

Non-PFC Plasma Chemistries for Patterning Complex Materials/Structures

(Task Number: 425.038)

PIs:

- **Jane P. Chang, Chemical and Biomolecular Engineering, UCLA**

Graduate Students:

- **Jack Chen, PhD student, Chemical and Biomolecular Engineering, UCLA**
- **Nathan Marchack, PhD candidate, Chemical and Biomolecular Engineering, UCLA**

Undergraduate Students:

- **Michael Paine, UG student, Chemical and Biomolecular Engineering, UCLA**

Other Researchers:

- **(TBD)**

Objectives

- **Assess the thermodynamic feasibility of patterning etch-resistant materials (complex materials and structures)**
- **Identify the non-PFC alternative for through silicon via**
- **Establish collaboration with industrial members to validate the theoretical assessment (etching experiments)**
- **Identify priority test cases (such as TSV)**

ESH Metrics and Impact

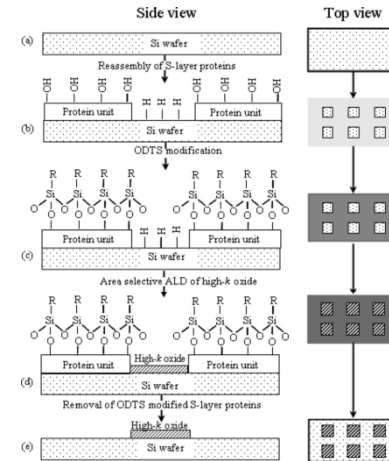
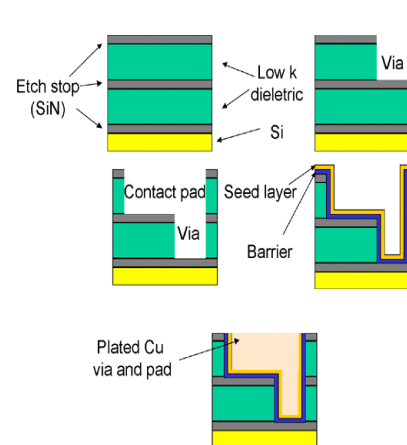
- 1. Reduction in the use of PFC gases by focusing on non-PFC chemistries**
- 2. Reduction in emission of PFC gases to environment**
- 3. Reduction in the use of chemicals by tailoring the chemistries to the specific materials to be removed**

Approach for Etch-Resistant Materials

- Use more aggressive etch chemistries such as PFC
 - Fluorine is the most electro-negative element
 - Carbon reaction products (CO and CO₂) are volatile

1970 → 1980 → 1990 → 2000 → 2010

IA																	0															
1																	2															
H																	He															
3	4															5	6	7	8	9	10											
Li	Be															B	C	N	O	F	Ne											
11	12	13	14	15	16	17	18								19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36
Na	Mg	Al	Si	P	S	Cl	Ar	K	Ca	Sc	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	Ga	Ge	As	Se	Br	Kr							
37	38	39	40	41	42	43	44	45	46	47	48	49	50	51	52	53	54															
Rb	Sr	Y	Zr	Nb	Mo	Tc	Ru	Rh	Pd	Ag	Cd	In	Sn	Sb	Te	I	Xe															
55	56	57*	72	73	74	75	76	77	78	79	80	81	82	83	84	85	86															
Cs	Ba	La	Hf	Ta	W	Re	Os	Ir	Pt	Au	Hg	Tl	Pb	Bi	Po	At	Rn															
87	88	89+	104	105	106	107	108	109	110	111	112	113																				
Fr	Ra	Ac	Rf	Ha	Sg	Ns	Hs	Mt	110	111	112	113																				
Lanthanide*		58	59	60	61	62	63	64	65	66	67	68	69	70	71																	
		Ce	Pr	Nd	Pm	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb	Lu																	
Actinide+		90	91	92	93	94	95	96	97	98	99	100	101	102	103																	
		Th	Pa	U	Np	Pu	Am	Cm	Bk	Cf	Es	Fm	Md	No	Lr																	

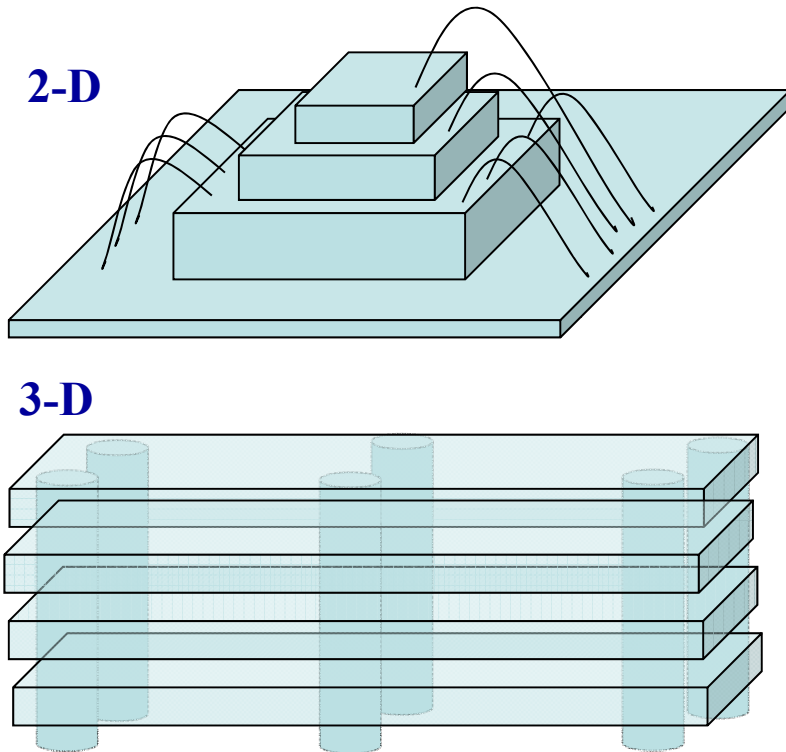


- Change processing approach to not etch these materials
 - Damascene process^[1] for Cu
 - Area selective atomic layer deposition

