Atomic layer deposition: a true and enabling nanotechnology

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Outline

1. Introduction
2. Atomic layer deposition (ALD)
3. ALD materials & Applications of ALD
4. Atomic layer etching (ALE)
5. Summary
Evolution of personal devices

- All-Weather Stereo
- AM/FM Clock Radio
- Stereo Phones
- Micro-Thin Calculator
- 284-Based PC
- Mobile Cellular Tele.
- Radar Detector
- Portable CD Player
- Desktop Scanner
- 3-Way Speaker
- VHS Camcorder
- Speed-Dial Phone
- Mobile CB
- Tape Recorder
- Phone Answerer
Scaling in semiconductor industry

- Integrated photonics
- Artificial intelligence
- Quantum computing

2020s
FinFET transistors

**Precise** and **conformal** deposition of high-\(k\) metal gate (HKMG) stack

Intel 22 nm
FinFET transistors

Precise and conformal deposition of high-k metal gate (HKMG) stack
Evolution of data storage

5 MB IBM hard disk - 1956
3D NAND memory

Precise and conformal deposition of oxide layers and charge trap layer

Samsung V-NAND 48L
3D NAND memory

Precise and conformal deposition of oxide layers and charge trap layer
Atomic layer deposition: an enabling technology

**Precise** and **conformal** deposition of ultrathin films with Ångstrom-level thickness control at (fairly) low temperatures

atomic-layer by atomic-layer
Outline

1. Introduction

2. Atomic layer deposition (ALD)
   • What is ALD?
   • Mechanism of ALD: the prototypical Al₂O₃ case
   • ALD process development, ALD history, ALD tools

3. ALD materials & Applications of ALD

4. Atomic layer etching (ALE)

5. Summary
What is ALD?

ALD is a **CVD-like technique** in which film growth takes place by repeating cycles. Each cycle consists of 2 (or more) “half-reactions” separated by purge/pump steps. The “half-reactions” are **self-limiting** leading to well-defined amount of added material.

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What is ALD?

Films grown by repeating cycles until desired thickness is reached
In every cycle 1 atomic layer is added with a perfect repeatability

Parameter for “growth rate” is “growth-per-cycle” (GPC)

Mostly expressed in thickness-per-cycle

Typically 0.01 – 0.15 nm/cycle

- Al₂O₃
- SiO₂
- Ta₂O₅
- ZnO
- TiO₂
Key merits of ALD

Key merits of ALD:  
1) Ångström-level growth control  
2) Unparalleled uniformity  
3) Excellent conformality  
4) Low substrate temperature (< 500 °C)

Uniformity and conformality

(a) Surface controlled

(b) Flux controlled

ALD

CVD
PVD
PECVD
Materials prepared by ALD

www.AtomicLimits.com
And here it is... the online ALD database

* A website where you can easily search, browse, and add ALD processes

March 20, 2019 / 3 Comments
### List of processes

<table>
<thead>
<tr>
<th>Z</th>
<th>Material</th>
<th>Reactant A</th>
<th>Reactant B</th>
<th>Reactant C</th>
<th>Further reactants</th>
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<td>Scarl et al. ADD</td>
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</table>
Self-limiting behavior for ALD Al\textsubscript{2}O\textsubscript{3}

(a) Growth per Cycle (nm/cycle) vs. Dose time (ms)

(b) Growth per Cycle (nm/cycle) vs. Plasma time (s)

Thermal ALD

Plasma ALD

\((\text{H}_2\text{O})\)

\((\text{O}_2\text{ plasma})\)
ALD of Al$_2$O$_3$ — Mass spectrometry

Gas phase reaction products

\[ \text{Mass spectrometry signal (A)} \]

\[ \begin{align*}
\text{Time (s)} & \quad \text{0} & \quad \text{25} & \quad \text{50} & \quad \text{75} & \quad \text{100} \\
\text{H}_2\text{O} & \quad \text{10}^{-11} & \quad \text{10}^{-9} & \quad \text{10}^{-7} & \quad \text{10}^{-5} & \quad \text{10}^{-3} \\
\text{CH}_4 & \quad \text{10}^{-11} & \quad \text{10}^{-9} & \quad \text{10}^{-7} & \quad \text{10}^{-5} & \quad \text{10}^{-3}
\end{align*} \]

\[ \begin{align*}
\text{Al(CH}_3\text{)}_3 & \quad \text{H}_2\text{O} & \quad \text{Al(CH}_3\text{)}_3 & \quad \text{H}_2\text{O} & \quad \text{Al(CH}_3\text{)}_3 & \quad \text{H}_2\text{O} & \quad \text{Al(CH}_3\text{)}_3 & \quad \text{H}_2\text{O} \\
\text{H}_2\text{O} & \quad \text{10}^{-11} & \quad \text{10}^{-9} & \quad \text{10}^{-7} & \quad \text{10}^{-5} & \quad \text{10}^{-3} & \quad \text{10}^{-11} & \quad \text{10}^{-9} & \quad \text{10}^{-7} & \quad \text{10}^{-5} & \quad \text{10}^{-3}
\end{align*} \]

