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“Formation of Aluminum Oxide Films from
Tris(hexafluoroacetylacetonato)aluminum(III)
and Tris(trifluoroacetylacetonato) aluminum(III)
in the Substrate Temperature Interval 320°C - 480°C
in an Argon Ambient Atmosphere”

by

Dorota Temple and Arnold Reisman

North Carolina State University
Department of Chemistry
Department of Electrical and Computer Engineering
Raleigh, North Carolina

July 31, 1989

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**Formation of Aluminum Oxide Films
from Tris(hexafluoroacetylacetonato)aluminum(III)
and Tris(trifluoroacetylacetonato) aluminum(III)
in the Substrate Temperature Interval 320°C - 480°C
in an Argon Ambient Atmosphere.**

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Abstract

A study of the thermally activated decomposition of Al(hfa)_3 (tris(hexafluoroacetylacetonato)aluminum) and Al(tfa)_3 (tris(trifluoroacetylacetonato)aluminum) in the gas phase is reported. The decomposition process was carried out in an open tube atmospheric pressure reactor in an inert atmosphere of argon for the substrate temperature interval 320°C - 480°C. In the case of Al(hfa)_3 , the chemical vapor deposition process resulted in the deposition of aluminum oxide films while the decomposition of Al(tfa)_3 led to the deposition of Al_2O_3 along with significant quantities of carbon. The breakdown strength of $\text{Al}/\text{Al}_2\text{O}_3/\text{Si}$ capacitors with aluminum oxide films prepared from Al(hfa)_3 was found to be higher than 3 MV/cm in most cases. The flat band voltage was positive, indicative of apparent negative oxide charge of an area density of approximately $3 - 10 \times 10^{11} \text{ cm}^{-2}$.

Introduction

Studies of the chemical and physical properties of various metal β -diketonates, in progress in this laboratory,¹⁻⁵ were extended to include an investigation of the vapor phase thermally activated decomposition of fluorinated β -diketonates of aluminum, namely, tris(hexafluoroacetylacetonato)aluminum(III) (Al(hfa)_3), and tris(trifluoroacetylacetonato)aluminum(III) (Al(tfa)_3). A previous paper discusses the deposition of copper by means of the thermal decomposition of Cu(hfa)_2 .⁴ The choice of Al(hfa)_3 and Al(tfa)_3 was prompted by their potential for formation of insulators in a low temperature CVD process and by the limited attention they have received in the literature. These and other metal acetylacetonates offer a promise for use in mixed insulator film formation from both the vapor and solid state. There are several reports⁶⁻⁸ dealing with the pyrolysis in the vapor phase of the non-fluorinated analog of the above compounds *i.e.* tris(acetylacetonato)aluminum(III). Thermally activated decomposition of this compound in nitrogen and nitrogen + oxygen is reported to result in the deposition of aluminum oxide films which is not surprising due to a strong affinity of aluminum to oxygen. The fluorinated acetylacetonates of aluminum which, to the authors' knowledge, have not been previously used for the aluminum oxide deposition, have potential advantages over the aluminum acetylacetonate in metal-organic chemical vapor deposition processes. They possess significantly higher vapor pressure⁹ and thus, they do not need to be heated to high temperatures in order to obtain an adequate concentration of their vapor in the reaction medium. Also, the temperature of the onset of decomposition is significantly higher than the required evaporation temperature, but still relatively low, which makes the deposition process attractive for microelectronic applications.

The object of the present study was to investigate the thermally activated decompositions of the Al(hfa)_3 and Al(tfa)_3 , varying the decomposition temperature, and examining the chemical composition and electrical properties of the obtained deposits.

