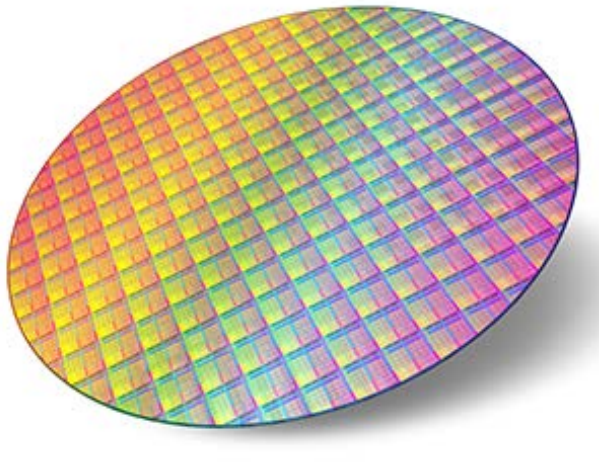
2017 Semiconductor Device Processing

# Oxidation



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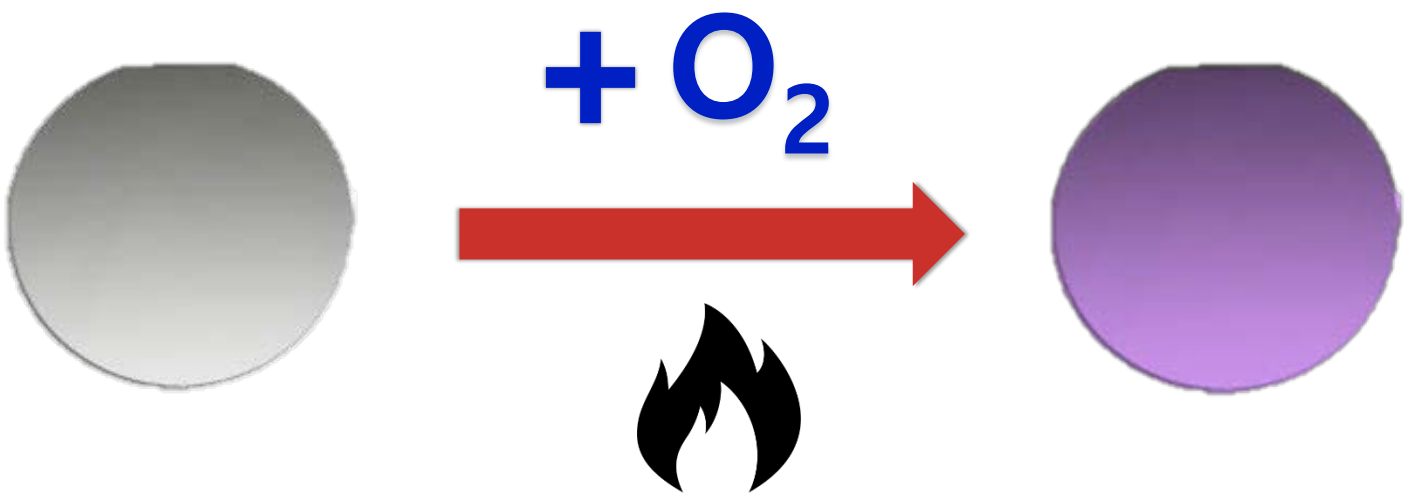
# 1. INTRODUCTION

# 1. Introduction

## 1) Definition of Oxidation

*"In microfabrication, thermal oxidation is a way to produce a thin layer of oxide (usually silicon dioxide) on the surface of a wafer."* Wikipedia "Thermal Oxidation"

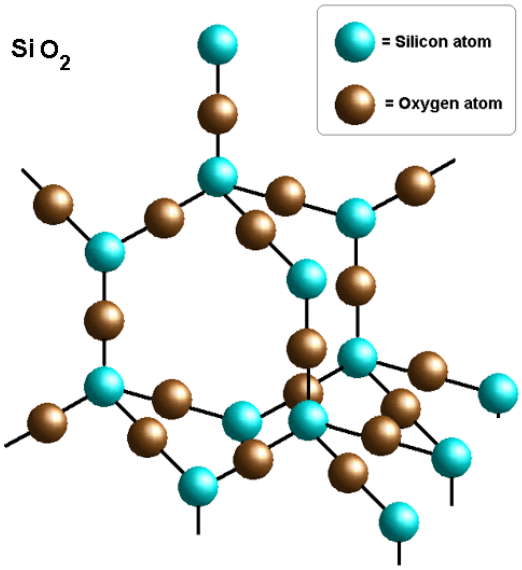


# 1. Introduction

## 2) Silicon Dioxide (Silicon oxide)

*"Silicon dioxide, also known as silica, is a chemical compound that is an oxide of silicon with the chemical formula $SiO_2$ ."*

*Wikipedia "Silicon dioxide"*




<Figure 1: Structure of $SiO_2$>
[http://pixshark.com/sio2-molecule.htm]

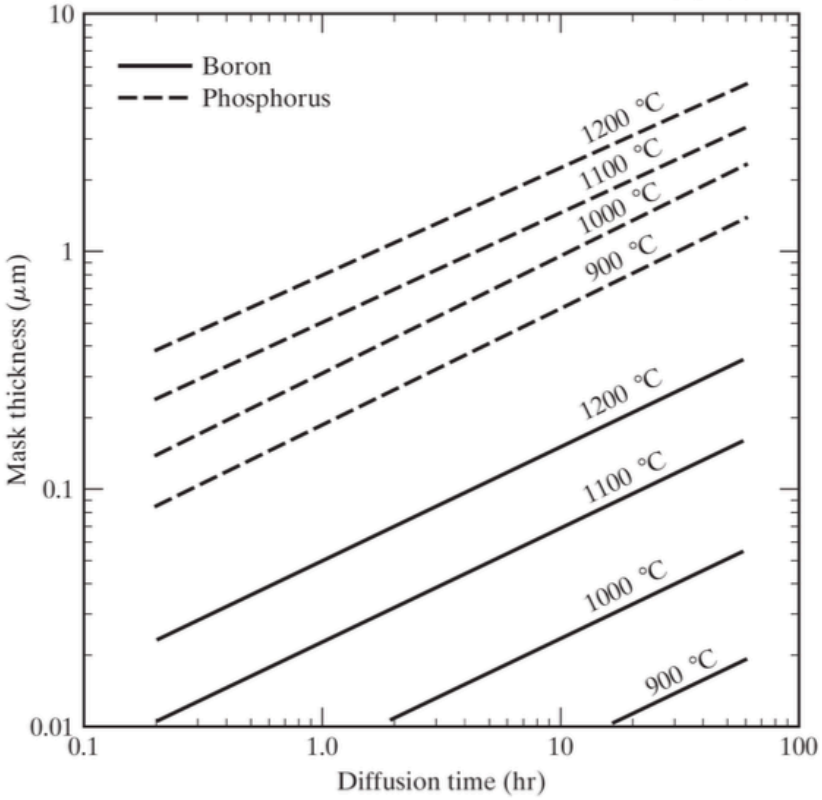
| | |
|---|---|
| Electrical Resistivity | 1 x $10^{13}$ (Ω·m) (20 ℃) |
| Relative Dielectric Constant | 3.9 |
| Index of Refraction | 1.46 |
| Breakdown Field | 6 x $10^{8}$ (V/m) |
| Melting Temperature | 1713 ℃ |

<Table 1: Properties of Silicon Dioxide>

## 3) Usage of oxide

① Mask against ion implantation or diffusion of dopant into Si.

✓ In ion implantation at same condition, silicon nitride is more effective(85 %) than silicon dioxide. On the other hand, photoresist needs 1.8 times than $SiO_2$ thickness.



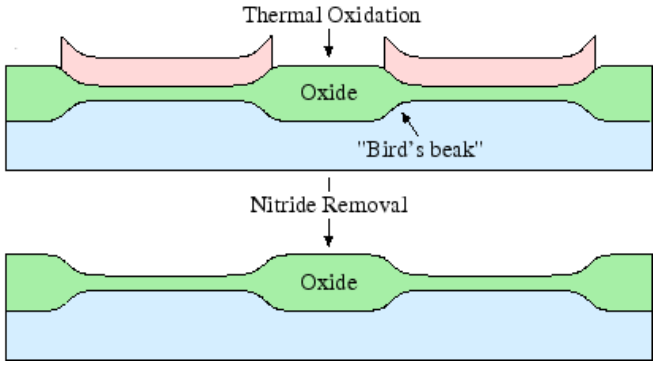

<Figure 2: Thickness of silicon dioxide needed to mask boron and phosphorus diffusions>
[Introduction to Microelectronic Fabrication 2nd - Richard C. Jaeger]

# 1. Introduction

## 3) Usage of oxide

② **Electrical isolation** for device isolation.

- ✓ Silicon dioxide is an excellent **insulator.**

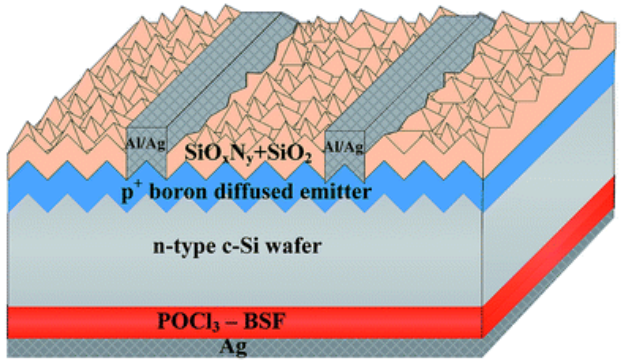



<Figure 3: Example of Isolation(LOCOS)>
[http://www.iue.tuwien.ac.at/phd/hollauer/node7.html]

③ **Surface passivation**

(corrosion, Impurity, stress)

- ✓ **Stable**
- ✓ **High melting Point.**
- ✓ **High breakdown Field**

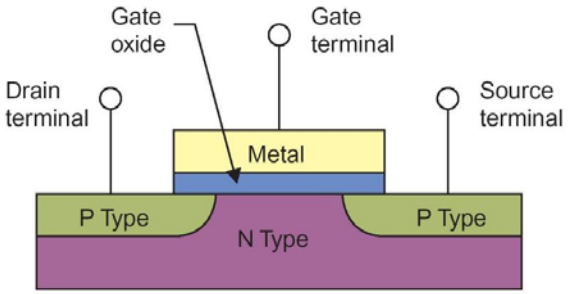



<Figure 4: Example of Surface Passivation>
[http://pubs.rsc.org/en/content/articlehtml/2016/ra/c6ra11043f]

## 3) Usage of oxide

④ Gate oxide in MOS structure & Dielectric material for capacitor in DRAM

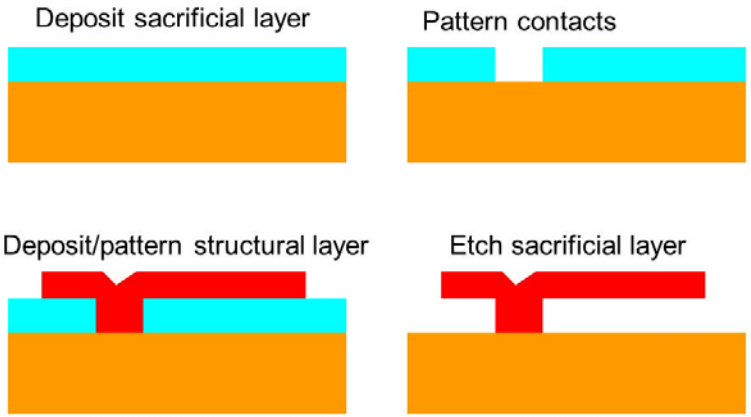
✓ Silicon dioxide is an insulator and has proper permittivity(k).




