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PREPARATION AND CHARACTERIZATION OF THIN FILMS OF
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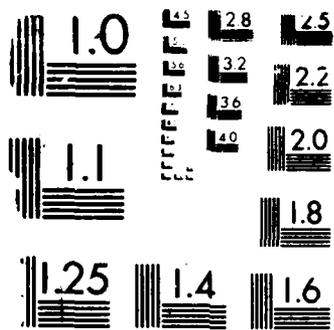
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TECHNICAL REPORT No. 10

Preparation and Characterization of Thin Films of Alumina
by Metalorganic Chemical Vapor Deposition

by

J. Fournier, W. DeSisto, R. Brusasco, M. Sosnowski, R. Kershaw,
J. Baglio, K. Dwight and A. Wold

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Brown University
Department of Chemistry
Providence, RI 02912

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PREPARATION AND CHARACTERIZATION OF THIN FILMS
OF ALUMINA BY METAL-ORGANIC CHEMICAL VAPOR DEPOSITION

by

J. Fournier, W. DeSisto, R. Brusasco, M. Sosnowski⁺, R. Kershaw,
J. Baglio⁺, K. Dwight and A. Wold*
Chemistry Department, Brown University
Providence, Rhode Island 02912

⁺Materials Research Laboratory, Brown University
⁺GTE Laboratories, Waltham, MA 02254

*Address all correspondence

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ABSTRACT

A simple novel horizontal reactor was used to prepare 2000 Å films of alumina on silicon substrates by the thermal decomposition of aluminum tri-isopropoxide at temperatures between 350° and 500°C. The films were annealed in oxygen to test stability towards shrinkage and were characterized as to porosity, the presence of OH absorption bands in the infrared transmittance spectra and breakdown voltage.

MATERIALS INDEX: Alumina, Aluminum Tri-isopropoxide, Silicon.

Introduction

In recent years there has been considerable research directed to the deposition of metal oxide films on a variety of substrates. Whereas it is relatively simple to prepare reasonably good films of SiO₂, these films are unsatisfactory for many electronic applications because of their relatively low dielectric constant, large sensitivity to the diffusion of Na⁺ ions and also their low radiation resistance. Among the possible alternatives, Al₂O₃ has been widely chosen because it has a higher dielectric constant, a lower sensitivity to Na⁺ diffusion and a higher radiation resistance (1).

Such oxide layers would be useful for numerous applications. Among such uses would be surface protection and selective masking during diffusion (2), in device isolation masking against impurities, junction passivation, and insulation between metal layers (3-6). Such oxide layers must be grown routinely and reproducibly with high yield and long-term reliability and must not be degraded by subsequent processing. Hence there has been a continual search for oxides with superior properties to those possessed by SiO₂.

Earlier efforts to prepare homogeneous films of Al₂O₃ on an InP support by a sol-gel method indicated that such films would be porous and result in low

19

breakdown potentials (7). Aboaf (8) obtained dense films of Al_2O_3 on silicon substrates by decomposing aluminum tri-isopropoxide at 420°C at ambient pressure. A horizontal reactor was used in his study. Duffy and Kern (9) prepared alumina films on silicon using a vertical reactor and Saraie (10) prepared alumina films on silicon substrates but at reduced rather than ambient pressures. Review papers by Ludowise (11), Westphal (12), Carlsson (13) and van den Ven et al. (14) have examined various reactor designs. In general, vertical reactors have a tendency to develop convection rolls which can trap reaction by-products in the gas stream and are inferior to horizontal displacement-type reactors. The horizontal reactors perform best at high flow velocities and under reduced pressure. In this study a horizontal-type reactor is described which emphasizes flexibility and versatility with respect to gas flow, reactor pressure, and substrate temperature and positioning. Films of alumina were prepared by decomposition of aluminum tri-isopropoxide at temperatures between $350\text{-}500^\circ\text{C}$. The resulting products were compared as to uniformity and stability. Further characterization involved shrinkage, porosity, breakdown voltage, and the presence of OH absorption bands in the infrared transmittance spectra.

Experimental

The MOCVD reactor used in this investigation is given in Fig. 1. Separate argon flows pass through the reactor and over the heated source material and are mixed before passing over the heated substrates. The substrate holder was made of spectroscopic grade carbon and heated by a Watlow Firerod cartridge heater. The bottom, sides, and leading edge of the holder were wrapped with aluminum foil to minimize infrared losses and improve heating efficiency. Substrates were placed flush in a groove milled out on the 45° face of the substrate holder.

