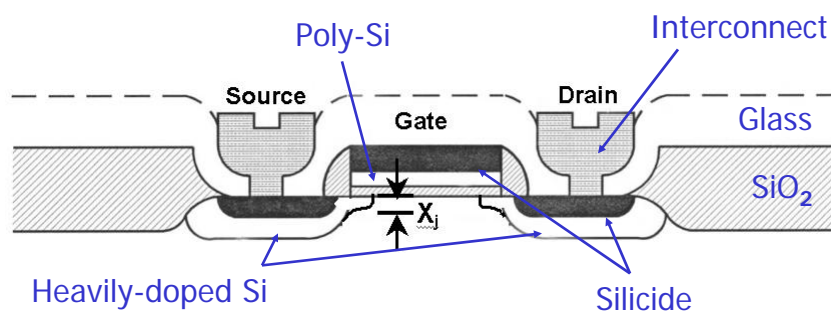


# An Interdisciplinary Laboratory Course for Microelectronics Fabrication


1

## MOSFET Schematic



- Hundreds of process steps
- Many materials
- Some components are nanoscale: finite-size effects

2




## International Technology Roadmap for Semiconductors

	1999	2001	2004	2007	2010	2013
<b>DRAM half-pitch (nm)</b>	180	130	90	65	45	32
<b>Transistors/chip at production (millions)</b>	61	97	193	386	773	1546
<b>MPU cost/function (<math>\mu</math>cents/transistor)</b>	120	60	30	15	5.3	1.9

- Rapid scaling requires skilled workforce for processing
  - Interdisciplinary, flexible

3

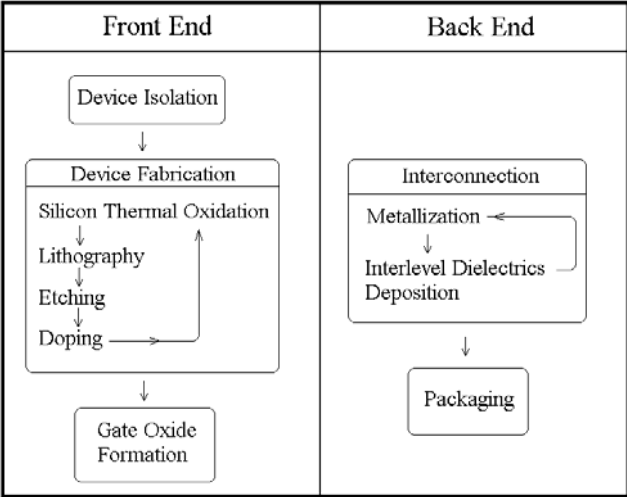


## Employment Patterns

- Typical US fab employs:
  - 25-30% chemical engineering
  - 25-30% electrical engineering
  - Remainder: materials science, mechanical eng, physics...
- Distribution of training differs outside US
- Fast-moving but high capitalization
  - Profit margins squeezed, patterns shifting
  - Market cycles
  - Time-to-market critical
- Technical staff must have working knowledge beyond their formal disciplinary training

4

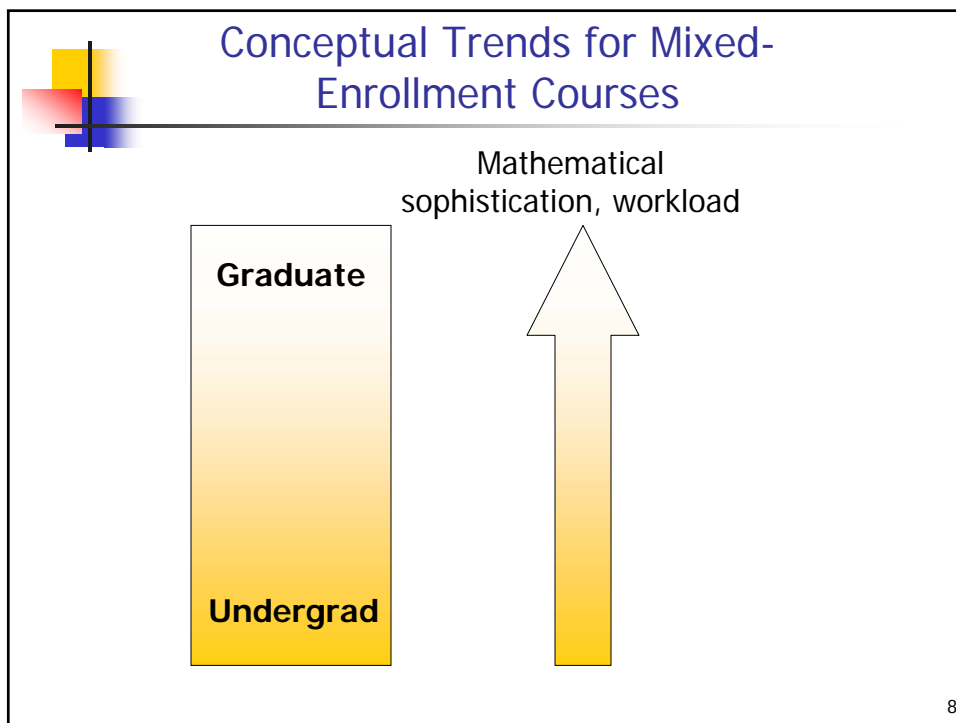
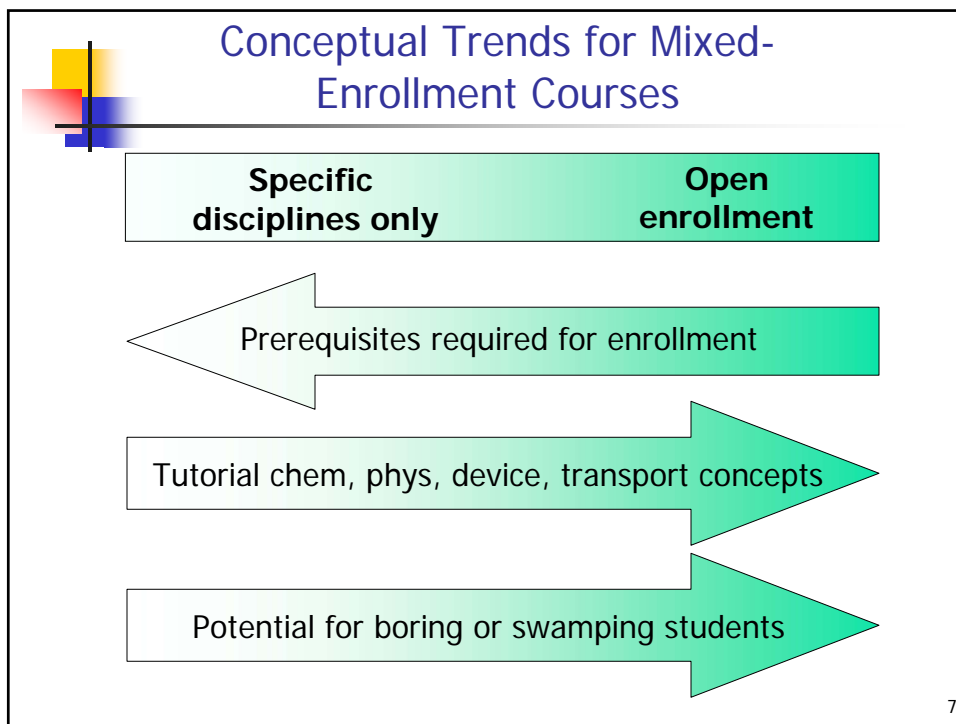
# Process Flow Schematic