[1] P.C. Andricacos, C. Uzoh, J.O. Dukovic, J. Horkans, H. Deligianni, IBM, J. Res. Develop., Vol. 42, No.5(1998).

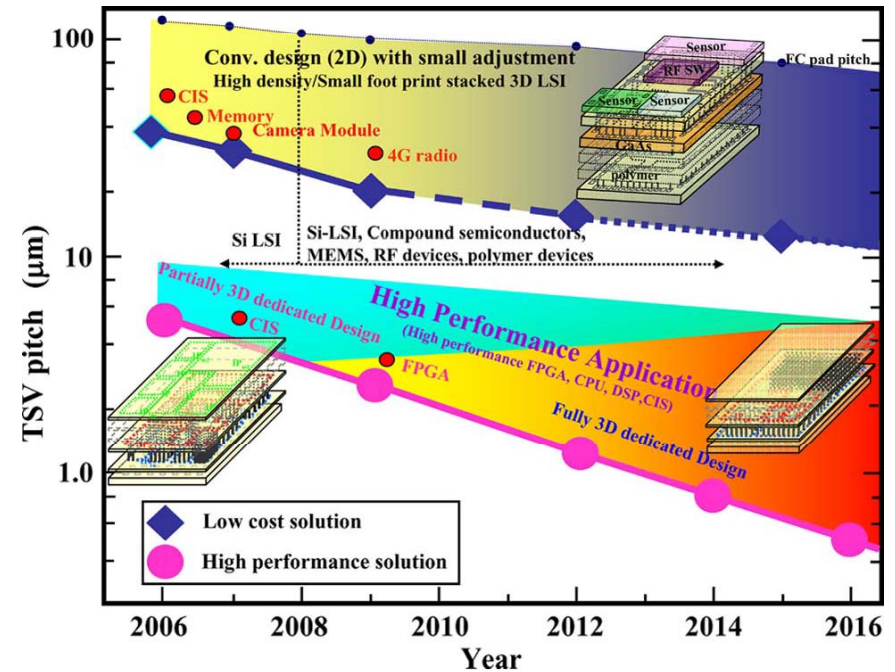
TSV (Through Silicon Via)

From 2-D to 3-D packaging



TSV for 3D-LSI technology road map

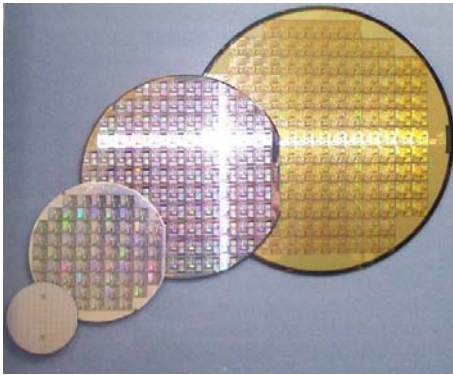
[M. Motoyoshi, IEEE, 2009]



- According to Moore's Law, the density of IC devices doubles every two years, the 3-D packaging system is required.

