

Real-Time Evaluation of Aluminum Borohydride Trimethylamine for Aluminum Chemical Vapor Deposition

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The chemical species in gas phase and on the surface of aluminum borohydride trimethylamine (ABHTMA) for aluminum chemical vapor deposition as a function of the hot-wall temperature and the chamber pressure were studied using two kinds of Fourier transform IR spectroscopes installed at the end of the chamber. The absorbance of Al–H, B–H, C–H, and C–N stretching features of ligands in ABHTMA in the gas phase and on the surface was sensitive to the variation of analysis conditions. The area ratio of integrated absorbance of Al–H and B–H stretching features located at the different position could estimate the dissociation rate of the ABHTMA, which was abruptly changed in the range of 140-160 °C. With these results, the temperature dependence of the film composition and quality could be explained. Additionally, the stabilities of the chemical species were investigated using density functional theory calculations. The ABHTMA was found to be the most stable molecule when trimethylamine was rich and borane and alane were close in their concentrations. Borane-trimethylamine was also found to be produced in alane-poor conditions as a by-product.

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Aluminum (Al) has been widely used as interconnect material in the fabrication of integrated circuit devices.^{1,2} Physical vapor deposition (PVD) techniques are often used to deposit Al films. However, successful Al interconnection becomes extremely difficult, because the size of the vias is decreasing and the aspect ratio is increasing. So, the chemical vapor position (CVD) of Al has attracted considerable attention as an alternative PVD metallization process. The Al CVD process must have a suitable metallorganic precursor; that is, a new precursor has to be designed for deposition. A variety of metallorganic precursors for Al CVD have been developed over the past two decades. Trimethylaluminum (TMA) and triisobutylaluminum were among the early Al precursors and were abandoned due to impurities like carbon incorporated to films, low deposition rate, and low reflective index. Dimethylethylamine (DMEAA), methylpyrrolidine alane (MPA),³ and trimethylamine alane (TMAA) including the amine-alane adduct have been found, and these are expected to yield high-purity (carbon-free) films due to no direct Al-C bonds in their molecular structures. However, DMEAA and MPA have low stability, and TMAA is a solid precursor. Normally, the solid precursor makes it difficult to control the vapor flow in terms of maintaining a constant surface area in the source.

Aluminum borohydride trimethylamine (ABHTMA, 99.999%)⁴ was recently developed for Al CVD. The ABHTMA was synthesized by the reaction among four kinds of molecules [LiAlH₄, AlCl₃, N(CH₃)₃, and NaBH₄], and the reaction equation is below

$LiAlH_4 + AlCl_3 + 2N(CH_3)_3 + 2NaBH_4 \rightarrow 2BH_3AlH_3:N(CH_3)_3$

ABHTMA is a clear, colorless liquid at room temperature, has no direct Al–C bond, and has a high vapor pressure (68 Pa at 30°C). The vapor pressure was measured by the homemade apparatus installed with a pressure gauge calibrated by international pressure standard system in Korea Research Institute of Standards and Science.

In order to study a newly developed precursor for device manufacturing, investigating chemical species under various conditions is important. Researchers in this field have been interested in the Fourier transform IR (FT-IR) system because it can observe accurately the concentration of chemical species in real time, is noninvasive to the process chamber, and has low maintenance costs.

More precursors have many bonds and vibrations which can be conjugated, leading to IR absorptions at characteristic frequencies that may be related to chemical groups (called ligands). If the metallorganic precursor is decomposed or reacted during the process, the stretching vibrations of ligands such as CH₃, N–(CH₃)₃ of a metallorganic precursor can be changed. With the spectrum measured by an FT-IR spectroscope, the reaction mechanism can be investigated. Using FT-IR spectroscopy, the real-time studies of the gas-phase species and adsorbed species on the surface have been reported for Cu deposition from hexafluoroacetylacetonate (hfac) Cu(vinyltrimethylsilane),^{5,6} Cu(bis-dipivaloylmethanato)₂⁷ and Cu(hfac)₂,^{8,9} Al deposition from the TMMA-NH₃ system,¹⁰ AlGaAs deposition from the tratethylorthosilicate–ozone system,¹² and diamond from a CH₄–H₂–O₂ gas mixture.¹³ In situ real-time FT-IR spectroscopy is a useful technique for monitoring the process and diagnosing a mechanism. Until now, ABHTMA has not been evaluated and reported with an FT-IR spectroscope.

