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CVD of SiC and AlN Thin Films Using  
Designed Organometallic Precursors

by

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## CVD OF SiC AND AlN THIN FILMS USING DESIGNED ORGANOMETALLIC PRECURSORS

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## INTRODUCTION

The chemical and physical properties of aluminum nitride have made it an attractive prospect for a wide range of applications in electronics, including substrates for integrated circuits, thin film dielectrics, protective coatings, and surface acoustic wave devices.(1,2) It is a good electrical insulator with a direct band gap of 6 eV. In addition, it has a high decomposition temperature (ca. 2400°C), good chemical stability, a thermal expansion coefficient closely matched to that of silicon, high thermal conductivity, and a dielectric constant close to that of  $Al_2O_3$ .

AlN is also of interest for optical devices and coatings, as it is optically transparent throughout the visible and near infrared regions.

Aluminum nitride films have been prepared by a variety of chemical vapor deposition methods.(1-20) For example, Pauleau and co-workers have explored the use of  $AlX_3$  (X=Cl,Br) and  $NH_3$  or the preformed  $AlX_3:NH_3$  adducts as CVD precursors to AlN.(3-5) Deposition temperatures from 400-1300°C have been studied, with temperatures from 900-1000°C being optimal. The low volatility of these precursors and problems with incorporation of halide, particularly at the lower temperatures, have limited the utility of this approach.

Manasevit,(11) Rensch,(12,13) and others have obtained AlN films using  $R_3Al$  ( $R=CH_3, C_2H_5$ ) and  $NH_3$  as precursors. Deposition temperatures from 800-1200°C and  $H_2$  carrier gas is required to obtain a crystalline deposit free from carbon. Epitaxial AlN films, 100 nm to 10µm. thick have been prepared on sapphire substrates at temperatures of 1200°C by this method.

Recently, Takahasi,(17) Gladfelter,(18) and co-workers have investigated the use of unimolecular precursors and low pressure CVD processes. Takahasi and co-workers deposited polycrystalline AlN films from  $[Al(NR_2)_3]_2$  and  $[Al(NR_2)_2]_2$ , where  $R=CH_3$  or  $C_2H_5$ , at total pressures of

30-40 torr and temperatures of 300-500°C, using N<sub>2</sub> or Ar as a carrier gas.

Few details were given regarding the composition, microstructure or electrical properties of these films, although high levels of carbon incorporation were suggested by Auger spectroscopy. Gladfelter and co-workers prepared AlN films by the CVD of [(CH<sub>3</sub>CH<sub>2</sub>)<sub>2</sub>AlN<sub>3</sub>] at total pressures of 50-100 Pa and temperatures of 480-650°C. Little information is given regarding the microstructure and properties of these films; however, the authors do present XPS data which indicate an aluminum to nitrogen ratio of 1.2-1.5 and significant contamination by carbon and oxygen.

SiC is both polymorphic and polytypic, with  $\alpha$  and  $\beta$  crystalline modifications. " $\alpha$ -SiC" is actually a series of hexagonal and rhombohedral phases which include a wurzite (2H) form that is isostructural to the only stable form of AlN. The  $\beta$  or cubic form has the zinc-blende structure and is therefore most closely related to that of elemental Si among the SiC polytypes, although the lattice mismatch is substantial ( $a_{\text{SiC}} = 0.436$ ,  $a_{\text{Si}} = 0.543$ ). Both forms can be considered as comprised of SiC layers which contain (SiC)<sub>3</sub> rings fused together in the all-chair conformation. SiC in either form exhibits excellent thermal stability, decomposing to Si and C at temperatures greater than 2700°C, a Moh's hardness of 9.5, and resistance to attack by most chemicals. As a refractory,  $\alpha$ -SiC combines great strength and chemical resistance with a low coefficient of thermal expansion ( $6 \times 10^{-6}$ ). Both  $\alpha$ - and  $\beta$ -SiC are semiconductors, with band gaps of 3.1 and 2.2 eV, respectively.  $\beta$ -SiC has been employed as a high temperature semiconductor with applications in transistors, rectifiers, and diodes.(21)