<Figure 5: Example of Gate oxide>
[http://info.tuwien.ac.at/theochem/si-srtio3_interface/si-srtio3.html]

⑤ Sacrificial layer & Microstructure

✓ Very great etching selectivity between Si and $SiO_2$. (HF)




<Figure 6: Example of Sacrificial layer & Microstructure>
[http://www-bsac.EECS.Berkley.EDU/~pister/245/]

# 1. Introduction

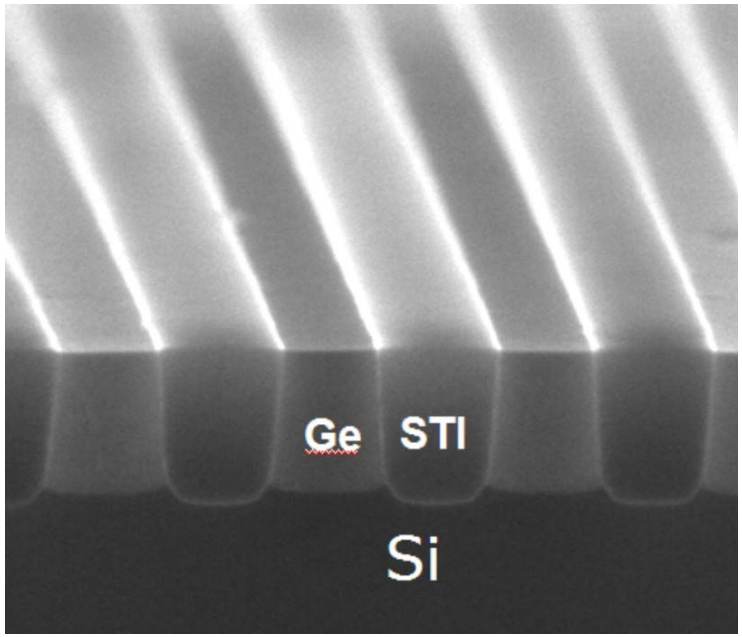
## 3) Usage of oxide

| Semiconductor Applications | Typical Oxide Thickness, Å |
|---|---|
| Gate oxide (0.18 μm generation) | 20 – 60 |
| Capacitor dielectrics | 5 – 100 |
| Dopant masking oxide | 400 – 1,200 |
| STI Barrier Oxide (Shallow Trench Isolation) | 150 |
| LOCOS Pad Oxide (LOCal Oxidation of Silicon) | 200 – 500 |
| Field oxide | 2,500 – 15,000 |

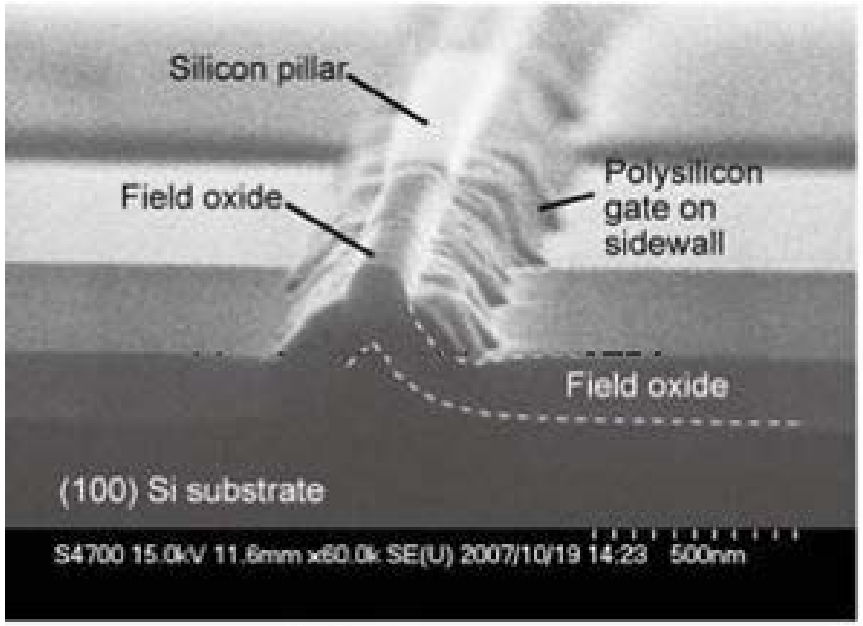
<Table 2: Oxide thickness ranges for applications>

# 1. Introduction

## 3) Usage of oxide




<Figure 7: SEM Image of STI>
[http://www.suggest-keywords.com/c3RpIHRyYW5zaXN0b3I/]




<Figure 8: SEM Image of Field oxide>
[https://www.intechopen.com/books/advances-in-solid-state-circuit-technologies/dimension-increase-in-metal-oxide-semiconductor-memories-and-transistors]

# 2. OXIDE GROWTH

# 2. Oxide Growth

## 1) Growth method

| Temperature | Method |
|---|---|
| 900 ~ 1200 ℃ | Thermal oxidation (Oxygen, Water) |
| 600 ~ 900 ℃ | CVD (pyrolysis of $Si(OC_2H_5)_4$ - TEOS) |
| 250 ~ 600 ℃ | CVD ($SiH_4 + O_2 \rightarrow SiO_2 + 2H_2$) |
| RT ~ 200 ℃ | Sputtering ($SiO_2$ Target, Oxygen) |

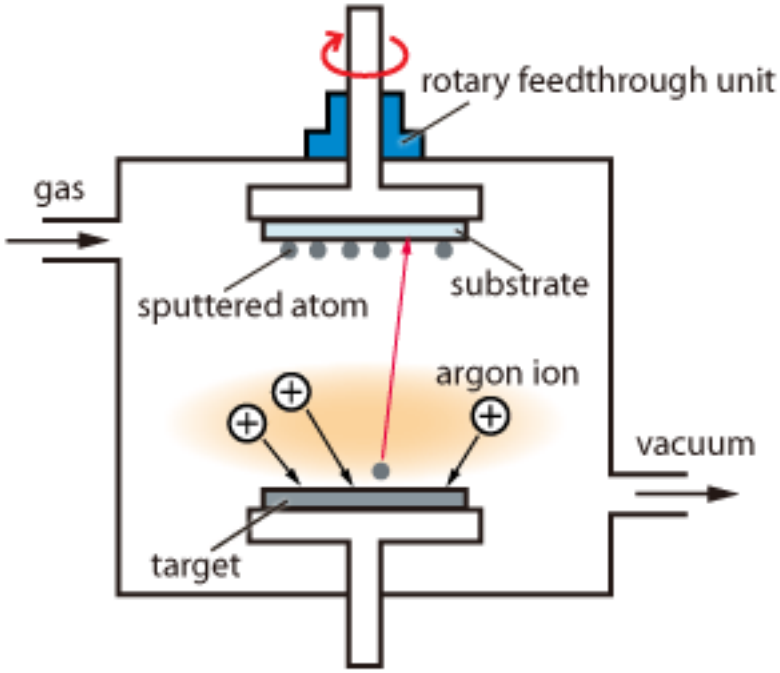
<Table 3: Silicon Oxide Growth Method>

## 2) Sputtering

### Sputter Deposition

✓ *Sputter deposition is a physical vapor deposition (PVD) method of thin film deposition by sputtering. That involves ejecting material from a "target" that is a source onto a "substrate" such as a silicon wafer.*

*Wikipedia "Sputter Deposition"*




<Figure 9: Schematic of sputtering principle>
[http://en.rigaku-mechatronics.com/case/sputtering-systems.html]

## 2) Sputtering

### Characteristics of Sputter Deposition

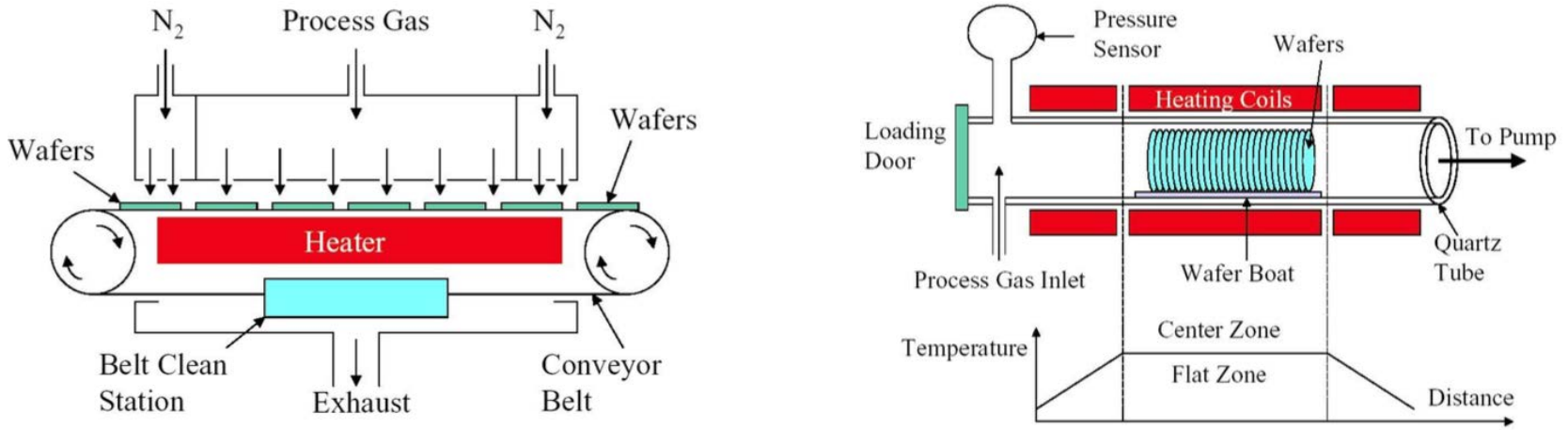
- ✓ Usually uses an argon plasma because argon, a noble gas, will not react with the target material.
- ✓ $SiO_2$ => 30% oxygen, 70% argon mixture from an $SiO_2$ target at a substrate temperature of 200°C
- ✓ Sputtering of dielectrics such as $SiO_2$, $Al_2O_3$ requires an RF power sources to supply energy to the argon atoms.
- ✓ Low temperature process

