

Trimethylamine Complexes of Alane as Precursors for the Low-Pressure Chemical Vapor Deposition of Aluminum

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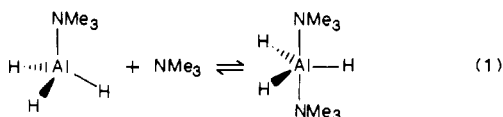
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Aluminum films were deposited in a hot-wall, low-pressure chemical vapor deposition reactor at temperatures as low as 100 °C by using $(\text{Me}_3\text{N})_2\text{AlH}_3$. Typical growth conditions were 180 °C substrate temperature and 25 °C for the precursor temperature. No carrier gas was used in any of the depositions, and the deposition rate on Si(100) under the above conditions was 0.9 $\mu\text{m}/\text{min}$. Changing the substrate temperature to 280 °C resulted in an increase in the deposition rate to 3 $\mu\text{m}/\text{min}$. Substrate materials included glass, polyimide films, and Si wafers. The surface morphology of the films was rough under most conditions. Pretreating the surface with TiCl_4 allowed smoother films to be deposited at lower temperatures. At 100 °C mirrorlike films were grown that exhibited a high degree of crystallite orientation. The (111) layers were found parallel to the surface. Resistivities of the as-deposited films ranged from 2.8 to 4.5 $\mu\Omega\text{ cm}$. A discussion of the deposition chemistry and a comparison to depositions using triisobutylaluminum are presented.

Introduction

Organoaluminum precursors, especially triisobutylaluminum (TIBA), have received a great deal of attention for their ability to generate high-quality Al films.¹⁻⁷ Typical deposition conditions for the low-pressure chemical vapor deposition (LPCVD) of Al on Si or other substrates using TIBA include substrate temperatures of 260 °C, TIBA temperature of 45 °C, and argon as the carrier gas at pressures up to 1 Torr. These conditions result in deposition rates of up to 0.15 $\mu\text{m}/\text{min}$.⁴ The films produced by such methods have low resistivities (2.8–3.5 $\mu\Omega\text{ cm}$) and other properties that compare well with Al films prepared by evaporation.⁶ A disadvantage observed in the use of TIBA is the rough surface morphology, which leads to poor reflective properties.¹ Particularly on nonmetallic surfaces such as silicon, improved Al film uniformity was obtained by pretreating the surface with TiCl_4 ,^{1,4-6} which is believed to provide additional nucleation sites. Recent success using a preheating treatment of the TIBA has achieved excellent reflectivities (90%) as well as epitaxial Al(111) growth on Si(111).⁷ Elegant studies of the surface reactions have led to a very thorough understanding of the mechanism of film growth.⁸ The rate-determining step in this chemistry involves the β -hydrogen elimination of isobutene from a surface-coordinated isobutyl group. This creates an Al-H bond which rapidly dissociates from the surface as H_2 .

A series of stable, volatile donor-acceptor complexes of alane (AlH_3) have been known for many years.⁹ They can be readily synthesized in one step from LiAlH_4 ,^{9,10} and they are less air-sensitive than the trialkylaluminums. The structures of these compounds are shown in eq 1, and among the known donors are Me_3N , Et_3N , Me_3P , Me_2S , and THF.⁹ Trimethylamine is unique among these donors in its ability to form a complex with two donors (eq 1).



As these complexes contain no Al-C bonds, the above studies⁸ on TIBA would suggest they might eliminate H_2 at lower temperatures to form Al films (assuming the relatively weak donor-acceptor bond is also readily

cleaved). In fact, from the early studies describing these compounds, it was known that they decomposed to form Al at temperatures >100 °C.⁹ Patents describing the use of amine-alane complexes for the vapor phase^{11,12} and electroless solution Al plating¹³ have appeared, as has a recent report of the laser-induced deposition of Al using these compounds.¹⁴ This paper describes details of using $(\text{Me}_3\text{N})_2\text{AlH}_3$ for thermally depositing high-purity aluminum films in a LPCVD reactor.

