

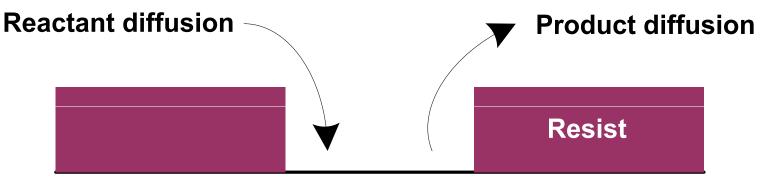
Non Silicon Wet Etching

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Basic Wet Etch Process

- Diffusion of reactants to the surface
- Reaction on the surface • (absorption, reaction, desorption)
- Diffusion of products from the surface •



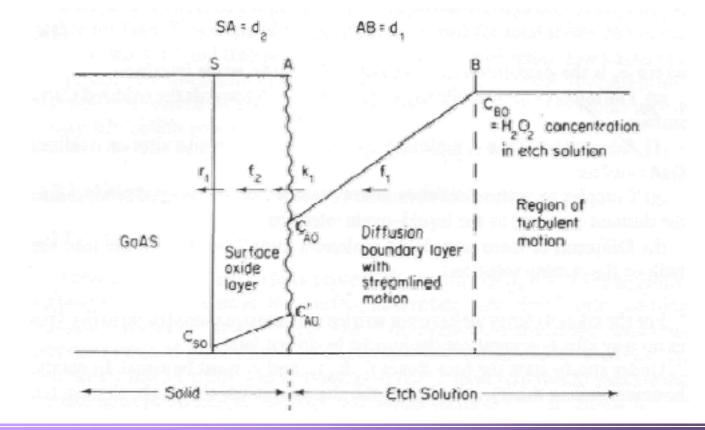
Surface reaction

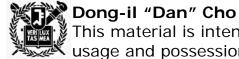
Oxide etching sequence in BOE



A Schematic View of the Etch Process

Concentration gradient of the oxidizing component, H_2O_2 in the etch solution close to the surface and inside the thin surface oxide, during a wet chemical etching process

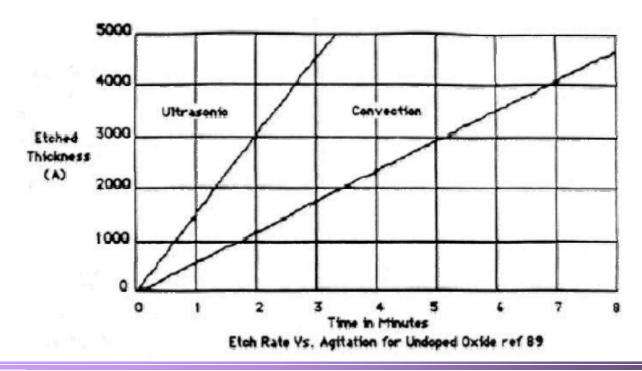


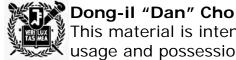


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Agitation in Etching Process

- Diffusion limited process controlled by reactant/product
- Increased agitation adds reactant, removes products
- Also remove hydrogen bubbles
- Ultrasonic sound generates controllable agitation •

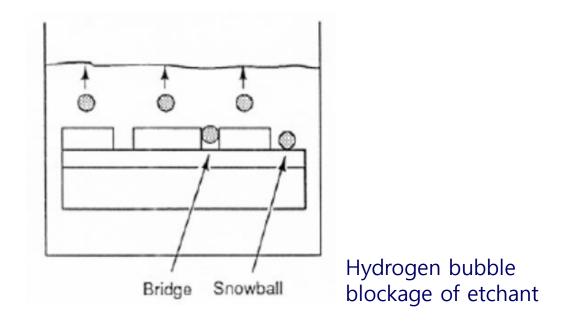


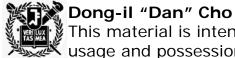


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Agitation and Bubbles

- Agitation necessary to remove hydrogen bubbles
- Bubbles become trapped in between lines/holes \rightarrow blocks them \rightarrow creates a bridge
- Bubbles on surfaces leave layer behind sometimes called • snowball

