Lecture #4

Basic Microfabrication (I)

Etching Process
Outline

• Examples of etching process in biomedicine
• Subtractive process: Removal of material (bulk substrate, metal, or oxide thin films)
  – Pattern transfer from lithography
  – Sacrificial process
• Wet Etching
  – Crystal orientation
• Dry etching
  – Plasma etching and Physical etching
• Deep RIE mechanisms
• Summary
Examples of Etching Process

Selective Etching in Dental Medicine

Wet Etched Si Microneedle Array
Examples of Etching Process

Microneedle Vaccine Patches
Examples of Etching Process

Microneedle Drug Patches

SOURCE: Hewlett Packard
Etched Transistor to Interface Neuron

Tune Ion Channels!!

Prof. Peter Fromherz Group
Max-Planck-Institute for Biochemistry

Signal Transmission from Individual Mammalian Nerve Cell to Field-Effect Transistor

Moritz Voelker and Peter Fromherz,
Small 1 (2005)
Si Etching for BioMEMS

Stable and Reproducible Bilayer Lipid Membranes Based on Silicon Microfabrication Techniques

Ayumi Hirano-Iwata et al., Advances in Planar Lipid Bilayers and Liposomes, 2010
Selective Etching in BioMEMS

Stable and Reproducible Bilayer Lipid Membranes Based on Silicon Microfabrication Techniques

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Selective Etching in Nanotechnology

Shape evolution of Pd concave polyhedra via an in situ facet-selective etching route

- Concave palladium polyhedra by selectively etching the \{100\} facets in situ by $I^-$ ions.

- Due to the presence of a high density of atomic steps and surface relaxation, the concave palladium polyhedra exhibit an enhanced electrocatalytic activity towards ethanol oxidation.

Z Zhang et al., ChemSusChem (2013)
Etching Process

• **Wet etching**
  – *Isotropic etching*
  – *Anisotropic etching*
  – *Crystal orientation*

• **Dry etching**
  – *Plasma etching*
  – *Reactive Ion Etching (RIE)*
  – *Ion Beam Etching (IBE)*
Wet etching

• Etching process which utilizes liquid chemicals to remove materials from the wafer or thin films.

• **Selectivity (S):** ratio between different etch rates of etchant for different materials (i.e. different oxides and metals)

• Advantages:
  – High throughput
  – Low cost
  – Excellent selectivity in most case

• Disadvantages:
  – Lack of anisotropy
  – Limited resolution
  – Poor process control
Isotropic Wet Etching of Si

- Isotropic etching: *etch all direction with same rate*

Peterson (1982)

Without Agitation

With Agitation

Hemispherical shape
Isotropic Wet Etching of Si

Examples:

- “HNA” @ 22°C: HF / HNO₃ / Acetic Acid (CH₃COOH)
  - Rate [10 ml /30 ml /8 ml]= 0.7 to 3 µm/min
    *Masking layer SiO₂ (30 nm/min)*

- Rate [25 ml /50 ml /25 ml]= 4 µm/min
  *Masking layer Si₃N₄ (30 nm/min)*

- Rate [ 9 ml /75 ml /30 ml]= 7 µm/min
  *Masking layer SiO₂ (70 nm/min)*
Anisotropic Wet Etching of Si

- Isotropic etching solution etch all direction with same rate

θ = 54.74°

Peterson (1982)
Figure 1. The drawings show the different v-grooves and shapes that are possible to etch on common silicon wafers, together with how the masks must be oriented.
Anisotropic Wet Etching of Si

• Hydroxides of alkali metals:
  – KOH, CeOH, RbOH, NaOH for etching with selective orientation
• KOH(44g), Water, Isopropanol (100 ml) @85 °C
  – Rate = 1.4 μm/min
  – (100)/(111) etch ratio=400:1
    – Masking layer SiO₂ (1.4 nm/min)
    – Masking layer Si₃N₄ (negligible)
• KOH(50g), Water, Isopropanol (100 ml) @50 °C
  – Rate = 1.0 μm/min
  – (100)/(111) etch ratio=400:1
Etching Rates vs. Orientation

Lateral under-etch rates as function of orientation

Seidal, et. al. J. Electrochem (1990)
Etch-Stop Layer

• Doped Silicon layer to control shape:

- Patterning SiO$_2$
- Diffusion Mask
- Boron Diffusion
- Selective Etching of Doped area
Selective Etching

EDP etching solution stop at the buried p++ Si layer:

Etylene diamine (750 ml)
pyrochatechol(120g)
Water(100ml)
Etch rate 1.25 mm/min
@115°C

Peterson (1982)
Wet Etching Thin Films

• Silicon dioxide:
  – HF (but it does not etch Si or GaAs)
  – Buffered HF (BHF)- 10:1 HF/NH3F “BOE”

• Silicon Nitride:
  – Phosphoric acid
    Selectivity $[\text{Si}_3\text{N}_4/\text{SiO}_2] = 40/1$

• Polysilicon:
  – KOH, EDP,
    TMAH(Tetramethyl ammonium hydroxide)
Wet Etching of Quartz

- Anisotropical etching by HF
  - with 10.9mol/l, Rate ~ 9.6 \( \mu \text{m/hr} \)

- Ammonium fluoride (NH\(_4\)F)

- Saturated ammonium bifluoride (NH\(_4\)HF\(_2\))

- *Need metal mask or oxide mask*

- Amorphous Si or Poly Si can be used as mask for deep etching
Dry Etching

- Plasma etching
  - easier to controls
- RIE
  - high selectivity
  - anisotropic etching
- Ion beam etching
  - low selectivity
  - no chemical reaction
Reactive Ion Etching (RIE)

Chlorine based plasma are commonly used for anisotropic etching of Si, GaAs, and aluminum based metalization.