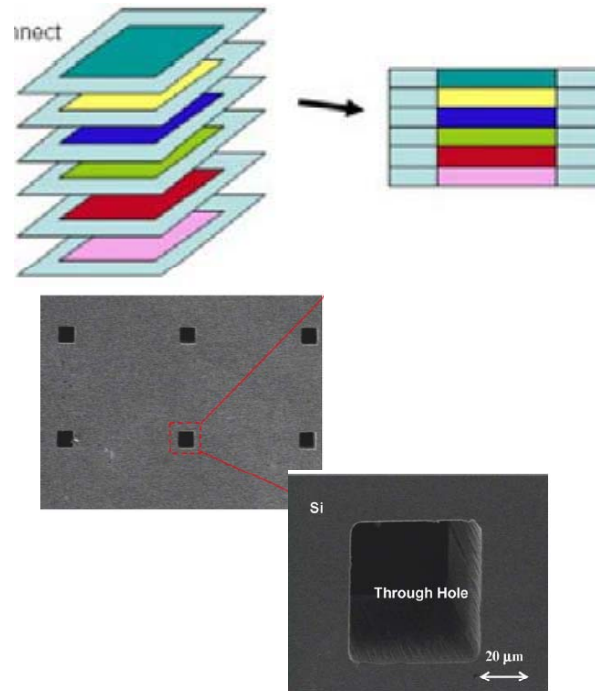
Challenges in TSV

High etch rate



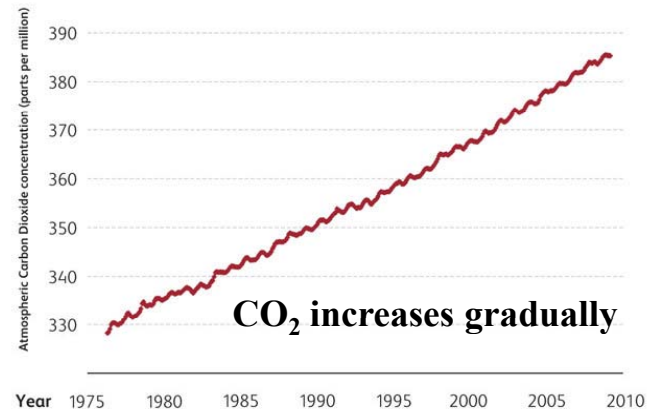
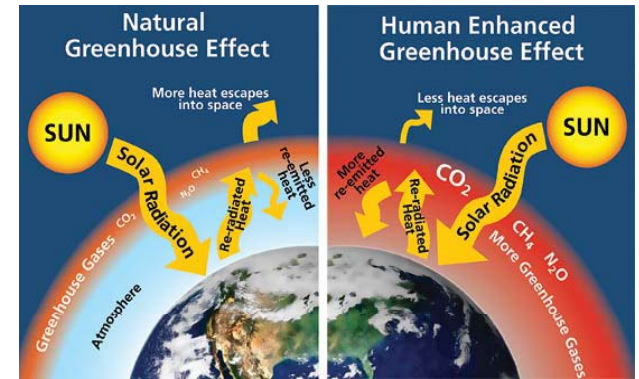
Silicon size (inch)	Thickness (um)
6	675
8	725
12	775
18	925

High aspect ratio



[M. Motoyoshi, IEEE, 2009]

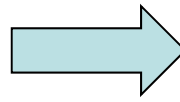
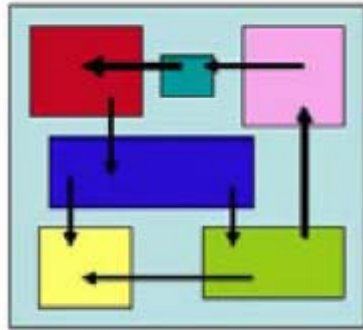
Greenhouse gas usage



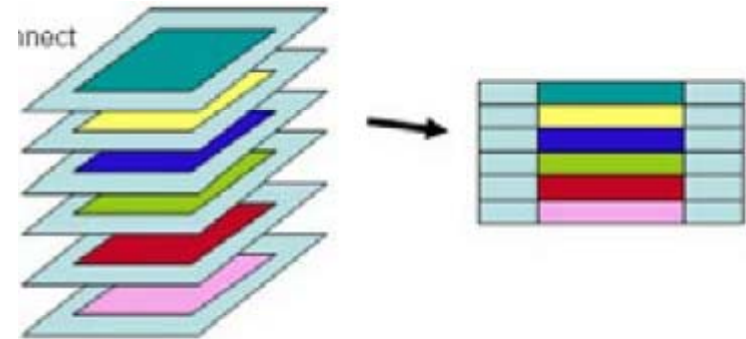
- Requirement to increase etch rate, increase aspect ratio and reduce the usage of global warming gases

3-D Stack with TSV

2D-Conventional



3D-TSV



[P. Garrou, Samsung website, 2010]

- **Advantages:**

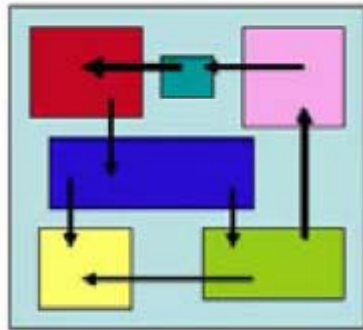
- Smaller footprint
- Smaller form-factor
- Less weight
- Less power consumption
- Potentially lower cost

- **Challenges:**

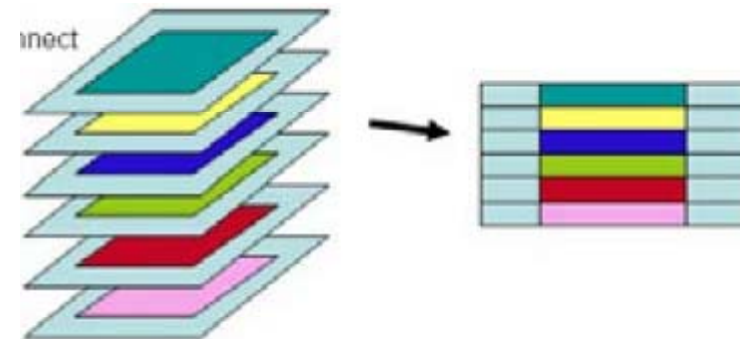
- High etch rate
- High aspect ratio
- Large Depth
- High yield
- GWP gases