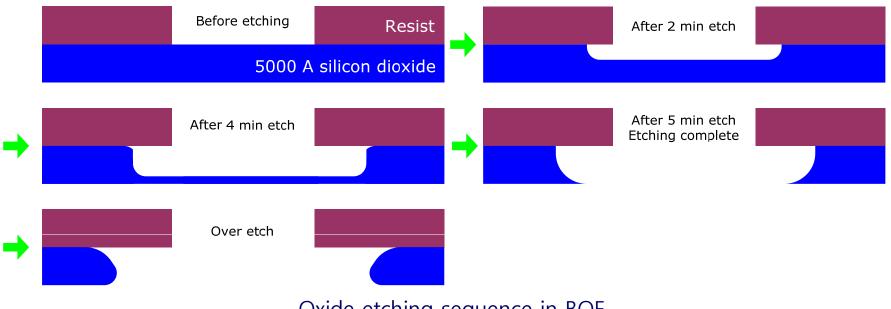




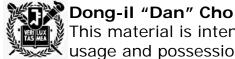
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Typical Wet Etch profile

- The etch process goes on both vertical and horizontal directions
- Undercut effect at the edge of the resist
- Etch process is slower than before at the etch stop
- Over etch process creates significant undercut



Oxide etching sequence in BOE



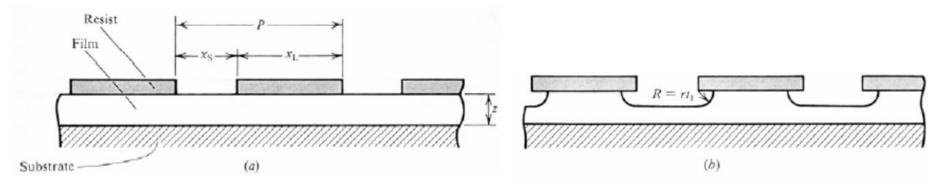
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Isotropic Wet Etching and Feature Size (1)

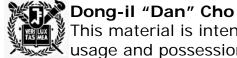
- Isotropic etch proceeds at same rate "r" in all directions
- Removes more at top edge than bottom
- Thus create circular profile of etch with radius

where t=time of etch (sec), r=etch rate (um/sec)

- Bottom of hole is flat, but edges curved
- Incomplete etch: film layer not fully removed



Isotropic etching in a wet bath. (a) unetched, mask film, showing parameters to be used, (b) Partially etched film



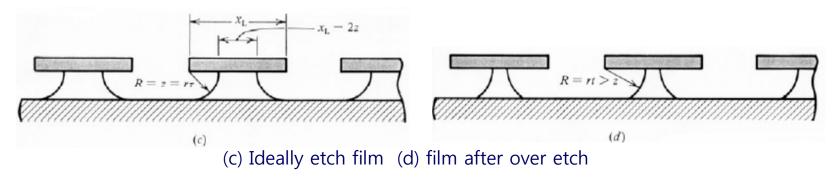
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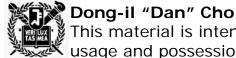
Isotropic Wet Etching and Feature Size (2)

- Perfect etch then just clear bottom
- Time of perfect etch is z/r (z=film thickness)
- This generates minimum undercut
- Side of lines measured at top/bottom
- For perfect etch get line size at top

 $x = x_1 - 2rt$

- But unevenness of etch over wafer means must have some over etch at points
- Over etch generate undercut at top

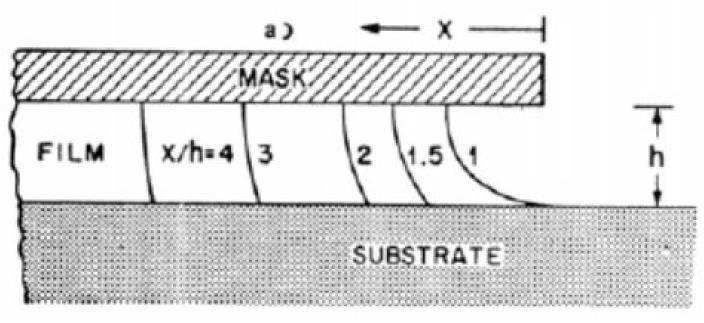




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Overetch Profile

- Initial undercut nearly circular •
- As proceeds undercut get more vertical

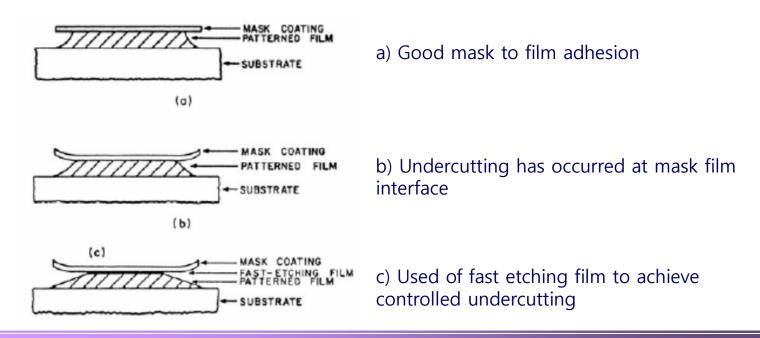


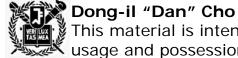
Wet etch profile



Sloped Sidewalls

- Sometimes wants sloped sidewalls: sloped edges
- Easier for layers stepping over edge
- As wet etch undercut resist begins to lift off
- May deposit thin fast etch layer under mask
- Generates shallow slope in undercut