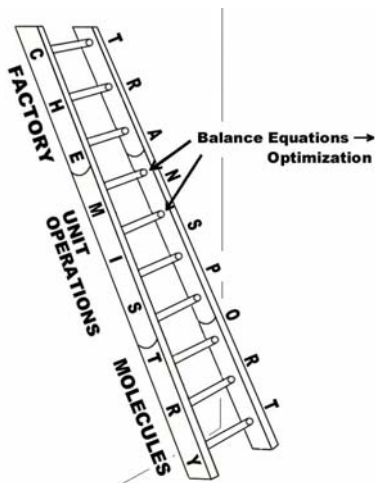
- Hundreds of steps
- Unit-ops concepts:
  - Lithography
  - Doping
  - Deposition
  - Etching
  - Cleaning/polishing
- Much chemistry, transport

# Student Enrollment

- Advantages of mixed course enrollment
  - Gives tast of work environment in fab
  - Broadens student base to justify large investment
- Two dimensions to mixed enrollment:
  - Multiple disciplines
    - Electrical/microelectronics engineering
    - Chemical engineering
    - Materials science
    - Chemistry
    - Physics
  - Multiple degree levels
    - MS, PhD
    - Upper-level undergraduate



## Purposes of Laboratory Courses



- Transfer specific facts
  - Technology details
  - Mathematical methods
- Offer an ordered framework for facts: knowledge
  - Unit operations  $\leftrightarrow$  process flow
  - Unity of form for balance eqns in mass, energy, momentum
- Offer experience in applying knowledge: wisdom
  - Necessarily experiential
  - Best done in lab course

9

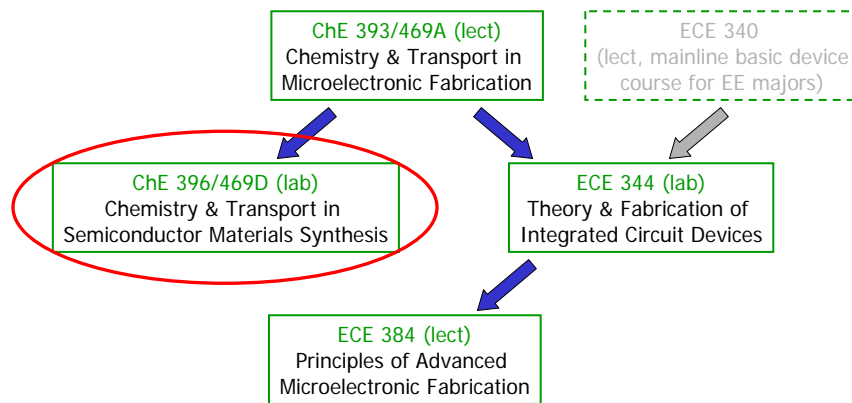
## UIUC: "Chemistry & Transport in Semiconductor Materials Synthesis"

- Lab course, up to 24 students per offering
  - Eight groups of 3 (mixed-discipline mandatory)
- Broad audience
  - ChE, EE, Chemistry, Materials Sci
  - Grad & Undergrad
- Limited number of in-depth experiments
  - Closely coupled to computational modeling
- Funded by Intel, Applied Materials, Illinois Board of Higher Education, 4 campus units

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## Informal Option in Processing at UIUC

- Up to 4 elective courses in chem eng, electrical eng
- Prerequisites kept minimal



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## Experiments

- Si oxidation kinetics
  - Dry etching of  $\text{SiO}_2$
  - Photoresist spin coating
  - Dry etching of photoresist
  - Principles of vacuum technology
  - Principles of surface chemistry
  - Atomic layer deposition of  $\text{TiO}_2$
  - Planned:
    - Chemical mechanical polishing
    - PECVD of amorphous Si
    - Cu electrodeposition
- ] "Process flow"  
 ] "Process flow"  
 ] Science-to-technology

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## Scope of Presentation

- Most equipment is custom-built
  - Often reduces expense
  - Tailored to specific goals
  - Details vary according to local circumstance
- For each experiment, outline:
  - Possible conceptual goals
  - Apparatus setup
  - Approach to data collection
  - Data analysis
- Offer suggestions for logistics of course setup

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## Silicon Oxidation Kinetics: Possible Goals

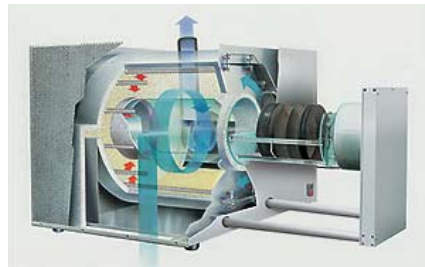
- Model a diffusion-reaction system with appropriate kinetics
- Show that different steps can be rate limiting depending upon conditions
  - Vary oxide thickness
- Determine an activation energy experimentally
- Observe spatial nonuniformity effects
  - Use observed kinetics to estimate nonuniformity in T
- Compare kinetics for different oxidants
- Provide material oxide for plasma etching experiment