In this work, we studied the behavior of ABHTMA at the gas phase and on the surface as a function of the temperature and the pressure of a hot-wall reactor with modified FT-IR spectroscopy after dosing ABHTMA for CVD. We also performed densityfunction-theory calculations to show the stable species and byproducts during the Al CVD process. ABHTMA was found to be a stable precursor for the Al CVD in the trimethylamine-rich and the near-stoichiometric condition of alane and borane. By the dissociation of ABHTMA and the recombination of the molecules dissociated, the borane-trimethylamine complex could be produced.

Experimental

The FT-IR system was installed at the end of the hot-wall reactor chamber to investigate the gas species coming out of the chamber during Al deposition (Fig. 1) with varying deposition temperatures and chamber pressures. The modified gas cell and the tube of the FT-IR system was heated to 40°C in order to avoid contamination of the gas cell, potassium bromide (KBr) windows, and pumping lines. A collimated IR beam from the FT-IR spectrometer (Nicolet Avata 360) was passed through the gas cell with two KBr windows, and a liquid-nitrogen-cooled Hg-Cd-Te detector was used. The IR band under examination covered wavenumbers ranging from 400 to 4000 cm⁻¹, and each IR spectrum was collected at 2 cm⁻¹ resolution using 62 scans to allow a good signal-to-noise ratio. The gas-cell pressure was controlled by the throttle valve. We used another FT-IR system called a transmission FT-IR spectroscope to obtain the information on the species adsorbed on the surface. This system requires high-surface-area particles. The ZrO₂ nanoparticles were obtained from Nanomaterials Research Corporation (Longmont, CO) and were spherical with an average diameter of 50 nm.

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Figure 1. (Color) Schematic of diagram of diagnosis system.

The ZrO_2 nanoparticles were supported by a photoetched tungsten grid. Before taking a spectrum of adsorbed species, the Al film was deposited on the pressed ZrO_2 nanoparticles for 10 min at 150°C with ABHTMA because of changing ZrO_2 surface to Al metal surface. The transmission FT-IR spectroscope system has been reported in previous studies.¹⁴⁻¹⁶ The ABHTMA was evaporated from a stainless steel bubbler maintained at an appropriate temperature of 25°C and transported to the reactor by Ar carrier gas at a rate of 20 sccm. The temperature of the feeding lines between the chamber and bubbler is 40°C, which is the same as the exhaust-line temperature.

We also performed density-functional theory calculations with ultrasoft pseudopotentials,¹⁷ as implemented in the Vienna Ab Initio Simulation Package code.¹⁸ A plane-wave cutoff of 400 eV and a cubic supercell containing a molecule with a volume of $10 \times 10 \times 10 \text{ Å}^3$ were used. The generalized gradient approximation for the exchange-correlation energy was used.¹⁹

Results and Discussion

First, we investigated the stabilities of molecules composed of the alane, borane, and trimethylamine molecular components. The molecular structures of ABHTMA are shown in Fig. 2. The calculated formation energies for various molecules are plotted in Fig. 3 as a function of the alane chemical potential $[\mu(AIH_3)]$. The high μ (AlH₃) represents the alane-rich condition in the chamber, while the low $\mu(AlH_3)$ is the borane-rich condition. When the trimethylamine concentration is rich (Fig. 3) and the concentrations of alane and borane are similar, the ABHTMA molecule is found to be the most stable among those composed of the alane, borone, and trimethylamine molecules. Thus, in the rich condition of the trimethylamine and the near-stoichiometric condition of the alane and borane, the ABHTMA can be a stable precursor. As the concentration of the alane component becomes reduced, the most stable species becomes the borane-trimethylamine complex, as shown in Fig. 3. The ABHTMA precursors used can also be degraded by the formation of the borane-trimethylamine complex. The schematic diagram about the chemical reaction in the gas phase and on the surface is shown in Fig. 4 to facilitate understanding.

Figure 5 shows the measured IR absorption spectrum for the chemical species coming out of the reactor at 40° C wall temperature. The stretching features of Al–H (1844, 1793, and 1007 cm⁻¹), Al–N (746 cm⁻¹), B–H (2485, 2443, and 2169 cm⁻¹), C–N (2828 and 2775 cm⁻¹), and C–H (2927, 2988, and 1484 cm⁻¹) from ABHTMA are clearly detected. Those peaks have been identified from similar molecules elsewhere.^{20,21} In this spectrum, we found the other B–H peaks (2394, 2382, and 2374 cm⁻¹) related to the borane-trimethylamine complex, which can be generated during the dissociation and recombination of ABHTMA passing the hot-wall reactor or contained little as the impurity of the synthesized

ABHTMA. The assignment of the borane-trimethylamine complex lines was confirmed by the IR spectrum library supported by the Nicolet Avata software.

