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A Model of the Gas-Phase Chemistry of Boron Nitride CVD from BCl_3 and NH_3

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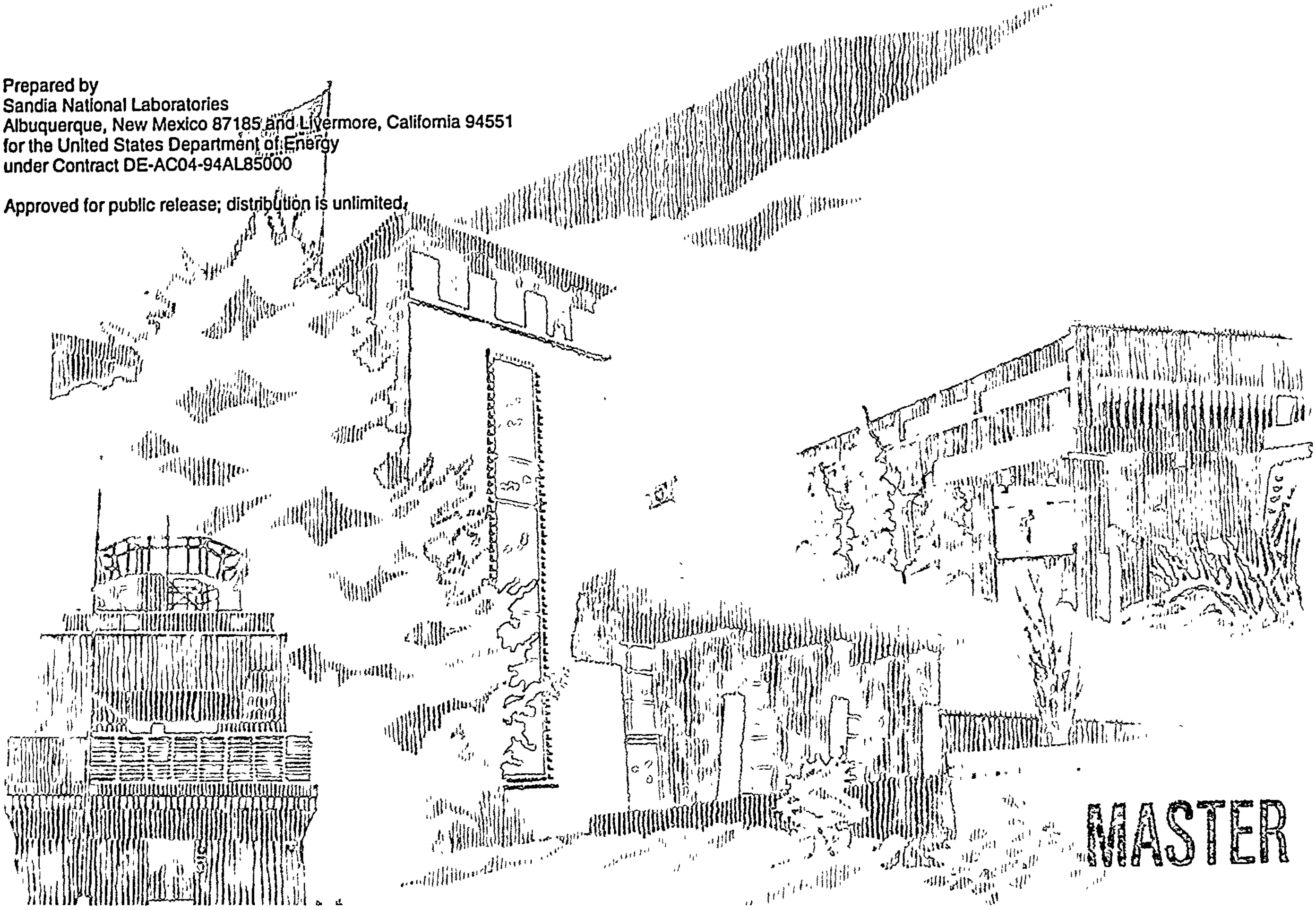