3-D Stack with TSV

2D-Conventional



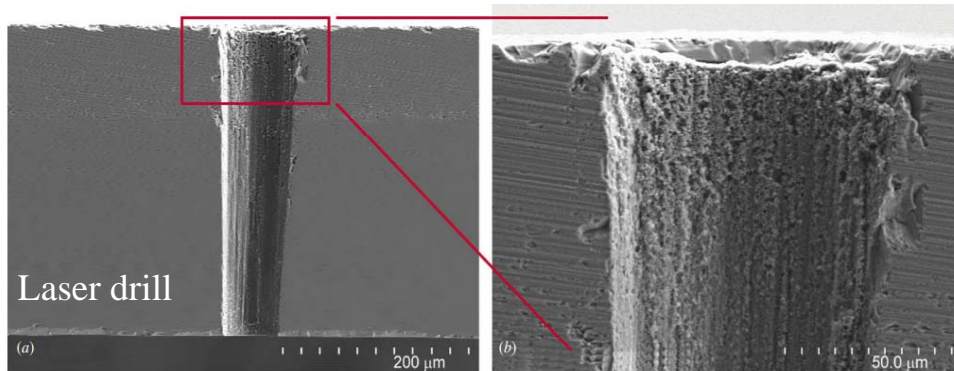
3D-TSV



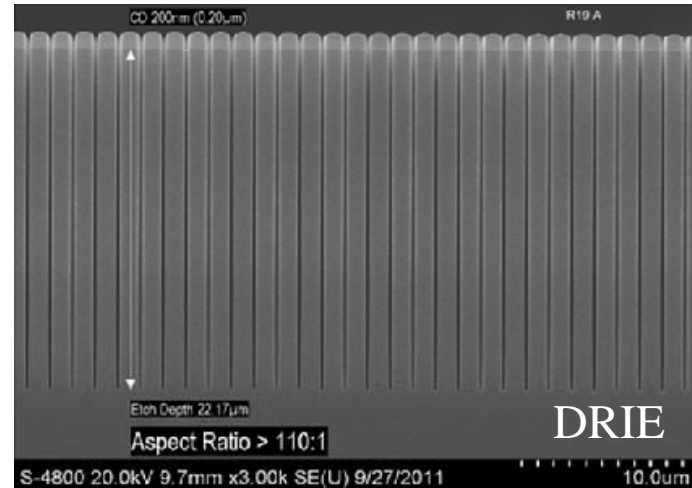
[P. Garrou, Samsung website, 2010]

	Features	Goal	Ref.
1	Etch rate	50 $\mu\text{m}/\text{min}$	I. Sakai, J. Vac. Sci. Technol. A, 2011
2	Aspect ratio (Anisotropy)	70-100	I. Sakai, IEEE, 2012
3	Side wall angle	$90^\circ \pm 10^\circ$	M. Hooda, J. Vac. Sci. Technol. A, 2010

TSV Process Options

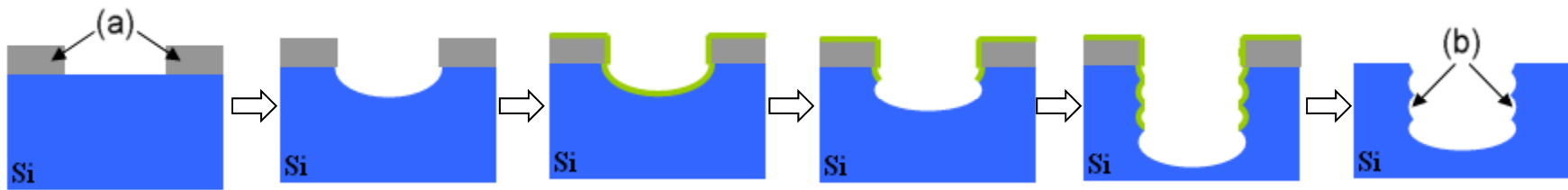


[C. Tang, J Micromech Microeng, 2012]



[Applied Materials Inc., website, 2012]

Bosch Process



[Gurgelgonzo, Public domain, 2007]