Experimental Procedures

Study of the thermally activated decompositions of the selected metal-organic compounds was carried out in an experimental open tube atmospheric pressure reactor, described in ref.4. The process con-

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sisted in the transport of the metal-organic compound vapor from an essentially equilibrium evaporation zone to the reaction chamber, where decomposition of the vapor to form films of Al_2O_3 on bare radiant heated Si substrates took place. Ultra-pure grade argon was used both as the carrier and diluent gas. P-type (100) silicon wafers were used as substrates. The substrates were cleaned before deposition by means of a standard technique¹⁰ and then dried by blowing filtered nitrogen across them. In the chemical vapor deposition process, the following parameters were controlled: temperature of the evaporation zone, flow rates of the carrier and diluent gases, and temperature of the substrates. In the experiments for a given compound, the evaporation zone temperature was maintained constant; it was typically 68°C for solid $\text{Al}(\text{hfa})_3$ and 150°C for liquid $\text{Al}(\text{tfa})_3$. These temperatures give vapor pressures of the compounds of 5 torr and 10 torr,⁹ respectively. The carrier and diluent gas flow rates were 20 sccm and 300 sccm, respectively. The temperature of the substrates was varied in the range 320°C - 480°C.

The metal-organic compounds: $\text{Al}(\text{hfa})_3$ (chemical formula $\text{Al}(\text{CF}_3\text{COCHCOCF}_3)_3$) and $\text{Al}(\text{tfa})_3$ (chemical formula $\text{Al}(\text{CF}_3\text{COCHCOCH}_3)_3$) were purchased from Strem Chemicals, Inc. They are solids at room temperature. The melting point of $\text{Al}(\text{hfa})_3$ is 73 - 74°C, while $\text{Al}(\text{tfa})_3$ melts at 122 - 123°C.¹¹

The chemical composition of thin film deposits obtained in the described process was analysed by means of X-ray Photoelectron Spectroscopy, XPS. Depth profiling was performed by means of argon ion sputtering. In the evaluation of the relative atomic concentrations of different elements in the deposits, sensitivity factors obtained for different elements for this particular spectrometer were used. This technique which has an atomic sensitivity of 1ppt - 1pph, depending on the element, appears to be accurate within 15-20%, the accuracy being limited by sample surface roughness effects. Thicknesses of the deposits were measured by a 3-wavelength ellipsometer. Presence of crystallographic structure was checked by X-ray diffraction techniques. Film surface morphology was examined using an optical microscope with a differential interference contrast capability and magnifications up to 440X. Electrical characteristics were determined by capacitance - voltage (C-V) and current - voltage (I-V) measurements on $\text{Si}/\text{Al}_2\text{O}_3/\text{Al}$ structures. The MOS capacitors were fabricated by vacuum deposition of Al through a metal mask. The area of the metal electrode was typically $1.4 \times 10^{-2} \text{ cm}^2$.

Results

A. Decomposition of Aluminum Hexafluoroacetylacetonate.

General Properties of Deposits- The thermally activated decomposition of Al(hfa)_3 under the process conditions specified in the previous section resulted in the deposition of transparent, continuous, well adherent films in the whole range of substrate temperatures studied 320°C - 480°C. Examination of film surfaces by means of an optical microscope did not reveal any surface roughening under the magnification used, 440X. The growth rate of films, as determined on the basis of the ellipsometric measurements of film thicknesses was in the range of 1 - 5 nm/min depending on the substrate temperature, and the position of the sample on the susceptor. Results of the examination of selected films by means of X-ray diffraction techniques indicated that the deposits were amorphous.

Chemical composition.- Fig.1 a and b show the XPS spectra for deposits obtained for the substrate temperatures, T_{sub} , equal to 320°C and 480°C, respectively. These spectra were taken after sputter-cleaning of the samples, performed in order to remove atmospheric contamination. Depth-profiling analyses were performed for the samples, and did not show any significant variations of the elemental concentrations with film thickness. The approximate values of the atomic concentrations of the elements detected in the spectra presented in Fig.1, along with the values of other analyzed samples are given in Table I. The main component of the deposits seems to be aluminum oxide as indicated by the relative atomic concentrations of aluminum and oxygen.

Examination of the data presented in Table I indicates also that the substrate temperature in the range studied affects the chemical composition of the deposits. The carbon concentration increases as temperature increases, as was also found in the study of copper film formation from Cu(hfa)_2 .⁴ The other impurity detected was fluorine and its concentration does not seem to depend on temperature. As indicated via sputter analysis, composition, including that of fluorine, was uniform throughout the oxide.