<Figure 10: $SiO_2$ Target for Sputtering>
[http://www.lesker.com/newweb/deposition_materials/]

**[Ultra-thin silicon-oxide films by sputter- deposition and their application to high-quality poly-Si TFTS** - T Serikawa, and S Shirai]

# 2. Oxide Growth

## 3) CVD (Chemical Vapor Deposition)




<Figure 11: APCVD and LPCVD>

### APCVD(Atmospheric Pressure)

- ✓ *High deposition rate*
- ✓ *Low temperature*
- ✓ *Poor step coverage*
- ✓ *Silicon dioxide passivation layer*

### LPCVD(Low Pressure)

- ✓ *Low pressure (0.1 ~ 1 Torr)*
- ✓ *Good uniformity, step coverage*
- ✓ *High Temperature(550 ℃ ~ 800 ℃)*
- ✓ *TEOS, LOCOS*

# 2. Oxide Growth

## 3) CVD (Chemical Vapor Deposition)

$$SiH_4 + O_2 \rightarrow SiO_2 + 2H_2$$

- ✓ A reaction between Silane (gas source) and Oxygen between 300 ℃ and 500 ℃ is commonly used to deposit $SiO_2$.

$$Si(OC_2H_5)_4 \rightarrow SiO_2 + \text{byproducts}$$

- ✓ Decomposition of the vapor produced from a liquid source, tetraethylorthosilicate(TEOS), can also be used in an LPCVD system between 650 ℃ and 750 ℃
- ✓ Excellent uniformity and step coverage (Ozone addition)

## 4) Thermal Oxidation

✓ Thermal oxidation of silicon is achieved by heating the wafer to a high temperature, typically 800 to 1200 ℃, in an atmosphere containing either pure oxygen or water vapor.

**Dry Oxidation**

$$Si(s) + O_2(g) \rightarrow SiO_2(s)$$

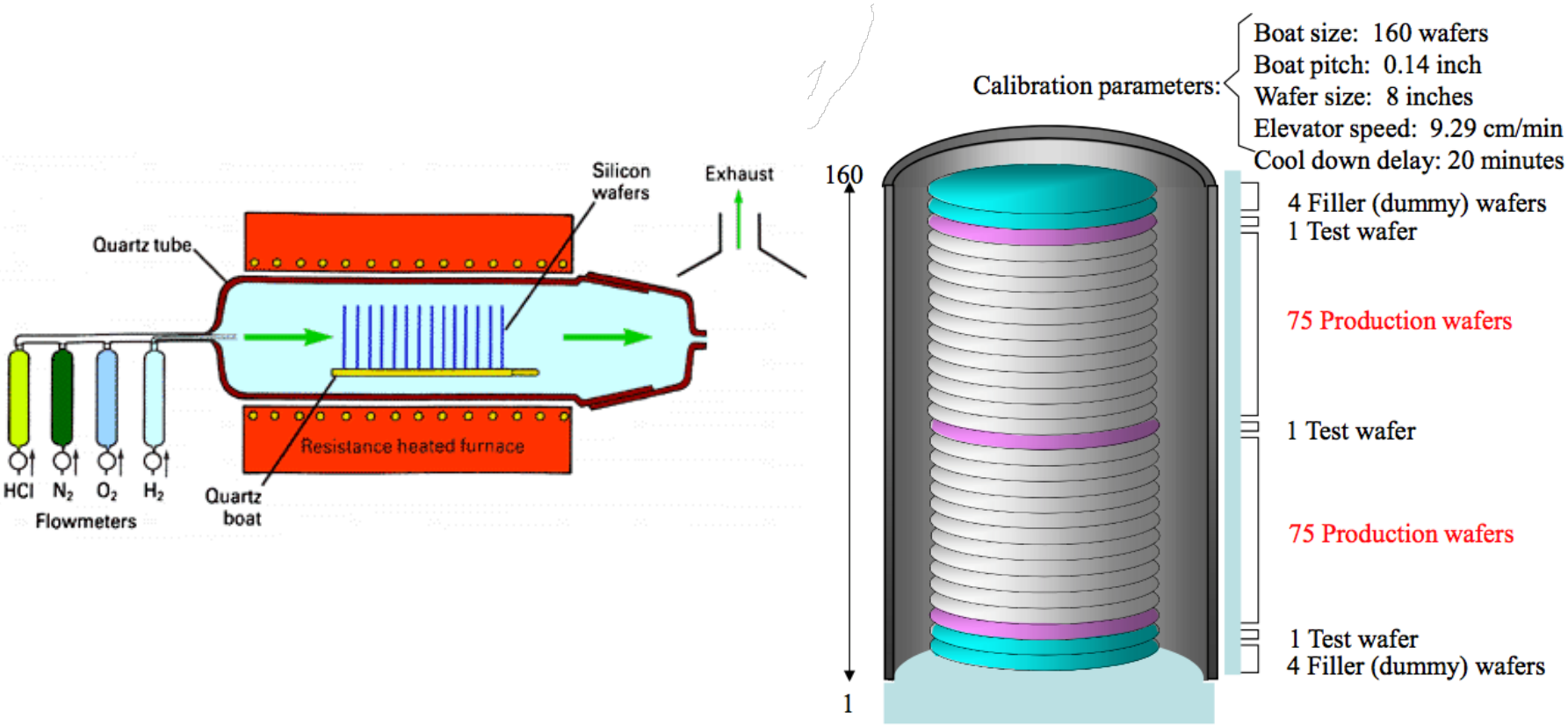
**Wet Oxidation**

$$Si(s) + 2H_2O(g) \rightarrow SiO_2(s) + H_2(g)$$

✓ Wet oxidation is used to grow thick oxides, and widely used for an impurity mask.

✓ Dry oxidation occurs slowly but results in a higher-density oxide than wet oxidation.

## 4) Thermal Oxidation



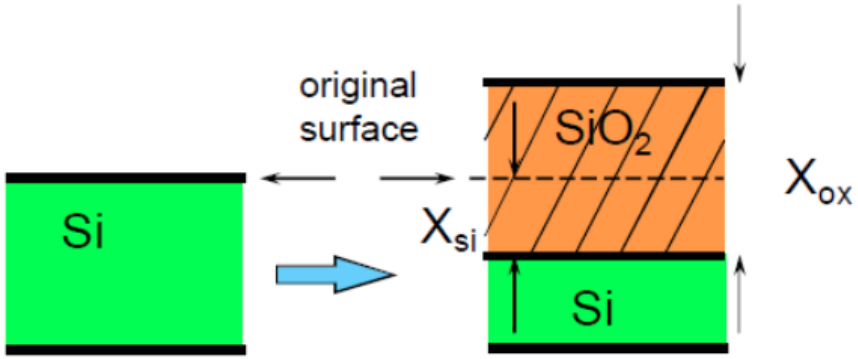

<Figure 12: Schematic of a thermal oxidation furnace and calibration example>
[http://web.eng.gla.ac.uk/groups/sim_centre/courses/oxidation/sigrowth_1.html]

## 4) Thermal Oxidation

<Figure 13: Vertical furnace>
[http://www.spts.com/products/lpcvd-diffusion/rvp-300plus]

<Figure 14: Rapid Thermal Process(RTP)>
[https://allwin21.com/rapid-thermal-processing-rtp/]

## 4) Thermal Oxidation

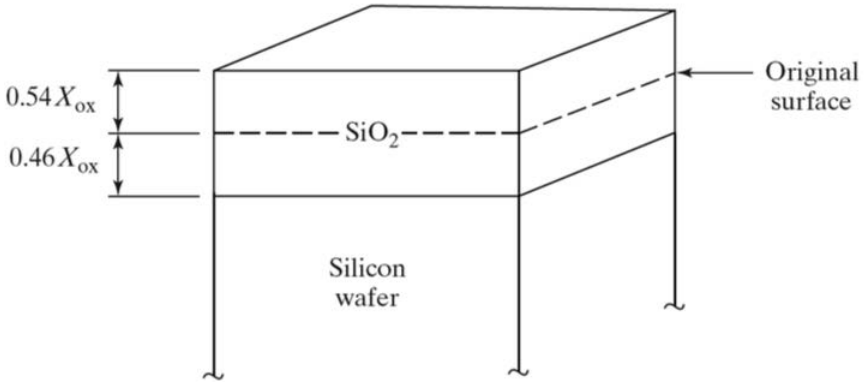


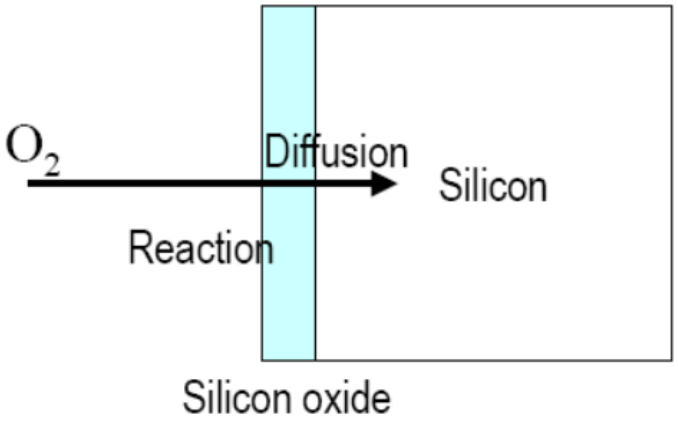

$$X_{si} = X_{ox} \cdot \frac{N_{ox}}{N_{si}}$$

$N_{ox} \Longrightarrow Molecular\ density\ of\ SiO_2$

$N_{si} \Longrightarrow Atomic\ density\ of\ Si$

$$X_{si} = X_{ox} \cdot \frac{2.3 \times 10^{22}\ molecules/cm^3}{5 \times 10^{22}\ atoms/cm^3} = 0.46\, X_{ox}$$