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## Silicon Oxidation Kinetics: Apparatus

- Programmable furnace: ATV PEO-601



- Use wafer pieces to reduce costs
- Temperature in furnace is not uniform

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## Primary Metrology: Optical Thickness Measurements



Sentech  
Model CER SE 500

- Two instrument capabilities
  - Single-wavelength ellipsometry (632.8 nm)
  - White light reflectometry
- Spatial resolution: 1 x 3 mm<sup>2</sup>
- *This instrument used for other experiments as well*

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## Optical Thickness Measurements

- Robust, multi-use instrument
  - Stands up to student mistakes
  - Combined ellipsometry, reflectometry needed for broad range of expts

### ■ Ellipsometry

- Advantages
  - High sensitivity (<10 nm)
  - Good for multifold stacks
- Disadvantages
  - Limited to transparent films (e.g., SiO<sub>2</sub>)
  - Slow data acquisition

### ■ Reflectometry

- Advantages
  - Fast data acquisition
  - Good for transparent & translucent films (e.g., photoresist)
- Disadvantages
  - Lower thickness sensitivity
  - Poorer for multifold stacks

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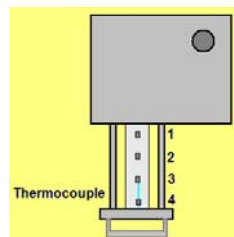
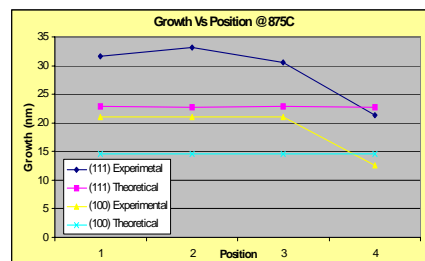
## Silicon Oxidation Kinetics: Data Collection

### ■ Experimental parameters

- Temperature
- Oxidant partial pressure
- Crystallographic orientation
- Position in furnace

### ■ Two possible oxidants

- Dry:  $\text{Si(s)} + \text{O}_2\text{(g)} \rightarrow \text{SiO}_2\text{(s)}$
- Wet:  $\text{Si(s)} + 2\text{H}_2\text{O(g)} \rightarrow \text{SiO}_2\text{(s)} + 2\text{H}_2\text{(g)}$ 
  - Gives exposure to bubbler behavior (thermodynamics)



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## Silicon Oxidation Kinetics: Data Analysis

- Use simplified Deal-Grove model

$$h = \frac{A}{2} \left[ \sqrt{1 + \frac{4B(t + \tau)}{A^2}} - 1 \right]$$

- Two regimes

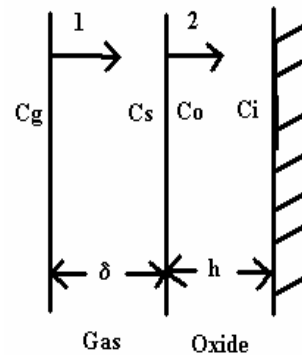
- Linear: rxn *and* diffusion matter

$$h \rightarrow 0 \Rightarrow h \approx \frac{B}{A}(t + \tau)$$

- Parabolic: diffusion dominates

$$h \rightarrow \infty \Rightarrow h \approx \sqrt{B(t + \tau)}$$

- Aggregated results from different groups yield better data set



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## Dry Etching of SiO<sub>2</sub>: Possible Goals

- Gain familiarity with concepts of dry etching
  - Capacitively* coupled plasma, sheath, plasma power...
  - Coupling between thermal, nonthermal chemistry
  - Spatial uniformity issues on wafer
- Gain exposure to spectroscopic diagnostics
  - Basic spectroscopy principles (emission)
  - Spatial uniformity in gas phase
- Use material from SiO<sub>2</sub> oxidation experiment
  - Process flow, integration

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## SiO<sub>2</sub> Dry Etching: Apparatus

- Capacitively coupled
  - Improved spatial uniformity
  - Chamber can also be used for PECVD

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## SiO<sub>2</sub> Dry Etching: Apparatus

Labels for the main apparatus:

- Gas inlet
- Plasma power supply
- Sample door
- Power leads
- Plasma tuner

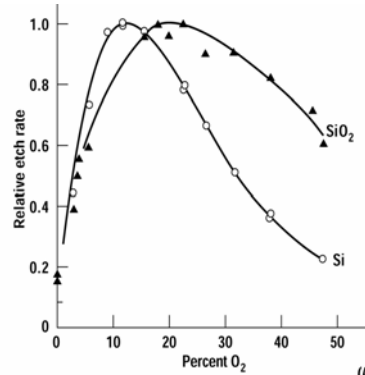
Labels for the detailed view:

- Gate valve
- Sieve trap
- Mechanical pump

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## SiO<sub>2</sub> Dry Etching: Data Collection

- Use CF<sub>4</sub> as etchant with added H<sub>2</sub> or O<sub>2</sub>
- P ~ 500 mTorr, V ~ 100V
- Selectivity of depends on additive concentration
  - CF<sub>4</sub>/H<sub>2</sub> increases C/F ratio, attacks SiO<sub>2</sub>
  - CF<sub>4</sub>/O<sub>2</sub> decreases C/F ratio, attacks Si



Mogab et al., *J. Appl. Phys.* 49: 3796 (1978)

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## Photoresist Spin Coating: Possible Goals

- Gain familiarity with concepts of spin-on techniques
  - Liquid application, spin effects
  - Soft bake, hard bake
  - Harmful effects of particulates
- Gain exposure to quantitative fluid mechanics
  - Coupled differential mass and momentum balances
  - Neglect of key terms
- Gain qualitative exposure to complex transport coupling
  - Solvent evaporation → skin formation, bubbling during bake
  - Surface tension → edge bead
- Create material for photoresist etching experiment
  - Process flow, integration

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## Photoresist Spin Coating: Apparatus