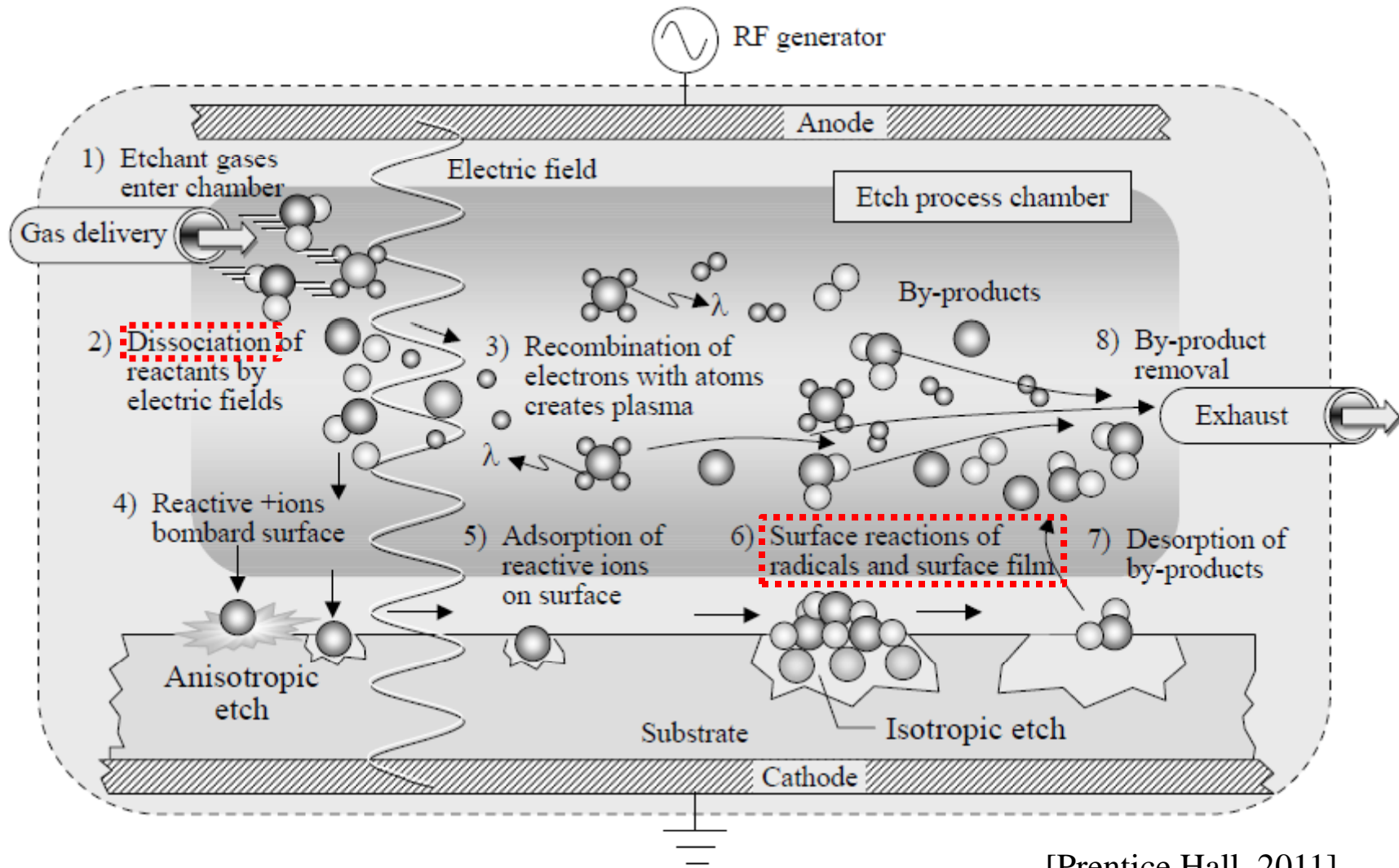
TSV Process Options

TSV Processes	Bosch DRIE*	Cryogenic DRIE#	Laser drill*
Width	<5 μ m	<5 μ m	<15 μ m
Aspect ratio	>20:1	>10:1	>7:1
Sidewalls	Vertical (90°)	Vertical & smooth	80°-90°
Temp. affection	Negligible	-110°C ~ -130°C	Yes
Process	ICP	ICP	Laser
Potential applications	High-end	High-end	Low-cost
Example	Microprocessors	Microprocessors	DRAM/Flash

[R. Landgraf, IEEE, 2008*; M. Walker, Proc. SPIE, 2001#]

- **As the critical dimension continues to decrease, DRIE becomes the most feasible technology for TSV**

Plasma Etch Process of a Silicon Wafer



[Prentice Hall, 2011]

Systematic Approach - Thermodynamics

- **Thermodynamic approach can be systematic**
 - **If such data is available**
 - NIST-JANAF Thermo-chemical tables
 - HSC Chemistry for windows, chemical reaction and equilibrium software with extensive thermo-chemical database
 - FACT, Facility for Analysis of Chemical Thermodynamics
 - Barin and Knacke tables (thermo-chemical data for pure substances and inorganic substances)
 - **Determination of dominant surface/gas-phase species**
 - **Assessment of possible reactions**
- **Graphical Representation of thermodynamic analysis**
 - **Richardson Ellingham diagram**
 - **Pourbaix diagram**
 - **Volatility diagram**

Important Chemistries for Silicon Etch

Chemistries	Radical	Volatile Product	Inhibitor
H ₂	H	HSiF ₄	Si _x F _y
CH ₄	CH ₃ CH ₂	SiH ₄ SiF ₄	Si _x C _y H _z
F ₂	F	SiF ₄	Si _x F _y
NF ₃	F, NF ₂	SiF ₄	Si _x N _y F _z
SiF ₄	F, SiF ₃	SiF ₄	Si _x F _y
CF ₄	F, CF ₃	SiF ₄	Si _x C _y F _z
SF ₆	F, SF ₅	SiF ₄	Si _x S _y F _z
S ₂ F ₂	F, S ₂ F	SiF ₄	Si _x S _y F _z
Cl ₂	Cl	SiCl ₄	Cl
Br ₂	Br	SiBr ₄	Br
CBr ₄	Br, CBr ₃	SiBr ₄	Br, Si _x C _y Br _z

Chemistries	Radical	Volatile Product	Inhibitor
CHF ₃	CF ₂	HF, SiF ₄	Si _x C _y H _z
CH ₂ F ₂	CFH C	HF	Si _x C _y F _z H _a
CH ₃ F	CH ₂ CFH	HF, H ₂	Si _x C _y F _z H _a
CF ₄ /O ₂	F, O, CF ₃	COF ₂ , O ₂ F, OF, F ₂ , SiF ₄	Si _x O _y F _z
CF ₄ /H ₂	F, H, CF ₃	CHF, HF, SiF ₄	Si _x C _y F _z
SF ₆ /O ₂	SF ₅ , O, F	SOF ₄ , SiF ₄	Si _x O _y F _z
SF ₆ /H ₂	SF ₅ , H, F	HF, SiF ₄	Si _x S _y F _z
SF ₆ /N ₂	SF ₅ , N ₂ , F	SiF ₄	Si _x S _y F _z
SF ₆ /CHF ₃	SF ₅ , F, CF ₂	HF, SiF ₄	Si _x C _y F _z
CBrF ₃	F, Br	SiBr ₄	Br, Si _x C _y F _z Si _x C _y Br _z
CCl ₄	CCl ₃ , Cl, C	SiCl ₄	Cl