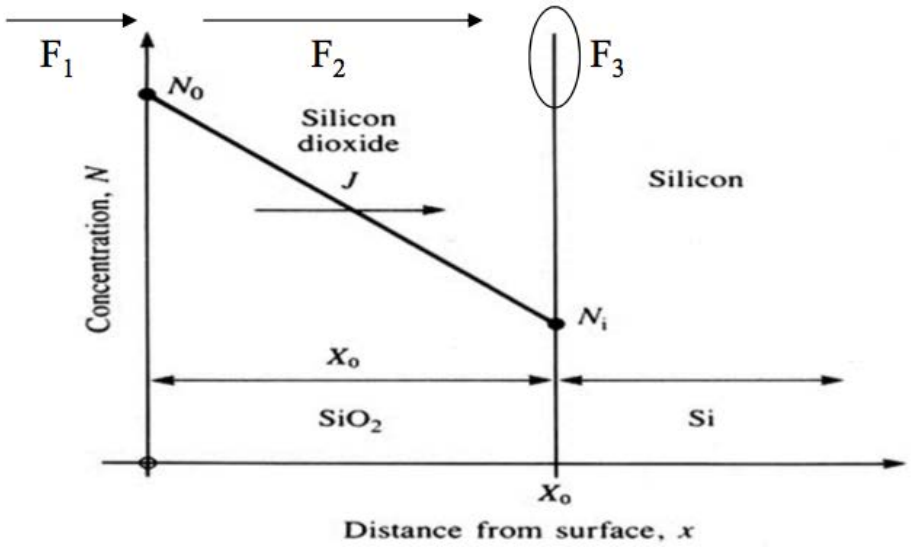




<Figure 15: Ratio of grown oxide>
[Introduction to Microelectronic Fabrication 2nd - Richard C. Jaeger]

# 3. MODELING OXIDATION

# 3. Modeling Oxidation

## 1) Flux Continuation (Steady State)




<Figure 16: Model for thermal oxidation of silicon>
[Introduction to Microelectronic Fabrication 2nd - Richard C. Jaeger]

$F_1$ = flux of oxidant from the gas to the surface

$F_2$ = flux of oxidant from the surface across the existing oxide toward the Silicon (Fick's Law)

$F_3$ = flux of oxidant reacting at the oxide-silicon interface

$$\text{Flux Continuation} : F_1 = F_2 = F_3$$

## 2) Fick's First Law of Diffusion

$$J = F_2 = -D \cdot \frac{\partial N(x,t)}{\partial x} = -D\frac{(N_i - N_0)}{X_0} \qquad \left[\frac{Number\ of\ particles}{cm^2 \cdot sec}\right]$$

D : diffusion coefficient

N : particle concentration

(-) : particles tend to move from a region of high concentration to region of low concentration

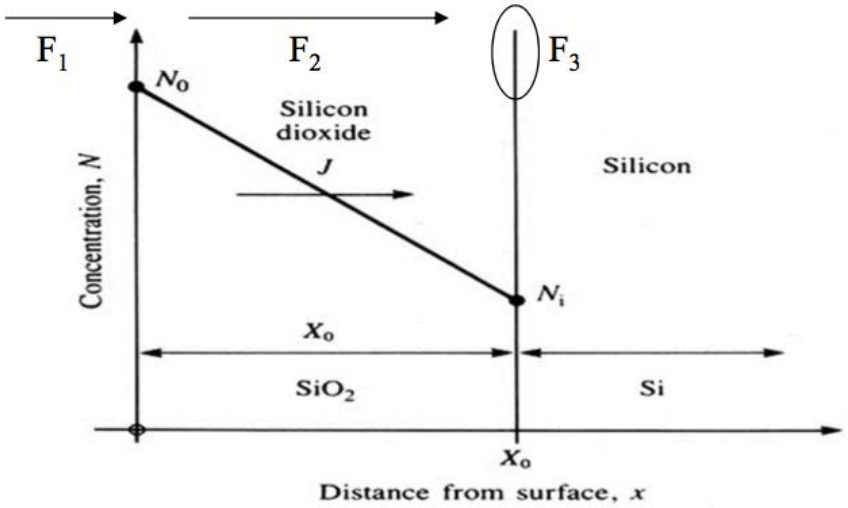
$X_0$ : thickness of the oxide at a given time

$N_0$, $N_i$ : concentration of the oxidizing species in the oxide at the oxide surface and SiO2-Si interface

## 3) The oxidation rate at the $SiO_2$-Si interface

$$J = F_3 = k_s \cdot N_i \qquad J = F_2 = -D\frac{(N_i - N_0)}{X_0}$$

$k_s$ : rate constant for the reaction at the $SiO_2$-Si interface




Eliminating $N_i$ using $F_2$ and $F_3$,

$$\therefore J = \frac{D \cdot N_0}{\left(X_0 + \frac{D}{k_s}\right)}$$

## 4) Thickness changing of the oxide layer with time

$$\frac{dX_0}{dt} = \frac{J}{M} = \frac{\left(\frac{D \cdot N_0}{M}\right)}{\left(X_0 + \frac{D}{k_s}\right)} \Longrightarrow \text{Differential Eq}$$

M : number of molecules of oxidizing species in a unit volume of oxide

**Boundary Conditions**

$$X_0 = X_i \ (t = 0)$$

## 5) Deal-Grove Model

$$t = \frac{{X_0}^2}{B} + \frac{X_0}{\left(\frac{B}{A}\right)} - \tau \quad \Longrightarrow \quad {X_0}^2 + AX_0 = B(t + \tau)$$

$$A = \frac{2D}{k_s} \qquad B = \frac{2DN_0}{M} \qquad \tau = \frac{{X_i}^2}{B} + \frac{X_i}{\left(\frac{B}{A}\right)}$$

$X_i$ : initial thickness of oxide on the wafer

$\tau$ : the time required to grow the initial oxide

A thin native oxide layer (10 to 20 Å) is always present on silicon due to atmospheric oxidation, or $X_i$ may represent a thicker oxide grown during previous oxidation steps.

## 5) Deal-Grove Model

$$\therefore X_0(t) = \frac{1}{2}A\left[\left\{1 + \frac{4 \cdot B}{A^2} \cdot (t + \tau)\right\}^{\frac{1}{2}} - 1\right]$$

***Short Times***

$$(t + \tau) \ll \frac{A^2}{4 \cdot B}$$

$$X_0(t) = \left(\frac{B}{A}\right) \cdot (t + \tau)$$

$\frac{B}{A}$ : Linear rate constant

***Long Times***

$$(t + \tau) \ll \frac{A^2}{4 \cdot B}, t \gg \tau$$

$$X_0(t) = \sqrt{B \cdot (t + \tau)}$$

$B$ : Parabolic rate constant

## 5) Deal-Grove Model

### *Short Times*

$$(t+\tau) \ll \frac{A^2}{4 \cdot B}$$

$$X_0(t) = \left(\frac{B}{A}\right) \cdot (t+\tau)$$

- ✓ Oxidation growth is proportional to time.
- ✓ In this region, growth rate is limited by the reaction at the Si interface.

### *Long Times*

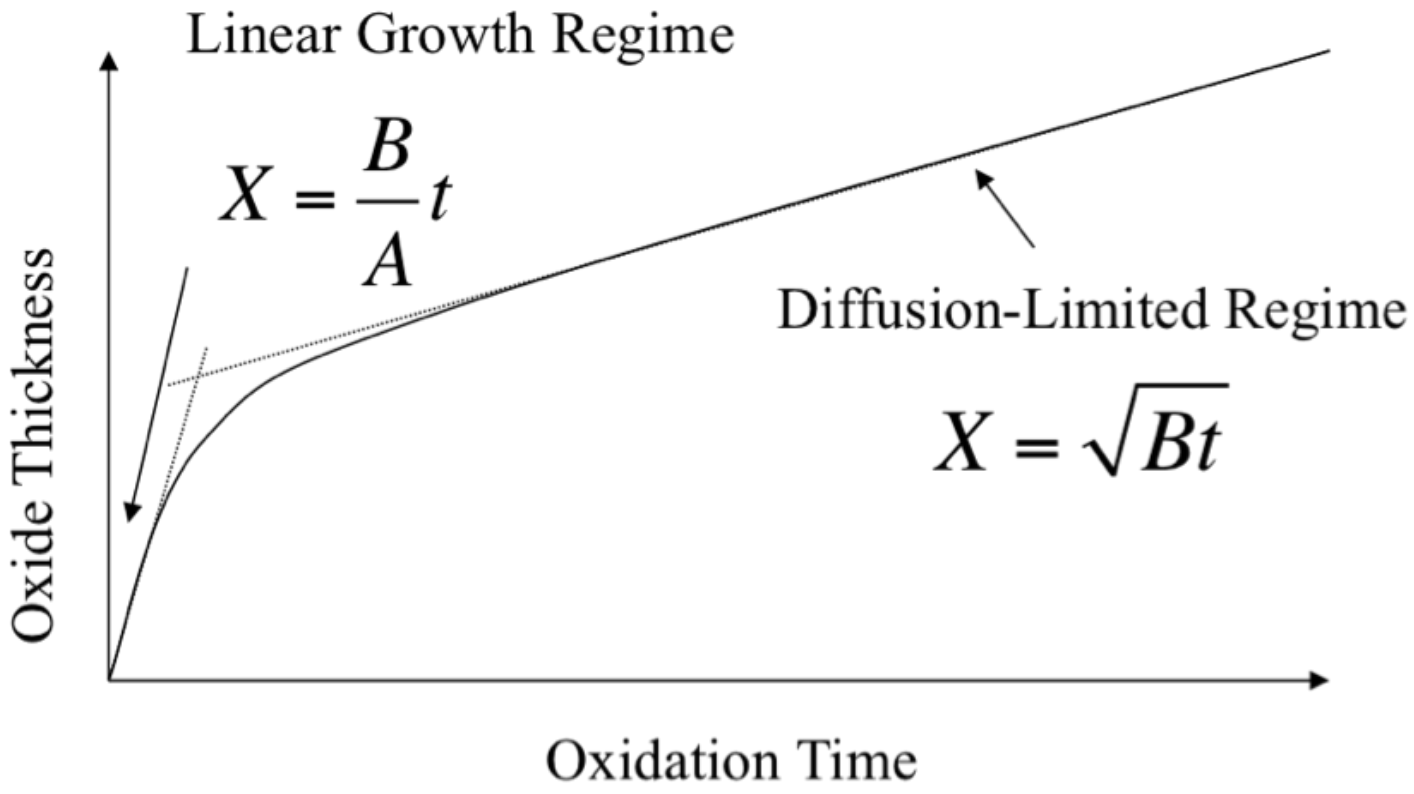
$$(t+\tau) \ll \frac{A^2}{4 \cdot B}, t \gg \tau$$

$$X_0(t) = \sqrt{B \cdot (t+\tau)}$$

- ✓ Oxidation growth is proportional to the square root of time.
- ✓ In this region, oxidation rate is diffusion limited.