- Small commercial unit
- Resist injected through hole in top using syringe
  - SPR3612
    - Positive resist for bump-plating
    - Low viscosity
    - Thickness 1-2  $\mu\text{m}$
  - BPR 100
    - Negative resist for i-line, g-line implantation
    - High viscosity
    - Thickness 150  $\mu\text{m}$
- Bake on hot plate



Si wafer  
Stage  
Controller

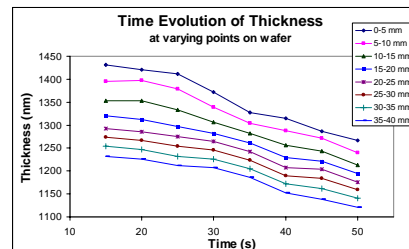
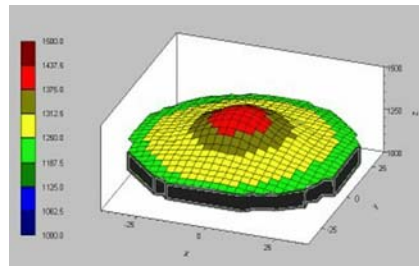
Photoresist drain

25



## Photoresist Spin Coating: Data Collection

- Experimental variables:
  - Spin rate ~ 0.0-5 krpm
  - Spin time ~ 15-60 s
  - Soft bake T ~ 70°C
  - Soft bake time ~ 3 min
  - Edge bead removal
  - Hard bake T ~ 70-120°C
  - Hard bake T ~ 3-10 min
  - Degree of dust removal
- Film thickness by reflectometry
- Examine thickness uniformity



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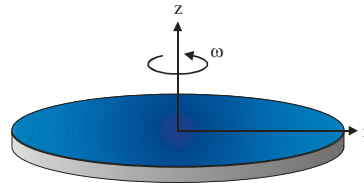
## Photoresist Spin Coating: Data Analysis

- Height, velocity relation:

$$\frac{\partial h}{\partial t} = -\frac{\rho\omega^2}{3\eta} \frac{1}{r} \frac{\partial}{\partial r} (r^3 h^3)$$

- For h independent of r:

$$h = \frac{h_0}{\left[1 + \frac{4\omega^2 h_0^2 t}{3\nu}\right]^{1/2}}$$



- Look at uniformity from ellipsometer measurements
- Compare results for different conditions to theory

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## Dry Etching of Photoresist: Possible Goals

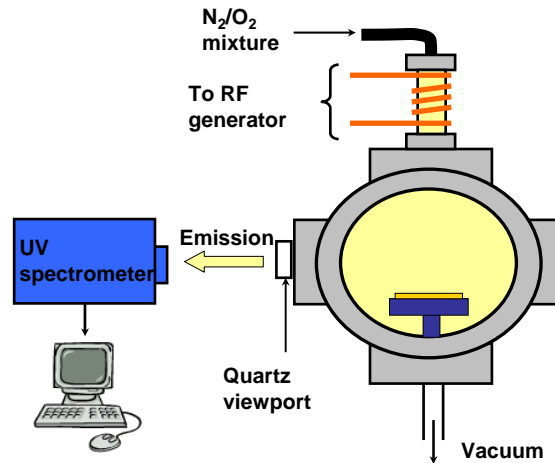
- Gain familiarity with concepts of dry etching
  - *Inductively* coupled plasma, sheath, plasma power...
  - Coupling between thermal, nonthermal chemistry
  - Spatial uniformity issues on wafer
- Gain exposure to spectroscopic diagnostics
  - Basic spectroscopy principles (emission)
  - Spatial uniformity in gas phase
- Use material from photoresist spinning experiment
  - Process flow, integration
- Couple experiments to simulations
  - Permits rapid testing/evaluation of different designs

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## Photoresist Dry Etching: Apparatus

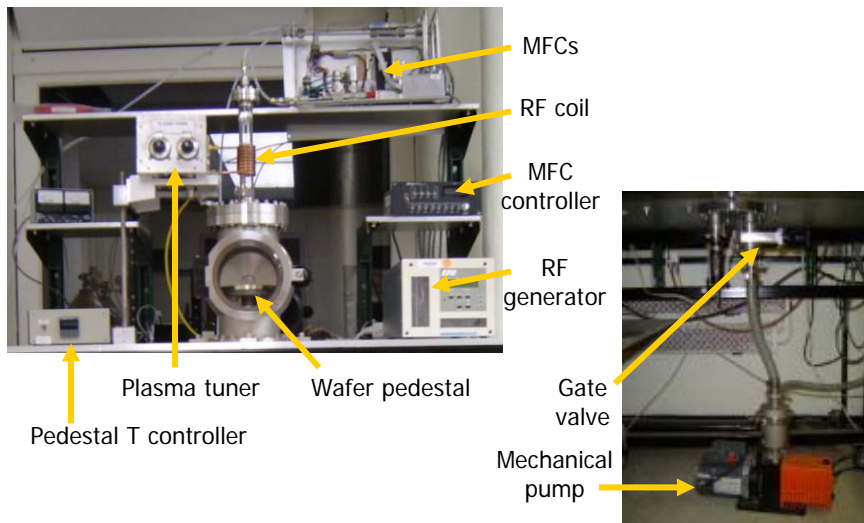
- Oxygen plasma (N added to prevent recombination)
- Spectrometer monitors plasma composition
- Rate data correlated to gas composition



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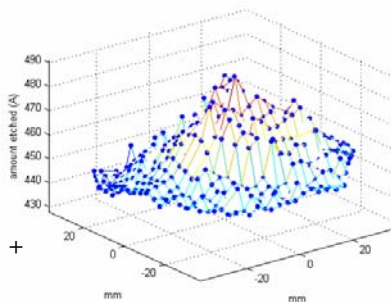
## Photoresist Dry Etching: Apparatus



30

## Photoresist Dry Etching: Data Collection

- Experimental parameters
  - Temperature
  - Oxidant partial pressure
  - Position in furnace
- Two possible oxidants
  - Dry:  $\text{Si(s)} + \text{O}_2(\text{g}) \rightarrow \text{SiO}_2(\text{s})$
  - Wet:  $\text{Si(s)} + 2\text{H}_2\text{O}(\text{g}) \rightarrow \text{SiO}_2(\text{s}) + 2\text{H}_2(\text{g})$
- Use ellipsometry for film thickness



