Thin Film Deposition at NUFAB

CVD, ALD and Beyond

John Ciraldo and William Mohr
Introduction

- Northwestern University Micro/Nano Fabrication Facility
- 6000 foot Class-100 cleanroom
- 4 Bays:
  - Characterization
  - Lithography
  - Wet Bench Processing
  - Etch & **Deposition**
- Staff of 6 scientist & engineers
- Full process & development support
- Located on ground floor of Tech F-wing
Thin Film Deposition

- **PVD**
  - Physical Vapor Deposition
    - Thermal Evap., MBE, e-Beam Evap., Sputter, PLD…

- **CVD**
  - Chemical Vapor Deposition
    - PECVD, LPCVD, MWCVD, MOCVD…

- **Some other stuff**
  - ALD, spin coating, electroplating…
PVD vs CVD

- Physical Vapor Deposition (PVD) relies on physical processes to vaporize solid materials.
- Chemical Vapor Deposition utilizes reactions between one or more precursors and in-situ gases.
  - Precipitate is deposited on substrate.
- CVD tends to be associated with higher temperature depositions and higher depositions rates.
Thermal Evaporation

- Chamber is evacuated to high vacuum
- Source material typically placed in W crucible
- Source material is heated to sublimation, allowing for deposition
- Reasonably good step coverage
  - Can be controlled somewhat through geometry
- Spitting is possible for some materials
- Deposition can be quite fast
- Poor process tunability
- Materials limited by thermal properties
Thermal Evap
At NUFAB

- Denton Vacuum Explorer 14
- Accommodates wafer sizes up to 6"
- Substrate heating up to 200°C
- Three 200kVA shielded sources
- Recipes proved for:
  - Multiple metals, high rate Cu and Au
Molecular Beam Epitaxy (MBE)

- Special case of thermal evaporation where Knudson (effusion) cells are used and throw distance is typically increased
- Generally limited to epitaxy
- Performed at UHV
- Not available at NUFAB
E-Beam Evaporation

- Special case of evaporation where the source is heated by an e\(^{-}\) beam
- Generally line-of-sight coverage
  - Efficient use of source materials
  - Well suited for lift-off processes
  - Not ideal for 3D structures
- Compound materials may decompose under high temperatures
E-Beam Evaporation At NUFAB

- AJA ATC-2036-E System
- Accommodates substrates up to 6”
- 6 pockets for source material, 2 user configurable
- Water-cooled, rotatable substrate holder
- Substrate heating to 200°C
- Staff-provided processes
  - Al, Ni, Cr, Au, Ag, Mo, Ti, W…
Sputtering

- Physical erosion via ion bombardment
- Comes in many ‘flavors’
  - RF, DC, Pulsed-DC, HiPIMS…
- Suitable for most solid materials
- Generally good conformality
  - Somewhat process tunable
  - Provides challenges for lift-off
- Processes are highly tunable
Sputtering

How does it work?

- Plasma ignition via electrical excitation and mechanical interactions
- Ions, typically argon, are accelerated towards the target material
- Near the target, the ions are ‘steered’ by a strong magnetic field
- Upon ion collision, atoms are ejected from target, with some depositing on substrate
Sputtering

How does it work?

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<tr>
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<th>RF</th>
<th>Pulsed DC</th>
<th>HiPIMS</th>
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<td>Smoothness</td>
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<td>Ease of Use</td>
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<td>√</td>
<td>X</td>
<td>XX</td>
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NUFAB has two sputtering systems

- Both systems:
  - AJA Orion 8 systems
  - Up to 8” substrate (4” above 350 °C)
  - Up to 850°C Substrate heating
  - RF substrate biasing
  - +/- 2% thickness uniformity

- Sputter I is ‘workhorse’
  - most flexibility in allowed materials
  - RF, DC, Pulsed DC and HiPIMS capabilities
  - User installed targets

- Sputter II is high purity system
  - RF, DC and Pulsed DC capabilities
  - Staff installed targets
  - Recently added gold and platinum sources

- Staff-written processes:
  - Ultra fast, multiple source processes for Cu and reactive SiO₂
  - ITO, Bi, Fe, Au, Pt…
Pulsed Laser Deposition (PLD)

- Sputtering, but with lasers
- Source material is ablated form target using high-power laser pulsed
- Can be prone to spitting, particulate formation and globular depositions
- Due to vapor quenching, can be used to study metastable material phases
- Not available at NUFAB
Thin Film Deposition

- **PVD**
  - Physical Vapor Deposition
    - Thermal Evap., MBE, e-Beam Evap., Sputter, PLD...