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A MODEL OF THE GAS-PHASE CHEMISTRY OF BORON NITRIDE CVD FROM BCl_3 AND NH_3*

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ABSTRACT

The kinetics of gas-phase reactions occurring during the CVD of boron nitride (BN) from BCl_3 and NH_3 are investigated using an elementary reaction mechanism whose rate constants were obtained from theoretical predictions and literature sources. Plug-flow calculations using this mechanism predict that unimolecular decomposition of BCl_3 is not significant under typical CVD conditions, but that some NH_3 decomposition may occur, especially for deposition occurring at atmospheric pressure. Reaction of BCl_3 with NH_3 is rapid under CVD conditions and yields species containing both boron and nitrogen. One of these compounds, Cl_2BNH_2, is predicted to be a key gas-phase precursor to BN.

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INTRODUCTION

Chemical vapor deposition (CVD) processes for depositing boron nitride (BN) have been of interest for some time for the production of structural ceramics and coatings [1-6]. Although a wide range of precursors have been used, boron trichloride (BCl_3) and ammonia (NH_3) are the ones typically used. Typically-used deposition conditions are 1073 - 1473 K and 1 - 150 torr, with or without a carrier gas consisting of either helium or hydrogen. Experimental studies have shown that these precursors react with each other in the gas phase at low temperatures (298-343 K) [7, 8]. In addition, mass-spectrometric measurements in a CVD reactor operating at 1373 K suggest that several gas-phase compounds containing boron and nitrogen are formed [3]. However, the identity of the gas-phase molecule(s) responsible for deposition is not clear; both BCl_3 [1, 2] and Cl_2BNH_2 [3] have been suggested.

Quantitative measurements of gas-phase reaction rates in this system are unavailable except for limited measurements of the reaction between BCl_3 and NH_3 performed at 343 K [8]. Heats of reaction for most of the potentially important gas-phase reactions are also unknown due to a lack of reliable thermochemistry. Theoretical studies have shown, however, that a donor-acceptor complex between BCl_3 and NH_3 can form at room temperature, with a B-N bond strength predicted between 21 and 36 kcal mol^{-1} [9, 10].

In this work we report a mechanism that describes the gas-phase reactions occurring during BN CVD. Rate constants for this mechanism were obtained from the literature or were estimated theoretically. To determine the importance of gas-phase reactions during BN CVD and to predict the distribution of species present under typical processing conditions, plug-flow calculations employing the mechanism were performed. The results show that BCl_3 and NH_3 are relatively stable thermally, but gas-phase reactions between the two precursors can cause the distribution of gas-phase species to vary widely depending upon the conditions used for deposition.

KINETIC MECHANISM AND THEORETICAL METHODS

The kinetic mechanism used here does not include all gas-phase species that could conceivably form since much of the needed kinetic data are unavailable. In particular, very few rate constants for reactions involving the BCl_2 and BCl radicals have been measured. Reactions of these molecules with hydrogen could, for example, form $BHCl_2$ under some deposition conditions, but this species is not included in the mechanism due to lack of kinetic data for the reactions that might produce it. Since the purpose of this investigation is to determine the importance of gas-phase chemical processes in various potential CVD operating regimes, we attempt to provide upper limits for the extent of gas-phase reaction by using upper limits for the rates of unknown reactions.

Selected reactions used in the mechanism are listed in Table I with the corresponding heats of reaction [9] and rate coefficients. Reactions 1 and 2 were not used in the simulations (see discussion below). Reactions that describe the decomposition of NH_3 [11], H_2 [12], and HCl [13], which are not listed in Table I with the exception of Reaction 11, were obtained from the literature. Rate constants for Reactions 3-8 are unknown and were estimated by applying high-pressure rate theory [14] or by comparisons with analogous reactions. Thermochemistry used here to determine bond dissociation energies (BDE), heats of reaction, and estimate reaction rate constants was obtained from *ab initio* calculations (performed at the level of fourth-order Møller-Plesset perturbation theory with empirical bond-additivity corrections, referred to here as the BAC-MP4 method) reported elsewhere [9]; these calculations are typically accurate within ± 3 kcal/mol. Plug-flow calculations were performed using the SENKIN program developed at Sandia [15].