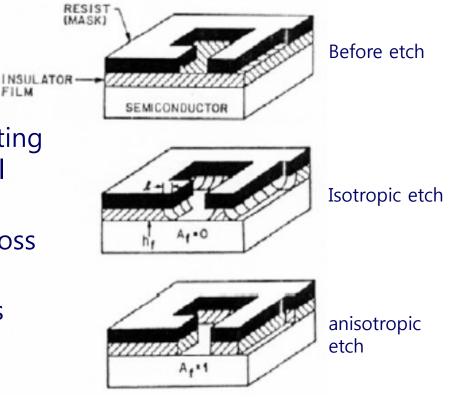




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Etching and Undercutting

- Perfect etching would be anisotropic
- Would generate vertical sidewalls
- Isotropic etch gets undercutting of resists removal of material under resist edge
- Because etch rate differs across wafer
 - undercutting different across wafer





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Wet Etch Chemicals

Concentration of various acids for wet etching ۲

Acid	Concentration	Property
Acetic acid	Pure	_
Sulfuric acid	96%	_
Phosphoric acid	85%	Deliquescent* solid at room temperature
hydrofluoric acid	49%	boiling point : 19.5 ° C
Nitric acid	70%	liquid in the range near room temperature

* Deliquescent : tending to melt or dissolve; *especially* : tending to undergo gradual dissolution and liquefaction by the attraction and absorption of moisture from the air



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Case Study – HF Glass Wet Etching

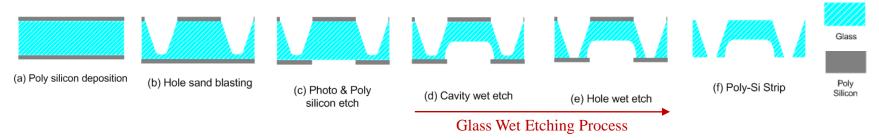
- Wafer-level Package using Silicon/Glass Anodic Bonding
 - Protect sensing element to enhance reliability
 - Reduce cost and production time
 - Optical transparency
 - Vacuum & hermetic sealing is possible

- Glass cap Glass scap Glass cap Glass cap Control of the scale of the s
- Chemical Composition of Pyrex 7740 Glass

Chemical component	SiO ₂	B ₂ O ₃	Na ₂ O ₃	K ₂ O	Al ₂ O ₃
Percentage [%]	80	13	3.5	1	2.5

Fig. Schematic diagram of Wafer-level Packaging

• Process Flow of Glass Wafer Fabrication





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Silicon Dioxide Wet Etchants (1)

- SiO_2 is an amorphous material which etches equally well in all directions (isotropic etching)
- HF-based etchants are used mainly for etching silicon dioxide, although they can also be used to remove silicon nitride
- HF-based solutions should be handled with Teflon containers \rightarrow not glass containers, which will be attacked
- Reaction
 - Pure HF etching

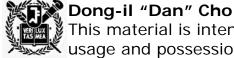
$$SiO_2 + 6HF \rightarrow H_2SiF_{6(aq)} + 2H_2O$$

The reaction in BHF solution

 $SiO_2 + 4HF + 2NH_4F \rightarrow (NH_4)_2SiF_6 + 2H_2O$

- The reaction involving the HF_2 ion

$$SiO_2 + 3HF_2 + H + \rightarrow SiF_6^2 + 2H_2O$$



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Silicon Dioxide Wet Etchants (2)

- Concentrated HF (49%)
 - Concentrated hydrofluoric acid (49% by weight, remainder water)
 - Etches oxides very rapidly \rightarrow often used to remove sacrificial oxide when micromachining
 - Concentrated HF tends to peel off photoresist, while lower concentrations (less then 3:1) do not
- 10:1 HF
 - 10:1 HF:H₂0: concentrated HF (49% HF)
 - Typically used for stripping oxide and for HF dips, diluted HF is cheaper than buffered HF
- 25:1 HF
 - 25:1 HF:H₂0: concentrated HF (49% HF)
 - This slow etch is used for HF dips to strip native oxide without removing much of the other oxides that may be on a wafer