Figure 6 shows the change of the C–H, C–N, and Al–H stretching features at the reactor pressure of 0.95 Torr as a function of the reactor wall temperature. The C–H stretching features located in the range of 1420–1520 cm⁻¹ (Fig. 6a) and 2850–3100 cm⁻¹ (Fig. 6b) gradually decreased with the increase of the reactor wall temperature. However, the C–H stretching feature at 2964 cm⁻¹ (CH₂ asymmetric stretch) increased with increasing the temperature over 175°C due to the decomposition of CH₃ groups at a trimethylamine ligand in ABHTMA. The C–H stretching vibration is normally strong and the intensity is higher. Thus, the C–H stretching features were used to investigate the decomposition of the precursor. For example, the β-hydride elimination between neighboring methyl



Figure 2. (Color) The calculated molecular structures of (a) boranetrimethylamine complex and (b) ABHTMA. The red, gray, and black balls are N, C, and H atoms in the ABHTMA. The dark green is Al in the alane component, and the bright green is B in the borane component.



Figure 3. (Color) The calculated formation energies of various molecules composed of alane, borane, and trimethylamine are plotted as a function of alane chemical potential $[\mu(AIH_3)]$. The trimethylamine-rich condition is considered, in which the ABHTMA molecule is stable.



Figure 5. The IR absorption spectrum of molecules coming out of the hot-wall reactor at 40° C.

groups on a trimethylamine ligand creates variation in stretching vibration of C–H features. Also, normally, the lowering of the intensity of CH₃ groups or the shift of peak location from CH₃ to CH₂ is caused by the precursor decomposition.²²

In the case of the C–N stretching features, the intensities of peaks at 2834 and 2775 cm⁻¹ increased with the increase in temperature up to 200°C. To investigate the reason, we have taken the IR spectrum of the similar molecules, DMEAA (Fig. 7a) and trimethylamine (Fig. 7b). In the case of the two molecules, the intensity of the C–N stretch mode is similar to that of the C–H stretch mode. However, after the trimethylamine was bonded with the alane-borane complex, the intensity of the C–N stretching feature of ABHTMA shown in Fig. 5 is much lower than that of the C–H. It is believed that the bonding of borane makes the intensity of the C–N stretching features in the range of 2750–2850 cm⁻¹ lower. So, the bond breaking between alane and borane by thermal energy would increase the intensity of the C–N stretching features. This can be identified in Fig. 6b.

Figure 6c shows the variation of peak intensity [1793 (peak A) and 1844 cm⁻¹ (peak B)] and generation of the peaks (1892 and 1882 cm⁻¹) related to Al–H stretching features over the reactor wall temperature of 150°C. This means that thermal energy from the hot



Figure 4. Schematic diagram of the chemical reaction in the gas phase and on the surface.



Figure 6. FT-IR spectra of (a and b) C–H, (b) C–N, and (c) Al–H stretching features as a function of the hot-reactor-wall temperature.

wall has an effect on the dissociation and decomposition of ABHTMA and the Al–H stretching features are sensitive to the transmission of thermal energy controlled by the wall temperature and the reactor pressure. Peak A only gives information about the Al–H vibrational feature of alane bonded with borane and trimethylamine. Peak B gives mixed information about Al–H features of alane itself and oligomerized forms of alane. And, the newly gener-



Figure 7. The IR absorption spectrum of (a) DMEAA and (b) trimethyl-amine.

ated two peaks only give information about oligomerized forms of alane. The alane dissociated from ABHTMA is easily oligomerized due to the high degree of intermolecular association of Al-H molecules.²³ When observing the two main peaks related to an Al-H vibrational feature, peak A was decreased and peak B was fluctuated as the deposition temperature was increased. Peak B showed a decline up to 140°C. However, the intensity at 150°C got higher than that observed at 140°C. In the temperature range of 160-200°C, the intensity was decreased again. Around 150°C, the abnormal behavior of peak B was observed, because this peak is correlated with the generated Al-H stretching feature. We found that new peaks at 1882 and 1892 cm⁻¹ started to be generated over 150°C, and then the intensity of peak B was increased. Thus, it is believed that the newly generated peaks are related to the oligomerized forms of Al-H molecules dissociated.²⁴ The intensity of the new peaks is much smaller than that of peaks A and B. This means that the amount of oligomerized forms of alane is small.

