# The Investigation of the Exposure Time Effects with Pressure in the Atomic Layer Deposition Process over Micro-Trench Surface

Olufunsho Oladipo Olotu

University of Johannesburg/ Mechanical Engineering Science, Johannesburg, South Africa Email: lotuzlynx@yahoo.com

Rigardt Alfred Maarten Coetzee, Peter Apata Olubambi<sup>a</sup> and Tien-Chien Jen University of Johannesburg/Mechanical Engineering Science|Metallurgy<sup>a</sup>, Johannesburg, South Africa Email: lotuzlynx@yahoo.com, rcoetzee@uj.ac.za, polubambi@uj.ac.za

and tjen@uj.ac.za

Abstract— Tremendous interest has been drawn towards the atomic layer deposition (ALD) as an ultrathin film deposition technique to deposit conformal quality films in the micro-semiconductor industry. However, a further grasp of the ALD process is mandatory to increase the probability of the industry in achieving the necessary improvement which can have a substantial impact on device performance. However, little has been done to investigate the specific effects of the operation pressure on the mechanistic, species transport and reaction rates. Moreover, the effects of these prior properties due to the change of pressure on a complex micro-trench substrate have not been studied yet. Hence, this study focuses on numerically investigating the effect between one and 10 torr operating pressure in the ALD process using the computational fluid dynamic approach. A two-dimensional numerical simulation of the Al<sub>2</sub>O<sub>3</sub> ALD thin film fabrication process over a surface with micro-trenches on a substrate is studied. Trimethyl-Aluminium and Ozone were utilized as the metal and oxidation source reactants. To assist the precursor reaction process a 2.5 second exposure time is added within the ALD sequence. The findings illustrated the fluid flow velocity, mass fraction, and growth of the thin-film process. The evaluations unveil close comparison to literature.

*Index Terms*—atomic layer deposition, computational fluid dynamic, exposure time, nanotechnology, semiconductor, thin film.

# I. INTRODUCTION

The fabrication of films with guaranteed control over the thickness, conformal, uniform and compactness on intricate topography have remained a critical task in present days. Atomic layer deposition (ALD) as a nanofabrication technique has attracted the awareness of the nanotechnology industry to the potency of this method to develop films with these desired features. A combination of the distinctive features results in the influence of some preferred material characteristics for the fabrication of the thin film over complex geometry typically found in semiconductors, sensors, photovoltaic cells, electronic display devices, fuel cells, among others.

Basically, ALD is a self-inhibiting and heterogeneous surface chemical reaction of precursors that is introduced sequentially into the reactor [1]. ALD technique fundamentally involves four steps, pulsing of reactive precursors that are separated temporally by purging inertgas to nullify homogenous reaction among the precursors [2, 3]. Additionally, an exposure time interval after a pulse can be used to improve the film growth quality.

While the miniaturization of electronic devices is progressively introduced and suitable for the modern-day use, it is, however, obligatory to comprehend the nanofilm deposition process with the anticipation to minimize defects in the product. The deposition of materials on surfaces with intricate geometry, such as trenches and pores, with unique features have been a major challenge in the microelectronic industries. The ALD process depends greatly on the parameters of time, sufficient precursor mass, velocity, pressure, and substrate temperature, especially around such complex structures. Considering that the future application of ALD would be found within these complex three-dimensional structures, the behaviour and time evolution within these complex topologies could be of great importance.

Higher operating pressure is reported to have a significant impact on the deposition rate [4, 5]. Growth rate is dependent on the system pressure which causes an increase in the gas velocity. This finding was illustrated to be as a result of the swift availability of the gases at the substrate surface [2]. It was suggested that in the ALD process that the velocity increase by virtue higher pressure could be responsible for the reduction in the boundary layer thickness formed by desorption species. Furthermore, more reactants are then allowed to react with available sites [3]. In spite of this, precursors are expensive, and wastage is necessarily needed to be

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abated which have so far led to the introduction of exposure time to allow more reacting duration without an increase of precursor dosage. The fundamental understanding of the exposure time influences in the ALD process has rarely been communicated.