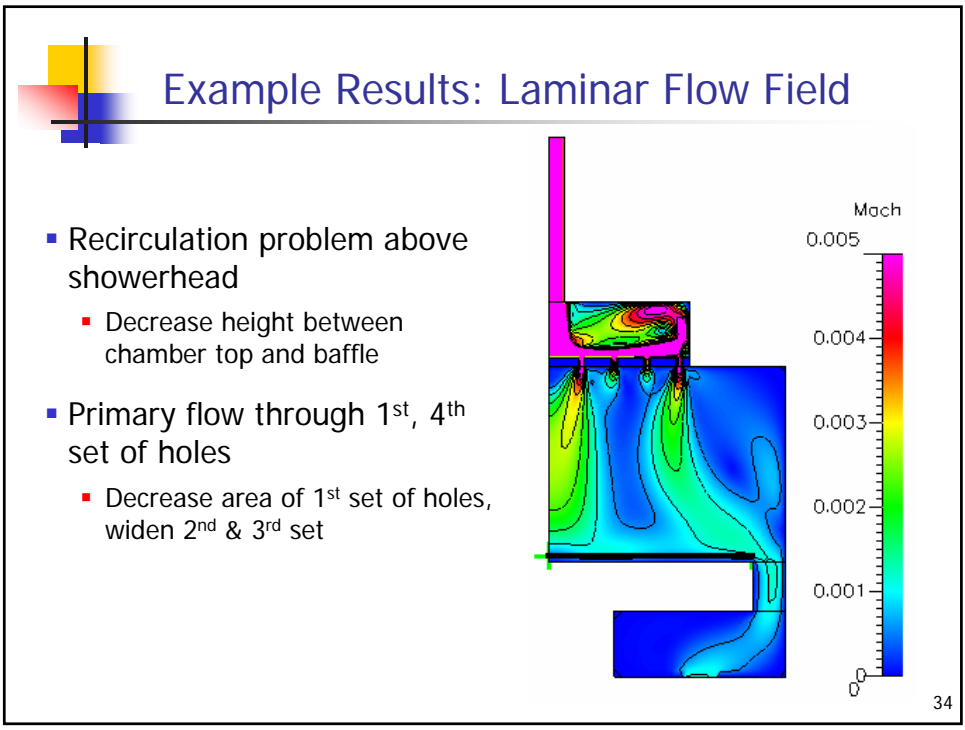
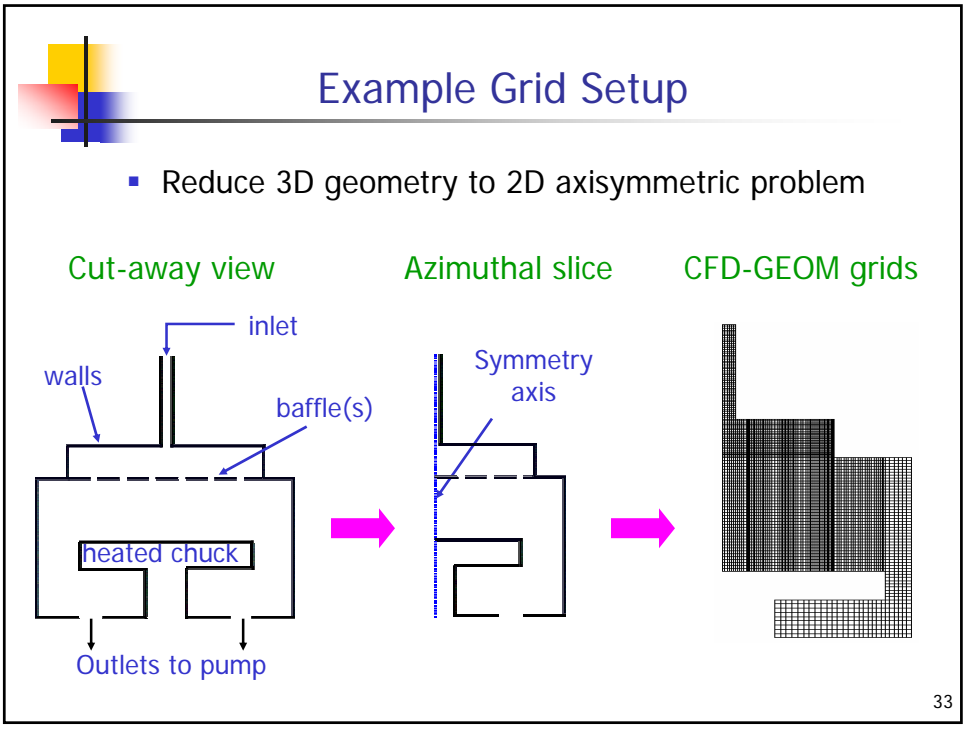
31

## Photoresist Dry Etching: Data Analysis

- Model with CFD-ACE+
- Three main modules:
  - CFD-GEOM – computational grid generator
    - 2-D or 3-D objects rendered using CAD-oriented user-interface
  - CFD-ACE – solver
    - User defines problem type (flow, heat transfer, chemistry) and boundary/initial conditions
  - CFD-VIEW – graphical solution viewer

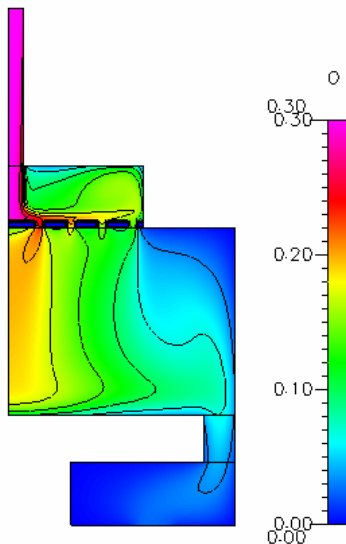
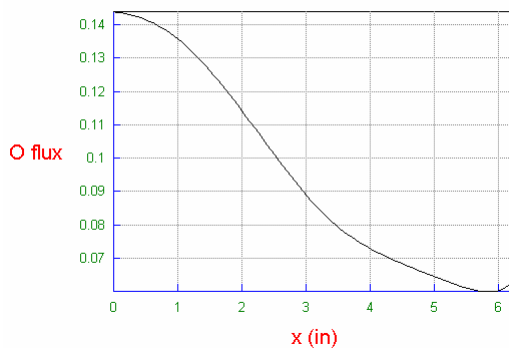
32