- Al(CH$_3$)$_3$ dosing: CH$_4$
- H$_2$O dosing: CH$_4$

ALD of Al$_2$O$_3$ — \textit{In situ} infrared spectroscopy

Monitoring surface groups

Al(CH$_3$)$_3$ dosing: \textit{-OH} replaced by \textit{-CH$_3$}

H$_2$O dosing: \textit{-CH$_3$} replaced by \textit{-OH}
ALD of $\text{Al}_2\text{O}_3$ — Ellipsometry & RBS

(a) Growth rate (nm/cycle)

(b) #Al atoms per cycle ($10^{14} \text{cm}^{-2}$)

Substrate temperature ($^\circ\text{C}$)


(Ideal) ALD temperature window

- Condensation
- Decomposition
- Low reactivity
- Desorption

Temperature

Growth per cycle

ALD
**Plasma-enhanced ALD**

**Merits** of plasma ALD:
- Improved material properties
- Deposition at reduced temperatures
- Increased choice of precursors/materials
- Good control of film composition
- Increase growth rate
- More versatility in general

**Challenges** of plasma ALD:
- Limited conformality
- Plasma-induced damage
- ...

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10 steps to successfully develop an ALD process

Atomic Layer Deposition Process Development

10 steps to successfully develop, optimize and characterize ALD recipes

10 steps to successfully develop, optimize and characterize ALD recipes

Atomic layer deposition (ALD) is a surface-controlled thin film deposition technique that can enable ultimate control over the film thickness, uniformity on large-area substrates and conformality on 3D (nano)structures. Each ALD cycle consists of at least two half-cycles (but can be more complex), containing a precursor dose step and a co-reactant exposure step, separated by purge or pump steps (see Figure 1). Ideally, the same amount of material is deposited in each cycle, due to the self-limiting nature of the reactions of the precursor and co-reactant with the surface groups on the substrate. By carrying out a certain number of ALD cycles, the targeted thin film thickness can be obtained.

Figure 1. Schematic illustration of a typical ALD cycle consisting of two half-cycles. Sequential precursor and co-reactant doses are separated by purge or pump steps, leading to self-limiting film growth. The ALD cycle consists of at least two half-cycles (but can be more complex), containing a precursor dose step and a co-reactant exposure step, separated by purge or pump steps. Ideally, the same amount of material is deposited in each cycle, due to the self-limiting nature of the reactions of the precursor and co-reactant with the surface groups on the substrate. By carrying out a certain number of ALD cycles, the targeted thin film thickness can be obtained.

Although the principle of ALD appears to be relatively simple, developing an ALD process is not necessarily a trivial task and it requires attention. Here, steps are suggested and described to successfully set up an ALD process. Although the steps are roughly ordered chronologically, repetition of some of the steps might be needed at a later stage. In summary, it is advised that the following steps are taken when developing an ALD process:

1. Reactant selection: Which precursor and co-reactant will be used for the process?
2. Composition: Is the deposited film of the expected material composition?
3. Thickness control: Does the film thickness proceed linearly with cycles?
4. Saturation: Are the precursor, co-reactant and purge steps in saturation?
5. Properties: Does the material have the desired material properties?
6. Temperature: Is ALD behavior observed for a range of deposition temperatures?
7. Uniformity: Does the film have the same thickness everywhere on the substrate table?
8. Conformality: Is the film thickness the same everywhere along 3D structures?
9. Nucleation: Does the initial growth on the substrate differ from steady state growth?
10. Other aspects: Are aspects such as safety, stability, reproducibility, etc. met?

1. Precursor and co-reactant selection

Before setting up an ALD process, a suitable combination of precursor and co-reactant has to be decided on. Most importantly, the precursor and co-reactant molecules should consist of the appropriate elements, to obtain a material of the desired composition. In addition, they need to be reactive towards the surface groups present after the preceding subcycle, and in turn result in reactive surface groups after dosing. Moreover, volatility, thermal stability and reactivity need to be sufficiently high. Other requirements include availability and safety of the chemicals. Furthermore, reactor constraints and the application of the ALD film have to be considered, since they can limit the choice of possible chemicals. In addition to selecting the precursor it has to be determined how the precursor will be delivered to the chamber: major stream, carrier gas assisted (i.e. carrier gas flowing over the precursor), bubbling (i.e. carrier gas flowing through the precursor), etc.