Silicon Dioxide Wet Etchants (3)

- 5:1 BHF
 - 5:1 buffered hydrofluoric acid (also know as buffered oxide etch, or BOE)
 - "5:1" refers to five parts by weight of 40-weight-percent ammonium fluoride (the buffer) to one part by weight 49 – weight-percent hydrofluoric acid
 - \rightarrow which results in a total of about 33% NH₄F and 8.3% HF by weight.
 - This etchant can be masked with photoresist
 - It is the often best choice for controlled etching of oxide
 - Note increase in the etch rate with temperature



Silicon Dioxide Wet Etchants (4)

- Concentration of HF & Etch Rate
 - Effect of HF concentration on the etch rate of thermally grown SiO_2
 - Significant species present in dilute HF solutions

25 °C

 1.30×10^{-3}

0.104

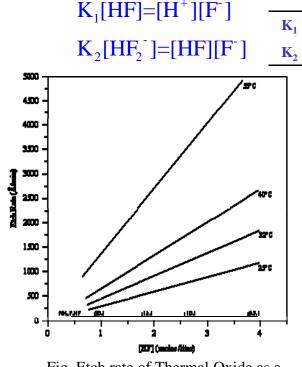
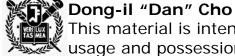


Fig. Etch rate of Thermal Oxide as a Function of HF Concentration



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Table. Concentrations and Associated Etch Rates

60 ℃

6.57 × 10⁻⁴

 3.66×10^{-2}

	SiO, Etch Ret (Ådmist)					
[HF]	M × 10 ³ ; [HF ₂]	ወጋ	(H ⁺)			
13	\$7	677	0.0255	4434		
41	159	409	0.129	87.12		
81	189	242	0.435	112.26		
109	191	182	0.778	124.08		
175	173	102	2.24	133.92		
266	128	49.4	7.01	118.26		

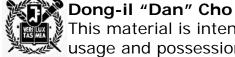
Silicon Nitride Wet Etchant (1)

Silicon nitride films can be etched by hydrofluoric acid (HF), buffered HF, and phosphoric acid (H_3PO_4) solutions

)	
Preservation method	hf	hf : Nh <i>f</i>	H_PO4
CVD, SH4+NH2	90	5	60
CVD, SICL + NH.	-	11	100
CVD, SH ₄ + N ₂ H ₄	300		-
CVD, SHICH + NH	600	150	75
RFOD, SLH, + NH,	-	50	-
RFOD, SiH4 + N2	-	100	-
LPCVD, SHL + NH2	150	-	-
LPCVD, SiH ₅ Ci ₂ + NH ₂	200	-	-
Direct of, sputtering	750	-	-
CVD, SizOyNg	>360	>600	>600
··· • -			

- Phosphoric acid (85%)
 - Phosphoric acid (85% by weight, remainder water) at 160 °C
 - Silicon nitride is etched at a significantly higher rate than silicon dioxide when hot phosphoric acid (H_3PO_4) is used as an etchant

Type of film	a ,n,	EO,	
Method of film preparation	Sill_+NH, \$00*C	51Cl_+O_+H_ \$\$0*C	AcidConsentration H ₃ PO ₆
Refuzed boiling at 180°C	105	10	91.5%



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Silicon Nitride Wet Etchant (2)

Phosphoric acid (85%)

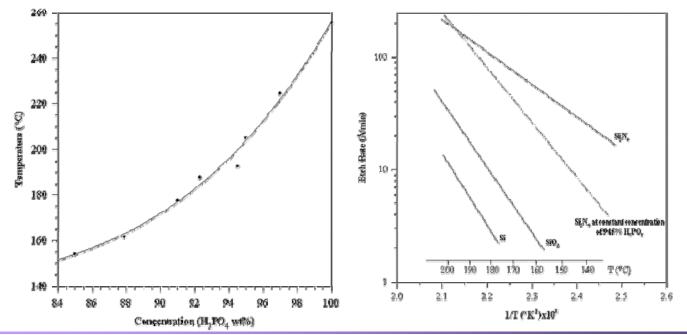
 $3Si_2N_4 + 27H_2O + 4H_3PO_4 \rightleftharpoons 4(NH_4)_3PO_4 + 9H_2SiO_3$