SiC thin films of widely varying composition and morphology have been prepared by a range of CVD techniques based on the pyrolysis of either volatile silicon and carbon compounds or organic compounds containing carbon and silicon in the same molecule. The literature in this area has been reviewed (22), complete with a table of precursors, carrier gases, and deposition conditions. As an example, consider the formation of SiC films using CH<sub>3</sub>SiCl<sub>3</sub> as the organometallic precursor. CH<sub>3</sub>SiCl<sub>3</sub> is a volatile liquid with the desired Si:C ratio. A carrier gas such as H<sub>2</sub>, He, Ar, or N<sub>2</sub> is generally employed; with H<sub>2</sub> assisting in the removal of chloride ion as HCl. Deposition temperatures range from 800-1800°C. The morphology and composition of the SiC deposits varies as a function of deposition temperature and the concentration of CH<sub>3</sub>SiCl<sub>3</sub>. Diefendorf and co-workers have shown that  $\beta$ -SiC is deposited at 1400°C. At lower temperatures, the deposit is silicon rich, while at higher temperatures it is carbon rich.(23)

Other organosilicon compounds have been employed in CVD processes; however, their application is limited to the preparation of SiC films with higher carbon contents and, in general, temperatures in excess of 1000°C are required. The thermolysis reactions of these compounds are quite complex, apparently involving the formation of separate silicon and carbon radical species in the gas phase, and a detailed explanation of the chemistry is still lacking. Few efforts to specifically modify or design precursors for SiC CVD have been reported.

A major limitation on the utility of these CVD approaches to AlN and SiC thin films is the relatively high temperatures (>900°C) which are required in order to obtain high quality material. In the few cases where

lower temperatures have been employed, problems with the control of the microstructure and purity of the films have been encountered. To be compatible with current device technology, low-temperature ( $< 500^{\circ}\text{C}$ ) CVD approaches to the preparation of high quality AlN and SiC films are needed.

The preliminary results of our studies of the cyclic dialkylaluminum amides  $[\text{R}_2\text{AlNH}_2]_3$  as a source of high purity AlN powder and thin films has been reported previously.(19) The  $\text{R} = \text{CH}_3$  derivative,  $[(\text{CH}_3)_2\text{AlNH}_2]_3$  (Figure 1a), has sufficient volatility (vapor pressure ca. 1 mm Hg at  $70^{\circ}\text{C}$ ) for use as a precursor for the low pressure CVD of AlN thin films. Based on thermogravimetric and gas analyses, on heating to  $400^{\circ}\text{C}$ , this precursor is largely converted to AlN and methane gas.(19) In this paper, we summarize the LPCVD of AlN from  $[(\text{CH}_3)_2\text{AlNH}_2]_3$  and present new information on the preparation of SiC in a low pressure thermal CVD process using  $[(\text{CH}_3)_3\text{HSiCH}_2]_3$  (Figure 1b).

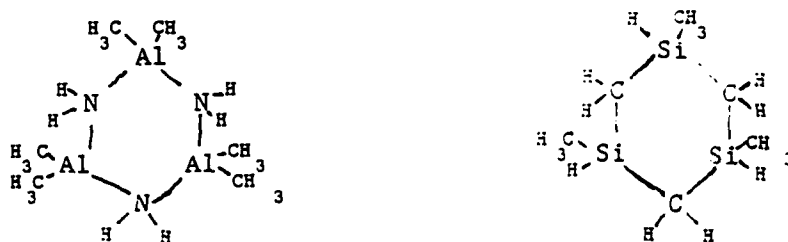


Figure 1. Molecular structures of  $[(\text{CH}_3)_2\text{AlNH}_2]_3$  (a) and  $[(\text{CH}_3)_3\text{HSiCH}_2]_3$  (b)

## EXPERIMENTAL

The organometallic precursors,  $[(\text{CH}_3)_2\text{AlNH}_2]_3$  (19) and  $[(\text{CH}_3)_3\text{HSiCH}_2]_3$  (24), were prepared and purified according to literature procedures. As  $[(\text{CH}_3)_2\text{AlNH}_2]_3$  is extremely air and moisture sensitive, all handling after preparation was restricted to a  $\text{N}_2$  filled glove box.