- **CVD**
  - Chemical Vapor Deposition
    - PECVD, LPCVD, MWCVD, MOCVD...

- **Some other stuff**
  - ALD, spin coating, electroplating…
Chemical Vapor Deposition

- Deposition typically occurs due to reactions at the substrate surface
- Not a single technique, but a family of techniques
- Prone to high rate depositions for thin and thick films
- Far too many variants for a single talk
Low Pressure CVD (LPCVD)

- Functionally similar to APCVD (atmospheric pressure CVD)
- Common technique for silicates
- Pressures on order of 75-750 mTorr
- Lower pressure dramatically improved mass transport characteristics (slows them)
  - Improved step coverage
- CVD is thermally driven
  - Typical temperatures in the range of 600-1100°C
Plasma Enhanced CVD (PECVD)

- Chemical interactions induced via electrical stimulation, rather than thermal
  - Allows for significantly lower substrate temperatures during deposition
  - Typical excitation is RF, but may be DC
- Very fast depositions realizable
  - 10s to 100s of nm/min possible
  - At very high rates, uniformity may be sacrificed
- Good conformality for structured wafers
# LPCVD vs PECVD

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<tr>
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<th>LPCVD</th>
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<tr>
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<td>Deposition Rate</td>
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<td>Substrate Damage</td>
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<tr>
<td>Uniformity</td>
<td>Good</td>
<td>Excellent</td>
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<tr>
<td>Film Density</td>
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<td>Excellent</td>
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<tr>
<td>Stress</td>
<td>Tends to be compressive (may be adjustable)</td>
<td>Typically low</td>
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<tr>
<td>Sides Coated</td>
<td>Single</td>
<td>Double</td>
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LPCVD
At NUFAB

- Recipes for standard and low-stress Si$_3$N$_4$
  - Both recipes stoichiometric
- Dichlorosilane (SiH$_2$Cl$_2$) based recipes, ammonia reducer
- 4” Wafer capability, with ability to load many wafers simultaneously
  - 25 wafers/carrier
- Processing temperature: 835°C
- Fully automated processes
PECVD
At NUFAB

- STS (SPTS) LpX CVD tool
- Configured for 4” substrates
  - Small substrates easily accommodated with carrier wafer
- Equipped with loadlock and automated transfers
- Fully automated processes
- Silane based processes
- Recipes for silicon oxide, silicon nitride and polysilicon
- Typical process temperature is 300°C
  - Can accommodate 200-400°C processes
- Dual excitation sources for stress control in nitride films
  - HF: 13..56 MHz, LF: 380kHz
- Staff-Provided recipes for Low stress SiO2, low stress Si3N4, polysilicon at multiple dopant concentrations, low temp processes…
Overview of Techniques

- Conventional deposition techniques
  - MOCVD
  - ALD

- Less Conventional
  - Parylene
  - Spin on Glass (SOG)
MOCVD Overview

- Metal Organic Chemical Vapor Deposition
  - Typically used for III-V or II-VI materials
    - GaN, InGaAs, AlGaAs, GaAs, InP, SiC, AlN
  - Homoepitaxy and heteroepitaxy

- Typically used in for creating photonic and power and RF devices

Pros:
- High throughput
- Epitaxy
- Large variety of materials
- Easy to change between materials
- Sharp interfaces
- Good uniformity

Cons:
- Expensive
- Can have extremely hazardous and or expensive sources
- Process needs to be closely monitored
- Not at NUFAB
MOCVD: Metal Organics

- Metal Organics
  - Ligand
  - Metal

- Typical Sources
  - Trimethyl Aluminum
  - Trimethyl Gallium
  - Triethyl Gallium
  - Trimethyl Indium
MOCVD: Hydrides

- Hydrides
- Typical Sources
  - NH₃
  - AsH₃
  - PH₃
  - SiH₄
  - Si₂H₆

![Chemical structures of Hydrides](image)
MOCVD: Principal of Operation

- Carrier Gas
- Hydrides
- Hydride manifold
- Wafer
- Gas Injection
- Chamber
- Susceptor planetary rotation
- Heater
- Pump and Exhaust system
- MO manifold
- Metal Organics
MOCVD: Principal of Operation
MOCVD: Principle of Operation

1. Metal Organics and Hydrides Mix in reaction chamber (500 – 1200°C)
2. Chemicals pyrolyze in chamber due to heat
3. Resulting metal lands on surface and random walks until a step is found
4. Organic is removed through the exhaust
ALD Overview