The rates of unimolecular reactions shown in Table I (Reactions 1, 2, 4, 7, and 8) typically depend on pressure. Predicting this dependence requires knowledge of intermolecular energy transfer rates, which are difficult to determine accurately without experimental measurements of rate constants. However, unimolecular reaction rates in the high-pressure (pressure-independent) limit can be predicted by RRKM and other theoretical methods with reasonable accuracy. Thus, the rate constants used for Reactions 2-4, 7, and 8 are those predicted by RRKM or estimated for the high-pressure limit of these reactions, providing an upper limit for the rate.

Table I: Gas-phase mechanism for BN CVD: selected reactions. All energies are in kcal mol^{-1}.

	Reaction	ΔH°_{reac} [f]	A [h]	E_a	Notes
1.	$Cl_3BNH_3 \leftrightarrow BCl_3 + NH_3$	24.0	--	--	f
2.	$Cl_3BNH_3 \leftrightarrow Cl_2BNH_2 + HCl$	11.1	--	--	f
3.	$BCl_3 + NH_3 \leftrightarrow Cl_2BNH_2 + HCl$	-12.9	1.66E+12	19.9	a,b
4.	$Cl_2BNH_2 \leftrightarrow ClBNH + HCl$	56.0	3.89E+14	79.0	a,b,c
5.	$Cl_2BNH_2 + NH_3 \leftrightarrow BCl(NH_2)_2 + HCl$	-3.9	1.66E+12	19.9	a
6.	$BCl(NH_2)_2 + NH_3 \leftrightarrow B(NH_2)_3 + HCl$	6.9	1.66E+12	19.9	a
7.	$BCl_3 \leftrightarrow BCl_2 + Cl$	117.7	9.86E+15	113.7	a,c,d
8.	$BCl_2 \leftrightarrow Cl + BCl$	79.7	2.25E+15	76.9	a,c,d
9.	$BCl_3 + H \leftrightarrow BCl_2 + HCl$	14.5	1.00E+14	12.0	a
10.	$BCl + HCl \leftrightarrow BCl_2 + H$	23.4	7.23E+13	24.2	e
11.	$NH_3 + M \leftrightarrow NH_2 + H + M$	109.1	2.50E+16	93.8	g

[a] This work. [b] Transition state from Ref. [9]. [c] High-pressure limit. [d] Rate estimated by RRKM calculation. [e] Ref. [16]. [f] Ref. [9], kcal mol^{-1}, 298 K. [g] Ref. [11]. [h] Units: cm, sec, moles.

RESULTS

Gas-phase reactions can be initiated by either unimolecular decomposition of the reactants or by a bimolecular reaction between BCl_3 and NH_3, each of which we examine in this investigation. We consider each case separately, using plug-flow calculations under typical CVD conditions to predict the evolution of gas-phase species as a function of reactor residence time. In addition, bond dissociation energies (BDE) are used as a guide to reactivity.

Decomposition of BCl_3 appears to be unlikely under CVD conditions given the strength of the B-Cl bond. The BDE predicted by the BAC-MP4 calculations is 118 kcal mol^{-1}, indicating that very high temperatures are required for Reaction 7 to reach significant rates. This is supported by plug-flow calculations, using a mechanism consisting of Reactions 7-10 and ten other reactions describing H_2 and HCl decomposition. As discussed above, there are no experimental data available for Reactions 7 and 8, so high-pressure rate constants were estimated using RRKM methods. Thus, the extent of BCl_3 decomposition and concentrations of BCl_2 and BCl predicted by the mechanism are expected to be upper limits.