The substrates were 3-4  $\text{cm}^2$  single crystal silicon pieces cut from (100) or (111) wafers or silica glass plates. Each substrate was cleaned before use by first removing surface organic contaminants with trichloroethylene, methanol, or ammonium hydroxide/hydrogen peroxide. In subsequent steps, metallic or inorganic contaminants were removed by hydrochloric acid/hydrogen peroxide, and the oxide layer on the silicon substrates was removed with dilute hydrofluoric acid.

The horizontal, hot-wall quartz CVD apparatus is shown in Figure 2. O-ring joints at both ends of the apparatus are used to connect the reactor to a vacuum system and a precursor container. Substrates were generally supported on a slightly inclined quartz ramp. The reactor was heated with an electrical resistance furnace, which was controlled by an external thermocouple probe. Reactor pressure was measured immediately downstream of the reactor using a thermocouple gauge. The temperature of the precursor was controlled by a thermostatted oil bath.

In a typical experiment, 0.5g of solid  $[(\text{CH}_3)_2\text{AlNH}_2]_3$  or liquid  $[(\text{CH}_3)_3\text{HSiCH}_2]_3$  was loaded into a precursor container, fitted with a high

vacuum greaseless stopcock and o-ring joint, in a  $N_2$  filled glove box.

After connecting the precursor container to the reactor and evacuating the system to  $10^{-5}$  torr, the substrates were heated at  $800^\circ\text{C}$  for several hours. The furnace was then set to the desired temperature, the precursor frozen with liquid nitrogen, and the precursor container opened to the reactor and vacuum system. Subsequently, the precursor was warmed to  $35-60^\circ\text{C}$  for  $[(\text{CH}_3)_2\text{AlNH}_2]_3$  or  $20^\circ\text{C}$  for  $[(\text{CH}_3)\text{HSiCH}_2]_3$  and vaporized into the reactor using a continuously pumping mechanical/diffusion pump. The average deposition rate was 5-25 Å/min. After the deposition, the reactor was cooled to room temperature and the substrates removed in air.

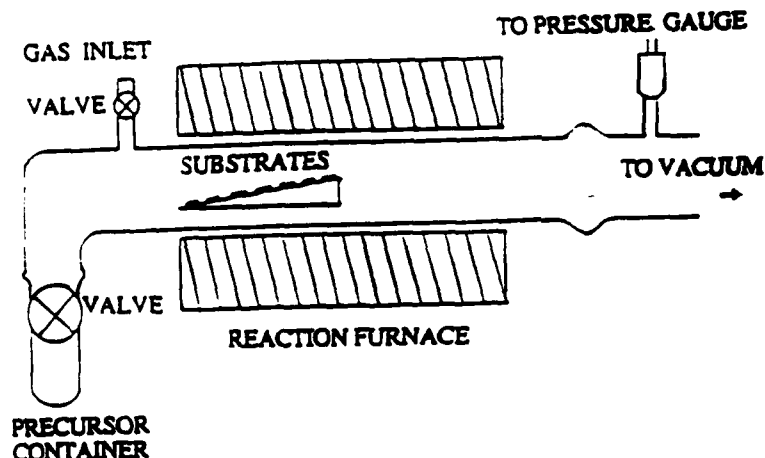


Figure 2. Schematic of CVD apparatus.