## Reactive Species Profile

O Flux Normal to Wafer Surface



- Higher uniformity of O flux desired
  - Solution: redesign showerhead

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## Uses for Computational Module

- Explore various showerhead designs to:
  - Reduce flow recirculation
  - Improve spatial uniformity of reactive species flux
- Reconfigure chamber to reduce nonessential volume
- Test selected cases experimentally

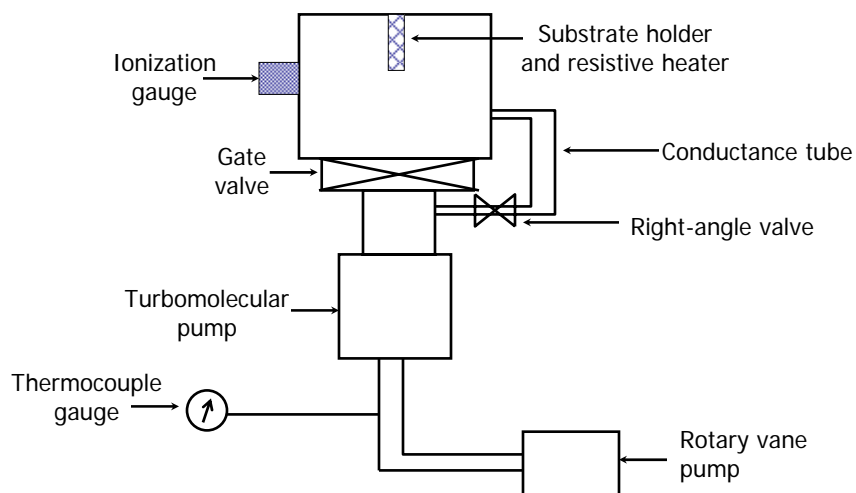
36

## Principles of Vacuum Technology: Possible Goals

- Gain familiarity with vacuum equipment, measurement
  - Pumps, gauges, flanges, gaskets, feedthroughs, valves...
- Learn key vacuum concepts
  - Pumping speed, conductance
  - Molecular vs. viscous flow
  - Measure & compare conductance of various tubes
  - Compare results with theory
- Learn simple mass balance equations
  - Compare steady-state, transient experimental results for mass flow

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## Vacuum Technology: Apparatus Schematic



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## Vacuum System: Upper Portion

Ion gauge

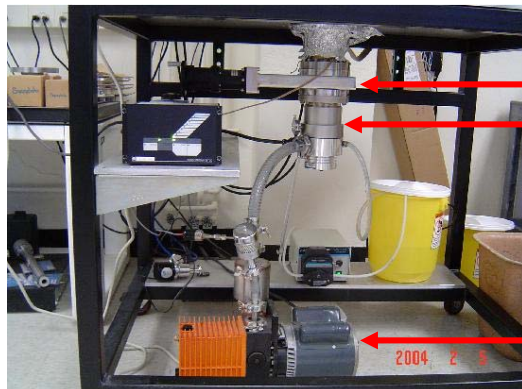


Base pressure  $\approx 1 \times 10^{-8}$  Torr

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## Vacuum System: Lower Portion



Gate valve

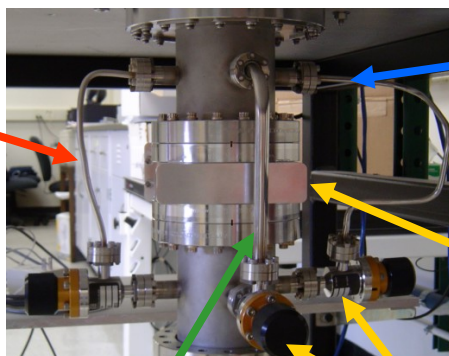
Turbomolecular  
pump

Mechanical  
backing pump

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## Accessories for Conductance Experiments

Tube 1:  
Diameter-  $\frac{1}{4}$ "  
Length- 8.5"



Tube 3:  
Diameter-  $\frac{1}{4}$ "  
Length- 16"

Gate valve

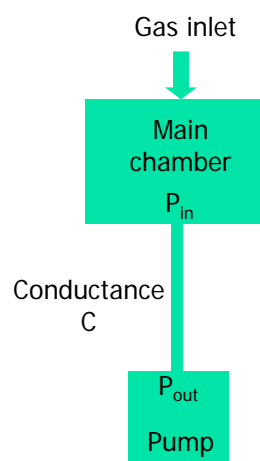
Tube 2:  
Diameter-  $\frac{1}{2}$ "  
Length- 8"

Right-angle valves

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## Vacuum Technology: Data Collection

- Admit air through leak valve,  $\sim 10^{-5}$  Torr
- Pump chamber through one of several long thin tubes
  - Diameter, length varies
  - Independent valving
- Measure pressures  $P_{in}$  and  $P_{out}$
- Snap leak valve shut, measure time constant for pressure decay



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## Vacuum Technology: Data Analysis

- Use transient mass balance to determine effective system pumping speed:

$$V \frac{dP}{dt} = -S_{eff} P$$

- Use pump specs to obtain tube conductance:

$$\frac{1}{S_{eff}} = \frac{1}{S_{pump}} + \frac{1}{C}$$

- Verify conductance relationship:

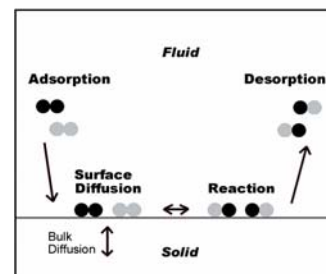
$$C_{tot} = C_1 + C_2 + \dots C_n$$

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## Principles of Surface Chemistry: Possible Goals

- Gain familiarity of surface kinetics
  - Adsorption, desorption
  - Concepts generalize to CVD, ALD
  - Results feed into ALD experiments
- Manipulate transient mass balances
  - Vacuum chamber
  - Surface
- Learn concepts of linear-ramp kinetics from temperature programmed desorption
  - Concepts generalize to RTP



