Atomic Layer Deposition at the Stanford Nanofabrication Facility

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NNIN ALD Workshop
## ALD Equipment @ SNF
### all Cambridge Nanotech

<table>
<thead>
<tr>
<th>Equipment</th>
<th>Online</th>
<th>Cleanliness</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>savannah</td>
<td>Fall 2009</td>
<td>MOS until summer 2012 now open</td>
<td>Metal oxides only currently. Previously a lot of metal nitride work</td>
</tr>
<tr>
<td>fiji1</td>
<td>Summer 2011</td>
<td>MOS clean</td>
<td>One half of a F202 system; metal oxides, metal nitrides, and BEOL MOS metals</td>
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<tr>
<td>fiji2</td>
<td>Summer 2012</td>
<td>Open to “all materials”</td>
<td>Heavy utilization (80% or more of 24/7); metal oxides, metal nitrides, metals</td>
</tr>
<tr>
<td>fiji3</td>
<td>Winter 2013</td>
<td>Open to “all materials”</td>
<td>Oxide only processing; also to meet capacity demand of fiji2</td>
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</tbody>
</table>
| Savannah-mvd    | Winter/Spring 2013| Open to “all materials”          | - Incorporated into a glovebox  
- Plasma cleaner also in glovebox  
- Molecular Vapor Deposition |

- Savannah-mvd - Incorporated into a glovebox
- Plasma cleaner also in glovebox
- Molecular Vapor Deposition
Films available @ SNF – Extensive Characterization

• $\text{Al}_2\text{O}_3$
  – Thermal (TMA + $\text{H}_2\text{O}$)
  – Plasma (TMA + $\text{O}_2$ plasma)

• $\text{HfO}_2$
  – Thermal (TDMA-Hf + $\text{H}_2\text{O}$)
  – Plasma (TDMA-Hf + $\text{O}_2$ plasma)

• $\text{TiO}_2$
  – Thermal (TDMA-Ti + $\text{H}_2\text{O}$)
  – Plasma (TDMA-Ti + $\text{O}_2$ plasma)

• $\text{ZrO}_2$
  – Thermal (TDMA-Zr + $\text{H}_2\text{O}$)
  – Plasma (TDMA-Zr + $\text{O}_2$ plasma)

• $\text{SiO}_2$ (Plasma 3DMAS + $\text{O}_2$ plasma)

• $\text{Pt}$
  – Thermal (Me(CpMe)Pt + $\text{O}_2$)
  – Plasma (Me(CpMe)Pt + $\text{O}_2$ plasma (+ $\text{H}_2$ plasma))

• $\text{TiN}$
  – Thermal (TDMA-Ti + $\text{NH}_3$)
  – Plasma (TDMA-Ti + $\text{N}_2$ plasma)
  – Plasma (TDMA-Ti + $\text{NH}_3$ plasma – early stages)

• $\text{ZnO}$
  – Thermal (DEZ + $\text{H}_2\text{O}$)

• $\text{Ta}_2\text{O}_5$
  – Thermal (TDEMATB-Ta + $\text{H}_2\text{O}$)
  – Plasma (TDEMATB-Ta + $\text{O}_2$ plasma)
Films available @ SNF – Demonstrated Deposition

- HfN (Hf₃N₄ really)
  - Thermal (TDMA-Hf + NH₃)
  - Plasma (TDMA-Hf + N₂ plasma)

- WOₓ
  - Thermal (BTDBMA-W + H₂O)
  - Plasma (BTDBMA-W + O₂ plasma)

- WN
  - Thermal (BTDBMA-W + NH₃)
  - Plasma (BTDBMA-W + N₂ plasma)

- Ru
  - Thermal ((CpEt)Ru + O₂)

- SnO
  - Thermal (TDMA-Sn + H₂O₂)

- InO
  - Thermal (CpIn + H₂O)

- ITO (see above)

- Y₂O₃ (!)
  - Thermal (Me(3MeCp)Y + H₂O or Me(2MeEtCp)Y + H₂O)

- YSZ (yttria + zirconia)

- AZO (see above)

- NiOₓ
  - Thermal (nickelocene + H₂O)
  - Ni from H₂ plasma reduction

- FeOₓ
  - Thermal (ferrocene + H₂O)
  - Fe from H₂ plasma reduction

- AlN
  - Thermal (MeCpSr + H₂O)

- SrO
  - Thermal (MeCpSr + H₂O)