- Atomic Layer Deposition
  - Typically used when:
    - High uniformity is required
    - Need to coat sidewalls of device
    - Very tight thickness tolerances
  - Typically lower temperature deposition as compared to other CVD processes

- Pros:
  - Large variety of materials
  - Easy to change between materials
  - Sharp interfaces
  - Good uniformity

- Cons:
  - Low throughput
  - Very slow
  - Can have extremely hazardous and or expensive sources
ALD: Applications

- Optical
  - Antireflection
  - Optical filters
  - Photonics
  - Transparent Conductors
- Electronics/MEMS
  - Gate dielectrics
  - Gate electrodes
  - Diffusion barriers
  - DRAM
  - Flexible electronics
- Other
  - Nanotubes
  - Powders
  - Wear/corrosion resistant coatings
  - Fuel cells/catalysis
ALD: Sources

- Same precursors as MOCVD
  - Additional precursors are also available based on material, temperature, cost restraints

- Typical reducers for ALD
  - $\text{H}_2\text{O}$
  - $\text{N}_2$ plasma
  - $\text{O}_3$ (Ozone)
  - $\text{O}_2$ plasma
  - $\text{NH}_3$
  - $\text{NH}_3$ plasma
ALD: Principle of Operation

- Discrete steps

"Thermal and Plasma-Enhanced Atomic Layer Deposition on Powders and Particles Geert Rampelberg, Véronique Cremers, Delphine Longrie, Davy Deduysche, Johan."
ALD: Principle of Operation

Carrier Gas

Oxidizer/Reducer

Oxidizer/Reducer manifold

Wafer

Metal Organics

Plasma Injection

Chamber

MO manifold

Pump and Exhaust system

Heated chuck no rotation needed
ALD: NUFAB Capabilities

- Arradiance GEMStar XT-P
  - Up to 6” substrates
  - Chamber Heats to 350C
  - Platten Heats up to 500C
  - Four slots for precursors
    - Can be changed by Staff with 48 hr notice
  - Can create Ar, O2, H2, NH3 plasma
  - Mechanical rotation for coating powders
  - Films currently available:
    - Al2O3, HfO2, TiO2, Pt, ZnO, SiO2
Parylene: Overview

- CVD process that creates biocompatible coating of devices for medical industry
- Also used to protect electronics from the environment

**Pros:**
- Directly coats surfaces at room temperature with high uniformity, high repeatability, and pinhole free
- Chemically stable and biocompatible
- Barrier to oxygen and moisture
- Fungal resistant
- Thermal mechanically stable
- Electrical isolation of samples
- Adheres well to most surfaces

**Cons:**
- Limited only to parylene
- Must be vacuum compatible
Parylene: NUFAB Capabilities

- SCS Labcoter2 Parylene Deposition System
  - Two systems: I and II
  - Fixture Rotation
  - Process parameters optimized for Parylene C
    - provided by NUFAB
  - 1g of Parylene yields ~ 1µm of film
Parylene: Principle of Operation

- Furnace heats to 690°C
- Dimer solid to gas
- Monomer gas to polymer
- Cold trap
- Pump

Chemical structure:

\[
\begin{align*}
\text{H}_2 & \quad \text{C} \\
& \quad \text{H}_2
\end{align*}
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\[
\begin{align*}
\text{Cl} & \quad \text{H}_2 \\
& \quad \text{C}
\end{align*}
\]
Spin On Glass (SOG): Overview

- Method for depositing SiO₂ without a vacuum chamber
- Used for barrier layers and for electrical isolation
- Biodegradable
- Able to deposit at a range of thicknesses depending on spin speed and viscosity
- Used in electronic industry as replacement for PECVD for electrical isolation and in medical field as biodegradable/biocompatible barrier
- Silicate (e.g. Na₂SiO₂ + water + ethanol + acetone + isopropanol)
Spin On Glass (SOG): Procedure

- Attach substrate to spin coater
- Apply SOG
- Set spinner to 3000 RPMs for 40 sec
- Bake at 200C for 60s
- Repeat procedure for a thicker coating
- Bake at 400C for 30min to solidify completely
Spin On Glass (SOG): NUFAB Capabilities

- Properties after baking for 200C for 60sec
  - Thicknesses: 200 nm
  - Index of refraction: 1.41

- Properties after baking for 400C for 30min
  - Thickness: 170nm
  - Index of refraction: 1.34
  - Volume Resistivity $5 \times 10^{13} \ \Omega \text{cm}$
  - Surface Resistance $2 \times 10^{12} \ \Omega/\square$
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<tr>
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