→ *Fluorine-based gases remains
the most effective chemistry*

[H. Jansen, 1996]

SRC/SEMATECH Engineering Research Center for Environmentally Benign Semiconductor Manufacturing

Global Warming Potentials (GWP)

Chemistries	Atmospheric conc. in 2005 (ppt)	Con. since 1994* & 1998 (ppt)	Annual emission in late 1990s (Gg)	Rafactive efficiency (W/m ² -ppbv)	Lifetime (year)	Global warming potential	Ref.
CO ₂	278x10 ⁶	358x10 ⁶ *	-	-	variable	1	b
CH ₄	7x10 ⁵	1721x10 ³ *	-	-	12.2	21	b
N ₂ O	275x10 ³	311x10 ³ *	-	-	120	310	b
CHClF ₂	-	105x10 ³ *	-	-	12.1	1400	b
CF ₄	74	-	~15	0.1	50,000	6500	a
CCl ₂ F ₂	-	503x10 ³ *	-	-	102	7100	b
C ₂ F ₆	2.9	3.4	~2	0.26	10,000	9200	a
CHF ₃	18	22	~7	0.19	270	11700	a
SF ₆	5.6	7.1	~6	0.52	3,200	23900	a
NF ₃	<0.1	-	~2.3	0.21	740	16800	a

[W. Tsai, J Hazard Mater, 2008^a; United Nations Environment Program(UNEP), 2010^b]

- GWPs are one type of simplified index based upon radiative properties which estimate the potential future impacts of gases
- At the annual review, industrial members suggested a study on NF₃

Is NF₃ a Greenhouse Gas?

- $\text{NF}_3 + 2\text{H}_2\text{O} \rightarrow 3\text{HF} + \text{HNO}_2$
- $2\text{NF}_3 + 3\text{H}_2\text{O} \rightarrow 6\text{HF} + \text{NO} + \text{NO}_2$
- $2\text{NF}_3 + 3\text{H}_2 \rightarrow \text{N}_2 + 6\text{HF}$
- **NF₃ and its reaction products are designated as toxic substances in Toxic Substances Control Act (TSCA)**

Title	Authors	Year	Journal	#Citation
NF ₃ , the greenhouse gas missing from Kyoto	Prather, M.J. and J. Hsu	2010	Geophysical Research Letters	55444
Environmental and health risk analysis of NF ₃ , a toxic and potent greenhouse gas	Tsai, W. T.	2008	Journal of Hazardous Materials	28060

Etch Rate of Si /poly-Si*

Etchant	Etch rate (nm/min)	Power (W)	Pressure (mtorr)	Ref
SF ₆ /O ₂ /CHF ₃	3000	500		[c]
HBr(10sccm), Cl ₂ (70sccm), and SF ₆ (6sccm)	1100*		100	[e]
SF ₆ /O ₂ (25%)	880	300	25	[d]
SF ₆ (80%), C ₂ Cl ₃ F ₃ (20%),	700	140	47	[i]
SF ₆ (40sccm), O ₂ (14sccm), CHF ₃ (17sccm)	670		60	[j]
SF ₆ /O ₂ (25%)	490		100	[d]
F/F ₂	460			[k]
CF ₄ /O ₂	460			[k]
CBrF ₃ (80%), SF ₆ (10%), Ar(10%)	410	190	50	[h]
CBrF ₃ (90%), SF ₆ (10%)	310	100	50	[h]
SF ₆	300		20	[g]
CF ₄ /O ₂	300			[k]
O ₂ (12%)/CF ₄ (90%)/N ₂ (8%)	260*			[e]
CBrF ₃	60	100	50	[h]
SiF ₄ /O ₂	44			[k]
CBrF ₃	40	100	20	[g]
CF ₄	30			[k]
CF ₄	20*			[f]

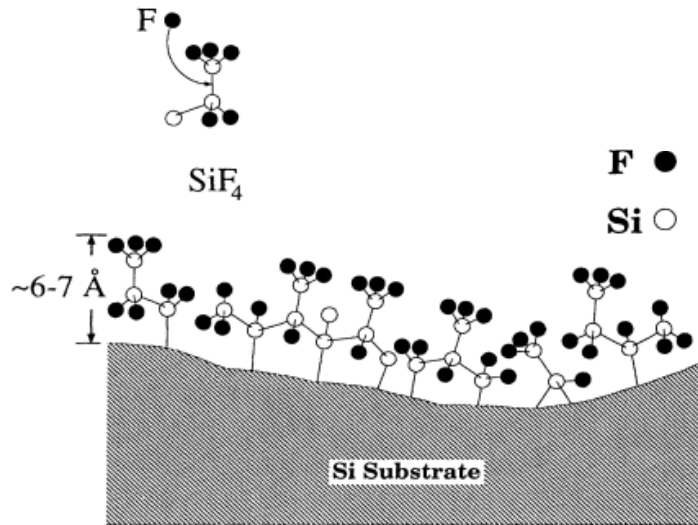
Etch Rate of SiO₂/Si₃N₄/SiC by NF₃

Etchant	Etch rate_SiO ₂ (nm/min)	Etch rate_Si ₃ N ₄ (nm/min)	Power (W/cm ²)	Pressure (mtorr)	Ref
NF ₃ (25%)/N ₂	860	7400	1.4	550	[1]
NF ₃ (25%)/Ar	670	8000	1.4	550	[1]
NF ₃ (25%)/He	560	7400	1.4	550	[1]
NF ₃ (25%)/O ₂	520	5200	1.4	550	[1]
NF ₃ (25%)/N ₂ O	280	3600	1.4	550	[1]
NF ₃ (100%)	90	1000	1.4	550	[1]