In an inert carrier gas such as helium or argon, virtually no BCl_3 decomposition is predicted to occur (< 5%) for a 1-second residence time at temperatures less than 1800 K (Figure 1, left). In a hydrogen diluent, decomposition is somewhat faster, but temperatures of 1400 K are still required to decompose more than 5% of initial BCl_3. Even at this temperature, unimolecular decomposition (Reaction 7) is not a factor; decompostion occurs by the reaction of BCl_3 with hydrogen atoms that form when the carrier gas dissociates. Thus, gas-phase decomposition of BCl_3 is of little importance except at very high temperatures, which are not typically used in BN CVD processes.

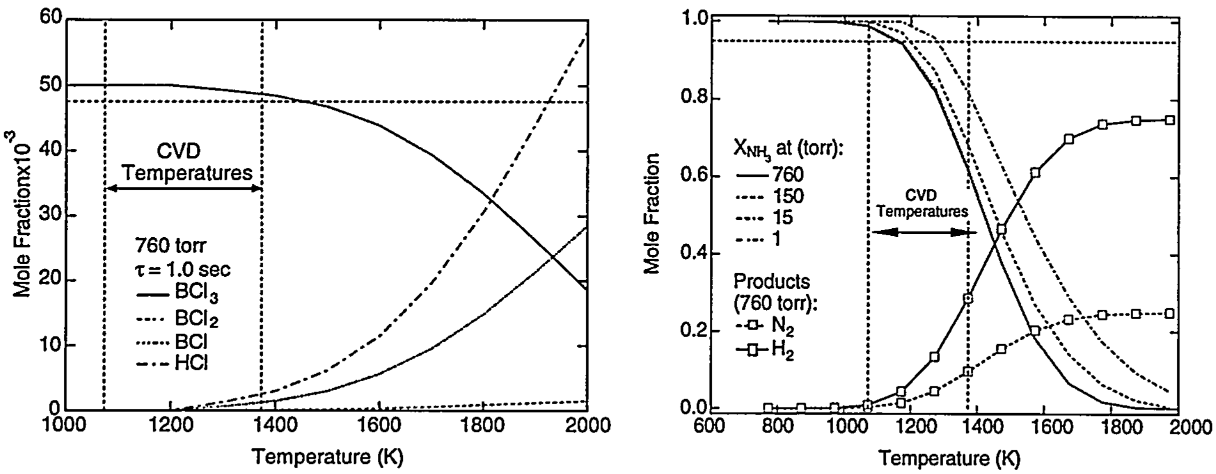

Figure 1: Plug-flow predictions of the decomposition of BCl_3 (left) and NH_3 (right) as a function of temperature. Residence time for all calculations is 1 second. Left: 5% BCl_3 in H_2, 760 torr. Right: 100% NH_3. Decomposition of NH_3 is shown as a function of reactor pressure.

In contrast, pyrolysis of NH_3 is more rapid under CVD conditions than decomposition of BCl_3. Such behavior is expected because the N-H bond (with a BDE of 109.1 kcal mol^{-1}) is weaker than the B-Cl bond. Since the high-temperature chemistry of NH_3 is far better understood than that of BCl_3, a detailed, 45-step reaction mechanism [11] including 11 species was used in the plug-flow calculations. Results of these calculations are shown in Figure 1 (right), where it is clear that NH_3 decomposition can be significant in the range of temperatures and pressures used for BN CVD. The unimolecular decomposition of NH_3 is in its bimolecular limit, meaning that the rate of Reaction 11 depends linearly on pressure. Thus, the amount of NH_3 decomposition is much smaller at 1 torr than at atmospheric pressure. Nevertheless, even at the lowest pressures reported for BN CVD (1 torr), as much as 18% of the NH_3 decomposes at a temperature of 1373 K, given a 1-second residence time. The principal products are N_2 and H_2, whose reactivity with the surface is expected to be considerably different than that of NH_3. This may be correlated with changes in microstructure that have been observed when deposition temperatures exceed 1523 K [5]. In addition, since NH_3 decomposition initially produces H atoms, BCl_3 decomposition could be accelerated by attack of these radicals on BCl_3 via Reaction 9. Clearly, the extent of NH_3

decomposition depends on the gas residence time and thus must be evaluated on a case-by-case basis. Nevertheless, these predictions indicate that models of the BN CVD process must account for the gas-phase decomposition of this reactant, especially since it is virtually always present in excess.