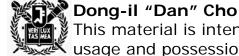
- Water is hydrolyzing the silicon nitride to form hydrous silica and ammonia
- Ammonia remains in the solution in the form of ammonium phosphate
- This stoichiometry suggests that water is an integral part of the chemistry involved in the etching of silicon nitride
- Mask material: Densified PSG (If the PSG mask is not densified, it is removed faster and may also have pores through which the acid can seep), poly-Si



Silicon Nitride Wet Etchant (3)

- Phosphoric acid (85%)
 - Temperature: 150 ~ 180 °C
 - At these temperatures the concentration of H_3PO_4 ranges from 85 ~ 92 wt% (water vaporization)
 - Use a reflux system or by replacing the water at the same rate at which it is evolved (Used in common semiconductor industry)

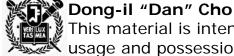




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Metal Wet Etchants (1)

- Aluminum Etchant Type A
- $80\% H_3PO_4 + 5\% HNO_3 + 5\% CH_3COOH + 10\% H_2O$
- This etchant is designed to etch AI at 6000 A/min at 50 °C
- It can be masked with PR
- Reaction •
 - Al is first oxidized by the nitric acid
 - The phosphoric acid and water simultaneously etch the resulting oxide
 - With the concentrations gives, these two processes occur at roughly the same rate
 - The phosphoric acid also removes the native AI oxide
 - \rightarrow no additional component is needed



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Metal Wet Etchants (2)

- Titanium etchant •
 - Mixed from 20:1:1 \rightarrow H₂O:HF (49%): H₂O₂ (30%)
 - HF is the active ingredient is this etchant, so it also etches oxides \rightarrow raising the fraction of HF in the solution increases the etch rate
 - It can be masked with PR



Metal Wet Etchants (3)

- CR-7 (Chromium etchant)
 - $-9\% (NH_4)_2 Ce(NO_3)_6 + 6\% HCIO_4 + H_2O$
 - This etchant is formulated to selectively etch chromium, and was also found to etch copper and silver at useful rates
 - It can be masked with PR
 - The chemical reaction $3Ce(IV)(NH_4)_2(NO_3)_{6(aq)} + Cr(s)$ \rightarrow Cr(III)(NO₃)_{3(aq)} + 3Ce(III)(NH₄)₂(NO₃)_{5(aq)}



Metal Wet Etchants (4)

- CR-14 (Chromium etchant)
 - $22\% (NH_4)_2 Ce(NO_3)_6 + 8\% CH_3 COOH + H_2O$
 - This etchant is formulated to selectively etch chromium
 - It was also found to slowly etch vanadium and copper
 - It etches chromium a little slower than CR-7, but have more a consistent etch rate, as well as much less undercut of PR
 - The etch reaction is the same as for CR-7



Metal Wet Etchants (5)

- Moly etch ٠
 - 180:11:11:150 \rightarrow H₃PO₄:CH₃COOH:HNO₃ (70%):H₂O
 - It can be masked with PR, which is etched at a moderate rate
- H₂O₂ at 50 °C (Hydrogen peroxide 30% by weight)
 - Etch target is tungsten, but etches it slowly
 - Heating increases the etch rate
 - Ultrasonic agitation aids in etch uniformity by helping to remove bubbles



Metal Wet Etchants (6)

- Cu FeCl₃ 200
 - Copper etchant: 30% FeCl₃ + $3 \sim 4\%$ HCl + H₂O
 - CE-200 can be patterned with PR
 - Reaction
 - FeCl₃ dissociates in water to generate Fe³⁺ and Cl⁻ ions $Cu_{(s)} + 2Fe_3 + + 3Cl^- \rightarrow CuCl_3 + 2Fe^{2+}$
 - The Fe³⁺ ions are reduced to Fe²⁺, which remain in solution, while the copper metal is oxidized to Cu²⁺
 - HCl assists in the dissolution of the ferric chloride, and also etches copper itself



Metal Wet Etchants (7)

- Cu APS 100
 - Copper etchant: $15 \sim 20\%$ (NH₄)₂S₂O₈ + H₂O at 30 °C
 - PR can be used as an etch mask
 - The overall reaction

 $Cu_{(s)} + (NH_4)_2S_2O_8 \rightarrow CuSO_{4(aq)} + (NH_4)_2SO_{4(aq)}$



Metal Wet Etchants (8)

- Dil. Aqua Regia ٠
 - 3:1:2 → HCI (37%):HNO₃ (70%):H₂O
 - This solution is self heating to about 30 °C for tens of minutes
 - The water was added to the standard aqua regia solution to reduce the attack of PR
 - Aqua regia targets noble metals