2. Chemical composition

Soon after deposition of the first ALD film, it is valuable to check if the grown material consists of the intended elements. Common ways to investigate the chemical composition are Xray photoelectron spectroscopy (XPS) and Rutherford backscattering spectroscopy (RBS), although XPS is typically more readily available. In case the material should be conductive, a simple four-point probe conductivity measurement can test the materials high purity. Furthermore, a quick assessment of the refractive index can also indicate whether the desired material is obtained. If the deposited material is very different from the expectation, it might be good to reconsider Step 0, since proceeding can be a waste of time. However, in many cases, optimization of the deposition temperature or dosing and purge times can lead to improvement of the material composition. It is important to realize that the chemical composition and stoichiometry will determine the final material properties.

3. Thickness control

An important characteristic of ALD is the deposition of the same amount of material in each cycle, allowing for ultimate thickness control. To confirm this, the thickness or material increase is to be determined as a function of cycles, which is called the growth per cycle (GPC). Determining the GPC can be done both in situ, by following the material increase during deposition, as well as ex situ, by depositing multiple samples with varying number of cycles. Typically, the film thickness is measured (e.g. by spectrometric ellipsometry), although alternative ways to check linear growth are by determining the number of deposited atoms (by Rutherford backscattering spectroscopy) or deposited mass (e.g. by quartz-crystal microbalances). Figure 2 shows a typical example of the film thickness increasing linearly with the number of ALD cycles. Note that the initial growth on the substrate might occur differently than at a later stage, as will be discussed in Step 8. For this reason the focus should lie on relatively thick films, with thicknesses over ~15 nm.
History of ALD

1965
Aleskovskii and Kolt’sov
First abstract on Molecular Layering

1983
Pilot production Electro-luminescent Displays

1984
Suntola
First patent on Atomic Layer Epitaxy

1985
Atomic Layer Epitaxy Work on III-V’s

1989
First report on (prototypical) ALD of Al2O3

1990
First International Atomic Layer Epitaxy conference

1991
First report on Molecular Layer Deposition

1998
Report on ALD films in DRAM devices

2007
Intel launches High-k metal gate with ALD Hf-based oxide

2008
Kodak reintroduces Spatial ALD

2012
Pilot production PERC solar cells with Al2O3

2016
First Workshop on Area Selective Deposition

Atomic Layer Deposition (ALD)
Atomic Layer Epitaxy (ALE)
Molecular Layering (ML)
Atomic layer CVD (ALCVD)

To appear on www.AtomicLimits.com
Atomic layer deposition (ALD) equipment

Research & Development

High volume manufacturing

ARRADIANCE
beneq
Lam RESEARCH
lotus
Veeco
CNT
NCD
ASM
LEVITECH
SENTECH
Picosun
Kurt J. Lesker Company
Applied Materials
ALD nanosolutions
OXFORD INSTRUMENTS
TEL
TOKYO ELECTRON
Ideal Energy Equipment (Shanghai) Ltd

Outline

1. Introduction
2. Atomic layer deposition (ALD)
3. ALD materials & Applications of ALD
   • Oxides, nitrides, metals, fluorides, sulfides
   • From nanoscale applications to large-area applications
4. Atomic layer etching (ALE)
5. Summary
ALD of metal oxides

1. $\text{Al(CH}_3\text{)}_3$
2. $\text{SiH}_2\{\text{N(C}_2\text{H}_5\}\}_2$
3. $\text{Ta}\{\text{N(CH}_3\text{)}_2\}_5$
4. $\text{Zn(CH}_2\text{CH}_3\}_2$
5. $\text{Ti(Cp^*)}(\text{OCH}_3\}_3$

$\text{H}_3\text{C} \quad \text{Al} \quad \text{CH}_3$

$\text{Si} \quad \text{H} \quad \text{H}_2\text{C} \quad \text{Zn} \quad \text{H}_2\text{C} \quad \text{CH}_3$

$\text{H}_3\text{C} \quad \text{Ti} \quad \text{CH}_3$

$\text{H}_3\text{CO} \quad \text{OCH}_3$

$\text{H}_3\text{CO} \quad \text{OCH}_3$

$\text{H}_2\text{O}$, $\text{O}_3$ or $\text{O}_2$ plasma
**FinFET transistors with high-\( k \) metal gate (HKMG)**