Unlike the unimolecular reactions of the BN precursors, which have high activation barriers, the bimolecular reactions between BCl_3 and NH_3 (Reactions -1 and 3) are exothermic, with relatively low activation barriers. These reactants constitute a Lewis acid-base pair, with BCl_3 acting as an electron acceptor and NH_3 as an electron donor. Such pairs are known to react readily with one another, although relatively limited data are available on the reactivity of the BCl_3/NH_3 pair. The energetics of these reactions and subsequent decomposition channels for the complex that initially forms were calculated using the BAC-MP4 method [9]. Though described in detail elsewhere, we present a brief discussion here of the relevant results.

Reaction (-1) between BCl_3 and NH_3 is exothermic by 24 kcal mol^{-1} and yields a stable complex, $Cl_3B{:}NH_3$; at room temperature. This complex can decompose by 1,2 elimination of HCl (Reaction 2) to form Cl_2BNH_2, but an activation barrier of 38 kcal mol^{-1} (at 298 K) must be surmounted. Little or no $Cl_3B{:}NH_3$ will form at CVD temperatures, however, because the energy distribution of the incoming reactants at CVD temperatures will allow most molecules to proceed over the barrier to form Cl_2BNH_2, rather than be collisionally quenched to form ground-state $Cl_3B{:}NH_3$. Thus, at the temperatures normally encountered during BN CVD, the process to form Cl_2BNH_2 is best described by Reaction 3. Loss of a second molecule of HCl to form ClBNH (Reaction 4) is inhibited by large barrier. However, Cl_2BNH_2 can react further with NH_3 to substitute additional amine groups for chlorine, yielding the Reactions 5 and 6 in Table I. Barriers for these reactions are expected to be similar to that predicted for Reaction 3. Substitution of the second amine group is energetically less favorable than the first (ΔH°_{reac} = -3.9 kcal mol^{-1} vs -12.9 kcal mol^{-1}), and substitution of the third amine group is predicted to be endothermic (ΔH°_{reac} = 6.9 kcal mol^{-1}) [9].

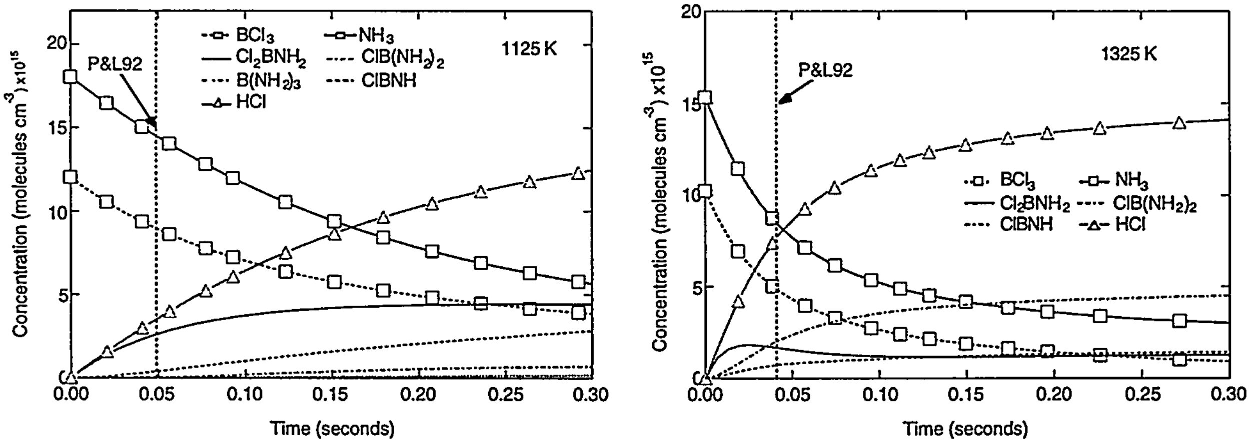

Figure 2: Plug-flow predictions of gas-phase concentrations at 1125 K and 1325 K. All calculations were performed for a total pressure of 2.0 torr and a 40% BCl_3, 60% NH_3 mixture.

