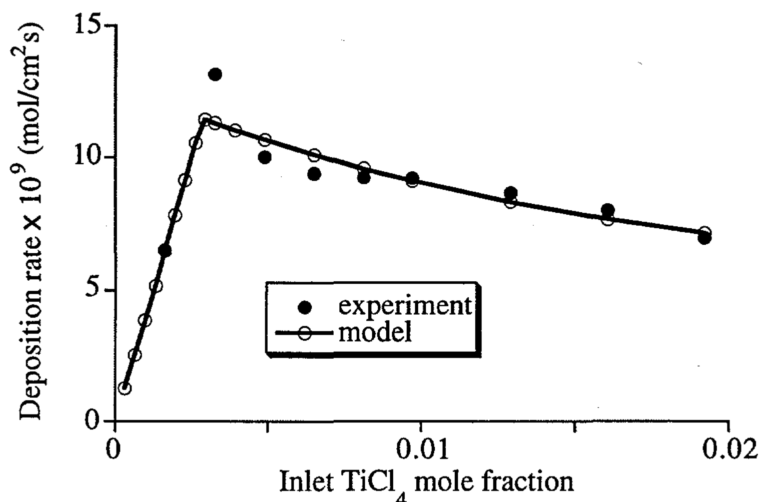
having a third-order rate constant $k$. The supposition is that an increasing inlet concentration of $TiCl_4$ causes additional $NH_3$ to be tied up in complexes and thus decreases the deposition rate, which is generally observed to have a strong positive order in $NH_3$.

In the Sandia study, the deposition rate was measured as a function of the inlet concentration of $TiCl_4$ but not $NH_3$. Furthermore, experiments were carried out at only one substrate temperature (903 K), so all computed surface rate constants will refer to this temperature. However, there was a substantial temperature gradient between the substrate and the inlet showerhead (at 623 K), so an activation energy $E$ will be used in conjunction with the gas-phase rate constant $k$. There are thus 7 adjustable parameters to be determined by optimizing the fit to the experimental deposition rates over the entire range of $TiCl_4$ concentrations. For any given set of parameters, the predicted deposition rates are computed from Sandia's SPIN code (12). The pressure is in all cases fixed at 20 Torr, the inlet $NH_3$ mole fraction varies slightly from 0.0196 to 0.0192, and the value of $\rho$, as computed from the bulk density of TiN, is $3.61 \times 10^{-9}\ \mathrm{mol/cm^2}$.

The optimized fit to the rotating disk reactor data is shown in Figure 1. The root-mean-square relative deviation between the experimental and theoretical values is 6.19%, which is probably within the uncertainty in the data. The near-kink in the model curve is highly unusual but seems to be real, as opposed to simply a numerical artifact. On the initial, rising portion of the curve, the $TiCl_4$ concentration at the substrate is reduced so severely that the intrinsic deposition rate is no longer in the zero-order regime, and the deposition is limited by the supply of this reactant. On the remainder of the curve, depletion of $TiCl_4$ is no longer an issue, and the deposition rate falls slowly due to the effect of the gas-phase complexing reaction.

Since the experimental data set does not span a wide range of process conditions, it is perhaps not surprising that the data fitting procedure does not provide definite values for all of the unknown rate parameters. The optimum value of $\alpha$ is essentially infinite, which suggests that Ti deposition is so efficient that it rarely limits the overall process. Likewise, the quality of the data fit is extremely insensitive to the value of $\gamma$, as long as it is large enough that $\phi_n$ is generally much smaller than unity, yet small enough that $\phi_t$ is generally large. The optimized values for the remaining rate constants are less uncertain: $\beta = (6.86 \pm 0.30) \times 10^{19}\ \mathrm{cm^5/mol^2 s}$, $\lambda = (5.77 \pm 0.29) \times 10^{10}\ \mathrm{cm^2/mol \cdot s}$, $\delta = (5.13 \pm 0.21) \times 10^{26}\ \mathrm{cm^6/mol^3 s}$, $k = (2.59 \pm 0.53) \times 10^{18}\ \mathrm{cm^6/mol^2 s}$, and $E = (76.0 \pm 13.2)\mathrm{kcal/mol}$. The quoted uncertainties are arbitrarily chosen to be those perturbations that cause the quality of the data fit to be degraded from 6.19% to 7%; this is enough to give rise to a noticeable change. The value given for $\beta$ can be shown to correspond to a reactive sticking coefficient of 0.0332 for $NH_3$.

Figure 1. Optimized fit of deposition data from Sandia/MRC rotating disk reactor




The value of $E$ is unrealistically large and has the effect of essentially confining the complexing reaction to the immediate vicinity of the substrate. This suggests that the source of the negative reaction order in $TiCl_4$ may actually be a heterogeneous reaction after all.

As noted earlier, lack of information about some reactor parameters makes it impossible to perform a rigorous analysis of the experimental data available in the literature. However, if it is assumed that the experiments were carried out under differential conditions, i.e., in the absence of significant reactant depletion (as claimed in Reference 4, for example), then the analysis becomes feasible. The data in References 3 and 4 are a natural choice to use, since they refer to temperatures close to that used in the Sandia study (although the pressures are lower by two orders of magnitude). Rather than re-optimizing the rate constants, however, we choose to keep those already computed and simply *predict* the deposition rates for the "new" data sets. In order to provide a fair comparison, the experimental values are first adjusted to 903 K by using the activation energies quoted in the respective articles.