- Intel 22 nm
- Intel 10 nm

- high-\( k \) gate dielectric = \( \text{HfO}_2 \)
- metal gate = \( \text{TiN} \)
FinFETs in Apple A11 and A12 chips

Topview of SRAM of A11 and A12

<table>
<thead>
<tr>
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<th>iPhone 8 (A11)</th>
<th>iPhone Xs (A12)</th>
<th>减减(%)</th>
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<tr>
<td>Length (nm)</td>
<td>315.2</td>
<td>244.3</td>
<td>-</td>
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<tr>
<td>Width (nm)</td>
<td>135.6</td>
<td>114.2</td>
<td>-</td>
</tr>
<tr>
<td>Gate pitch (nm)</td>
<td>65.62</td>
<td>55.76</td>
<td>15.1%</td>
</tr>
<tr>
<td>Unit cell (um²)</td>
<td>0.0427</td>
<td>0.0278</td>
<td>34.9%</td>
</tr>
</tbody>
</table>

A11 – Iphone 8 - TSMC 10 nm process
A12 – Iphone Xs - TSMC 7 nm process
The biggest ALD market - patterning

Self-aligned double patterning (SADP) – since 22 nm node

Pitch

Line-width

ALD of SiO₂ (50 °C)

Double pitch

Line-width = thickness ALD film

1 Litho step!

(a) Patterned photoresist
(b) Spacer deposition
(c) Anisotropic etch step
(d) Photoresist removal
(e) Anisotropic etch step
(f) Spacer removal

See blog about patterning on www.AtomicLimits.com
**SADP – SAQP – SAOP  ***  193i or EUV litho**

**Self-aligned quadruple patterning (SAQP)** – since 10 nm node for finFETs

193 nm immersion with **self-aligned octuple patterning (SAOP)** or EUV with SADP

SAOP (with 3 ALD steps) outperforms EUV-based litho in terms of roughness.

Arstechnica.com & IBM research
ALD market development

ALD market size has grown tremendously over last decade, even beating predictions!
Excellent conformality of ALD

Encapsulation of defects by $\text{Al}_2\text{O}_3/\text{TiO}_2$ “nanolaminate”
Excellent conformality of ALD

Collaboration with Erik Bakkers et al. (TU/e)
ALD for nanowire devices

Surface passivation of semiconducting nanowires (for LEDs, nanolasers, solar cells)

ALD for MEMS-type devices

Mechanically tunable photonic crystal with two membranes suffering from stiction

15 nm of Al₂O₃ prevents stiction of membranes when actuated

Petruzell et al., Optics Express 28, 3882 (2018)
Advanced ALD cycles (beyond AB-type cycles)

(a)  Regular (AB)$_m$

(b)  Multistep (ABC)$_m$

(c)  Supercycle ((A1B1)$_m$(A2B2)$_n$)$_x$

ALD of doped materials: e.g., Al-doped ZnO

Increasing the carrier density by decreasing the cycle ratio (more supercycles)

TEM image of ZnO:Al using TMA (3 supercycles)

Many more doped materials proven, for example TCOs:
- ZnO:X (X = Al, B, Ga), In$_2$O$_3$:X (X=H, Sn), TiO$_2$:X (X=Nb)

Digital doping control

\[ N_e = 7 \times 10^{19} - 1 \times 10^{21} \text{ cm}^{-3} \]
ALD of metals: initial growth and nanoparticles

ALD of Pt from MeCpPtMe$_3$ and O$_2$ on Al$_2$O$_3$ substrate (300 °C)

Mackus et al., Chem. Mater. 25, 1905 (2013)
ALD of nanoparticles

ALD of Pd from Pd(hfac)$_2$ and H$_2$ plasma and O$_2$ plasma (ABC-type cycle, 100 °C)

Nanoparticle size distribution (monodisperse, 1 - 5 nm diam.)

ALD of core-shell nanoparticles

HAADF-STEM imaging

EDS mapping

Weber et al., Chem. Mater. 24, 2973 (2012)
ALD for catalysis

Nanoparticles with ALD overcoat do not sinter and don’t suffer from no heavy coke formation

Catalytic oxidative dehydrogenation of ethane
\[ \text{C}_2\text{H}_6 + \text{O}_2 \rightarrow \text{C}_2\text{H}_4 + \text{CO} + \text{CO}_2 + \text{H}_2\text{O} + \text{H}_2 \]
ALD of 2D metal sulfides

ALD of MoS$_2$ from (N$^{t}$Bu)$_2$(NMe$_2$)$_2$Mo and H$_2$S plasma at < 450 °C

Initially formation of horizontal layers and subsequent formation of vertical fins
ALD of 2D metal sulfides