# 3. Modeling Oxidation

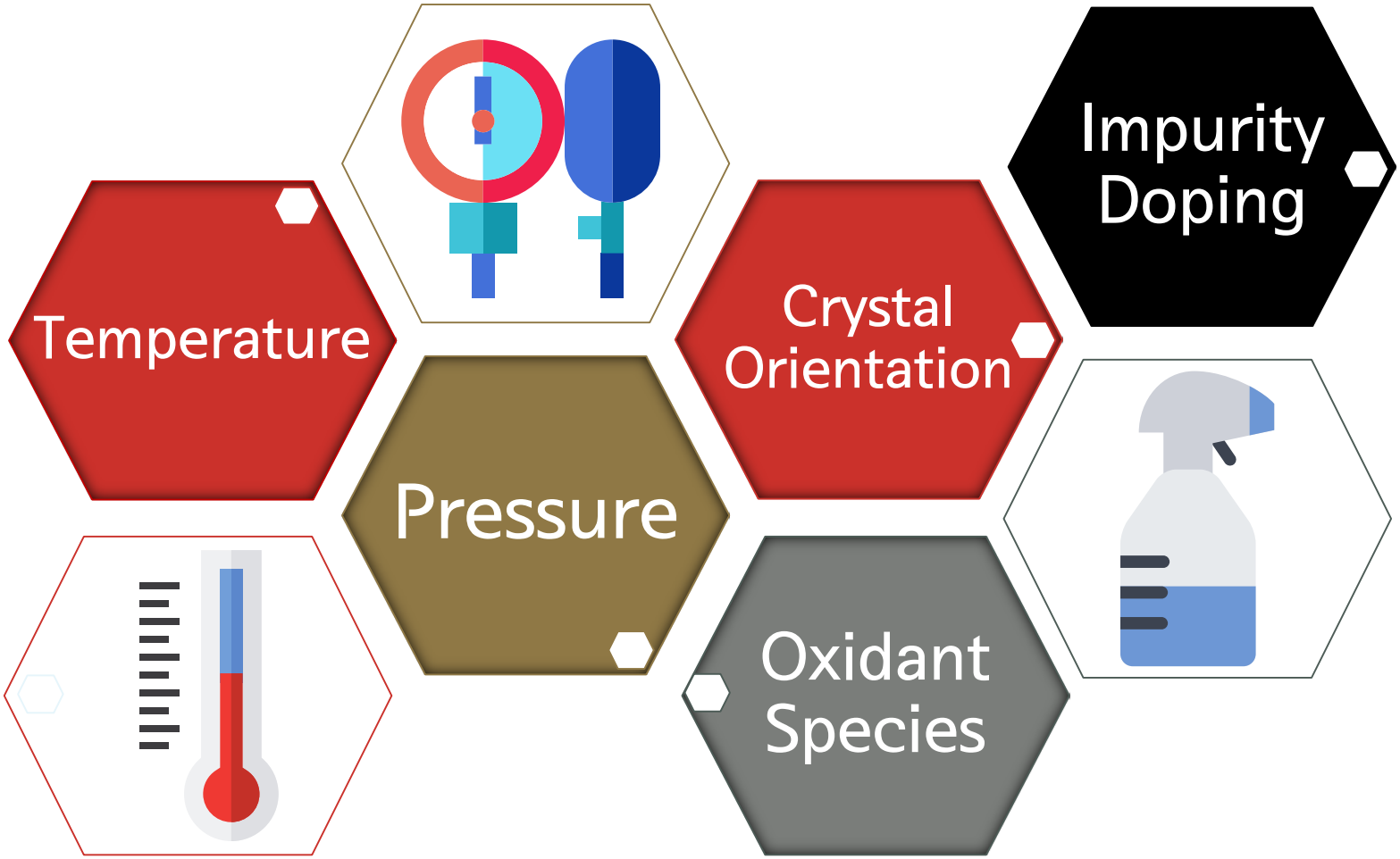
## 5) Deal-Grove Model




<Figure 17: The Basic Deal-Grove Model>
[http://www.semitracks.com/newsletters/october/2014-october-newsletter.php]

# 3. Modeling Oxidation

## 6) Factors Influencing Oxidation Rate



# 3. Modeling Oxidation

## 6) Factors Influencing Oxidation Rate

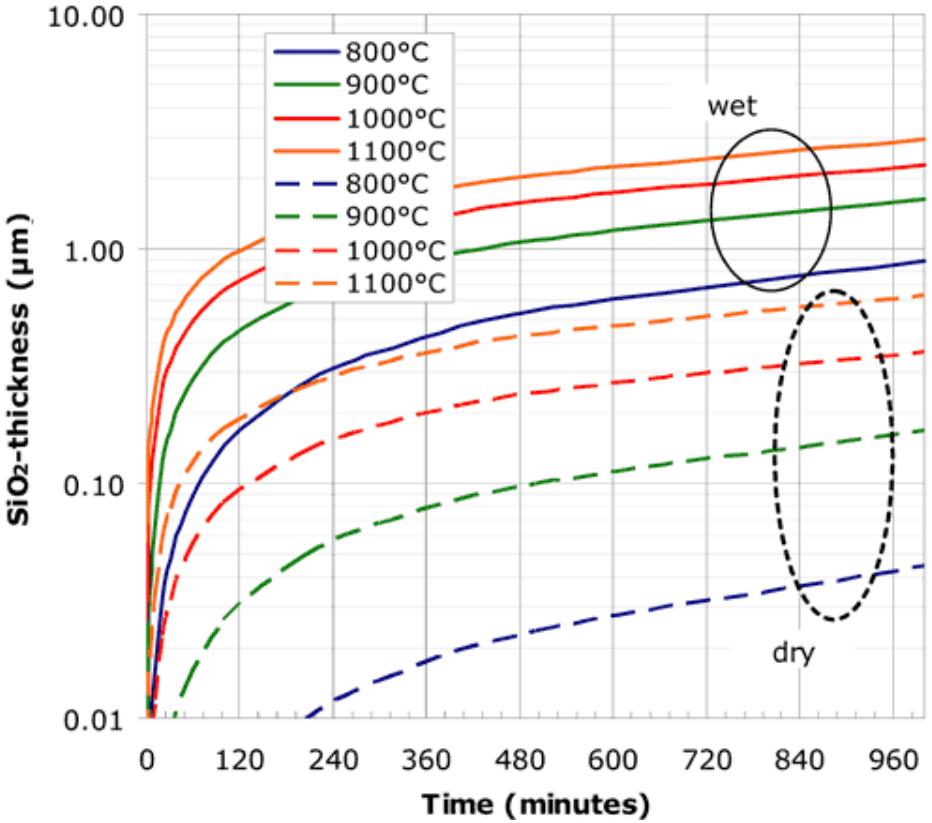
① **Temperature** influences both linear and parabolic rate constant.

**Arrhenius equation**

$$k = A\exp\left(\frac{-E_A}{k_B T}\right)$$

② **Oxidant Species**

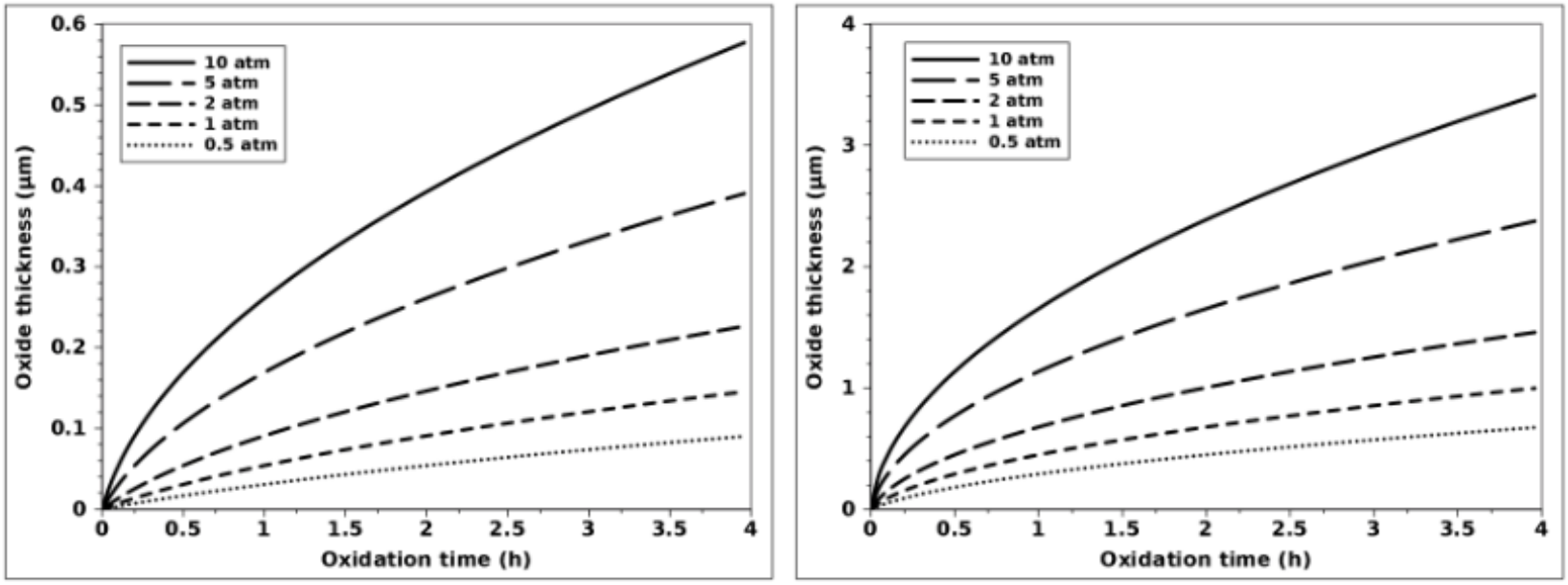
✓ **Water vapor has a much higher solubility than $O_2$** in SiO2, which accounts for much higher oxide growth rate in a wet atmosphere.