The results of this procedure are shown in Figures 2–4. It can be seen that the predicted deposition rates are always within a factor of two of the observed values and usually much closer than that; in fact, for the near-differential reactor experiments of Srinivas et al. (4), the deviation never exceeds 40%. Since this kind of treatment necessarily ignores the depletion effect of the complexing reaction, the observed negative reaction orders in $TiCl_4$ cannot be reproduced; the predicted order is essentially zero. Not surprisingly, this is a more serious problem for the experimental conditions of Buiting et al. (3). On the other hand, the observed trends in the deposition rate with $NH_3$ concentration are predicted quite well. This kind of agreement may be partly fortuitous if the reactor experiments were not truly differential, as suggested by the $TiCl_4$ results. Nevertheless, the overall success of the model gives considerable support to the proposed surface reaction mechanism.

Figure 2. Predicted vs. observed deposition rates (adjusted to 903 K) in experiments of Buiting

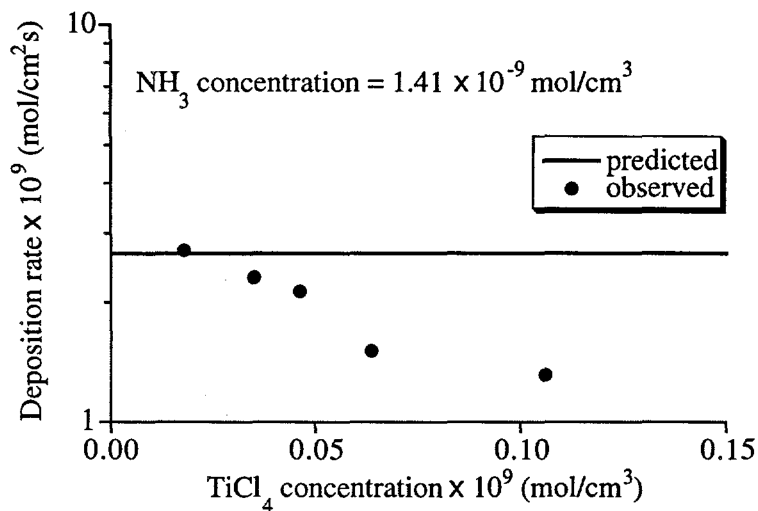



Figure 3. Predicted vs. observed deposition rates (adjusted to 903 K) in experiments of Srinivas

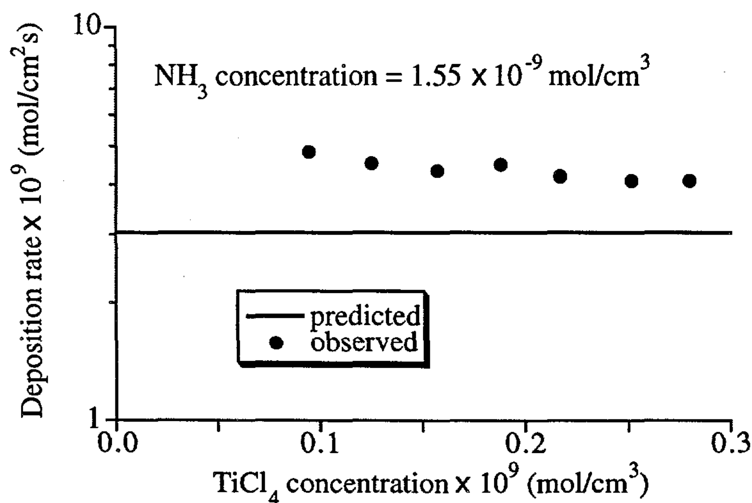
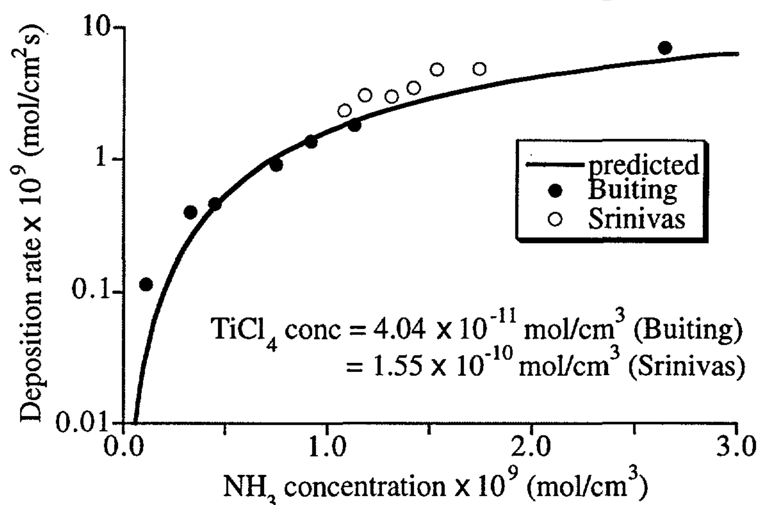


Figure 4. Predicted vs. observed deposition rates (adjusted to 903 K) in two sets of experiments

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