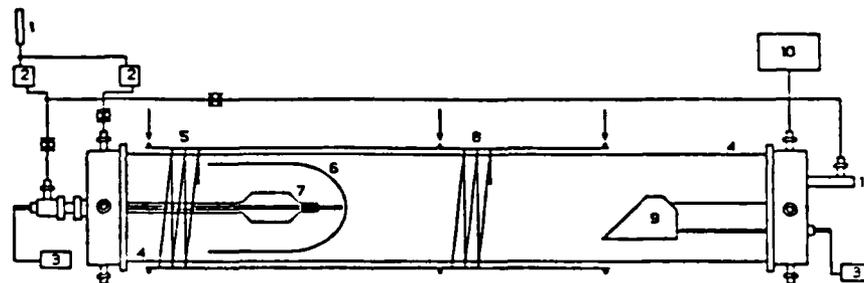
Deposition of alumina was performed by volatilizing and thermally decomposing aluminum isopropoxide (Chattem Chemical Co., melting point 118°C) in an argon gas stream at approximately 1 torr pressure. In all cases, the source material was premelted and the temperature then lowered to the desired value. Under a dry argon atmosphere, aluminum tri-isopropoxide remains a melt (supercooled liquid) even at room temperature. Substrate material consisted of 1 cm squares cut from n-type (100) silicon wafers obtained from Wacker Chemitronic. The silicon had a resistivity of $8.5 \Omega\text{-cm}$ and the number of carriers was 4.5×10^{14} .

Cleaning and etching were carried out just before deposition in accordance with the following procedure.

ETCHING PROCEDURE

- 1) Clean substrates in trichloroethylene for 5 min. at 80°C.
- 2) Clean in acetone for 1 min. at room temperature
- 3) Clean in methanol for 1 min. at room temperature.
- 4) Etch in 1% v/v bromine/methanol for 15 min. at room temperature.
- 5) Rinse in H₂O, then methanol.
- 6) Etch in 30% w/w H₂O₂ for 2 min. at 80°C.
- 7) Rinse in H₂O, then methanol and finally air dry.

This oxidative etch by hydrogen peroxide was found to be compatible for etching silicon and III-V semiconductors such as InP. Post deposition annealing treatments were performed in a horizontal tube furnace under flowing oxygen. The gas was dried by passing through a P₂O₅ dessicant, and the temperature was programmed at 50°C hr⁻¹ from room temperature to 350°C. The samples were maintained at this temperature for four hours, and the furnace was then cooled at 50°C hr⁻¹.



LEGEND

1. MATHIESON UHP ARGON
2. BROOKS MASS FLOW CONTROLLERS
3. OMEGA TEMPERATURE MONITOR
4. SILICA REACTOR TUBE 50 x 910 mm
5. EXTERNAL KANTHAL HEATING ELEMENT (ZONE 1)
6. MIXING TUBE 41 x 120 mm
7. SOURCE MATERIAL CHAMBER
8. EXTERNAL KANTHAL HEATING ELEMENT (ZONE 2)
9. SUBSTRATE HOLDER (SEE TEXT)
10. THELAL TEMPERATURE CONTROLLER
11. GAS EXIT TO EDWARDS ROTARY PUMP

Fig. 1. Schematic Diagram of MOCVD Reactor

The thickness of the films was determined by ellipsometry using a Rudolph Research Auto EL-II ellipsometer. The wavelength of the laser beam was 632.8 nm. Values of the two ellipsometric angles, Δ and Ψ were recorded and input to a Hewlett Packard 9825A computer to calculate the thickness. Additional input parameters for the thickness calculation were the real ($n_S=3.877$) and complex ($k_S=0.019$) values for the index of refraction of the substrate and a trial value for the index of refraction of the film ($n_D=1.5$); the values were taken from the data of Aspnes and Studna (15).

Characterization of the surface porosity was achieved using a two-step replication procedure. Parlodion was applied to the surface of the film in a dry atmosphere and allowed to dry for 24 hours. Carbon-platinum was shadow evaporated onto the stripped Parlodion plastic at an angle of 60° ; the overall thickness of the replica was 250Å. The carbon replicas were liberated using methyl acetate and mounted on 200 mesh copper grids. The replicas were examined in a Philips Optics Electron Microscope (420 STEM) operating at 80 kV. Magnification calibration consisted of photographing spherical particles of known diameter (0.312 μm).