Experimental Section

General Techniques. The synthesis of $(\text{Me}_3\text{N})_2\text{AlH}_3$ was accomplished using literature procedures.^{9,10} X-ray diffraction studies were conducted by using a Siemens D500 diffractometer with monochromatic (graphite) $\text{Cu K}\alpha$ radiation. Film thicknesses were measured by stylus profilometry (Tencor Alphastep), and resistivities were measured by using a Veeco FPP-5000 four-point probe. Scanning electron micrographs were obtained on a JEOL 840 II, and Auger electron spectra were measured on a Perkin-Elmer Corp./Physical Electronics Division Model 555 electron spectrometer. Reflectivity measurements were made on a Perkin-Elmer Lambda 9 UV-vis-NIR spectrophotometer.

Reactor Configuration and Procedures Employed for Thin-Film Depositions. The reactor employed in the low-pressure chemical vapor deposition of Al was an all-glass system equipped with an oil diffusion pump capable of base pressures of 3×10^{-5} Torr. The reaction tube itself was made of quartz and had an inside diameter of 2.6 cm. The temperature of the tube furnace was monitored by thermocouples placed above and below the quartz tube at 4.5 cm (exactly in the middle of the region where the substrates were always placed) from the edge of the heating

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Table I. Typical Deposition Conditions and Results

$T(\text{precursor}),$ °C	$T(\text{reactor}),$ °C	TiCl ₄	press., ^a Torr	growth rate, μm/min	resistivity, μΩ cm	grain size, μm
25	280	no	0.210	2.9	3.9	4.5 ^b
25	180	no	0.194	0.8	3.3	2.5 ^c
25	180	yes	0.190	1.0	4.5	2.0 ^b
25	100	yes	0.102	0.05	3.4	0.15 ^c
25	180	yes	0.237	1.0	3.2	2.7 ^b
25	180	yes	0.184	0.6	3.8	1.5 ^b

^a Measured as described in the text after 2 min of deposition. ^b Measured by using optical microscopy. ^c Measured by using SEM.

coils (on the precursor entry side of the tube). The substrate temperatures in the tube were not monitored during a deposition; the numbers quoted as the deposition temperatures were obtained by a separate calibration of the internal temperature against the external thermocouple readings.

Si(100) wafers were degreased/etched by immersion in each of the following baths for 10 min each: tetrachloroethylene, ethanol, deionized water, dilute HF, and deionized water. Glass microscope slides and strips of polyimide film (Du Pont Kapton Type H) were cut to fit in the tube and were treated in the same fashion as the silicon wafers with the exclusion of the HF etching treatment. The substrates were placed in the tube, which was evacuated and heated to the reaction temperature for a minimum of 1 h. In depositions that included a pretreatment of TiCl₄ the entrance to the quartz tube was fitted with a Y-joint leading to a vessel containing the TiCl₄ and another containing (Me₃N)₂AlH₃. The TiCl₄ containing vessel was cooled to 10.0 °C with a Haake A81 circulating bath and opened to the reactor for 1.0 min while pumping was maintained. The tube was evacuated for 20–30 min before the (Me₃N)₂AlH₃-containing vessel, which was also maintained at a constant temperature, was opened. Upon completion of the deposition the system was allowed to return to its original pressure before opening. The pressure was monitored with an Inficon capacitance manometer placed between the pump and the liquid nitrogen cooled trap located at the exit of the furnace.

Results

Description of the Al Deposition. In a typical deposition the precursor vessel was opened to the reactor system for 4 min during which time the inside of the quartz tube and the substrates were coated with an Al film. The extent of the film down the length of the furnace and the appearance of the film were dependent on the specific reaction conditions. At the exit of the furnace a small amount of a crystalline deposit of precursor (or (Me₃N)-AlH₃) was observed. Unreacted precursor was also found in the liquid nitrogen cooled trap placed between the reactor and the diffusion pump. As the trap was located between the capacitance manometer and the reactor, the pressures measured resulted from the H₂ expelled during the deposition. This appeared to be a sensitive measure of the reproducibility of a given set of reaction conditions. The behavior usually observed is best described with a specific example. With the precursor vessel at 25 °C and the furnace at 180 °C, the pressure would stabilize at a constant value of approximately 0.2 Torr. Both lower furnace and precursor vessel temperatures lowered the observed pressures.