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## Surface Chemistry: Apparatus

Feedthrough for  
resistive heating,  
thermocouple

Ion gauge



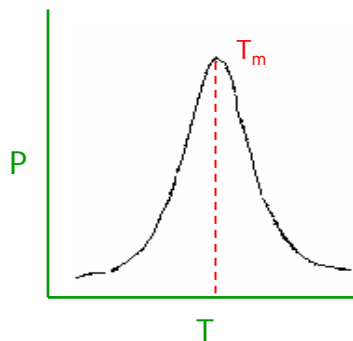
Base pressure  $\approx 1 \times 10^{-8}$  Torr

Optical  
pyrometer

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## Surface Chemistry: Data Collection

- Perform temperature programmed desorption experiments with  $\text{TiCl}_4$  or  $\text{H}_2\text{O}$ 
  - Expose surface to gas for fixed  $t$ ,  $P$
  - Apply linear temperature ramp  $\sim 10\text{K/s}$
  - Measure  $\Delta P$  vs  $t$  in ion gauge
    - $\Delta P \propto$  desorption rate
- Vary exposure, heating rate  $\beta$ 
  - Peak area  $\propto$  initial coverage
  - $T_m$  increases with  $\beta$



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## Surface Chemistry: Data Analysis

- Mass balance on chamber shows  $P \propto$  desorption rate

$$-A \frac{d\Theta}{dt} = \frac{V}{k_B T} \left( \frac{dP}{dt} + \frac{S}{V} P \right) \approx \frac{S}{k_B T} P$$

thus  $P \propto -\frac{d\Theta}{dt} = \frac{k_n^0 \Theta^n}{\beta} \exp(-E_{des}/k_B T)$

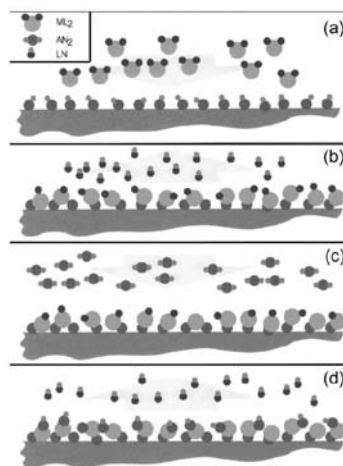
- Line shape analysis gives all kinetic parameters
- Peak temperature  $T_m$  varies with heating rate

$$\frac{k_1^0}{\beta} \exp\left(-\frac{E_{des}}{k_B T_m}\right) = \frac{E_{des}}{k_B T_m^2}$$

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## Atomic Layer Deposition of $\text{TiO}_2$ : Possible Goals

- See gas-solid interaction kinetics used in practice
  - Use concepts, data from surface chemistry experiment
  - Learn connection between CVD, ALD
- Manipulate transient mass balances
  - On surface
  - In deposition reactor (gas transport greatly simplified)



Seidel et al., *Thin Sol. Films* 402: 248 (2002)

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## Safety Problems in Deposition Experiments

- Toxicity, flammability of “interesting” source gases, products
  - $\text{SiH}_4$ ,  $\text{Si}_2\text{H}_6$  (pyrophoric)
  - $\text{SiH}_2\text{Cl}_2$
  - $\text{H}_2$
  - Dopants
- Alternative: deposition of  $\text{TiO}_2$  from  $\text{TiCl}_4$ ,  $\text{H}_2\text{O}$ 
  - HCl product no worse than standard chem lab



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## Aspects of $\text{TiCl}_4$ , $\text{H}_2\text{O}$ Chemistry

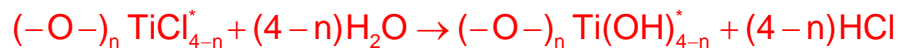
- Advantages
  - Low substrate T ( $\sim 200^\circ\text{C}$ )
    - Less strain on equipment (heaters, o-rings, etc.)
  - High rate (easier to measure)
  - Low P (less source gas used)
  - Good for both CVD, ALD
- Disadvantages
  - Not a conventional semiconductor material

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## ALD From $\text{TiCl}_4$ , $\text{H}_2\text{O}$

- Operating Conditions:
  - $P_{\text{TiCl}_4} \approx 0.2$  Torr
  - $P_{\text{H}_2\text{O}} \approx 2$  Torr
  - $T \approx 250$  °C
- Half Reactions (\* indicates surface species):



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## Atomic Layer Deposition of $\text{TiO}_2$ : Data Collection

- Experimental parameters
  - Temperature
  - Source gas partial pressure
  - Number of cycles
  - Length of cycles
  - Length of purge time
    - Observe switch to CVD if purge too short
- 300 cycles, estimated depth ~45 nm
- Thickness from ellipsometry
- Possible supplementary experiment:  
standard low-pressure CVD

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## Atomic Layer Deposition of $\text{TiO}_2$ : Data Analysis

- Use film thickness, number of cycles to compute thickness/cycle
- Compare for various conditions and cycle times
- Model growth using adsorption, desorption parameters from surface chemistry experiment
  - Compare with experimental results

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## Chemical Mechanical Polishing: Possible Goals

- Gain familiarity with concepts of CMP
  - Relative angular velocity, down-pressure, slurries
  - Physics-based governing equations used phenomenologically
- Gain familiarity with quantifying spatial nonuniformity effects

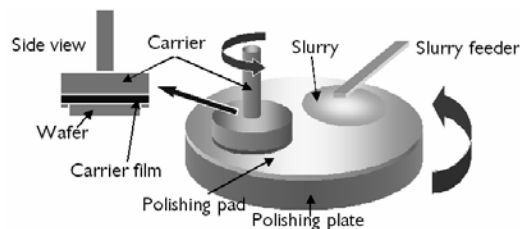
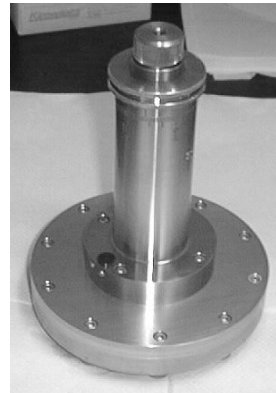
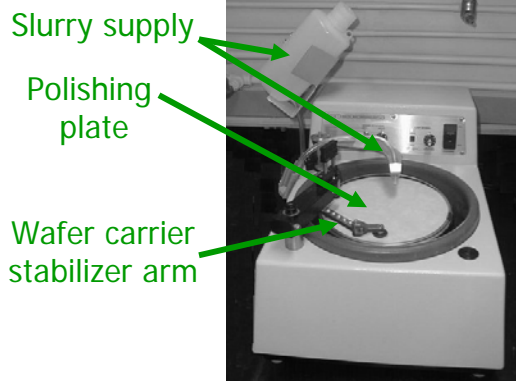