Figure 8 shows the A-H peak variation as a function of the pressure at a fixed temperature. At 125°C, both peaks (A and B) increased with the increase in reactor pressure. It means the additional decomposition of ABHTMA is not happening in the gas phase at temperatures as low as 125°C. In the temperature range of 140-200°C, peaks A and B decreased gradually with increasing pressure due to the decomposition of ABHTMA. However, the decrease rates of peaks A and B were different. The decrease rate of peak A was faster than that of peak B, because the mixed information about various Al-H stretching features is shown together at the position of peak B. The intensity of peaks generated at 1882 and 1892 cm⁻¹ in the range of 150-160°C was increased with the increase in reactor pressure. However, in the range of 175-200°C, the peaks fluctuated with the pressure range. The peak increased at the pressure of 0.65-0.95 Torr but decreased at the pressure of 0.95-2.5 Torr. This means that oligomerized forms are sensitive to the pressure and the reactor temperature.

It is believed that the Al-H stretching peaks give us valuable information on the reaction mechanism in the gas phase. With the area ratio of integrated absorbance of peaks A and B at various temperatures and pressures, the degree of dissociation of ABHTMA can be known. The variation of the area ratio of Al-H stretching features as a function of the reactor pressure is shown in Fig. 9. The initial value of the area ratio (peak B/peak A) is 0.36, and the ratio does not change because additional decomposition of ABHTMA does not happen under 125°C at the full range of the reactor pressure. However, at 140°C, the ratio is gradually increased over 0.95 Torr. This means that the decrease rate of peak A was faster than that of peak B and the dissociation of ABHTMA took place fast over 0.95 Torr. At 150°C, the increase rate of the ratio over 0.7 Torr got faster. And, the rate increased rapidly even at pressure as low as 0.45 Torr at 160°C. With these results, we can see that the variation of Al-H stretching features are sensitive and reactive to the analysis conditions, especially in the range of 150-160°C.

Figure 10 shows the B-H stretching features as a function of the pressure at the fixed temperature. At temperatures as low as 125°C, the pressure increase resulted in an increase in absorbance for B-H of ABHTMA (peak C) and borane-trimethylamine (peak D). The increase rate of the integrated absorbance ratio raised slightly with the increase of pressure as shown in Fig. 11. This is not due to additional decomposition with increasing pressure but to the additional recombination between borane and trimethylamine. If additional decomposition of ABHTMA was happening, the intensity of the Al-H stretching features shown in Fig. 8 should be decreased together. At 140°C, peak C was decreased by lowering the ABHTMA concentration. Meanwhile, peak D was increased rapidly by the recombination of borane and trimethylamine. In the case of B-H stretching features observed at 175°C, the peaks related to a borane-trimethylamine complex were only observed in the full pressure range. Over 175°C even at low pressure, the ABHTMA was fully dissociated and recombined. So, the Al-H and B-H stretching features of ABHTMA located at the original position disappeared







Figure 9. Area ratio of integrated absorbance of Al–H stretching features (peak B at 1844 cm^{-1} /peak A at 1799 cm^{-1}).

and then were generated at the new position, which is related to oligomerized forms of an alane molecule and a boranetrimethylamine complex, respectively.

Figure 11 shows the variation of the area ratio of integrated absorbance of peaks C and D as a function of the reactor pressure. The ratio was changed abruptly over 140°C, and the increase rate was faster over 0.95 Torr. Over 160°C, the ratio increased rapidly at pressures even as low as 0.65 Torr. This means that peak D is more sensitive to the change of ambient conditions than peak C. And, the peak is valuable to monitor the deposition process and the status of the precursor, because the ratio represents the decomposition rate of ABHTMA or the recombination rate of the borane and trimethylamine molecules, and the borane-trimethylamine complex (peak C) is the by-product (or the complex) generated after decomposition or dissociation of ABHTMA.