The rate of Al deposition was determined by masking part of the wafers prior to the deposition and measuring the step height created for a given deposition time. With a precursor temperature of 25 °C and the reaction tube at 180 °C, the deposition rate was 0.9 μm/min. The results of single depositions under different conditions are given in Table I. In depositions where the reactor and its contents were treated with TiCl₄ prior to the alane precursor, a more even distribution of the Al layer was observed. On the edges of these films on the quartz reactor tube, a gray, nonreflective portion was observed. We be-

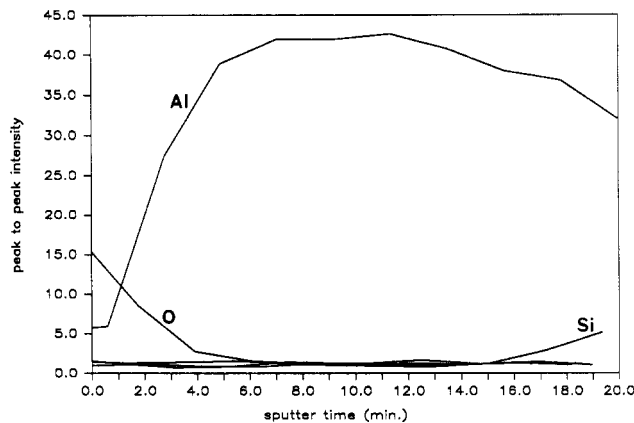


Figure 1. Auger electron intensity as a function of sputtering time (the C and N lines are shown but not separately labeled).

lieve this to be small particles of Al that have not yet formed a continuous film.

Characterization of the Al Films. Figure 1 shows the results of the Auger electron spectral profile as a function of sputtering time. The top layers of the film exhibit the usual oxide coating, and the carbon and nitrogen content is also adsorbed from the atmosphere. All of these elements decrease rapidly to within the detection limits of the method as the sputtering proceeds. In the films where TiCl₄ was used to pretreat the surface, no Ti or Cl was detected in the Al films or at the interface with the silicon wafer. This is consistent with the suggestion made regarding the TiCl₄-promoted deposition of aluminum from TIBA that less than a monolayer of TiCl₄ actually adsorbs during the pretreatment.⁴

X-ray diffraction shows the formation of polycrystalline Al films on the surface of Si(100) or glass slides (Figure 2a). Thicker films deposited without any TiCl₄ pretreatment gave nearly the expected¹⁵ intensity distributions, while an increasing deviation from the polycrystalline Al pattern was observed for thinner films. Figure 2b, however, shows the striking effect caused by pretreating the surface with TiCl₄ and growing the films at 100 °C. The films show nearly complete preference for growing with the (111) face parallel to the surface. While Figure 2b was taken from a film grown on Si(100), similar patterns are obtained on simple glass slides. Figures 3 clearly illustrates this same orientation preference on TiCl₄ treated polyimide films.

The microstructure of the films was examined by using scanning electron microscopy (SEM), and, once again, a dramatic effect of pretreating the film with TiCl₄ was observed. Figure 4 shows the surface of a rough film typical of those deposited without TiCl₄ pretreatment. The grain size observed averages 2–3 μm, and the photograph shows the large gaps between grains that cause the roughness of the surface. In the regions near the edge of

(15) Powder Diffraction File, International Center for Diffraction Data, Swarthmore, PA, Card No. 4-0787.