Refractive index.- The refractive index at 6328 Å of the deposits was determined by means of ellipsometry and was found to vary with temperature within the range 1.75 - 1.95. Higher substrate temperature

favorable films with higher index of refraction. The value for polycrystalline stoichiometric $\alpha\text{-Al}_2\text{O}_3$ is 1.76.¹² The values of refractive index of the films reported in this paper are comparable to the values for aluminum oxide films obtained from aluminum acetylacetonate⁶ but higher than the values for aluminum oxide films deposited from aluminum isopropoxide (1.62)¹³ and from trimethylaluminum (1.54-1.67).¹⁴

Electrical Properties. - In order to evaluate electrical properties of the obtained aluminum oxide films metal - oxide - semiconductor (MOS) capacitors were prepared by depositing aluminum dots on the metal - oxide structure. The capacitance voltage measurements were done at 1 MHz, using a C-V plotter. The voltage was swept from negative 5 V to positive 5 V and back to the initial negative 5 V in order to detect hysteresis in the C-V trace. After post-metal annealing, pma, in forming gas (10% H_2 - 90% N_2) at 400°C for 30 min, the value of the flat band voltage, V_{FB} , was found to be in most cases positive, indicative of apparent negative charge in the dielectric film. V_{FB} ranged typically from 0.5 - 1.9 V for film thicknesses in the interval 90 nm - 130 nm. These values of V_{FB} correspond to the net negative charge density in the range $3 - 10 \times 10^{11} \text{ cm}^{-2}$, if the assumption is made that the oxide charge is located at the Si - oxide interface. If, in fact, the charge is uniformly distributed in the oxide, the values of the density need to be multiplied by the factor of 2. Hysteresis was not generally observed in the C-V curves of capacitors which received only a pma cycle.

For some samples annealing in dry oxygen at 450°C for 2 hrs. was carried out on specimens which were stripped of their capacitor Al dots. New Al dots were evaporated and the capacitors were pma, as before. The flat band voltage was found to be smaller than in the case of the non-treated samples. However, the samples annealed in oxygen exhibited hysteresis in their C-V traces.

The C-V measurements served also to determine the dielectric constant of the aluminum films. The dielectric constants were calculated using the values of capacitance in the accumulation region and were found to be in most cases in the interval 6.0 - 7.9 which is comparable with values obtained for aluminum oxide films deposited from other compounds.^{6, 13, 14}

The breakdown field of the oxide films was determined by applying a voltage across the oxide and observing on an I-V curve tracer the voltage at which the current suddenly increased to the point of no return. The voltage scan direction was toward accumulation in the silicon substrate. The breakdown field was generally better than 3MV/cm.

B. Decomposition of Aluminum (III) Trifluoroacetylacetonate.

XPS analyses of the deposits obtained by the thermally activated decomposition of $\text{Al}(\text{tfa})_3$ indicate a much higher concentration of carbon than the concentration in the deposits obtained in the same temperature range from the hexafluoroacetylacetonate compound. Fig.2 shows XPS spectra obtained for T_{sub} equal to 350°C and 450°C, respectively. Again, the depth profiling analysis showed uniform concentration of the different species throughout film thickness. While the concentration of the carbon is high, the results indicate that the films contain both aluminum and oxygen, aluminum being bonded to oxygen in Al_2O_3 , as indicated by the position of Al 2p peak. The carbon content of the films increases with temperature of deposition. Due to the high concentration of carbon in the deposits obtained from $\text{Al}(\text{tfa})_3$, no attempts were made to further characterize the films.

Summary and Conclusions

Thermally activated decomposition of aluminum hexafluoroacetylacetonate carried out at atmospheric pressure in an inert atmosphere of argon resulted in the deposition of aluminum oxide films. Films deposited in the substrate temperature range 320-400°C contained the smallest amount of carbon impurity. Flat band voltage of $\text{Si}/\text{Al}_2\text{O}_3/\text{Al}$ capacitors was found to be positive indicative of negative charge concentration of $3 - 10 \times 10^{11} \text{ cm}^{-2}$ and post-deposition annealing of the films in dry oxygen at 450°C was found to lead to the reduction of the oxide charge density. In addition, following oxygen annealing the C-V curves exhibited hysteresis.

Decomposition of aluminum trifluoroacetylacetonate resulted also in the deposition of aluminum oxide, however the carbon content of the films was much higher than in the case of the deposits obtained

from aluminum hexafluoroacetylacetonate under similar process conditions.