<Figure 18: Oxide thickness-time graph>
[http://www.microchemicals.com/products/wafers/wafers_sinx_si3n4_sio2.html]

# 3. Modeling Oxidation

## 6) Factors Influencing Oxidation Rate



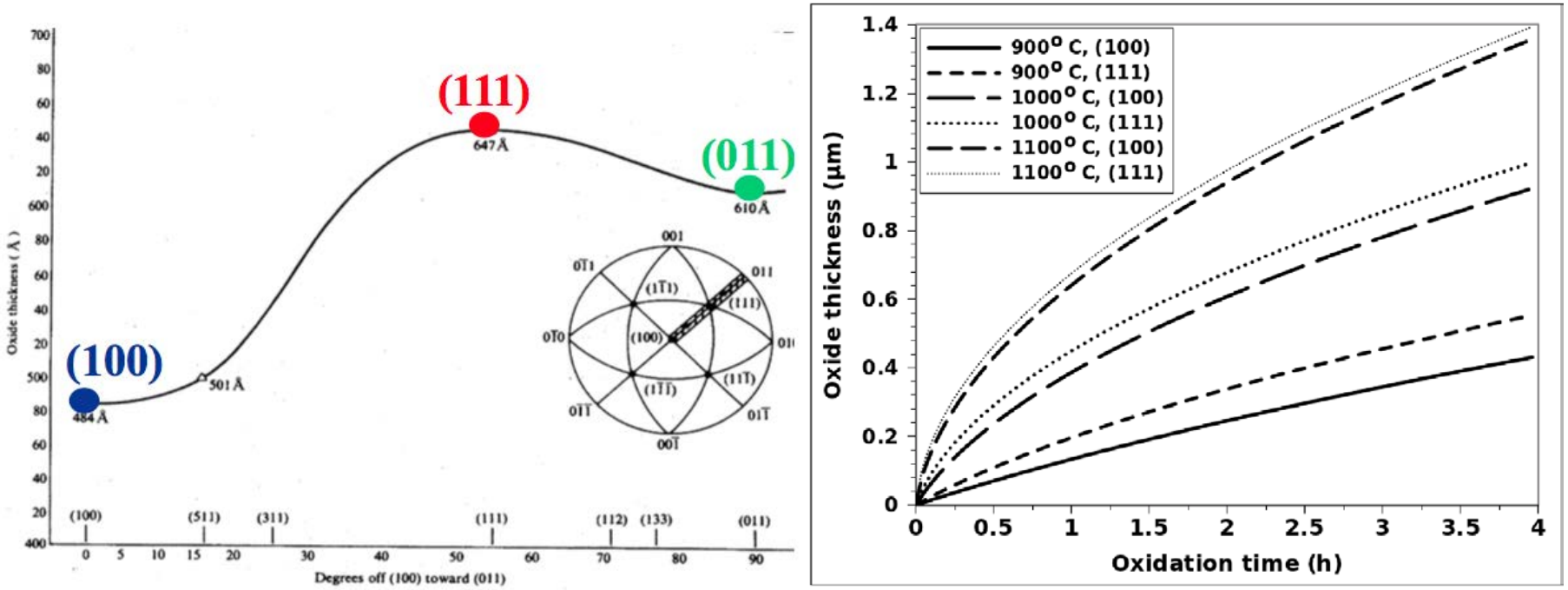

(a) Dry oxidation (b) Wet oxidation

<Figure 19: Effects of pressure on thermally grown oxide thickness >
[http://www.iue.tuwien.ac.at/phd/filipovic/node29.html]

③ Pressure is proportional the partial pressure of the oxidizing species, so pressure can be used to control oxide growth rate.

# 3. Modeling Oxidation

## 6) Factors Influencing Oxidation Rate



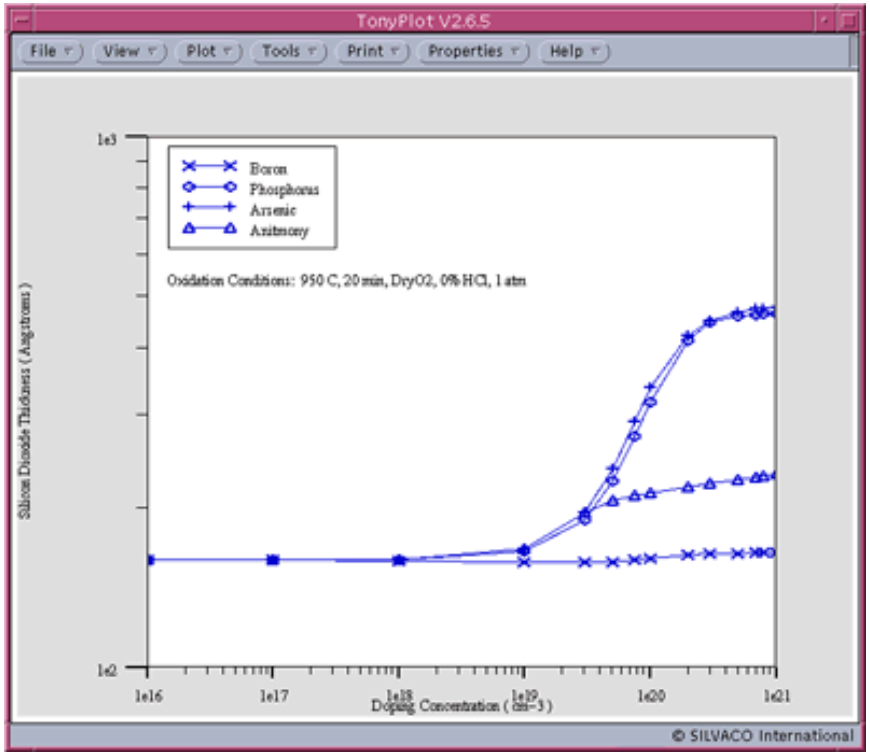

<Figure 20: Oxide thickness versus oxidation time for (100) and (111) oriented silicon>
[http://www.iue.tuwien.ac.at/phd/filipovic/node29.html]

④ Crystal Orientation changes the number of silicon bonds available at the silicon surface, which influences the oxide growth rate.

## 6) Factors Influencing Oxidation Rate

⑤ **Impurity doping :** Heavy doping of silicon changes oxidation characteristics.

- ✓ **Phosphorus** doping increases the **linear rate constant.**
- ✓ **Boron** doping increases the **parabolic rate constant.**
- ✓ Impurity redistribution during oxidation

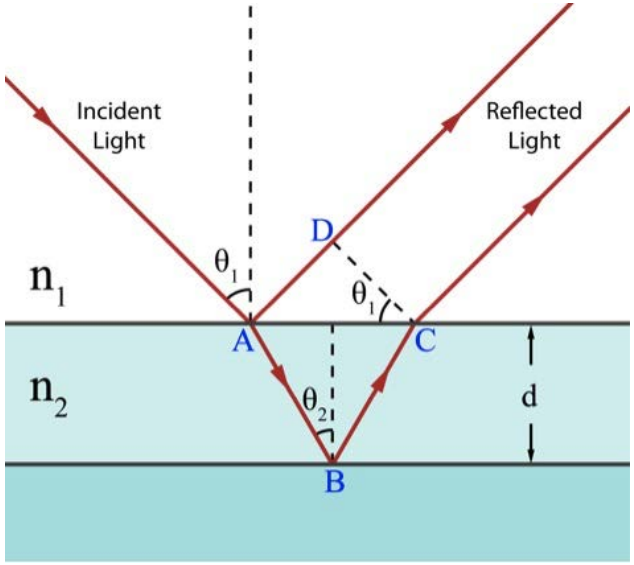



<Figure 21: Simulated Silicon Dioxide Thickness Versus Doping Concentration for Common Silicon Dopants.>
[http://www.silvaco.com/tech_lib_TCAD/simulationstandard/1997/aug/a3/a3.html]

# 4. INSPECTION

# 4. Inspection

## 1) Oxide Thickness Color Chart




<Figure 22: Constructive interference in thin film>
[http://www.tohotechnology.com/nanospec-6500.php]

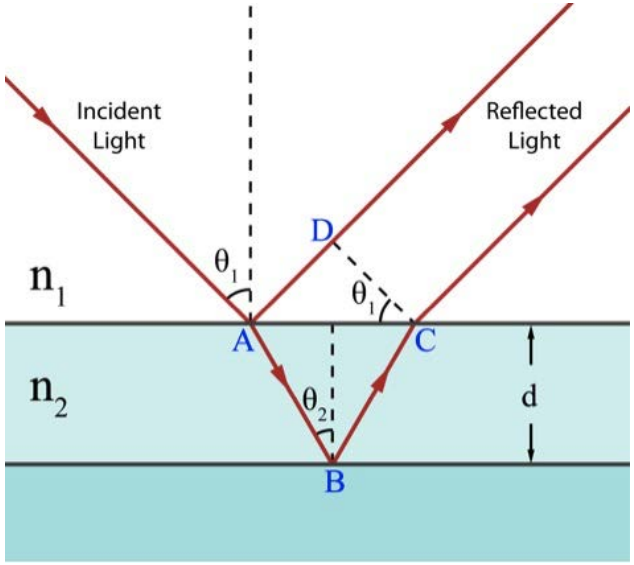
| Film Thickness (Å) | Color of Film (those shown are only indicative) |
|---|---|
| 500 | tan |
| 700 | brown |
| 1000 | dark violet to red violet |
| 1200 | royal blue |
| 1500 | light blue to metallic blue |
| 1700 | metallic to very light yellow-green |
| 2000 | light gold or yellow - slightly metallic |
| 2200 | gold with slight yellow-orange |
| 2500 | orange to melon |
| 2700 | red-violet |
| 3000 | blue to violet-blue |
| 3100 | blue |
| 3200 | blue to blue-green |
| 3400 | light green |
| 3500 | green to yellow-green |
| 3600 | yellow-green |
| 3700 | green-yellow |
| 3900 | yellow |
| 4100 | light orange |
| 4200 | carnation pink |
| 4400 | violet-red |
| 4600 | red-violet |
| 4700 | violet |
| 4800 | blue-violet |
| 4900 | blue |
| 5000 | blue-green |
| 5200 | green |
| 5400 | yellow-green |
| 5600 | green-yellow |
| 5700 | yellow to "yellowish" (at times appears light gray or metallic) |
| 5800 | light orange or yellow to pink |
| 6000 | carnation pink |
| 6300 | violet-red |
| 6800 | "bluish" (appears between violet-red and blue-green - overall looks grayish |
| 7200 | blue-green to green |
| 7700 | "yellowish" |
| 8000 | orange |
| 8200 | salmon |
| 8500 | dull light red-violet |
| 8600 | violet |
| 8700 | blue-violet |
| 8900 | blue |
| 9200 | blue-green |
| 9500 | dull yellow-green |
| 9700 | yellow to "yellowish" |
| 9900 | orange |

① Oxide thickness color chart is one of the simplest methods for determining the thickness of an oxide.