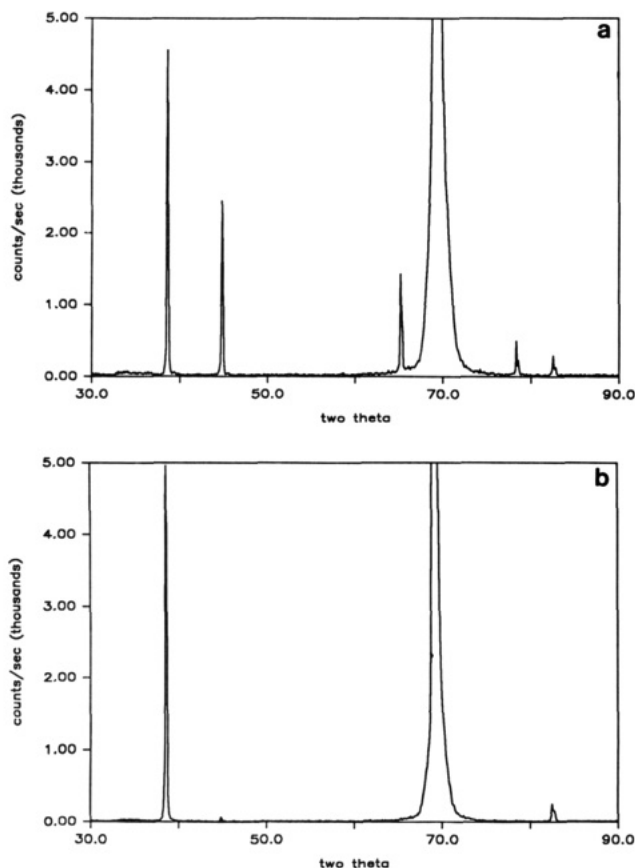


Figure 2. X-ray diffraction of thin films deposited (a) on Si(100) at 280 °C without any TiCl_4 pretreatment and (b) on Si(100) at 100 °C following treatment of the substrate with TiCl_4 . The 2θ values (degrees) with their relative intensities and assignments for polycrystalline aluminum are 38.47 (1.00) (111), 44.74 (0.47) (200), 65.13 (0.22) (220), 78.22 (0.24) (311), 82.43 (0.07) (222).¹⁵ The reflection at 69.17° is due to the substrate.

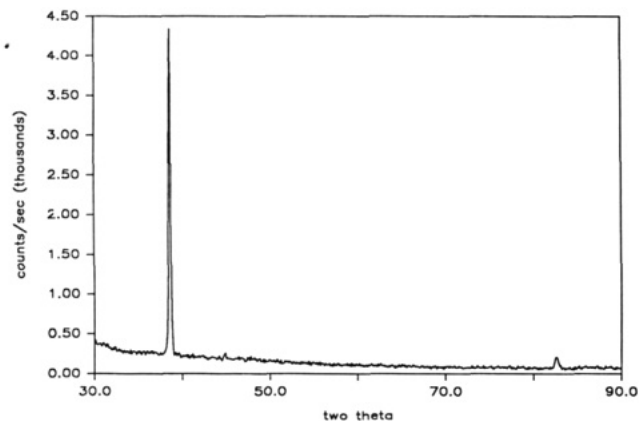


Figure 3. X-ray diffraction of an aluminum film deposited on a TiCl_4 treated polyimide film at 100 °C.

the Al created by the mask, small crystallites of Al were visible on the Si. Scanning toward the Al film reveals an increase in crystallite size but not a corresponding increase in the number of small crystallites. This suggests that the rate of crystallite growth is greater than the rate of nucleation, which undoubtedly contributes to the surface roughness of the final film. Those films grown on Si(100) wafers or glass slides that were pretreated with TiCl_4 exhibited a much higher number of crystallites in this same near-edge region. On those films grown on pretreated surfaces at 180 °C the grains averaged 1 μm , whereas the films grown on pretreated surfaces at 100 °C exhibited an average grain size of 0.15 μm (Figure 4).

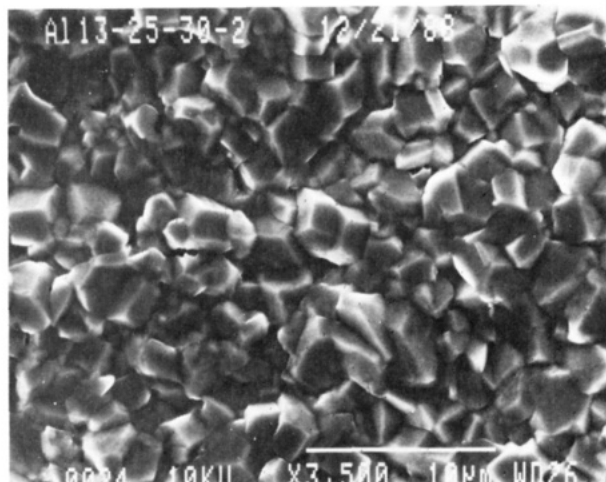


Figure 4. SEM of the surface of an aluminum film grown at 180 °C on Si(100) without any TiCl_4 pretreatment. The bar in the lower right corner represents 10 μm .