- The overall reaction (Au)

$$Au_{(s)} + 4H^+ + NO_3^- + 4Cl^-$$

 $\rightarrow AuCl_4^- + NO_{(g)} + 2H2O_{(l)}$



Metal Wet Etchants (9)

- AU-5
 - Gold etchant: 5% I_2 + 10% KI + 85% H_2O
 - Such iodine solutions are the color of dark coffee, making it impossible to observe a submerged sample
 - Rinsing with running water must be done to remove the solution from the surface \rightarrow isopropanol or methanol can be used for a faster rinse
 - PR can be used as an etch mask
 - The overall reaction

$$2Au_{(s)} + I_{2(aq)} \rightarrow 2AuI_{(aq)}$$



Metal Wet Etchants (1)

- NiCr TFN •
 - Nichrome etchant: $10 \sim 20\%$ (NH₄)₂Ce(NO₃)₆ + $5 \sim 6\%$ HNO₃ + H_2O
 - It is formulated to etch nickel-chromium alloy
 - It etched pure nickel slowly, but etched chromium, copper, and molybdenum faster than the NiCr alloy
 - PR can be used as an etch mask



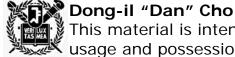
GaAs Wet Etch (1)

- Unique Advantages of GaAs over Si ٠
 - High resistivity
 - High electron mobility
 - High saturated drift velocity
 - Wide direct bandgap

Operability over a wide temperature range

Table. Electrical and mechanical properties of silicon and GaAs

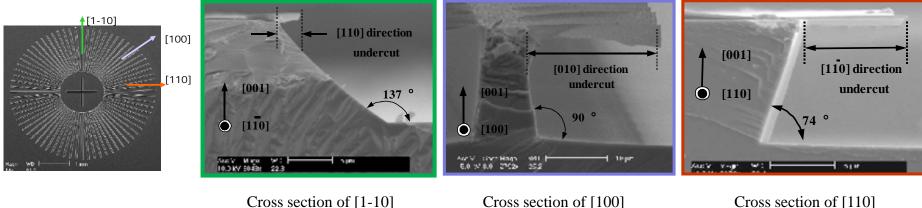
Properties	Si	GaAs
Atoms (cm ⁻³)	$5.0 imes10^{22}$	4.42×10^{22}
Atomic weight	28.09	144.63
Breakdown field (V/cm)	${\sim}3 imes 10^5$	${\sim}4 \times 10^5$
Crystal structure	Diamond	Zincblende
Density (g/cm ³)	2.33	5.32
Dielectric constant	11.9	13.1
Energy bandgap (eV)	1.12	1.424
Intrinsic resistivity (Q.cm)	$2.3 imes 10^5$	1×10^8
Electron mobility (drift) $(cm^2 \cdot V^{-1} \cdot s^{-1})$	1500	8500
Young's modulus (GPa)	£y	$E_{(100)} = 82.3$ $E_{(110)} = 121.3$ $E_{(111)} = 141.2$
Piezoelectric coefficient (pm/V)	0	$d_{14} = 2.69$



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GaAs Wet Etch (2)

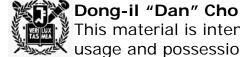
- **Etch conditions** •
 - Etchants: 30%wt NH₄OH + 30%wt H₂O₂ + H₂O
 - Wafer: (001) GaAs substrate
 - Mask material: PECVD oxide, PECVD low stress nitride ____
 - Temperature: 18 ℃
- (001) GaAs wet etch results ٠



directional beam

Cross section of [100] directional beam

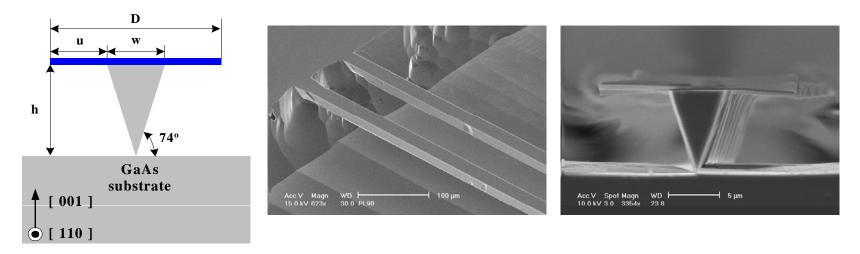
Cross section of [110] directional beam



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GaAs Wet Etch (3)