Etchant	Etch rate_SiC (nm/min)	Bias (W)	Power (W)	Pressure (mtorr)	Ref
NF ₃	437	150	900	12	[m]
NF ₃ /O ₂	350	250	750	2	[n]
NF ₃ (60%)/CH ₄ (40%)	111	150	800	6	[m]

- **Not much is found on NF₃ for silicon etching but it has been used for etching other silicon containing materials**

Silicon Etching by Fluorine



	Reactions	G(eV)	log(K)
1	$\text{Si(c)} \rightarrow \text{Si(g)}$	4.2	-70.5
1'	$\frac{1}{2}\text{F}_2(\text{g}) + \text{Si(c)} \rightarrow \text{SiF(g)}$	-0.54	9.0
2	$\text{F(g)} + \text{Si(c)} \rightarrow \text{SiF(g)}$	-1.18	19.8
3	$\text{F(g)} + \text{SiF(g)} \rightarrow \text{SiF}_2(\text{g})$	-6.31	105.9
4	$\text{F(g)} + \text{SiF}_2(\text{g}) \rightarrow \text{SiF}_3(\text{g})$	-5.57	93.4
5	$\text{F(g)} + \text{SiF}_3(\text{g}) \rightarrow \text{SiF}_4(\text{g})$	-5.82	97.7

- Fluorine is the most effective etching chemistry for silicon, especially atomic fluorine, as produced by plasma

Production of Fluorine from SF₆ and NF₃

	Reactions	G(eV)	Log(K)
S1	SF₆(g) → SF₅(g) + F(g)	3.52	-59.1
S2	SF₅(g) → SF₄(g) + F(g)	1.85	-31.0
S3	SF₄(g) → SF₃(g) + F(g)	3.07	-51.5
S4	SF₃(g) → SF₂(g) + F(g)	2.56	-42.9
S5	SF₂(g) → SF(g) + F(g)	3.64	-61.1
S6	SF(g) → S(g) + F(g)	3.25	-54.6

	Reactions	G(eV)	Log(K)
N1	NF₃(g) → NF₂(g) + F(g)	2.17	-36.4
N2	NF₂(g) → NF(g) + F(g)	2.58	-43.3
N3	NF(g) → N(g) + F(g)	2.84	-47.7

[CRC handbook, 2010]

[CRC handbook, 2010*; Y. Tanaka, IEEE, 1997]

- Under equilibrium conditions, the production of atomic F from SF₆ and NF₃ are comparable

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[CRC handbook, 2010]

[CRC handbook, 2010*; Y. Tanaka, IEEE, 1997]

	Reactions	G(eV)
S1	SF₆(g) + e → SF₅(g) + F(g) + e	4.00
S2	SF₅(g) + e → SF₄(g) + F(g) + e	2.27
S3	SF₄(g) + e → SF₃(g) + F(g) + e	3.47
S4	SF₃(g) + e → SF₂(g) + F(g) + e	2.92
S5	SF₂(g) + e → SF(g) + F(g) + e	4.01
S6	SF(g) + e → S(g) + F(g) + e	3.52

- For SF₆, electron impact dissociate energy is comparable and slightly higher than that from thermal equilibrium data (~0.4eV)

Production of Fluorine from SF₆ and NF₃

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S4	SF₃(g) + e → SF₂(g) + F(g) + e	2.92
S5	SF₂(g) + e → SF(g) + F(g) + e	4.01
S6	SF(g) + e → S(g) + F(g) + e	3.52

- For SF₆, electron impact dissociate energy is comparable and slightly higher than that from thermal equilibrium data (~0.4eV)

- It is possible that the available equilibrium data on NF₃ can be used as a guide to the production of fluorine in a plasma