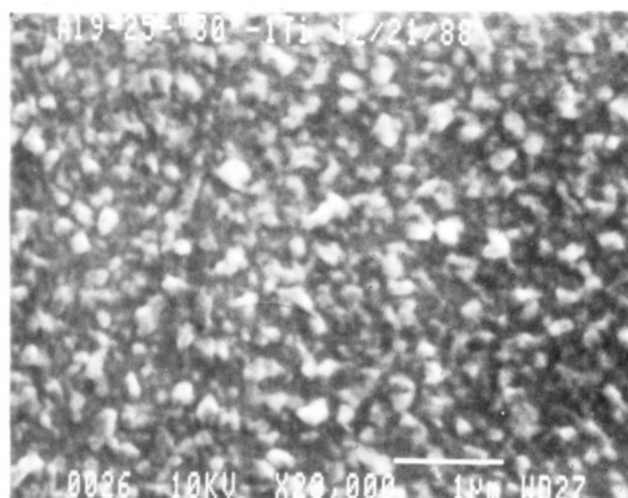


Figure 5. SEM of the surface of a mirrorlike aluminum film grown at 100 °C on Si(100) that was treated with TiCl_4 prior to the aluminum deposition. The bar in the lower right corner represents 1 μm .

The resistivities of the films were evaluated by using a four-point probe and are summarized in Table I. The low resistivities observed are virtually identical with those reported for Al films grown using TIBA, and they are near that of bulk Al.¹⁻⁷ The resistivities of the films reported in Table I are the same given the experimental limits of our thickness measurements.

The surface roughness of many of the films meant that their specular reflectivity was very low. Typically, the total reflectivity was greater than 90% at a wavelength of 550 nm, but the specular contribution to this value was less than 5%. For the films grown at 100 °C on TiCl_4 -treated surfaces, the relative contributions of the diffuse and specular components to the total reflectivity were reversed. Figure 6 illustrates the high reflectivity of these films (the actual value of the specular reflectivity of these mirrorlike films at 550 nm was 85%). Similar reflectivities are also obtained from Al deposited on treated polyimide films.

The adhesive property of films to their substrates is very important, but it is a property that is difficult to quantify. The qualitative test used in this study to evaluate the adhesive nature of these Al films was the Scotch tape test. All of the films grown on Si(100), glass, or polyimide at 180 °C or above remained intact as the tape was peeled away from the Al film. Some of the films grown at the

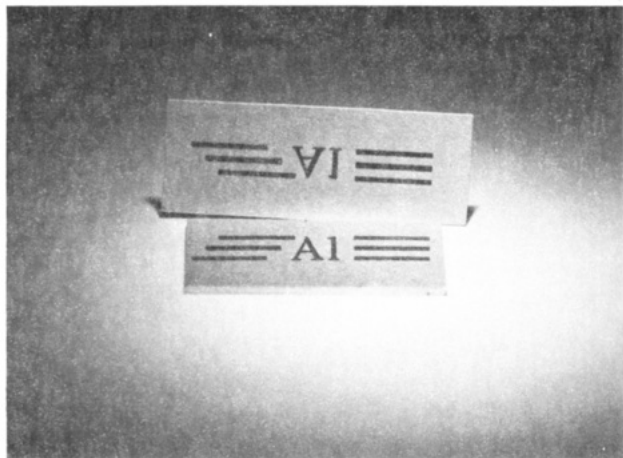


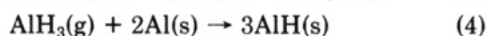
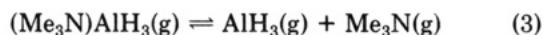
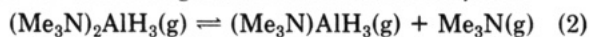
Figure 6. Photograph illustrating the reflectivity and smoothness of the LPCVD aluminum films grown at 100 °C with TiCl_4 pretreatment. The top image was printed on paper and is reflected off the Al film on the lower wafer.

lower temperatures on Si(100) were partially removed as the tape was peeled away. These films were also the ones grown on the pretreated surface, and the reason for the lower adhesion is not apparent. The adhesion of the Al films to polyimide was always good.

Discussion

$(\text{Me}_3\text{N})_2\text{AlH}_3$ is an effective precursor for the formation of thin films of high-purity, low-resistivity aluminum in a low-pressure chemical vapor deposition reactor. The microstructure of the film is very dependent on the reaction conditions, especially the temperature and the pretreatment of the surface. In the following discussion we will comment on a possible mechanism for the deposition and offer some suggestions on the role of TiCl_4 . We will then compare the deposition reactions of $(\text{Me}_3\text{N})_2\text{AlH}_3$ with TIBA.