Complete details of the characterization of the AlN films is presented elsewhere. (21)

## RESULTS AND DISCUSSION

### CVD of Aluminum Nitride Films

Polycrystalline aluminum nitride films were deposited on silicon and silica substrates at  $400-800^\circ\text{C}$  and pressures of  $0.2-1.0 \times 10^{-2}$  torr. Under appropriate conditions of substrate temperature ( $>500^\circ\text{C}$ ) and growth rate (5-25 Å/min), the films were transparent, smooth, and highly adherent to the substrate. Auger electron spectroscopy indicated that these films contained close to a one-to-one ratio of aluminum to nitrogen with carbon and oxygen  $<1\%$  and  $<5\%$ , respectively (Figure 3). X-ray and electron diffraction studies showed that the 2H (hexagonal,  $a = 3.114 \text{ Å}$ ,  $c = 4.9792 \text{ Å}$ ) phase of aluminum nitride was the only crystalline product. Scanning and transmission electron microscopy indicated a dense, polycrystalline microstructure with an AlN grain size range of  $0.05-1.00 \mu\text{m}$ , depending on deposition conditions. In the case of the films which contained larger AlN grains (ca.  $1 \mu\text{m}$ ), evidence for texture was obtained from TEM studies with a preferred orientation of the AlN grains with their c-axis perpendicular to the Si (100) surface.

Films deposited on silica at or above  $600^\circ\text{C}$  were transparent in the visible and near infrared with an absorption edge of ca. 6 eV. Transmission FTIR spectra of the films on silicon showed the characteristic

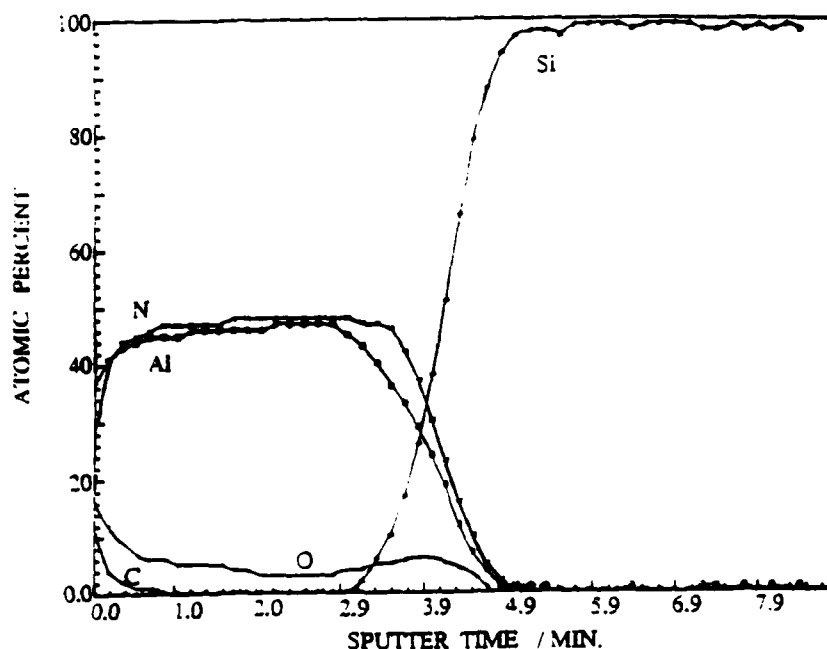


Figure 3. Auger depth profile of AlN film on Si (100)

Al-N stretching vibration at  $ca. 680\text{ cm}^{-1}$  with relatively weak, broad bands in the N-H and C-H stretching region. Current-voltage and capacitance-voltage measurements were performed on aluminum nitride films deposited on Si (100), yielding values for the dielectric constant, field breakdown strength, and resistivity which suggest that films obtained at temperatures greater than  $600^{\circ}\text{C}$  have properties suitable for microelectronic applications.