Figure 12 shows FT-IR spectra of ABHTMA adsorbed on the sample coated with Al metal deposited by CVD as a function of the temperature and dosing time. At temperatures as low as 80°C (Fig. 12a), ABHTMA dosed to the grid was stacked on the surface. Though the intensity of the peaks increased with dosing time, the peaks disappeared after 12 min without dosing. This is because most of the ABHTMA molecules were physisorbed on the Al metal surface at temperatures as low as 80°C. We also observed that the

H337



Figure 10. IR spectra of B–H stretching features as a function of the pressure at a fixed temperature.

absorbance peaks of physisorbed ABHTMA were similar to ABHTMA in the gas phase, as shown in Fig. 13. Thus, the physisorbed ABHTMA was purged, and the peaks disappeared as time passed. Figure 13b and c shows the IR spectra of ABHTMA adsorbed on the surface at 80°C and ABHTMA taken in gas cell at 80°C, respectively. The peak positions of Al–H, C–H, and C–N



Figure 11. Area ratio of integrated absorbance of B–H stretching features (peak D/peak C).

stretching features were observed at a similar position when compared with two spectra (Fig. 13b and c). Especially, the B-H stretching features were shifted to a lower frequency, and the ratio of intensity of B-H and Al-H stretching features was bigger than the ratio in Fig. 13c. We believe that it is on account of the interaction among the borane-trimethylamine, of which the interaction between Al metal surface and the adsorbed molecule could be causing the change in frequencies and intensities. When ABHTMA was dosed at 125°C, the Al-H stretching features in the range of 1795–2000 cm⁻¹ and C–H stretching features in the range of 1400–1500 cm⁻¹ were observed. The Al–H and C–H stretching features remained on the surface due to the incomplete reaction of the dosed ABHTMA, which represent the source of the carbon contamination incorporated into the films. Meanwhile, the peak related to B-H was not observed. This is because of the high vapor pressure (0.8 Torr at 23°C) and high stability of borane complex (easy de-The decomposition characteristics of sorption). boranetrimethylamine were already reported. The borane-trimethylamine complex was used to deposit the boron-carbonitride films in the temperature range of 500-700 °C.²⁵ When the ABHTMA was dosed at 150°C (Fig. 12c) the peaks were not observed. This means that all kinds of molecules were adsorbed and then all kinds of by-products were desorbed at this temperature, which affected the film quality. The film deposited in this region (around 150°C) has the best film properties with respect to surface roughness, reflectance, and resistance.⁴ It is good evidence that there is a strong relationship between deposition characteristics and the inspection result of molecules adsorbed and desorbed.

Conclusion

The study on the behavior diagnosis of ABHTMA in the gas phase and on the surface was successfully carried out using a modified FT-IR spectroscope. The alane-borane-trimethylamine complex was found to be the most stable when the trimethylamine concentration was high and the alane and borane concentrations were similar to each other. By reducing the alane concentrations, the boranetrimethylamine complex could be generated as a by-product, as observed in the FT-IR measurements. It was observed that the C-H, C-N, Al-H, and B-H stretching features were sensitive to change in measurement conditions such as wall temperature and pressure. The intensity of most C-H stretching features was decreased with the increase in wall temperature due to the partial decomposition of ligands. The intensity of C-N stretching features showed different behavior with temperature change compared to that of C-H features. The intensity of C-N peaks was increased with the increase in temperature by the dissociation of borane. Especially, the B-H and



Figure 12. IR spectra of ABHTMA adsorbed on the Al metal surface at the surface temperature of (a) 80, (b) 125, and (c) 150°C.

Al-H stretching features gave information about the status of ABHTMA. The Al-H stretching features were fluctuated, and the new peaks related to the oligomerized forms of alane molecules were generated over 150°C. In the case of the B-H stretching features, the peaks showed the shift of the peak position by the reaction of borane with trimethylamine after consuming the alane of ABHTMA in the hot-wall reactor. From the variation of the Al-H and B-H stretching features, we found that the reaction behavior of ABHTMA was abruptly changed in the range of 140-160°C. The surface chemistry of Al CVD on the Al metal surface with the transmission FT-IR spectroscope was investigated. The species phys-



Figure 13. Comparison of spectrum taken (a) at the surface temperature of 125°C, (b) at the surface temperature of 80°C, and (c) at the gas-cell temperature of 80°C.

isorbed at temperatures as low as 80°C was almost purged from the surface, so the deposition did not happen. The species adsorbed at 125°C was chemisorbed and, by surface reaction the Al film was deposited. But, the film purity was not good due to the incorporation of carbon by the imperfect desorption of the molecules or byproducts adsorbed on the surface. We also observed the C-H stretching feature around 1500 cm⁻¹ acting as a carbon source. At temperatures as high as 150°C, any stretching features were not observed.

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