This work investigates the ALD process in different operating pressure over micro-trenches with a depth 300  $\mu m$  and an aspect ratio of 2:1, along with the introduction of exposure time. The deposition of Al<sub>2</sub>O<sub>3</sub> film is investigated using Trimethyl-Aluminium (TMA) as the metal source and Ozone (O<sub>3</sub>) as the oxidation source precursors, and Argon (Ar) as the inert purging gas. The ALD reaction phase, half reactions and reaction data are illustrated in Table I.

TABLE I. GASEOUS, SITE AND BULK REACTION MECHANISMS AT THE SUBSTRATE SURFACE

ALD Chemical Reaction	
Phase	Formula
G1	$O_{3} + M_{kb}^{kf}O_{2} + O + M$ $A_{r} = \frac{4.51e^{9}m^{3}}{mol}, s, E_{r} = 100  416 \frac{J}{mol}, \beta = 0$
G2	$O_{3\frac{kf}{kb}}O_{2} + O$ $A_{r} = \frac{2.97e^{7} m^{3}}{mol} \cdot s, E_{r} = 25104 \frac{J}{mol}, \beta = 0$
<b>S</b> 1	$Al(CH_3)_3 + O^* \xrightarrow{kf} Al(CH_3)_2^* + 0.5C_2H_6$ $\gamma = 0.1$
<b>S</b> 2	$2Al(CH_3)_2^* + 0 \xrightarrow{kf} Al(CH_3)OAl(CH_3)^* + C_2H_6$ $\gamma = 1$
<b>S</b> 3	$\begin{array}{l} 0.5Al(CH_3)OAl(CH_3)^* + O \xrightarrow{k_f} O^* + 0.5C_2H_6 + 0.5(Al_2O_3)^B \\ \gamma = 1 \end{array}$

#### II. SIMULATION ENVIRONMENT

The precursors and purge inert-gas are introduced in sequence into the reactor flow region through the inlet. The flow resembles a typical top-to-bottom type ALD reactor. The flow of precursors and inert purging gases are evenly distributed over the inlet and allowed to fully develop before reaching the substrate and the reactive trenched surface. After the reactive and inert-gases have flown and penetrated into the trenched substrate the excess and by-products are then exhausted past the edges of the trenched substrate towards the outlet of the reactor. The operating pressure within the reactor and pressureoutlet for the process are changed between 1 torr and 10 torr. The gases are assumed to enter at an inlet temperature of  $150 \,^{\circ}$  while the substrate, reactor walls and outlet temperature of 250 °C is used. The TMA and O<sub>3</sub> precursors are both pulsed separately, according to the sequence, into the reactor at 0.085 m/s for 0.2 and 1 second, respectively. While inert-purge gas (Ar) is used to purge the reactor domain at 0.17 m/s for 5 seconds between the pulse and exposure times. For this work the ALD sequence follows in a pulse-exposure-purgeexposure-pulse-exposure-purge manner to form a complete ALD cycle. The velocity ranges used in this investigation are the same as the velocity value in previous literature [6, 7]. The exposure time is simulated by discontinuing the inlet flow for 2.5 seconds.



Figure 1. (A) The designed geometry of the arbitrary reactor (LEFT), and the meshed arbitrary reactor (RIGHT). (B) The designed geometry of half of the substrate and its trenches. It illustrates the points of interest at the top, middle and bottom of the trenches.

The reactor flow region and its 67023 nodes meshed domain is shown in Figure 1. The ALD process within the arbitrary reactor is investigated by numerical simulating the reactor using computational fluid dynamics (CFD) within commercial software packages ANSYS FLUENT and CHEMKINPRO. This transient process is implemented by the coupled algorithm approach to solve for the pressure-velocity coupling of the mass, momentum, energy and species transport. The full mathematical model is similar to that of Pan, *et al.* [3]. The governing equations of the process are hereby expressed as:

$$\frac{\partial \rho}{\partial t} + \nabla \rho \vec{v} = S_m \tag{1}$$

$$\frac{\partial \rho \vec{v}}{\partial t} + \nabla (\rho \vec{v} \vec{v}) = -\nabla P + \nabla \bar{\bar{\tau}} + \rho \vec{g} + \vec{F}$$
(2)

$$\frac{\partial \rho E}{\partial t} + \nabla (\vec{v} (\rho E + \rho)) = \nabla (k_{eff} \nabla T - \sum h_i J_i) + R_r \quad (3)$$