Illustration courtesy of Prof. David B. Graves 54



## Chemical Mechanical Polishing

- Use conventional tabletop polishing tool



Wafer carrier

Photos courtesy of Prof. David B. Graves 55



## Chemical Mechanical Polishing: Data Collection

- Polish wafers with grown thermal oxide
- Experimental parameters
  - Spin rate
  - Pressure on wafer
  - Polishing time
- Thickness/uniformity from reflectometry
  - 9-point inspection pattern
  - Surface tension → edge bead
- Use material from photoresist etching experiment
  - Process flow, integration

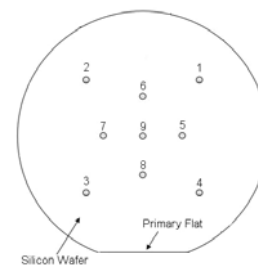


Illustration courtesy of Prof. David B. Graves 56

## Chemical Mechanical Polishing: Data Analysis

- Quantify Planarization: With-In-Wafer-Non-Uniformity

$$WIWNU(\%) = \text{Max} \left[ \frac{\text{oxide thickness}_{i+1} - \text{oxide thickness}_i}{\text{mean of total oxide thicknesses}} \times 100 \right]$$

- Examine Preston's wear equation:

$$\left. \frac{dh(x)}{dt} \right|_P = C \left. \frac{dL(x)}{dA} \frac{ds(x)}{dt} \right|_P$$

- Quantify Preston's coefficient C:

$$C = \Delta h \left( \frac{A}{L} \right) \left( \frac{1}{v_{avg} \Delta t} \right)$$

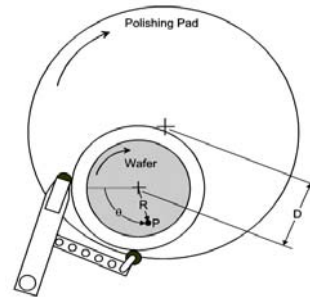


Illustration courtesy of Prof. David B. Graves 57

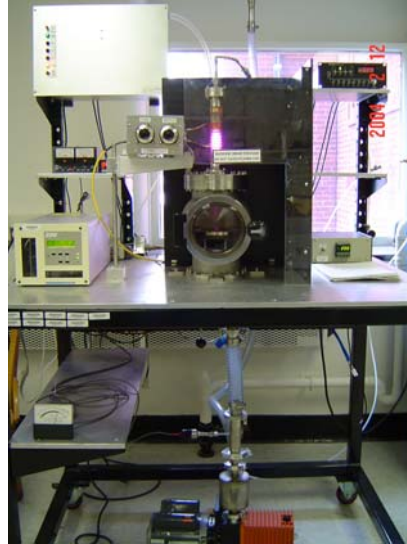
## PECVD of Amorphous Si: Possible Goals

- Gain familiarity with concepts of plasma deposition
  - Inductively* coupled plasma, sheath, plasma power...
  - Comparison to plasma etching
  - Spatial uniformity issues on wafer
- Potential for CFD modeling

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## Amorphous Si PECVD: Apparatus

- Inductively coupled plasma
  - Home-build tuner
  - Need to keep close to wafer surface
- Bottled  $\text{SiH}_4$  diluted in Ar (<0.5% by volume) – below flammability limit



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## Amorphous Si PECVD: Data Collection

- Parameter space explored experimentally over semester with aggregate data from all groups
  - Process P (~500 mtorr)
  - Gas flow rate (~100 sccm  $\text{SiH}_4/\text{Ar}$ )
  - Wafer T (~200°C)
  - RF power (~4 W)
- Film thickness by ellipsometry
- $\text{SiH}_4$  plasma chemistry can be modeled

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## Copper Electrodeposition: Possible Goals

- Gain familiarity with concepts of electrochemistry
  - Redox potentials, buffers...
  - Electrochemical kinetics
  - Comparison to CVD
- Experiment still in planning stages

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## Laboratory Course Challenges

- Initial investment of time  
(several years)



- Initial investment of \$\$\$
- Ongoing expenses \$\$\$
- Experienced staffing
- Setting proper pace, depth
- Creating faculty, student constituency

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## Techniques to Mitigate Challenges (Faculty)

- Design experiments that dovetail to research
  - Permits equip exchange, leverages expertise of support staff
  - UIUC examples
    - CVD/ALD of  $\text{TiO}_2$
    - Surface chemistry/vacuum technology (for equip exchange)
    - Thin film metrology tool
- Engage faculty from several disciplines
  - Adds depth to experiments
  - Offers access to additional financial resources
  - Expands pool of students likely to enroll
  - Aids in staffing

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## Techniques to Mitigate Challenges (Students)

- Add discussion section early in course development
  - Presentations given by students
    - 1 "primary" (25-min) presentation
    - Several "secondary" (5 min)
  - Provides future lecture material
  - Q&A teaches students how in interdisciplinary conversation
  - Presentations, Q&A calibrates what students understand
- Use enrolled students to help build, debug equip in pilot course offerings
  - Less support staff required
  - Design orientation benefits students
  - Expt robustness: typical student mistakes emerge early

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## Summary

- Interdisciplinary lab course with nonstandard equipment
  - Focuses of processing fundamentals
  - Gives exposure to:
    - Elements of process flow, integration
    - Conversation in interdisciplinary groups
- Mixed enrollment simulates dynamics of industrial teams
- Challenges to setup are significant
  - Need multidisciplinary faculty constituency
    - Intellectual fertilization
    - Resource development