<110> beam release •



$$w = D - 2u = D - 2hS = D - \tan 74^{\circ} \cdot w$$

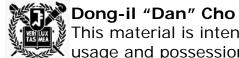
$$w = \frac{D}{\left(1 + \tan 74^{\circ} \cdot S\right)} = \frac{\tan 16^{\circ} \cdot D}{\left(\tan 16^{\circ} + S\right)}$$

$$h = \tan 74^{\circ} \cdot \frac{w}{2} = \frac{\tan 74^{\circ} \cdot \tan 16^{\circ} \cdot D}{2\left(\tan 16^{\circ} + S\right)} = \frac{D}{2(\tan 16^{\circ} + S)}$$

$$\left(S = \frac{u}{h}$$

$$h = \tan 74^{\circ} \cdot \frac{w}{2}$$

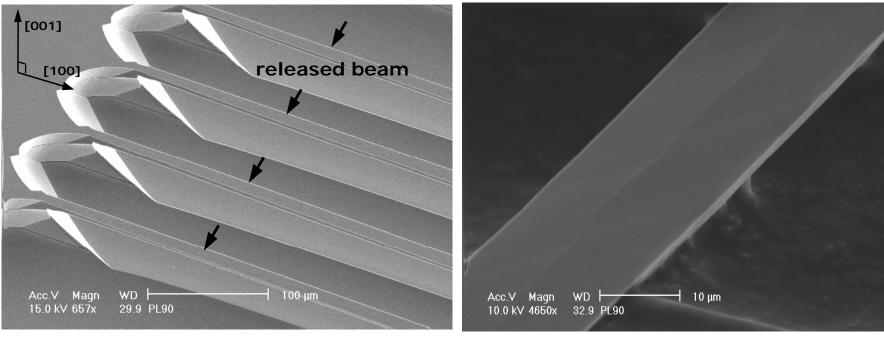
$$\tan 74^{\circ} = \frac{1}{\tan 16^{\circ}}$$



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GaAs Wet Etch (4)

<100> beam release •



(a) released [100] direction beams

(b) backside of the released beam



Other Wet Etchants (1)

- Piranha •
 - The piranha (also known as sulfuric-peroxide) is a mix of H_2SO_4 (96%):H₂O₂ (30%) → 4:1
 - It is heated 120 °C
 - It is used as a cleaning solution that strips organics and some metals



Other Wet Etchants (2)

- Acetone
 - Pure acetone, also known as dimethyl ketone.
 - It is used to strip photoresist and for cleaning off organics.
 - It removed photoresists rapidly, as expected, but did not etch polyimide.
- Methanol
 - Pure methanol
 - It is used for cleaning and in supercritical drying.
 - Methanol removed photoresist, but did not etch polyimide.
- Isopropanol
 - Pure isopropanol, also known as isopropyl alcohol (IPA) and 2propanol.
 - It is used for cleaning off organics.
 - In contrast to methanol, isopropanol was found to remove photoresist at a moderate rate.



Dong-il "Dan" Cho This material is intended Nano/Micro Systems & Controls Lab.

Etch Rate Table (1)

	What Each Dense for Minner a history and IC Dense in $(\hat{\lambda}/2)$																
Wet Etch Rates for Micromachining and IC Processing (A/min)																	
he top etch rate was measured by the authors with fresh solutions, etc. The center and bottom values are the low and high etch rates observed by the authors and others in our lab under less carefully controlled conditions.																	
	MATERIAL																
		SC Si										locc	Olin				
		<100		Poly not	Wet Ox	· ·	LTO not	PSG not	PSG annea	Stoic	Low Stress			Sput Ti	Sput Ti/W	OCG 820	Hunt
ETCHANT EQUIPMENT	TARGET	>		dope	0.			annea		e		SI	rung	"		PR	PR
CONDITIONS	MATERIAL			d			d	led			e						
Concentrated HF (49%)	Silicon oxides	-	0	-	23k	F	>14k	F	36k	140		42	<50	F	-	P	F
Wet Sink Room Temperature					18k 23k						30 52	0 42				0	
10:1 HF Wet Sink Room Temp	Silicon oxides	-	7	0	230	230	340	15k	4700	11	3	2500 2500 12k		11k	<70	0	(
25:1 HF Wet Sink Room Temp	Silicon oxides	-	0	0	97	95	150	W	1500	6	1	W	0	-	-	0	(
5:1 BHF Wet Sink Room Temp	Silicon oxides	-	9	2	1000 900 1080	1000	1200	6800	4400 3500 4400		4 3 4	1400	<20 0.25 20		1000	0	(
Phosphoric Acid (85%) Heated Bath with Reflux 160°C	Silicon nitrides	-	7	-	0.7	0.8	<1	37	24 9 24	28	19		-	-	-	550	390
Silicon Etchant (126 HNO₃:60 H₂:5 NH₄F) Wet Sink Room Temp	Silicon	1500	3100 1200 6000		87	W	110	4000	1700	2	3	4000	130	3000	-	0	(