#### CVD of Silicon Carbide Films

Initial experiments indicate that SiC films can be deposited using  $[(\text{CH}_3)\text{HSiCH}_2]_3$  at temperatures of  $700\text{--}800^{\circ}\text{C}$  or less and 0.9 torr pressure. The deposited films are transparent, with a yellow shiny color and smooth, uncracked surface. Scanning electron micrographs show a uniform, fine-grained surface morphology. SiC films deposited on Si or  $\text{SiO}_2$  were non-crystalline or nanocrystalline according to XRD analyses. Film thicknesses varied from  $0.25\text{--}1.0\text{ }\mu\text{m}$ , depending on the location of the wafer piece in the furnace.

Auger electron spectroscopy studies show that the films contain 49% Si, 51% C, and  $<0.8\%$  O (Figure 4). These results were derived using sensitivity factors for the Si and C Auger peaks obtained from measurements on a 6H silicon carbide single crystal of known 1:1 stoichiometry.

#### Surface Kinetics Studies of $[(\text{CH}_3)\text{HSiCH}_2]_3$ Pyrolysis

Preliminary investigations into the mechanism and kinetics of the interaction of organometallic precursors to AlN and SiC with solid surfaces are being carried out in conjunction with Dr. John Hudson and co-workers at Rensselaer Polytechnic Institute. The research involves the controlled study of surface reaction on a well-characterized surface in ultrahigh

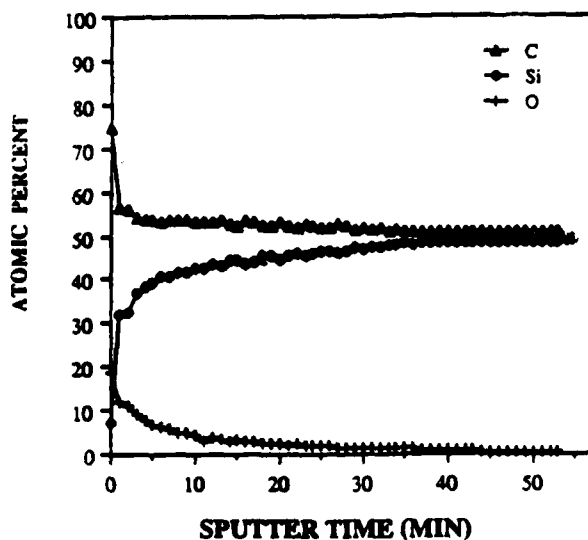


Figure 4. Auger depth profile of SiC film on Si (100)

vacuum by mass spectrometry, thermal desorption spectroscopy, molecular beam relaxation spectroscopy, Auger electron spectroscopy, and scanning electron microscopy.

These studies indicate that at temperatures below ca 500 °C,  $[(CH_3)HSiCH_2]_3$  adsorbs on the Si (100) surface, then desorbs without decomposition. The desorption behavior indicates two distinct states or species on the surface, as might result from the two different isomers known to be present in the sample of the  $[(CH_3)HSiCH_2]_3$  precursor employed. Above ca. 550 °C, attenuation of the mass spectral peaks associated with the  $[(CH_3)HSiCH_2]_3$  precursor is observed indicating the onset of a surface reaction. The solid reaction product of this decomposition was identified as SiC by Auger spectroscopy and subsequent examination by SEM indicated that the growing SiC films consisted of small crystallites, epitaxially oriented on the substrate surface.

#### SUMMARY

High purity, crystalline AlN and SiC thin films have been prepared by the chemical vapor deposition of  $[(CH_3)_2AlNH_2]_3$  and  $[(CH_3)HSiCH_2]_3$ , respectively, at temperatures under 800 °C. The use of these "designed precursors" results in film stoichiometries of nearly one-to-one and the evolution of non-corrosive reaction by-products. In addition, no carrier gas is required. Preliminary studies of the interaction of the  $[(CH_3)HSiCH_2]_3$  system with a clean Si (100) surface indicate interesting precursor adsorption and decomposition behavior with epitaxial growth of SiC on Si(100) at or below 700 °C. It is anticipated that information from these studies will be used to improve the deposition processes and aid in the design of new precursors.

#### ACKNOWLEDGEMENTS

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