Plug-flow calculations confirm that Reactions 3-6 are much faster at CVD temperatures than any of the pathways initiated by unimolecular decomposition of the inital reactants. This is shown in Figure 2, in which gas-phase concentrations are shown as a function of reactor residence time at 1125 K and 1325 K. Several key results are illustrated by Figure 2. First, it is clear that, even on these relatively short time scales, the reaction between BCl_3 and NH_3 is quite fast, with 68% of the BCl_3 reacted within 300 msec at 1125 K and >90% consumption at 1325 K. Second, a range of species containing both boron and nitrogen is present. At 1125 K, all three amine-substituted boranes ($Cl_nB(NH_2)_{3-n}$, n=0-3) are predicted to form, as well as small amounts of ClBNH (< 1%, which is consistent with the high activation barrier for Reaction 4). At 1325 K, much more ClBNH forms, with its concentration exceeding that of all other B-N-containing molecules for residence times greater than 160 msec. We reemphasize that the concentration of ClBNH is expected to be an upper limit that is attained only at very high pressures; at the pressures typically used for CVD, the concentration is likely to be lower. Finally, the relative product concentrations change dramatically with time, with the mixture dominated by BCl_3 at early times, then shifting to Cl_2BNH_2 and other species at later times. At 1325 K, Cl_2BNH_2 is only an intermediate that decomposes to form ClBNH at residence times greater than 25 msec.

The plug-flow predictions shown in Figure 2 can be correlated with deposition conditions reported by previous investigators. Accurate predictions, however, require knowledge of the time-temperature history of the gases in the reactor, which is often difficult to determine because of complex reactor geometries and mixing effects. A reasonable estimate of the residence time can be made in some cases by assuming that mixing and heat transfer are rapid compared with the time required for the gas to travel to the substrate, and axial diffusion is inconsequential. If the additional assumption is made that radial concentration gradients produced by wall reactions are negligible, then the plug-flow model can be applied. Such assumptions are most likely to be reasonable in cases where low reactor pressures are used and/or the gases are premixed prior to entering the reactor. Two studies of the BN deposition kinetics, by Patibandla and Luthra (P&L) [2] and Lee et al. [1], are good candidates for application of the plug-flow model. In the the P&L experiments, pressures between 1-3 torr were used, ensuring rapid mixing of the reactants, which were also preheated prior to mixing. We estimate that the residence time ranged between 40 and 50 msec in these experiments. In the experiments of Lee et al., somewhat higher pressures were used (40 torr), but the reactants were mixed prior to entering the reactor. Because of the more complex reactor geometry used in this case, however, we can only estimate an upper limit (5 msec) for the residence time. The predictions shown in Figure 2 correspond to the pressures used in the study by P&L and cover the range of temperatures they accessed (1125-1325 K), while Figure 3 shows results for the conditions used by Lee et al. In each figure, the dashed line indicates the estimated experimental residence time.

Figure 2 suggests that the composition of the gas-phase varies significantly over the range of temperatures used by P&L. At the lower end of their temperature range (1125 K), 75% of the original BCl_3 remains when the gases reach the substrate. The boron-containing species in highest concentration is BCl_3, with a significant amount of Cl_2BNH_2 and $ClB(NH_2)_2$ also present. At the highest temperature used by P&L (1325 K), less than half of the BCl_3 remains unreacted, with roughly equal amounts of ClBNH, Cl_2BNH_2, and $ClB(NH_2)_2$ present. Thus, the calculations suggest that the identity (and potentially the reactivity) of the molecules interacting with the surface varies substantially as a function of temperature.