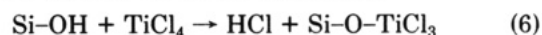
Studies of eq 1 in the gas phase suggest¹⁶ that by 80 °C the equilibrium lies mostly to the left, making the predominant species in the gas phase $(\text{Me}_3\text{N})\text{AlH}_3$. Although less well documented, higher temperatures were reported to cause dissociation of the second Me_3N releasing AlH_3 . Alane itself is known to form an intractable polymeric solid at room temperature.⁹ In the above study¹⁶ precipitation of Al metal or of solid alane did not occur as evidenced by the reported reversibility. This earlier study differs from that described here in that it was conducted in a sealed vessel where volatile products were not continuously removed. Studies of the thermal decomposition of liquid $(\text{Me}_3\text{N})_2\text{AlH}_3$ and $(\text{Me}_3\text{N})\text{AlH}_3$ have also suggested that dissociation of the Me_3N precedes hydrogen loss.¹⁷ On the basis of these studies, the following sequence is proposed for thin-film growth on aluminum surfaces:



Equations 2 and 3 are based on the previous studies, whereas eq 4 is based on the observed surface chemistry of TIBA.⁸ It was found that upon adsorption of TIBA on Al surfaces, the three isobutyl groups behaved identically. That coupled with the fact that molecular TIBA desorp-

tion was never observed suggested that upon adsorption the alkyl groups became equivalent by migration to adjacent surface aluminum atoms. The similarity between alkyl and hydride ligands is the basis for proposing the step shown in eq 4. Finally, it is known that aluminum surfaces do not dissociatively adsorb H_2 (the reverse of eq 5) at low pressures. Studies of eq 5 have been accomplished by reacting atomic hydrogen with aluminum surfaces at low temperatures. Temperature-programmed desorption studies showed that H_2 desorbs from Al surfaces around 60 °C.^{8,18} We note that this is between the lowest temperature where Al films were observed (100 °C; lower temperatures were not examined) and room temperature, where no Al films formed. While it is tempting to suggest that eq 5 is the rate-limiting step, we point out that the temperature range required for deposition is also the range where eq 2 and 3 become significant. It is important to note that our data do not rule out other possible steps, such as direct adsorption of $(\text{Me}_3\text{N})_2\text{AlH}_3$ or $(\text{Me}_3\text{N})\text{AlH}_3$. Kinetic studies of the deposition are planned that should help delineate the mechanism.

The mechanism outlined in eq 2–5 highlights the difference between growth of the Al on Al and initial nucleation of the Al crystallites, the latter being the slower process. Some consideration of the role of TiCl_4 can be addressed in this light. Alane, a powerful reducing agent, would undoubtedly react rapidly with TiCl_4 . Evidence of this can be observed in the trap placed at the exit of our CVD reactor. Even at very low temperatures (–100 °C) dark green to blue coloration is observed, and as the trap is further warmed toward room temperature, a very exothermic reaction takes place. While no spectral data regarding the products are yet available, we would anticipate Ti–H [or perhaps aluminohydride, $\text{Ti}(\text{AlH}_4)$] complexes to form initially. Reductive elimination of H_2 from the Ti would be expected to be facile, thus providing a route to metallic Al. It should be kept in mind that in our procedures both the Si(100) and glass surfaces are coated with hydroxyl groups which offer a route (the first step is shown in eq 6) for binding the Ti to the surface:



Another factor that could contribute to the surface roughness is gas-phase nucleation of particles of Al or $(\text{AlH}_3)_n$. The degree to which this is important for the film growth could be addressed by examining the kinetics of the deposition.