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Table 1

Chemical composition of deposits obtained by thermally activated decomposition of Al(hfa)_3 as determined by XPS analyses.

Temp. of Substrates [°C]	Composition of Deposits			
	rel. at. %			
	Al	O	C	F
320	39	53	3	5
340	38	53	3	6
380	40	52	4	4
440	39	46	6	9
480	36	49	11	4

Figure Captions

Figure 1.

XPS spectrum of the film obtained by decomposition of Al(hfa)_3 for the substrate temperature

a) $T_{\text{sub}} = 320^\circ\text{C}$ and

b) $T_{\text{sub}} = 480^\circ\text{C}$

(after sputter-cleaning of the film).

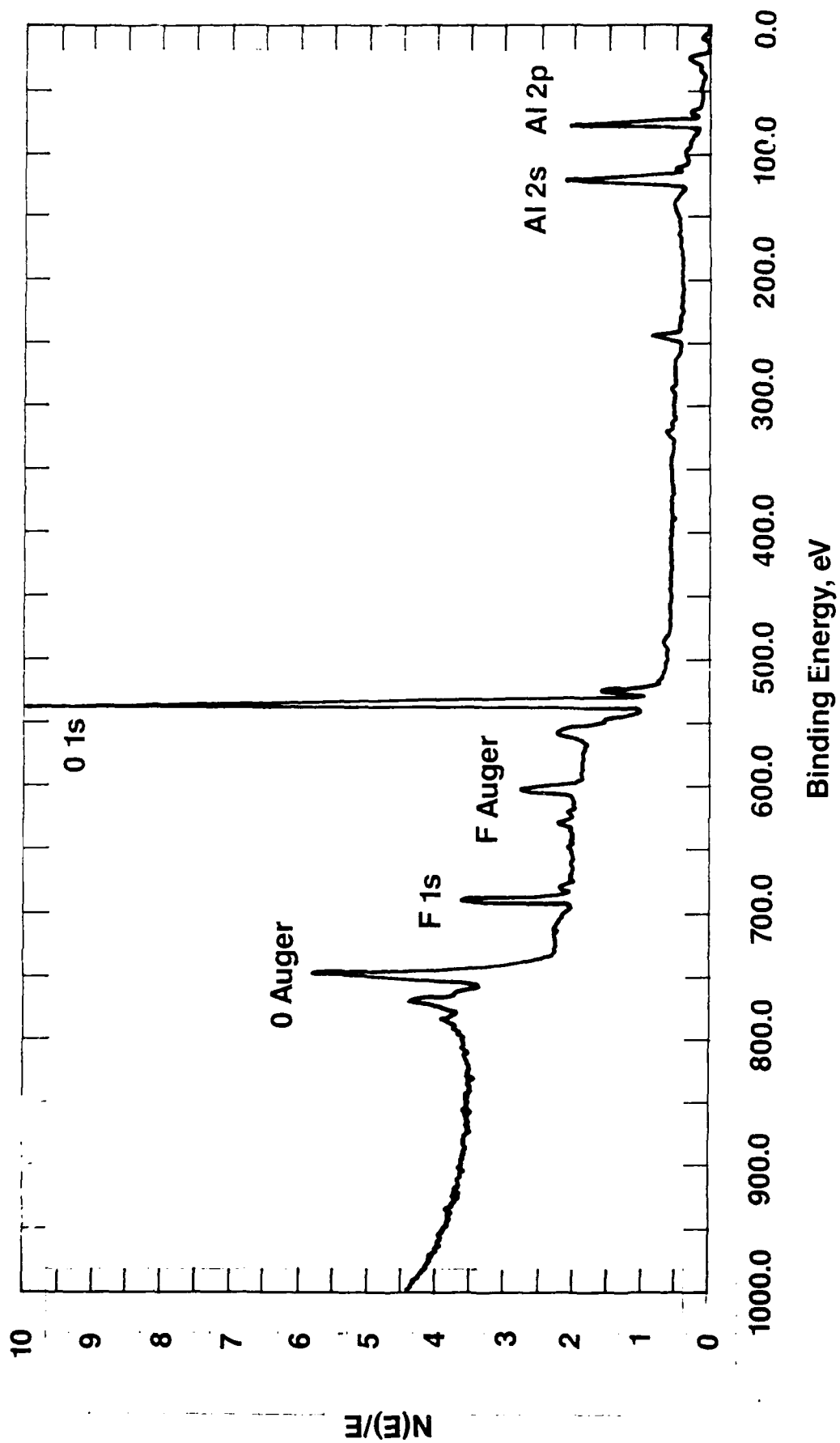
Figure 2.