<Figure 23: $SiO_2$ Thickness Color Chart.>

# 4. Inspection

## 1) Oxide Thickness Color Chart



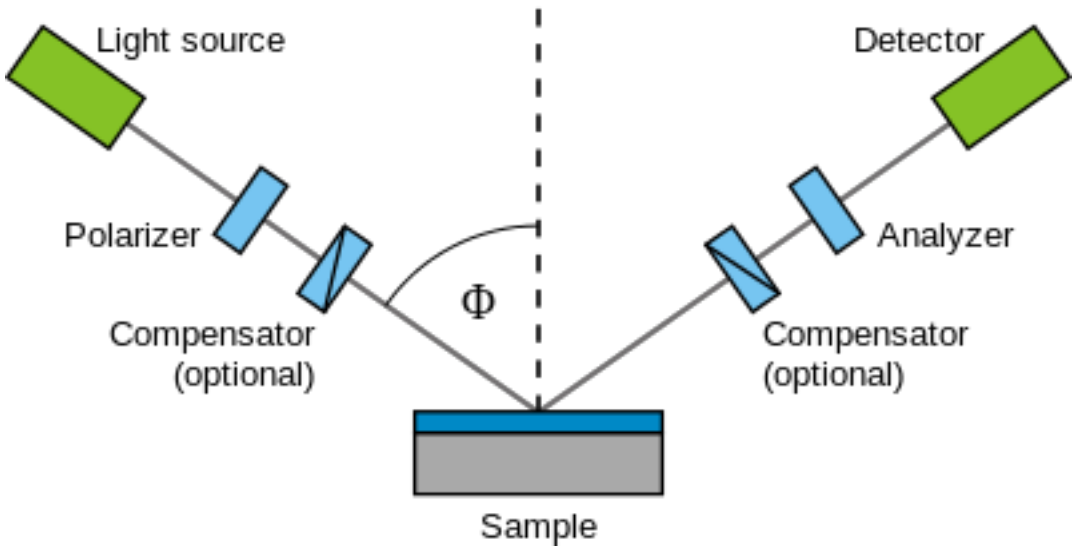

$$2X_0 = \frac{k\lambda}{n}$$

*k is any integer greater than zero.*
*n is the refractive index of oxide*
*($n = 1.46$ for $SiO_2$)*

- ✓ When the wafer is illuminated with white light perpendicular to the surface, the light penetrates the oxide film and is reflected by the underlying silicon wafer.
- ✓ Constructive interference causes enhancement of a certain wavelength in the reflected light, and the color of wafer corresponds to the enhanced wavelength.

# 4. Inspection

## 2) Ellipsometry




<Figure 24: Schematic setup of an ellipsometry experiment>
[https://en.wikipedia.org/wiki/Ellipsometry]

② *"Ellipsometry is an optical technique for investigating the dielectric properties of thin films. Ellipsometry measures the change of polarization upon reflection or transmission and compares it to a model."* *Wikipedia "Ellipsometry"*

# 4. Inspection

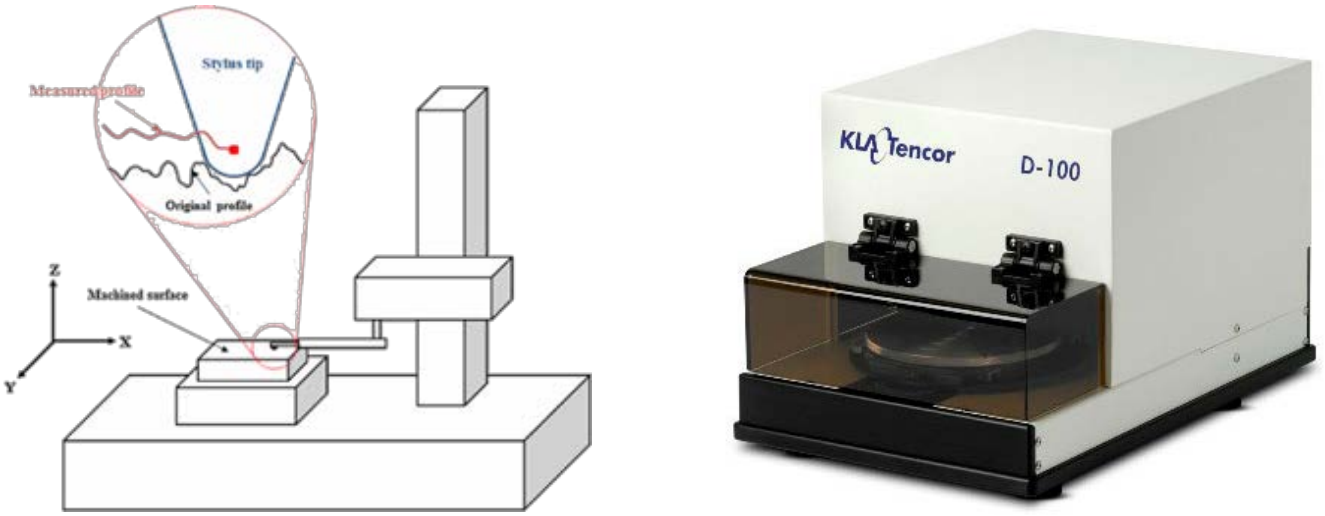
## 2) Ellipsometry

<Figure 25: Ellipsometer>
[http://mmrc.caltech.edu/Ellipsometer/Ellipsometer.html]
[http://www.azom.com/materials-equipment.aspx?cat=14]

- ✓ *It can be used to characterize composition, roughness, thickness (depth), crystalline nature, doping concentration, electrical conductivity and other material properties.*

# 4. Inspection

## 3) Stylus Profiler (Alpha-Step)




<Figure 26: Schematic of a stylus profiler and Alpha-Step>
[http://www.nanoscience.com/technology/optical-profiler-technology/how-profilometer-works/]
[http://www.millice.com.sg/products/kla-tencor/kla-tencor-alpha-step-d-100-stylus-profiler/]

③ *"Profiler is a measuring instrument used to measure a surface's profile, in order to quantify its roughness. Critical dimensions as step, curvature, flatness are computed from the surface topography"* *Wikipedia "Profilometer"*

# 5. SUMMARY

# 5. Summary

## 1. Introduction
1) Definition of oxidation
2) Silicon Dioxide
3) Usage of oxide

## 2. Oxide Growth
1) Growth method
2) Sputtering
3) CVD
4) Thermal oxidation

## 3. Modeling Oxidation
1) Flux continuation
2) Fick's First Law of Diffusion
3) Deal-Grove Model
4) Factors Influencing Oxidation Rate

## 4. Inspection
1) Color chart
2) Ellipsometry
3) Stylus Profiler

# 6. Reference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2. https://en.wikipedia.org/wiki/Silicon_dioxide
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4. https://en.wikipedia.org/wiki/Thermal_oxidation
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14. **VLSI Technology 2nd** - S.M. SZE
15. ELECTRONIC MATERIALS, DEVICES, AND FABRICATION Section III: Fabrication

# Thank You
# Any questions?

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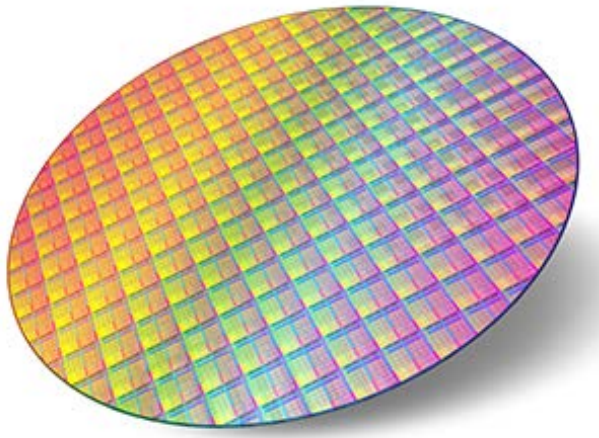
# Supplementary Materials



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Gwangju Institute of Science and Technology

# 1. Thermal Oxidation for the Other Materials.

## GaAs Oxidation

**Thermal Oxidation of III-V Materials and Heterostructures**

R. J. Hussey, R. Driad,[a] G. I. Sproule, S. Moisa, J. W. Fraser, Z. R. Wasilewski, J. P. McCaffrey,[b] D. Landheer,* and M. J. Graham*,[z]

*National Research Council of Canada, Institute for Microstructural Sciences, Ottawa, Canada K1A 0R6*

*GaAs*.—Thermal oxides formed on **GaAs(100) at 500°C in oxygen or moist air** (air bubbled through $H_2O$ at 25 or 95°C) **or moist nitrogen** ( $N_2$ bubbled through $H_2O$ at 95°C) comprise principally $Ga_2O_3$ to a level of *ca.* 97%.
There are small enrichments of As at both the gas/oxide and the oxide/substrate interface, the latter probably as a result of the disproportionation reaction

$$As_2O_3 + 2GaAs \rightarrow Ga_2O_3 + 4As$$

# 1. Thermal Oxidation for the Other Mateials.

## Cupric Oxide (CuO)

**Thermal Oxidation of Copper for Favorable Formation of Cupric Oxide (CuO) Semiconductor**

Uma Nerle and M. K. Rabinal*
*(Department of Physics, Karnatak University Dharwad - 580 003, Karnataka, India)*
**Corresponding author email: mkrabinal@yahoo.com*

Open air thermal oxidation of copper has been studied to obtain photovoltaic active cupric oxide semiconductor (CuO).