There is a striking similarity in physical properties and microstructure between the aluminum films grown from $(\text{Me}_3\text{N})_2\text{AlH}_3$ and those grown from TIBA. The difference in using these two precursors comes in the processing parameters. Although there is an emerging understanding of the surface growth mechanism,⁸ detailed kinetic studies of film growth under typical CVD conditions are not available for TIBA. The conditions reported by Green and co-workers⁵ are typical and involve heating the precursor to 45 °C and the substrate to 220–300 °C. With deposition pressures in the range 0.2–0.5 Torr, growth rates of 0.02–0.08 $\mu\text{m}/\text{min}$ were observed. While the pressures in our reactor were not measured, the H_2 pressure after the trap was in the vicinity of 0.2 Torr for a reactor temperature of 180 °C. We would expect the pressure in the reactor to be somewhat higher but less than the equilibrium vapor pressure of $(\text{Me}_3\text{N})_2\text{AlH}_3$, which is 1.8 Torr at 25 °C.¹⁶ The growth rate observed, 0.9 $\mu\text{m}/\text{min}$, is over an order of magnitude greater than that found for TIBA.

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The other difference comes in the threshold temperature for aluminum deposition, which is below 100 °C for $(\text{Me}_3\text{N})_2\text{AlH}_3$ and around 180 °C for TIBA. The high rates and low temperatures of deposition should make $(\text{Me}_3\text{N})_2\text{AlH}_3$ especially attractive for growing aluminum on temperature-sensitive substrates.

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Registry No. Al, 7429-90-5; $(\text{Me}_3\text{N})_2\text{AlH}_3$, 92818-52-5; Si, 7440-21-3.

Preparation of Polymer Composites. A Colloidal Pathway

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The preparation of a new type of polymer blend by a two-step procedure is described. The composite consists of hydrophilic (hydrophobic) polymer particles dispersed in a hydrophobic (hydrophilic) polymer. In the first step, polymer latex particles are obtained by the concentrated emulsion method by polymerizing the dispersed phase. The volume fraction of the dispersed phase in these emulsions can be as large as 0.995. In the second step, the polymerized concentrated emulsion is dispersed in another monomer (or a solution of the monomer in water when the latter monomer is hydrophilic) that has characteristics (in regard to hydrophilicity or hydrophobicity) of the same type as those of the continuous phase of the concentrated emulsion. This ensures the formation of a stable colloidal dispersion of latex particles in the monomer, without introducing any additional dispersant. The obtained dispersion yields upon polymerization a composite with a homogeneous microstructure. Composites of poly(styrenesulfonic acid) salt latexes dispersed in cross-linked polystyrene matrices as well as polymer composites of polystyrene latexes dispersed in cross-linked polyacrylamide matrices are thus obtained. The composites prepared by the two-step method are compared with those obtained in a single step from concentrated emulsions, emulsions, or microemulsions.

Introduction

Polymer composites are prepared to control the mechanical properties of the polymeric materials as well as for obtaining permselective membranes for separation processes. Many important polymer composites are, however, blends of incompatible polymers.¹ In spite of their high impact strength² or high liquid permselectivity,^{3,4} the large-scale application of these conventional polymer composites is sometimes limited due to the difficulty of controlling their morphology. Indeed, because of the tendency for segregation caused by the incompatibility, the materials will have a nonuniform structure. It is, therefore, useful to develop blends whose structures can be more easily controlled and, hence, can be more uniform.

The composites developed here constitute a new type of polymer blend in which the incompatibility of the polymers is taken advantage of. They are prepared by a two step polymerization method. In the first step, hydrophilic (hydrophobic) polymer latex particles are prepared. They are then dispersed in a hydrophobic (hydrophilic) (to increase the hydrophilicity of the hydrophilic phase, the hydrophilic monomer is in general replaced by

its solution in water)) monomer containing a thermal initiator, and the mixture is subsequently polymerized. In this work, the concentrated emulsion polymerization method^{5,6} is employed to produce relatively uniform polymer latex particles of submicron sizes. As in the case of the conventional emulsions, the dispersed phase in concentrated emulsions is either hydrophobic or hydrophilic, and the continuous phase is the opposite (hydrophilic or hydrophobic). However, in contrast to conventional emulsions, the volume fraction of the dispersed phase in the concentrated emulsions is very large⁷ (>0.74, which represents the volume fraction of the most compact arrangement of spheres of equal radius, and as large as 0.995). In concentrated emulsions, the spheroidal globules of the conventional emulsions are replaced by polyhedral cells separated by thin films of the continuous phase. The thin films form a network, similar to that in a foam, whose stability is ensured by the adsorption of the dispersant from the continuous phase onto the cell-film interface. The concentrated emulsions have the appearance of gels and are prepared in the present experiments at room temperature. A suitable initiator is introduced in the dispersed

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