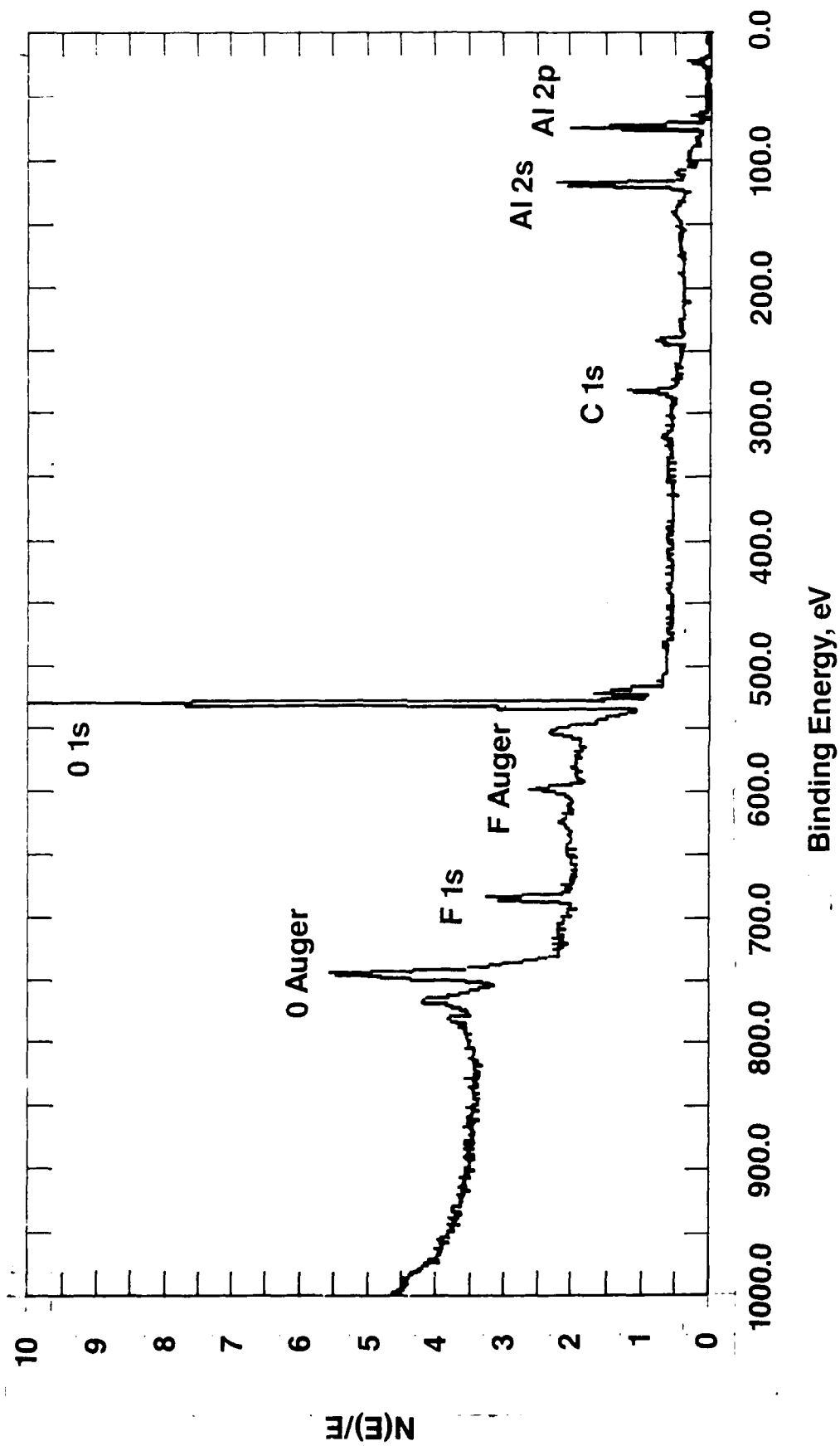
XPS spectrum of the film obtained by decomposition of Al(tfa)_3 for the substrate temperature