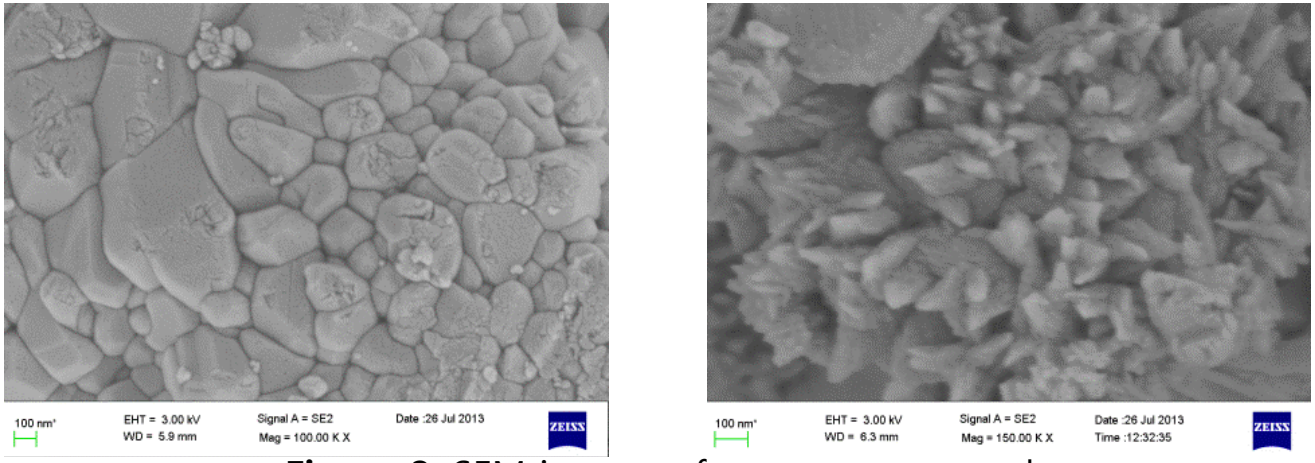


**Figure 3**: SEM images of pure copper powder,
(a) oxidized at 500 °C and (b) oxidized at 700 °C for 60 minutes.

# 1. Thermal Oxidation for the Other Mateials.

## $Al_2O_3$ Passivation for AlGaN/GaN

**Effect of $Al_2O_3$ Surface Passivation by Thermal Oxidation of Aluminum for AlGaN/GaN Structure**

Jeong-Jin Kim[1,2], Ho-Kyun Ahn[2], Seong-Bum Bae[2], Young-Rak Pak[2], Jong-Won Lim[2], Jae-Kyung Moon[2], Sang-Chun Ko[2], Kyu-Hwan Shim[1], and Jeon-Wook Yang[1,a]

[1] Department of Semiconductor and Chemical Engineering, Chonbuk University, Jeonju 561-756, Korea

[2] Electronics and Telecommunications Research Institute, Daejeon 305-700, Korea

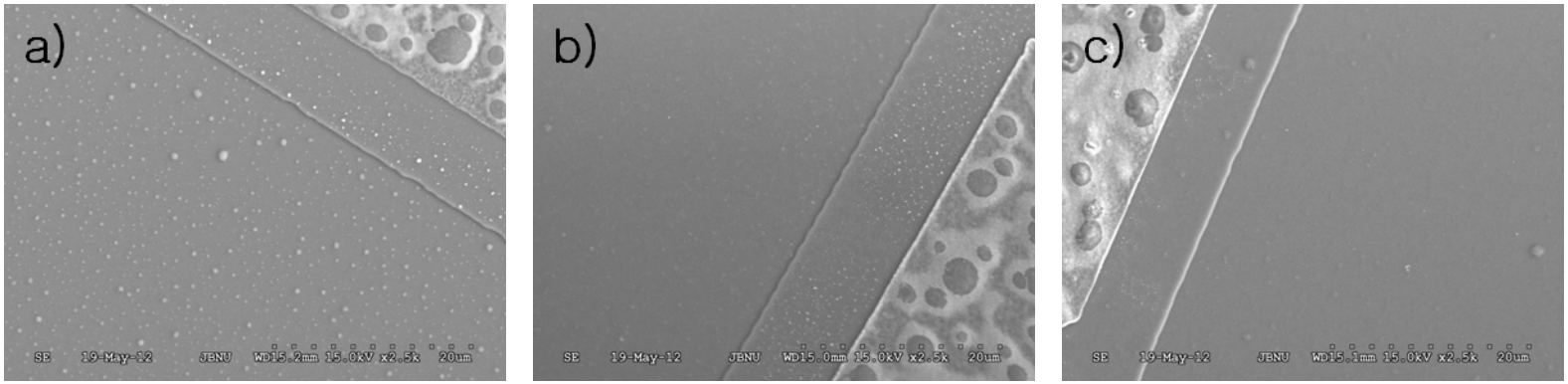


**Fig. 1**. Surface morphology of Al2O3 films (a) annealed at 525℃, (b) annealed at 600℃, (c) annealed at 675℃.

# 2. steady state

**Steady state is the theoretical assumption.**

Flux Continuation : $F_1 = F_2 = F_3$

In steady state

The Deal-Grove model assumes that each of these stages proceeds at a rate proportional to the oxidant's concentration. In the first case, this means Henry's law; in the second, Fick's law of diffusion; in the third, a first-order reaction with respect to the oxidant. It also assumes steady state conditions, i.e. that transient effects do not appear.

*Wikipedia "Deal-Grove model"*

# 2. steady state

## Steady state is the theoretical assumption.

The Deal-Grove model works very well for single-crystal silicon under most conditions. However, experimental data shows that very thin oxides (less than about 25 nanometers) grow much more quickly in O2 than the model predicts.

Deal-Grove also fails for polycrystalline silicon ("poly-silicon"). First, the random orientation of the crystal grains makes it difficult to choose a value for the linear rate constant. Second, oxidant molecules diffuse rapidly along grain boundaries, so that poly-silicon oxidizes more rapidly than single-crystal silicon.

*Wikipedia "Deal-Grove model"*

## How to measure surface roughness using Ellipsometry

**Experimental Study of Polarization Properties of Rough Surface**

S. N. Svitasheva

Rzhanov Institute of Semiconductors Physics of RAS, ac. Lavrentiev Av. 13, Novosibirsk, 630090, Russia

**ERRORS ARISING FROM SURFACE ROUGHNESS IN ELLIPSOMETRIC MEASUREMENT OF THE REFRACTIVE INDEX OF A SURFACE**

CARL A. FENSTERMAKER and FRANK L. McCRACKIN

Institute for Materials Research, National Bureau of Standards, Washington, D.C. 20234, U.S.A.

**Roughness measurements by spectroscopic ellipsometry**

J. R. Blanco, P. J. McMarr, and K. Vedam

# Thank You
# Any questions?

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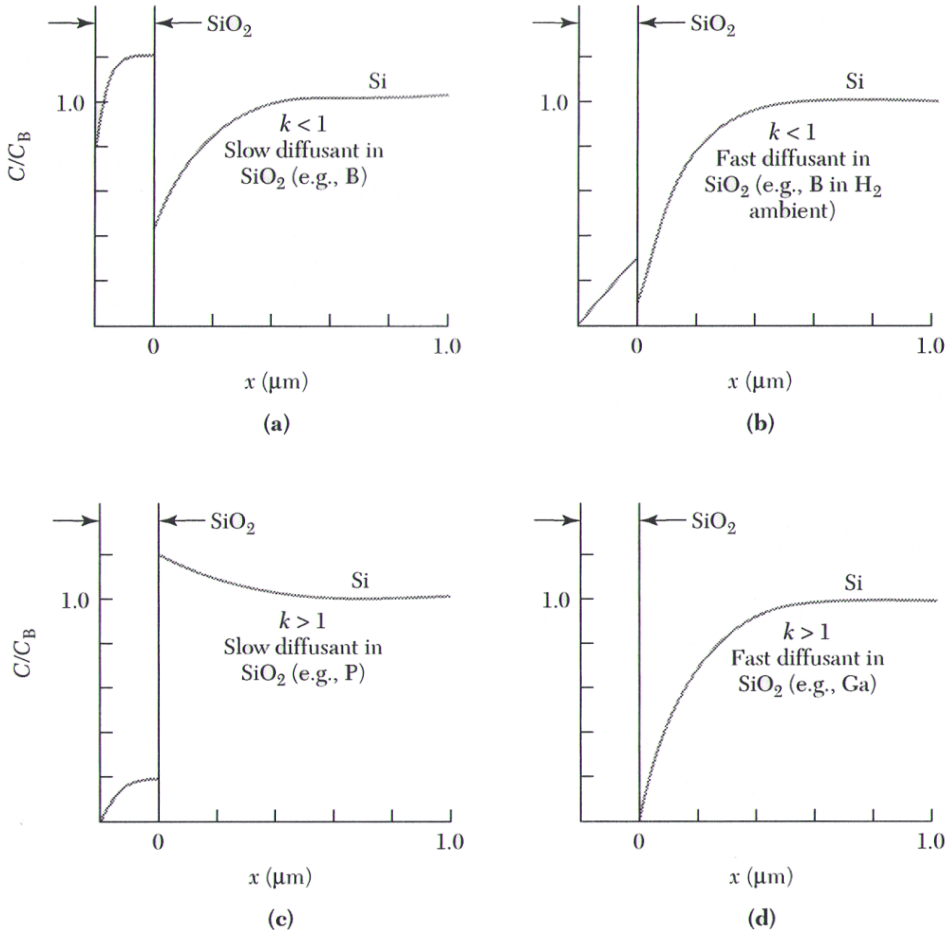
# Appendix

## Impurity redistribution during oxidation




$$\kappa = \frac{\text{Impu. concen. in Si}}{\text{Impu. concen. in } SiO_2}$$

*Segregation*