Notation: - = test not performed; W = Not performed, but known to Work (100 A/min); F = not performed, but known to be Fast (10 kA/min); P = some of film Peeled during etch or when rinsed; A = film was visibly Attached and roughened. Etch areas are all of a 4-inch wafer for the transparent films and half of the wafer for single-crystal silicon and the metals. Etch rates will vary with temperature and prior use of solution, area of exposure of film, other materials present (e.g., photoresist), film impurities and microstructure, etc. Some variation should be expected.

Ref.) K. R. Williams, and R. S. Muller, JMEMS, Vol. 5, No. 4, pp. 256-269, 1996



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Etch Rate Table (2)

											_						
		SC Si	Poly	Poly	Wet	Dry	LTO		PSG		Low	Alum	Sput	0.000		OCG	Olin
		<100	n+	not	Ox	Ox	not	not	annea	Nitrid	Stress	2%	Tung	Ti	Ti/W	820	Hunt
ETCHANT EQUIPMENT	TARGET	>		dope			dope	annea	led	e	Nitrid	SI				PR	PR
CONDITIONS	MATERIAL			d			d	led			e						
KOH (1 KOH: 2 H₂O by weight) Heated Stirred Bath 80℃	<100>Silicon	14k	>10k	F	77 41 77	-	94	W	380	0	0	F	0	-	-	F	F
Aluminum Etchant Type A (16 H₃PO₄:1 HNO₃:1 HAc:2H₂O) Heated Bath 50°C		-	<10	<9	0	0	0	-	<10	0	2	6600 2600 6600	-	0	-	0	0
Titanium Etchant (20 H ₂ O:1H ₂ O ₂ O:1 HF) Wet Sink Room Temp	Titanium	-	12	_	120	W	W	W	2100	8	4	W	0 0 <10		-	0	0
H ₂ O ₂ (30%) Wet Sink Room Temp	Tungsten	-	0	0	0	0	0	0	0	0	0	<20	190 190 1000		60 60 150	<2	0
Piranha (-50 H₂SO₄:1 H₂O₂) Heated Bath 120℃	Cleaning off metals and organics	-	0	0	0	0	0	-	0	0	0	1800	-	2400	-	F	F
Acetone Wet Sink Room Temp	Photoresist	-	0	0	0	0	0	-	0	0	0	0	-	0	-	>44k	>39k

Notation: - = test not performed; W = Not performed, but known to Work (100 A/min); F = not performed, but known to be Fast (10 kA/min); P = some of film Peeled during etch or when rinsed; A = film was visibly Attached and roughened. Etch areas are all of a 4-inch wafer for the transparent films and half of the wafer for single-crystal silicon and the metals. Etch rates will vary with temperature and prior use of solution, area of exposure of film, other materials present (e.g., photoresist), film impurities and microstructure, etc. Some variation should be expected.

Ref.) K. R. Williams, and R. S. Muller, JMEMS, Vol. 5, No. 4, pp. 256-269, 1996



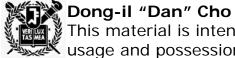
Dong-il "Dan" Cho

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Etch Rate Table (3)

substrate	etch solution	temperature(℃)	end point
SiO ₂	7 NH ₄ F : 1 HF	22	Sheet-off
Pyrolyc oxide	7 NH ₄ : 1 HF	22	Sheet-off
PSG • Insulator • Passivation	7 NH ₄ F : 1 HF 6H ₂ O : 5HC ₂ H ₃ O ₂ : 1NH ₄ F	22 22	Sheet-off none
Si ₃ N ₄	H ₃ PO ₄	155	none
Poly-silicon •Doped •Undoped	200HNO ₃ : $80HC_2H_3O_2$: 1 HF 20HNO ₃ : $20HC_2H_2O_2$: 1 HF	22 22	Color change
AI	$80H_3PO_4 : 5HNO_3 : 5HC_2H_3O : 10 H_2O$	40 - 50	Color change

Ref: J. D. Lee, "Silicon Integrated Circuit microfabrication technology," 2nd edition



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