External energy (RF) drives chemical reaction
Ion Beam Etching (IBE) & Deposition

Advantages:

- Easy to control etching profile
- Physical & chemical etching

Three different types:

1. IBE
2. RIBE (Reactive IBE)
3. CAIBE (Chemically Assisted IBE)
Focused Ion Beam (FIB) Etching

**Beam spot diameter**: ~100 nm.  
**Applications**: implantation, sputtering, deposition, micro-machining, ion beam lithography, *nanostructure machining*  
**Range of Ions**: Ga, Si, Au, Co, Y, Pr
Dry Etching of Thin Films

• Silicon dioxide:
  – CF$_4$ (Freon$^{\text{TM}}$ 14) + O$_2$
  – CHF$_3$ (Freon$^{\text{TM}}$ 23)
  – C$_2$F$_6$ (Freon$^{\text{TM}}$ 116)
  – C$_2$F$_8$ (Freon$^{\text{TM}}$ 118)

• Silicon nitride:
  – CF$_4$ (Freon$^{\text{TM}}$ 14) + O$_2$
  – CHF$_3$ (Freon$^{\text{TM}}$ 23)
  – C$_2$F$_6$ (Freon$^{\text{TM}}$ 116)
  – SF$_6$ +He

• Photoresist:
  – O$_2$

• Polysilicon:
  – CClF$_3$ (Freon$^{\text{TM}}$ 13) + Cl$_2$
  – CF$_4$ (Freon$^{\text{TM}}$ 14) + H$_2$
  – CClF$_5$ (Freon$^{\text{TM}}$ 15)
High-Aspect-Ratio Etching

• Deep and anisotropic structures in silicon is very important in MEMS applications:
  – Comb-drive based actuators
  – Microfluidics
  – Microsurgical devices
  – Optical MEMS

• Deep Reactive Ion Etching (DRIE)
DRIE for MEMS

30 µm

Courtesy of John Evans
Deep RIE

- Based on high-density plasma source by inductive coupling method
- By alternating process of etching and protective polymer layer deposition
- High aspect ratio of 30:1
- Vertical etched wall control (~ 90°)
- Etching rate 2-3 mm/min

The Mechanism of Deep RIE

• The key concept:
  Alternating between etching and protective polymer deposition

1. Etching step: SF$_6$/Ar with $V_{sb} = -5$ to $-30$ (V)
   - Substrate biasing ($V_{sb}$) is for the vertical acceleration of cations generated in the plasma into vertical motion

2. Polymerization step:
   Trifluoromethane (CHF$_3$)/Ar or C$_4$F$_8$/SF$_6$ → polymerization

Teflon like polymer (polymerized CF$_2$)
SEM of High-Aspect Ratio Structure
Combinational Anisotropic/Isotropic Etching

Example 1

1. PECVD mask oxide

2. Cl₂/BCl₃ RIE etch

3. PECVD oxide

4. CF₄ (oxide) & Cl₂/BCl₃ (Si)

5. Anisotropic SF₆ etch

6. Metal deposition: Al

Shaw, et al. (1993)
Anisotropic/Isotropic Etching

Example 2

Prefabricated CMOS: 3-level Al

1

Anisotropic SF$_6$ / O$_2$ RIE etch

3

Anisotropic CHF$_6$ / O$_2$ RIE etch

2

Isotropic SF$_6$ / O$_2$ RIE etch

4

Fedder, et al. (1996)
Lift-off Process

- Simple pattern transfer
- Require re-entrant profile
- Poor step coverage
- Low yield
- Best for prototype
Howe (1988): CVD phosphosilicate glass (PSG) as sacrificial layer since it etch faster in HF than thermal or undoped oxides
Polysilicon Applications

- MOSFET’s gate material
- High-value resistors
- Conductors
- Ohmic contacts for crystalline silicon
- Piezoresistors
- Selective etching layer
- MEMS structural material
Polysilicon Thin Film

- LPCVD by pyrolyzing silane in low pressure reactor @ 600-650 °C
  \[ \text{SiH}_4 \rightarrow \text{Si} + 2\text{H}_2 \]
- PECVD with low temperature
- Structures of poly Si are influenced by dopant and thermal processing condition.
- Doping: phosphine, diborane, or arsine by diffusion or implantation
- Sputtering with annealing for low stress fine grain films for MEMS
Si etching

Lap poly 1

Sac layer

Poly dep & etching

Poly dep

HF etching
Sealed Cavity for Microfluidics

- Oxide or Nitride Sealing
- Thermal Oxide Sealing

PSG
Sacrificial Layer (PSG) for Hinge

K. Pister et al. (1992)
Selective Etching for Optical MEMS
Summary of Etching

- Wet Etching
  - Crystal orientation
  - Selectivity
- Dry etching
  - RIE & DRIE
  - Ion beam etching
- Sacrificial Layer
- Selective Etching