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$$\frac{\partial(\rho Y_i)}{\partial t} + \nabla \rho \vec{v} Y_i = -\nabla \vec{J}_i + R_i \tag{4}$$

The governing equations are temporally and spatially discretized making use of first-order implicit and secondorder upwind schemes, respectively. The transport equation source term is linearized.

The convergence condition of the solution is set to be that the residual values of continuity, energy, and momentum are below the absolute criteria of  $1 \times 10^{-5}$  while the gaseous species residual values are below the absolute criteria of  $1 \times 10^{-4}$ . The transient period of each case is subjected to a time step of  $5 \times 10^{-5}$  s. The sequential pulse-exposure-purge-exposure steps are

controlled by the implementation of a user-defined function (UDF) to pilot the simulation in harmony with the anticipated sequence of the ALD process.

## III. RESULTS AND DISCUSSION

## A. Velocity Flow

The velocity among other parameters plays a vital role to achieving successful ALD process. A refined combination of flow behaviours and available precursor mass can result in conformal step and trench coverage. This section analyses the influence of the pressure on the species flow on and within the trenched substrate.

Nevertheless, the interest of the flow phenomena lies near the substrate surface and the effects the flow is penetrating into the trenches. Similar to the overall velocity flow pattern, the streamlines, Figure 2 shows the velocity increasing as it moves to the substrate edge, away from the stagnant midpoint. The pulsing inlet flow causes a stagnant flow allow diffusion controlled flow into trench 3 (midway). At this trench, flow is subjected to diffusion without a form of recirculation flow. As the flow is redirected towards the substrate edge, the other trenches (trench 1 & 2) is subjected to some form of recirculation flow region, typically seen within sudden volume expansion scenarios [8]. This recirculation region size is increased as the velocity penetration into the trench is increased. At 10 torr the recirculation regions are of more influential than that of one torr. However, at lower pressure the mass fluid flow of TMA penetrates the trenches at a higher velocity. Contrary, the O<sub>3</sub> reveals the opposite, as 10 torr velocity flow penetrates the trenches more than 1 torr system. This is significantly seen within the trench subjected to stagnation flow. This behaviour can be due to the density dependency of the mass species potential to penetrate or resist penetration in its transport in regard to the reaction process [4]. This occurrence can have negative influences towards conformal thin film growth as the transport of the oxygen species is transported at a pace that can cause uneven surface reactions. A similar finding is reported by Mousa, et al. [4]. Purging is seen to create overwhelming recirculation zones when penetrating the trenches at a higher velocity than that of the pulsing sequences. However, the penetration into the trenches in the pulsing period is mostly diffusion dominate due to it lower inject velocity and a shorter time.



Figure 2. Velocity streamlines in trenches at end of TMA pulse for (a) 1 torr, (b) 10 torr, at end of O<sub>3</sub> pulse (c) 1 torr, (d) 10 torr, and at the end of purging (e) 1 torr, (f) 10 torr.

# B. Mass Fraction

In order to be able to evaluate the influence of pressure change in the ALD process, it is necessary to study the transient species mass fraction at specific location on the substrate surface. The TMA dosage increase drastically after the 0.2 second pre-purge sequence of inert-gas Argon. The mass fraction of the TMA and  $O_3$  species at the points shown in Figure 1 (B) is illustrated in Figure 3. It is seen that little change in mass fractions has been observed between the points of the selected top surface and bottom of trench points. Note that these points are chosen to study the mass

fraction distribution during the ALD process. The mass fraction of TMA at all points are observed to peak at about 26% in one torr operating pressure case. While, in the 10 torr test, a maximum of 76.3% TMA mass fraction was reached. A difference of about 50.3% is realized between the peaks of both tests. The significant difference in the TMA mass fraction is that within the 10 torr case, an indication that more proportion of the TMA was delivered to the reactive points, and in a shorter time duration. However, the TMA is also rapidly removed from the reactive sites. This may be beneficial to avoid intermixing, but not if more time is necessary for the precursor to react.



