Novel Reactor Design and Metrology Study for Tungsten ALD process

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OUTLINE

- Design of novel ALD reactor
  - Motivations for new reactor design
  - Our approach
  - 2 ALD operational modes

- Integration of real-time mass-spectrometry
  - Nucleation kinetics revealed in real-time by MS
  - “First wafer effect”: understanding effects on film thickness metrology of competing wall and wafer surface reactions

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MS-based real-time sensing in ALD processes
2 main approaches for ALD reactor designs

Tube furnace (University approach)

- Low cost
- Reliability (simplicity of design)
- Fast cycle times
- QCM integration
- Small wafer size (1-2”)
- High wall to wafer surface ratio
- Hot walls => wall reactions
- Contamination issue
- Low wafer throughput

Substrate-heated production-scale tool (manufacturing approach)

- Large wafers (up to 300 mm)
- Cluster tool integration
  - High throughput
  - Low contamination
  - Automation
- Small wall to wafer surface ratio
- Cold wall
- High cost
- Not compatible with QCM
Embedded mini-reactor concept

- Differentially pumped MS
- 35 µm orifice
- 10^5 torr
- 5 torr
- Z-axis pneumatic actuator
- UHV chamber
- Moveable cap
- 100 mm wafer
- Substrate heater
- Gas Outlet
- Gas Inlet

ALD mini-reactor
Benefits of embedded mini-reactor concept

- 100 mm substrate heater
- 0.2 L reactor volume
- Load-lock
- On-the-shelf UHV components
- Mass-spectrometry

- Wafer scale
- Low wall/wafer ratio
- Fast cycling time
- Low contamination
- In-situ diagnostic
- High wafer throughput
- "Low" cost

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Multiple Operation Modes

Dynamic mode: steady-state flow with alternating gas species

- Exposure:
  - Reactant + carrier gas
  - Throttle Valve
  - 5 torr carrier gas
  - 10^{-5} Torr

- Purge:
  - Fast gas switching
  - 10^{-5} Torr

Small reactor volume
Short gas residence time
Viscous flow condition
Multiple Operation Modes

Static mode: Fill-and-pumpout cycling of each gas

Precursor A exposure

10^{-5} Torr

Purge

Precursor B exposure

5 Torr

Pumpout is accelerated by lifting the cap to employ the larger vacuum chamber as ballast
Real-time MS Process Sensing

ALD process dynamics are directly observed by in-situ mass spectrometry

Precursors: WF$_6$, SiH$_4$
SiH$_4$ exposure $\rightarrow$ H$_2$ product
WF$_6$ exposure $\rightarrow$ SiF$_4$ product
MS response time: < 1 sec
Deposition kinetics can be directly observed by integrating byproduct MS signal over each cycle and plotting it against cycle number.
High process temperature accelerates the nucleation process

Effect of temperature on nucleation kinetics

High process temperature accelerates the nucleation process

Integrated $H_2$ Mass Spec Signal Per Cycle

- $325 \degree C$
- $250 \degree C$
- $175 \degree C$

$\int H_2$ during $SiH_4$ half cycle

Cycle Number

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Real-time identification of nucleation kinetics for different wafer preparations

Integrated SiF$_4$ MS Signal Per Cycle

Cycle Number

Nucleation region for HF-last treated surface

Nucleation region for H$_2$O-last treated surface

HF Treated Surface

H$_2$O Treated Surface
Characterization of first wafer effect

1. Integrated byproduct MS signal starts at much higher level for non-1\textsuperscript{st} wafer cases than 1\textsuperscript{st} wafer case.
2. Integrated byproduct MS signal tends to the same level in linear growth region for both 1\textsuperscript{st} wafer and non-1\textsuperscript{st} wafer cases.
Physically based model to understand 1st wafer effect

Nucleation occurs on wafer as well on walls

1st wafer case

Reaction on reactor surface

Reaction on wafer surface

Total reaction detected by MS

Nucleation occurs on wafer as well on walls
Effect of wall conditioning on in-situ chemical diagnostic

For non-1st wafers, nucleation occurs only on clean wafer surface and not on pre-conditioned wall surfaces.

Non-1st wafer cases

Reaction on reactor surface

Reaction on wafer surface

Total reaction detected by MS
• Linear correlation between total integrated byproduct MS signal and ALD film thickness
• 1st wafer effect will make the 1st wafer data deviate from the linear relationship
• With proper pre-process chamber treatment, 1st wafer effect can be reduced
Conclusion

1. A novel wafer-scale ALD system has been implemented by embedding a mini-ALD reactor (0.2L) inside a UHV chamber

2. Integration of in-situ mass spectrometry to ALD reactor has been optimized to achieve real-time process sensing

3. Deposition kinetics are observed in real-time using integrated MS signal data, but chamber history effect poses a challenge on the interpretation of MS results

4. The 1st wafer data can not be used for ALD film metrology because of chamber history effects, and pre-process chamber treatment can help to reduce the effect of chamber history