The infrared spectra was obtained using a Bomen (Model # BA3.02) F.T.IR Spectrometer with a mercury cadmium telluride detector. Measurements were made on both coated and uncoated silica substrates. The final data were obtained from the ratio of these measurements. The plotted output corresponds to the properties of the film.

Electrodes for the DC current-voltage measurements consisted of indium alloy (Indalloy #9, Indium Corporation of America) ultrasonically bonded to the back surface of the silicon substrate. The indium alloy contacts were checked for ohmic behavior before measurement of the DC current-voltage behavior. Gold was evaporated through a mask to obtain an array of circular electrodes on the surface of the alumina film of area $1.1 \times 10^{-2} \text{cm}^2$. Contact to a gold electrode was obtained by touching a gold tipped micromanipulator to the surface.

A DC potential was applied via a voltage follower having an output impedance less than 0.1Ω , and the resulting response was measured with a current amplifier, which inserted a negligible potential drop (less than $1 \mu\text{V}$) in the external circuit.

Results and Discussion

The novel reactor design shown in Fig. 1 gave alumina films using the deposition conditions summarized in Table 1. To avoid gas depletion and ensure uniformity, a substrate tilt angle of 45° was used. Results reported by Aboaf (8) and Brusasco (16) indicate that films grown at a rate of 100Å min^{-1} or higher produce highly porous films. Therefore, growth rates considerably less than this value were used in the present study.

TABLE 1

DEPOSITION PARAMETERS

Substrate Temperature:	350°C	400°C	450°C	500°C
Source Flow:	3 sccm	3 sccm	3 sccm	3 sccm
Carrier Flow:	57 sccm	57 sccm	57 sccm	57 sccm
Pressure:	0.75 torr	0.72 torr	0.71 torr	0.71 torr
Source Temp. (Zone 1):	118°C	91°C	78°C	78°C
Wall Temp. (Zone 2):	225°C	225°C	225°C	225°C
Deposition Rate (Å min ⁻¹):	35	36	46	45

From ellipsometry measurements, the thickness of each film was determined, uniformity established, and the refined value of each refractive index was obtained. These results are given in Table 2.

TABLE 2

FILM THICKNESS AND INDEX OF REFRACTION

Substrate Temp. °C	Thickness Å	Refractive Index
500	1850	1.629
450	1917	1.606
400	1914	1.596
350	1915	1.580

From Table 2 it can be seen that the refractive index of the film prepared at 500°C approaches the value reported (9) for low temperature alumina films (1.64)

From the IR data a distinct absorption band was evident from 2700-3700 cm⁻¹ in the films prepared at 350°C. This band was absent in films prepared at 500°C. The presence of the OH absorption band is related to residual hydration of the films. This is consistent with the known tenacity of alumina to hold water.

The samples prepared at 350° and 400°C showed shrinkages of 6.5% and 5.0%, respectively, after annealing in flowing oxygen for 4 hours at 350°C. Those prepared at 450° and 500°C showed less than 1% shrinkage. The large shrinkage obtained for the 350°C film is consistent with the observed presence of hydrated species as indicated by the IR data.

Photomicrographs were taken of oxygen annealed films prepared at 350° and 450°C. The number of defects per cm² for films prepared at 350°C was approximately 18 x 10⁶. Films prepared at 450°C contained approximately half this number of defects per cm². The average size of the pits in films prepared at 350° and 450°C were 0.14 μm and 0.08 μm, respectively. It can be seen that the preparation of the alumina film at increasing deposition temperatures results in less shrinkage and pitting upon annealing.

It appears from these results that decomposition of aluminum tri-isopropoxide must be carried out above 400°C in order to obtain films which are thermally stable.

For films prepared at all the temperatures indicated above, negligible DC-current was observed with increasing applied potential until a "breakdown" voltage of approximately 4 volts was reached. Above this voltage, the measured current increased sharply.

The density of defects and their sizes observed for films prepared at 350°C, while higher than those prepared at 450°C, are not sufficient to perforate the film and lower the breakdown potential. Characterization of alumina films prepared by the thermal decomposition of aluminum tri-isopropoxide at 450° and 500°C indicates no shrinkage upon thermal annealing. The use of oxygen annealing at 350°C has been shown to be a method of determining stability of alumina films after preparation. Attempts to prepare films under similar conditions using aluminum tert-butoxide failed to yield uniform films at 500°C. Higher decomposition temperatures are necessary if this organometallic compound is to be used as a source material.

Acknowledgments

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Brown University
Providence, Rhode Island 02912

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