After the TMA injection sequence was completed, the exposure sequence was introduced. It is noticed in both scenarios that the exposure time introduction prompted a linear decrease of the mass fraction at all points. However, the mass fraction linear gradient in the 10 torr test is observed to be steeper than in the other. This behaviour confers prolonged reaction times for the surface reaction in the presence of high amount of the gaseous reactant. While the steeper linear decrent signifies the rapid depletion of reactive TMA availability in the process.

The exposure time appears to play a major role in the process as the  $AlMe_2$  half-reaction takes place within the TMA pulsing-exposure sequence, and the purging is saddled solely with the responsibility of removing the excess reactants and by-products. It is observed that TMA purging was a lot faster in the 10 torr tests compared to the one torr test. This is due to the increased velocity induced by the higher operating pressure.

During the  $O_3$  pulsing sequence, about 38% mass fraction was realized at all the points operating at 1 torr, and about 92% mass fraction was achieved at 10 torr, respectively. Similar to the observation during the TMA pulse and exposure sequence, the  $O_3$  pulse and exposure sequence show similar trends at each point over the trenched surface, respectively. More so, the exposure time is initiated after the  $O_3$  pulse, which happens to induce a linear decline of the  $O_3$  mass fraction. This is seen as favourable, as it gives more reaction time for the O-atoms to react with the reactive absorbed layer by allowing the necessary quantity of the reactant to reach the surface.

In comparison of the mass fraction during the exposure window in both tests, the linear drop in mass fraction is discerning. However, the steep decline after the exposure time in the 10 torr case is concerning. The rapid decline in the mass fraction within the  $O_3$  exposure time in the 10 torr test is as a result of fast transport of the reactant momentum (within the exposure time) and purge velocity. This allows less time for further film deposition to occur where needed and may cause non-conformal growth.

At the end of the cycle, the reactants and by-products are expected to be completely removed from the reactor, and ready for the next cycle. From Figure 3, this is realized with the 10 torr case. However, the 1 torr case shows approximately 2.5% of  $O_3$  on the surface at the end of the cycle. This observation indicates that high operating pressure has a beneficial influence on the process. Moreover, the purging time can be reduced to optimize the ALD cycle duration. Contrary, the 1 torr test displays that additional time is required to ensure no homogeneous interaction between reactive precursors.

## C. Growth and Conformal Analysis

The transient film growth analysis of the ALD process along one cycle period on predefined locations of the trenches is illustrated in Figure 4. From close examination of the growth pattern, it can be observed that superior growth is achieved utilizing a 10 torr case. Similar results from previous experimental literature can validate this scenario [4, 5].

Within this study, the prior parameters of mass fraction have resulted in the 10 torr case to have a much more dominant result than that of the 1 torr case. By observation, the growth can be estimated to have increased to more than twice between the two pressure cases, similarly reported from previous experimental literature [4, 5]. In both cases a self-limiting state has been rapidly achieved. The film growth attained climax at 11.9 Å at point A and the least growth of 1.72 Å for the 10 torr case while it climaxes at 1.15 Å and minimum growth of 0.56 Å in one torr test. In this case it is seen that the near-edged topology of the substrate will be influenced by excessive non-uniformity in respect to the remaining substrate. As such, future applications of ALD should avoid depositing film close to substrate platform edges in which effects such as bottle necking may occur. For this study purposes, these locations trends were purposely withheld.