ALD of $\text{MoS}_2$ from $(\text{N}^t\text{Bu})_2(\text{NMe}_2)_2\text{Mo}$ and $\text{H}_2\text{S}$ plasma at $< 450 \, ^\circ\text{C}$

Initially formation of **horizontal layers** and subsequent formation of **vertical fins**

For 10 cycles (~10 Å nominal) **monolayer signature** (Raman & photoluminescence)

ALD for silicon solar cells

Al$_2$O$_3$ nanolayers (\(~5\) nm) lead to excellent passivation of silicon surfaces


International Technology Roadmap for Photovoltaic, 9th ed. – September 2018
High-volume ALD in photovoltaics

World market share (%)

- PECVD AlOx + capping layer
- ALD AlOx + capping layer
- Other

Batch ALD Al₂O₃

Spatial ALD Al₂O₃

PECVD Al₂O₃

Spatial ALD for photovoltaics

Wafer move from one side to the other (up to 6000 wafers/hr)

ALD process at atmospheric pressure

Levitech B.V. - www.levitech.nl

Note: spatial ALD also provided by SolayTec B.V. – www.solaytec.com
Other potential large application areas

- Silicon & perovskite solar cells
  - Flexible solar cells...
  - Tandem cells...

- Displays
  - From TFEL displays (mid 80’s)
  - To flexible (smartphone) displays...

- Li-ion batteries
  - ALD on particles...
  - Solid-state batteries...

- Catalysis
  - Model studies...
  - Microreactors...?
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4. Atomic layer etching (ALE)
   - What is ALE?
   - Anistropic and isotropic ALE
5. Summary
Atomic layer deposition & etching (ALD & ALE)

Atomic layer deposition:

Deposit or etch layer-by-layer

Atomic layer etching:

Deposit or etch layer-by-layer
Atomic layer deposition & etching (ALD & ALE)

Half-reaction A
1. Adsorption (Self-limiting)
2. Purge

Half-reaction B
3. Activation (Self-limiting)
4. Purge

One ALD Cycle

One ALE Cycle

ALE with ion bombardment (anisotropic etching)

- Example, Si etching with Cl\textsubscript{2} gas and Ar plasma (Ar\textsuperscript{+})

- Precise ion energy control to be within ALE window
Atomic layer deposition & etching (ALD & ALEt)

(a) Uniformity → Conformality → Growth control

(b) Uniformity → Isotropic Etch → Etch control → Anisotropic Etch → Atomic layer removed

History of ALE

1988
ALE of diamond

1989
Directional ALE III-V (GaAs)

1990
Directional ALE Si with F

1991
Directional ALE Si with Cl₂

1993
Isotropic ALE III-Vs

1994
Isotropic ALE SiO₂ with Salts

2001
ALE with neutral beams

2007
Concept of directional ALE SiO₂ with fluorocarbon deposition

2008
Directional ALE High-k Dielectrics

2013
ALE of Si in commercial reactor

2014
First ALE Workshop

2015
Al₂O₃ First isotropic thermal ALE process

2016
ALE of SiO₂ adopted in production

2017
ALE of oxides, nitrides, metals. Many more isotropic ALE processes

Atomic Layer Etching (ALE)
Atomic Layer Etching (ALEt)
Digital etching
Layer by layer etch
Molecular layer etching...

To appear on www.AtomicLimits.com
FinFET transistors

Precise and conformal deposition of high-k metal gate (HKMG) stack
Gate-all-around FETs – sub-5 nm node

IBM sub-5 nm

IBM, Global Foundries, Samsung – IBM News Room, June 5, 2017

See also blog about atomic-scale processing on www.AtomicLimits.com
Gate-all-around FETs – sub-5 nm node

IBM sub-5 nm

Si/SiGe stack

Fin etching (anistropic)

SiGe etching (istropic)
Summary

- Atomic layer deposition (ALD)
  - Oxides, nitrides, metals, sulfides, fluorides, ...
  - Thin films, nanoparticles, 2D materials, ...

- ALD as true & enabling nanotechnology

- ALE an “etch-counter” part for ALD – still in its infancy but rapidly emerging!
ALD Academy (www.ALDacademy.com)

Mission: educate students and professionals on the principles, applications and future advancements of ALD and related atomic-scale processes.

Prof. Gregory Parsons  
Dept. of Chemical and Biomolecular Engineering  
North Carolina State University

Prof. Erwin Kessels  
Dept. of Applied Physics  
Eindhoven University of Technology

New event:

ALD Academy on Atomic Layer Etching & Deposition

January 14/15, 2020 - Eindhoven

Register at www.ALDacademy.com
Acknowledgments

PMP group – January 15, 2019