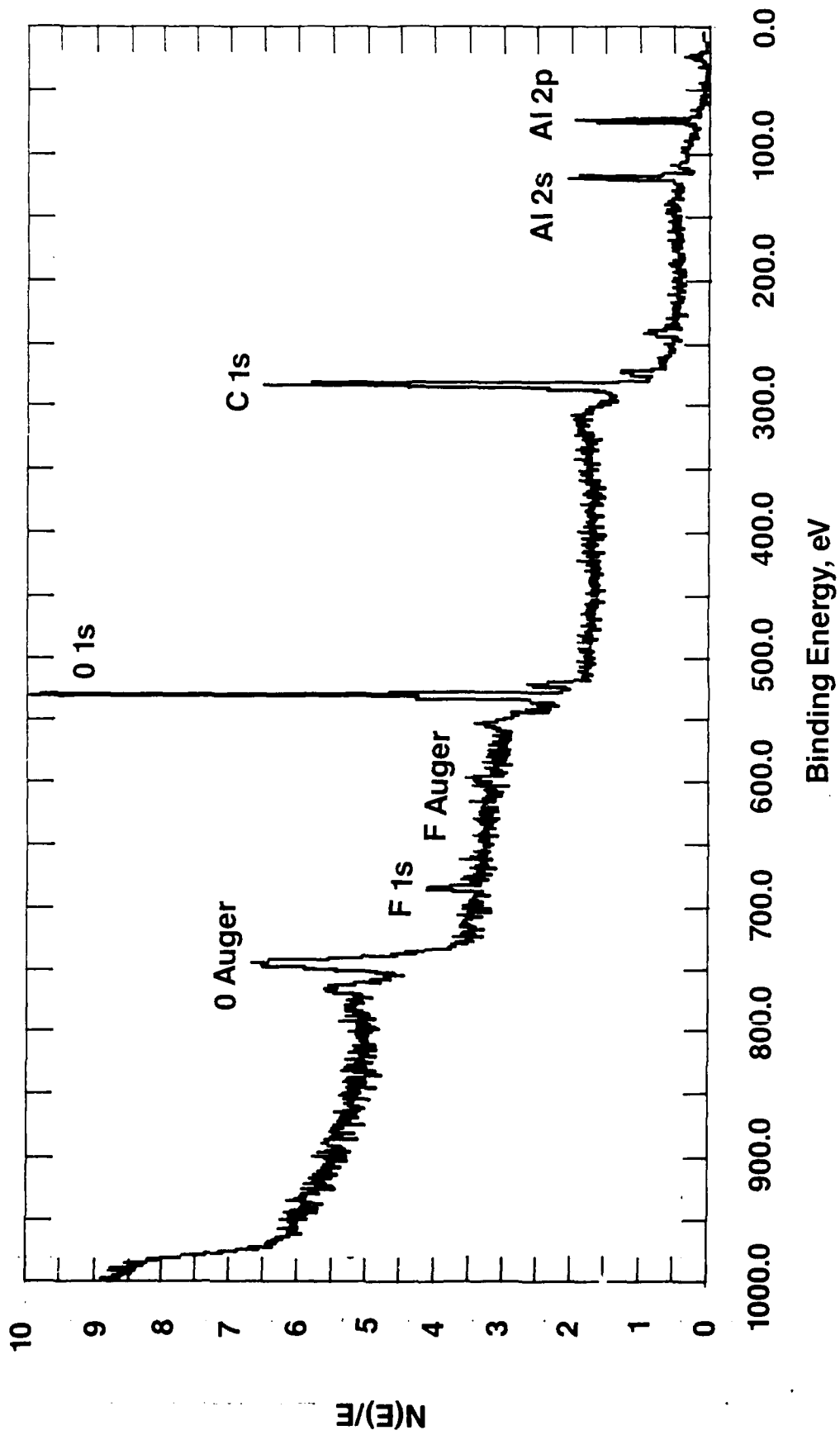
a) $T_{\text{sub}} = 350^\circ\text{C}$ and