Although the 10 torr shows superior growth, it can also be deduced that it also converts to inferior conformal growth than that of the 1 torr case considering the margin in the growth achieved at the analysed locations. It is seen that from trench 1, 2 and 3, the conformal growth maximum difference (being wall-to-wall, bottom-to-wall, or bottom-to-surface of the trenches) for the 1 torr case is between 0.141Å, 0.296Å, and 0.356Å, respectively. In contrary, conformal maximum growth difference at 10 torr resulted in a conformal difference of 0.284Å, 0.386Å, and 0.371Å, respectively.



Figure 4. Transient growth trends over the trenched substrate specified points at 250 °C with an operating pressure of 1 torr and 10 torr, respectively.

### IV. CONCLUSION

This study numerically investigates the introduction of 2.5 seconds exposure time in an  $Al_2O_3$  ALD process along with the change of operating pressures between one and 10 torr over micro-trenched substrate in a top-tobottom flow type arbitrary ALD reactor. Using TMA and  $O_3$  reactants,  $Al_2O_3$  thin film is deposited with Ar as the purging gas. The velocity flow, precursor mass fractions, and conformal growth is investigated. It is clearly observed that parameters such as temperature, pressure, precursor concentration, reactor geometry, sequence timing, substrate structure, play a vital role in a successful ALD process, and the combination of the parameters influences the deposition quality.

In cases where deposition is required on surfaces of complex structures such as trenches and holes, the precursors are required to be transported to the targeted surface despite the interference the complexity it possesses. The gas flow is observed to be the key to resolving the issue. An alternating flow pattern with recirculation zones is noted in the tests. The flow into the trenches is dependent on the angle of penetration of the gases and trench position on the substrate. Two distinctive flow behaviours are seen to influence the deposition in the trenches. Velocity and diffusioncontrolled flow both plays a role within the trenches.

Higher pressure displays greater advantage over the mass fracture of the precursor in the process. 76.30% TMA and 92%  $O_3$  peak is realized with higher pressure at all positions on the trenched substrate. The pressure is revealed to have significant influence on the gas transport to reach the trench surface in a much shorter duration. By doing so, giving a higher potential for the reactions to take place. The one torr case is found to be taking much longer for the gas to get to the trench surface. Moreover, an approximate of 2.5%  $O_3$  precursor gas is still lingering in the trenches after purging, which

is an indication that extended purge time is required at the last purge of the cycle. This is mandatory to circumvent mixing of the chemical gases which can result in undesired deposition. In the 10 torr case, the growth is seen to sharply grow within a short window and reaches a self-limiting state just after the  $O_3$  pulse. The growth of the one torr test exhibit gradual development within  $O_3$  pulse and exposure time. Selflimiting state was attained within the exposure time. Higher deposition rate may, however, be achieved with a higher operating pressure, but the conformal growth will be converted inferior to that of a lower operating pressure.

#### CONFLICT OF INTEREST

The authors declare no conflict of interest

### AUTHOR CONTRIBUTIONS

Olufunsho Oladipo Olotu, Main Author, contributed towards conducting the research, wrote the paper, data analysed the data.

Rigardt Alfred Maarten Coetzee, Conducted the research, wrote the paper, data analysed the data, Assisted in research guidance and research idea.

Peter Apata Olubambi research guidance, gave resource, and supervise the research.

Tien-Chien Jen research guidance and research idea, gave resources, wrote the paper, and supervise the research; all authors had approved the final version.

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**Olufunsho O Olotu** is a Doctor of Philosophy candidate in Engineering: Mechanical at the University of Johannesburg (UJ). His research is focused on active materials and thin film Fabrication process (Atomic Layer Deposition) for smart energy applications. He studies closely with a large number of researchers in JENano research group of the ALD Centre of Competence, department of mechanical

engineering Science, School of Mechanical and Industrial Engineering, UJ.