In contrast, the much shorter residence times achieved in the experiments by Lee et al. provide little opportunity for the precursors to react prior to reaching the substrate. As can be seen in Figure 3, even with an upper limit of 5 msec for the residence time, less than 7% of the BCl_3 reacts at the highest temperature (1275 K) used in these experiments. At the lowest temperature used to determine the deposition kinetics (1075 K), essentially no gas-phase reaction occurs. This is consistent with the analysis by Lee et al., who concluded, based on deposit morphology, that gas-phase reactions were insignificant.

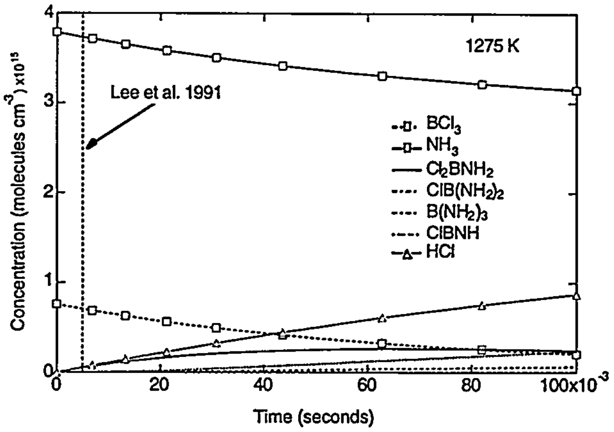

Figure 3: Gas-phase concentrations predicted by the plug-flow model for the conditions used in Ref. [1].

Thus, in one case (P&L), gas-phase reactions appear to be very important and may affect deposition rate and microstructure, while in the other case (Lee et al.), they should have little or no influence. This is surprising, since the two studies obtained very similar deposition rates. In fact, the activation barriers measured by the two groups differ by only 4 kcal mol^{-1}. One hypothesis explaining this similiarity is that deposition is limited by a surface reaction involving a common intermediate. The intermediate is created by the adsorption of gas-phase species and its decomposition to form BN is the rate-limiting step. For example, the intermediate Cl_2BNH_2(surf) could be formed by Reactions 3 and 12, or by Reactions 13 and 14, with BN formation occurring by Reaction 15:

$$Cl_2BNH_2 \rightarrow Cl_2BNH_2(surf) \qquad (12)$$

$$BCl_3 \rightarrow BCl_3(surf) \qquad (13)$$

$$BCl_3(surf) + NH_3 \rightarrow Cl_2BNH_2(surf) + HCl \qquad (14)$$

$$Cl_2BNH_2(surf) \rightarrow BN(bulk) + 2HCl \qquad (15)$$

Alternatively, the surface reactivity of the various gas-phase species may be sufficiently similar that no variation in mechanism is observed as a function of temperature, even though the gas-phase composition varies considerably. In either case, the mechanism must be consistent with the observation [1, 2] that BN deposition is first-order in the BCl_3 concentration and independent of the NH_3 concentration. Since NH_3 was present in excess in the experiments of both P&L and Lee et al., the gas-phase reactions should be pseudo-first order. The deposition rate derived from Reactions 13-15 is also independent of the NH_3 concentration, if the concentrations of BCl_3(surf) and Cl_2BNH_2(surf) are at steady-state (note that Reactions 13-15 are not unique; other mechanisms can be imagined that give the same reaction orders). Thus, the two hypotheses appear to be equally plausible.

CONCLUSIONS

We conclude that gas-phase chemical reactions occur at significant rates on the timescales and at the temperatures and pressures employed in BN CVD. Except at the shortest residence times, these reactions produce a mixture of species that contain both boron and nitrogen. The relative efficiency with which these species deposit BN is unknown, although the similarity of the BN deposition kinetics obtained by various research groups suggests that there may not be great differences. Alternatively, a rate-limiting surface reaction proceeding from a common (surface) intermediate can account for the observed kinetics. Additional experiments that correlate gas-phase concentrations with deposition rates must be performed to identify the correct mechanism.

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