- SiO₂
  - Thermal (tert-butoxy silanol + TMA)
  - YIKES!!! – discontinued.
Films @ SNF – Wish List

- TaN
- Ti
- Plasma Ru (better nucleation)
- W
- $\text{Y}_2\text{O}_3$
- Ideally want to support anything requested
ALD Issues Pareto (last 6 months)

ALD Issues- last 6 months
Pareto Analysis

- Load Arm (user)
- Load Arm
- Computer
- Chuck Heater
- Heater Jacket
- Plasma
- ALD Valve
- Elbow connection
- Pump leak

Frequency
Cumulative %
Primary Issues

• Primary issues related to Fiji load arms.
  • Most issues related to user error.
    • Fiji2 loading arm required refurbishment after being bent by user.
    • After summer break there was a large spike in errors. Once users re-familiarized themselves with loading process, error frequency dropped.
  • Setting screws to secure loading arm height also tend to drift and re-calibration of arm height is second most common error.
  • Fortunately these errors are relatively easy to recover from and result in minimal equipment downtime.
Maintenance Schedule

• Savannah
  – Reset pressure gauge (2x/yr)
  – Pump rebuild (1/yr); change oil (2x/yr)
  – Kalrez O-Ring (1/yr)
  – Chamber CO$_2$ clean: 1/yr (during shutdown)
  – Manifold clean: 4µm of film (coming soon)

• Fiji
  – Sample holder clean: As needed (~2X/year)
  – Load Arm refurbishment: Recommended 1/year
  – Chamber clean: haven’t done yet
  – Manifold clean: 4µm of film
Additions to system

• We added a boost system in fall 2012 to aid with low volatility precursors
  – Similar to a bubbler
  – Very positive results (SrO and $Y_2O_3$)
  – Desire to add to more chambers

• Wish list
  – In situ film measurement
    • QCM
    • Ellipsometry (not really possible in several of our systems)
  – Ozone
Material Characterization: Al$_2$O$_3$

- Found deposition rate @ 200 °C: ~0.99 A/cycle
Material Characterization: $\text{Al}_2\text{O}_3$

- Deposition rate decreases with temperature increase
Material Characterization: $\text{Al}_2\text{O}_3$

- Dielectric constant is extracted from accumulation capacitance & measured dielectric thickness

![Graph showing capacitance vs. DC bias for different cycles]

<table>
<thead>
<tr>
<th>Cycles</th>
<th>Dielectric Constant (k)</th>
</tr>
</thead>
<tbody>
<tr>
<td>50</td>
<td>6.30</td>
</tr>
<tr>
<td>100</td>
<td>6.77</td>
</tr>
<tr>
<td>150</td>
<td>6.74</td>
</tr>
<tr>
<td>200</td>
<td>6.76</td>
</tr>
</tbody>
</table>

- Deposition temperature: 200 °C

Extracted dielectric constant: $\sim 6.75$
Material Characterization: $\text{Al}_2\text{O}_3$

![Breakdown Voltage vs BV for AlOx]
Other well characterized metal oxides

- Similar data is available at the tool’s website
  - Example HfO₂

- Average Dielectric Constant: 15.27
- Average Doping Concentration: \(1.5 \times 10^{16}/\text{cm}^3\)
- Hysteresis Range: 230mV-285mV
- Mobile Charge Range: \(8 \times 10^{11}/1.4 \times 10^{12}/\text{cm}^2\)

- Dielectric Constant (k), doping type and concentration were extracted at 1MHz.
Pt and Nucleation

Thermal Pt from MeCpPtMe$_3$ and O$_2$
Pt and Nucleation

Plasma Pt from MeCpPtMe$_3$ and O$_2$
Pt and Nucleation

Plasma Pt from MeCpPtMe$_3$ and O$_2$

Plasma ALD Pt on Al$_2$O$_3$

Plasma ALD Pt on thermal SiO$_2$
TiN: Thermal vs Plasma

- Oxygen content kills conductivity of TiN
- With savannah we never saw less than 15% atomic weight in oxygen (mΩ-cm level resistivity)
- Switching to plasma in fiji 1% oxygen and best result is 10µΩ-cm resistivity
- How well can the load locked, higher temp fiji do for thermal TiN?
Open questions of interest

• How to handle ZnO?
• General contamination concerns...
  – Sulfide ALD for instance
• Utilizing the tool for non-ALD projects
• Coating of powders and loose material
• Training and theory background for users
• Anyone with a fiji: load transfer arms
Thank you.
Any questions?