**Rigardt A.M. Coetzee** is currently a PhD candidate under the supervision of Prof Tien-Chien Jen. He was awarded meritorious student of 2014 in the field of Mechanical Engineering from SAIMeCHE. His research is based on the understanding of the ALD process, reactor design and thin film simulation methods. Furthermore, his research expertise also extends to renewable energy systems. He has published peerreviewed journals concerning atomic

layer deposition, computational fluid dynamics, mass transfer, reactor designs, solar thermal systems and multi-purpose solar thermal systems in accredited journals. Mr. Coetzee also contributes to the renewable energy projects and advance manufacturing processes such as the solar car challenge, cold gas dynamic spray, dry drilling, among others at the

University of Johannesburg. He also assisted with the development of South Africa's first ALD Laboratory situated in the University of Johannesburg, Johannesburg, Mr. Coetzee's Email: <a href="mailto:rcoetzee@uj.ac.za">rcoetzee@uj.ac.za</a>.



Professor Peter Olubambi obtained B.Eng (Hons) and M.Eng degrees in Metallurgical and Materials Engineering from the Federal University of Technology, Akure, Nigeria in 1998 and 2002, respectively and a PhD in Metallurgical Engineering from the School of Chemical and Metallurgy Engineering University of the Witwatersrand, South Africa in 2008. His research activities are focused on three key and interrelated fields materials processing; "Powder Metallurgy",

of advanced materials processing; "Powder Metallurgy", "Nanomechanics" and "Tribocorrosion". He is involved in the utilization of innovative powder-based techniques for developing high strength, high-temperature and tribocorrosion resistance advanced metallic alloys and metal-ceramics composites for applications in the aerospace, mines, energy, chemical and allied industries as well as for biomedical applications. He has published above 170 articles in reputable international journals and over 100 peer-reviewed conference proceedings. He is an editorial board member of two international journals, reviewer to many high impact factor journals, and has served as organizing committee members for many local and international conferences. He is a Professor of Materials Engineering, and currently the Head of the School of Mining, Metallurgy and Chemical Engineering at the University of Johannesburg.



**Professor Tien-Chien Jen** joined the University of Johannesburg on August 2015, before that Prof Jen was a faculty member at University of Wisconsin, Milwaukee. Prof Jen received his Ph.D. in Mechanical and Aerospace Engineering from UCLA, specializing in thermal aspects of grinding. He has received several competitive grants for his research, including those from the US National Science Foundation, the US Department of Energy and the EPA. Dr. Jen

has He has brought in \$3.0 million of funding for his research, and has received various awards for his research including the NSF GOALI Award. Prof Jen has recently established a Joint Research Centre with Nanjing Tech University of China on the "Sustainable Materials and Manufacturing." Prof Jen is also the Director of Manufacturing Research Centre of the University of Johannesburg. Meanwhile, SA National Research Foundation has awarded Prof Jen a NNEP grant (National Nano Equipment Program) worth of USD 1.5 million to acquire two state-of-the-art Atomic Layer Deposition (ALD) Tools to be housed in a 220m<sup>2</sup> 10000 level (ISO 7) clean room facility for ultrathin film coating. This ALD Research facility, which is named ALD Center of Competence, will be the first in South Africa and possibly the first in Africa continent.

In 2011, Prof Jen was elected as a Fellow to the American Society of Mechanical Engineers (ASME), which recognized his contributions to the field of thermal science and manufacturing. As stated in the announcement of Prof Jen Fellow status in the 2011 International Mechanical Engineering and Congress Exposition, its states "Tien-Chien Jen has made extensive contributions to the field of mechanical engineering, specifically in the area of machining processes. Examples include, but not limited to, environmentally benign machining, atomic layer deposition, cold gas dynamics spraying, fuel cells and hydrogen technology, batteries, and material processing." Prof Jen has written a total of 300 peer-reviewed articles, including over 130 peer-reviewed journal papers, published in many prestigious journals including International Journal of Heat and Mass Transfer, ASME Journal of Heat Transfer, ASME Journal of Mechanical Design and ASME Journal of Manufacturing Sience and Engineering, Journal of Additive Manufacturing. He also has written eleven (12) chapters in special topics book. For example, Prof Jen has a chapter in Numerical Simulation Proton Exchange Membrane Fuel Cell, published by WIT Press, and another chapter in Application of Lattice Boltzmann Method in Fluid Flow and Heat Transfer in Computational Fluid Dynamics -Technology and Application.