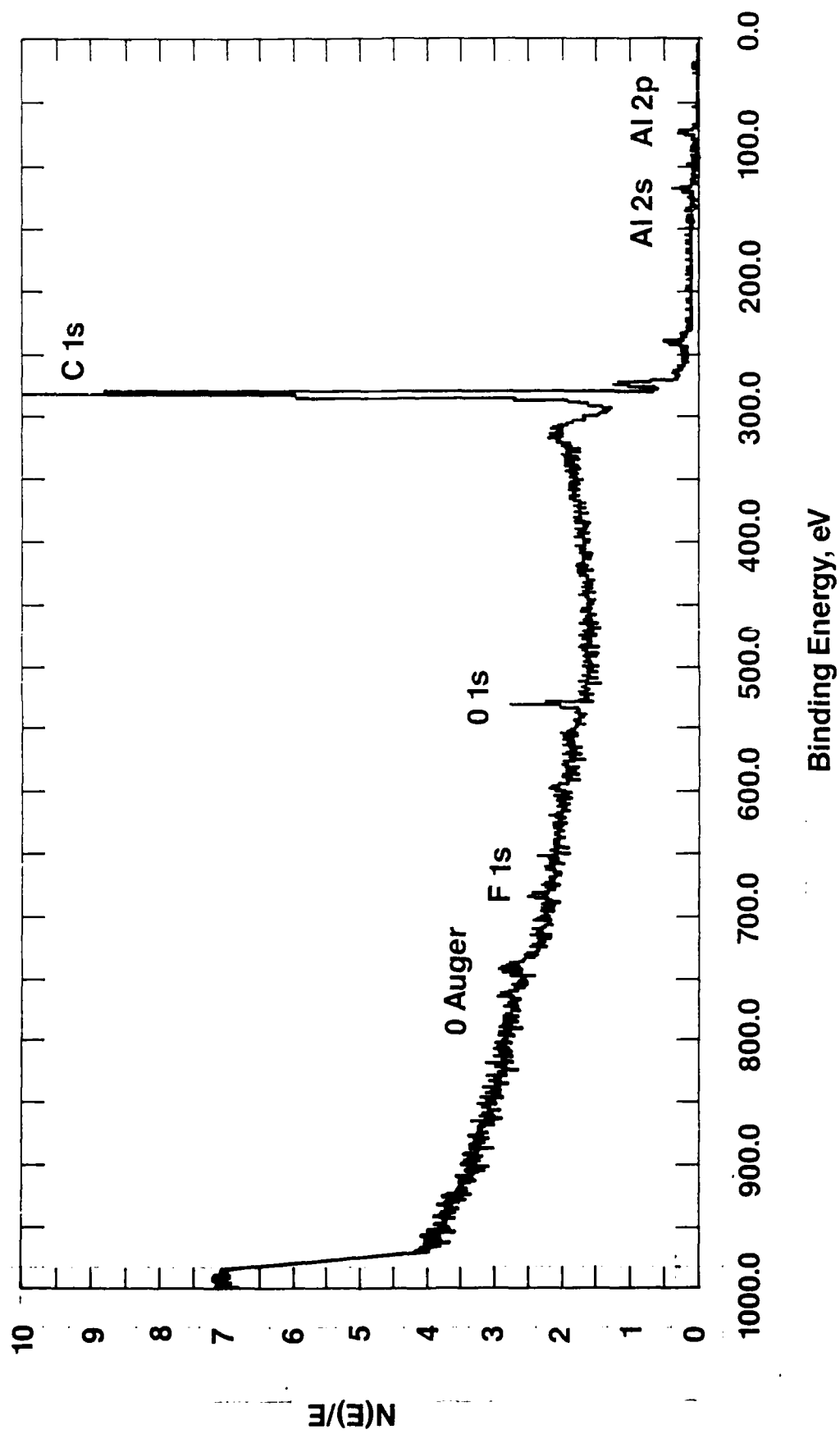
b) $T_{\text{sub}} = 450^\circ\text{C}$

(after sputter-cleaning of the film).









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