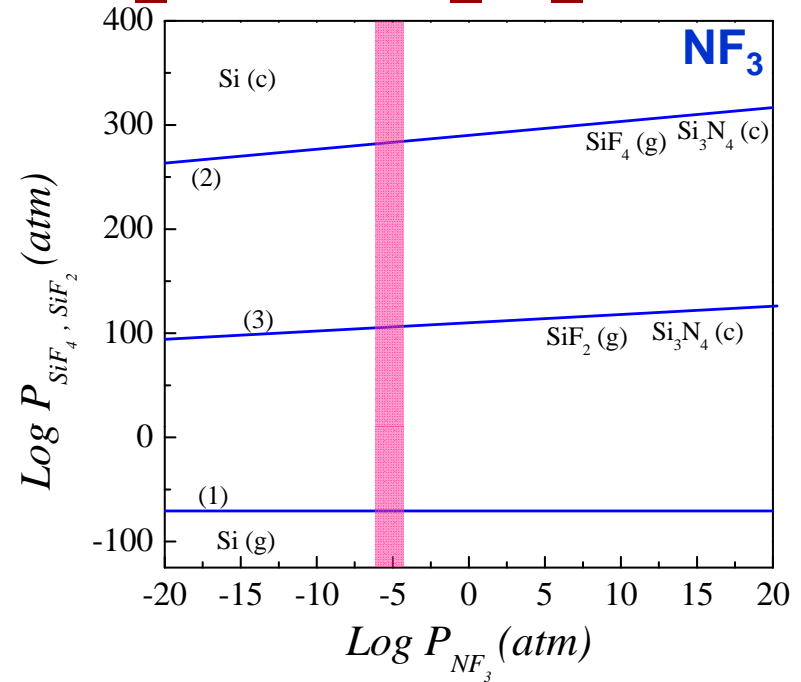
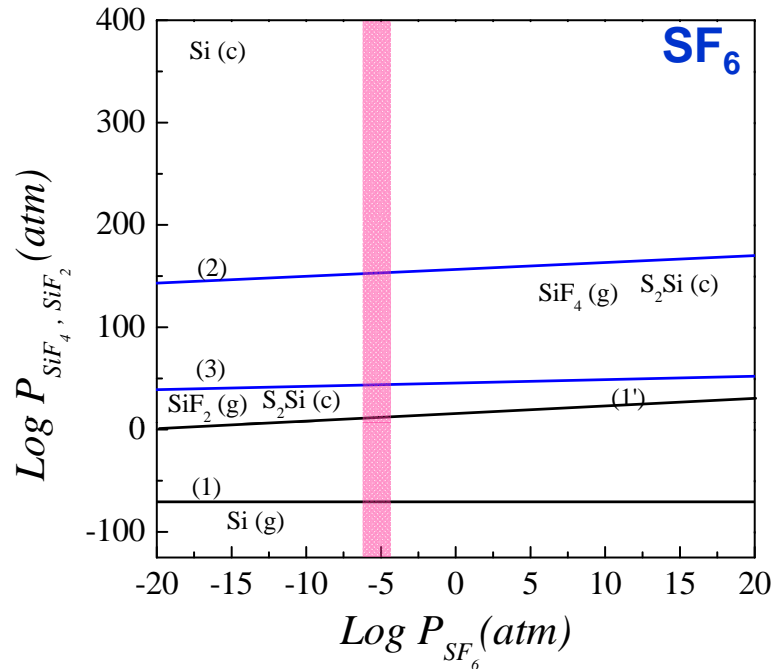
Can SF₆ be Replaced by NF₃?



Formation of non-volatile byproduct	G(eV)	Formation Si₃N₄(c)	G(eV)
$4\text{Si(c)} + 2\text{SF}_6(\text{g}) \rightarrow 3\text{SiF}_4(\text{g}) + \text{S}_2\text{Si(c)}$	-28.0	$6\text{Si(c)} + 4\text{NF}_3(\text{g}) \rightarrow 3\text{SiF}_4(\text{g}) + \text{Si}_3\text{N}_4(\text{c})$	-51.8
Removal of non-volatile byproduct	G(eV)	Removal of non-volatile byproduct	G(eV)
$\text{S}_2\text{Si(c)} + 16\text{F(g)} \rightarrow \text{SiF}_4(\text{g}) + 2\text{SF}_6(\text{g})$	-47.5	$\text{Si}_3\text{N}_4(\text{c}) + 24\text{F(g)} \rightarrow 3\text{SiF}_4(\text{g}) + 4\text{NF}_3(\text{g})$	-61.4

- From the thermodynamics analysis, NF₃ is more capable of removing silicon via the formation of SiF₄
- However, another significant reaction product from reaction with NF₃ is Si₃N₄, which has to be removed and competes for fluorine

Comparison of SiS₂ and Si₃N₄



	Reactions	G(eV)	log(K)
1	Si(c) → Si(g)	4.2	-70.5
1'	1/2F ₂ (g) + Si(c) → SiF(g)	-0.54	9.0
2	2SF ₆ (g) + 4Si(c) → S ₂ Si(c) + 3SiF ₄ (g)	-28.0	469.8
3	2SF ₆ (g) + 7Si(c) → S ₂ Si(c) + 6SiF ₂ (g)	-16.3	273.5

	Reactions	G(eV)	log(K)
1	Si(c) → Si(g)	4.2	-70.5
1'	1/2F ₂ (g) + Si(c) → SiF(g)	-0.54	9.0
2	4NF ₃ (g) + 6Si(c) → N ₄ Si ₃ (c) + 3SiF ₄ (g)	-51.8	870.2
3	4NF ₃ (g) + 9Si(c) → N ₄ Si ₃ (c) + 6SiF ₂ (g)	-40.1	673.97

	BP(°C)	H(kJ/mol)	S(J/mol/K)	G(kJ/mol)
S ₂ Si(c)	1090	-213.36	80.812	-212.61

	BP(°C)	H(kJ/mol)	S(J/mol/K)	G(kJ/mol)
Si ₃ N ₄ (c)	1900	-744.8	113.5	-646.7

A Strategy to Remove Si₃N₄

NF₃ - O₂

Formation Si ₃ N ₄ (c)	G(eV)
6Si(c) + 4NF ₃ (g) → 3SiF ₄ (g) + <u>Si₃N₄(c)</u>	-51.8
Removal Si ₃ N ₄ (c) by NF ₃	G(eV)
<u>Si₃N₄(c)</u> + 24F(g) → 3SiF ₄ (g) + 4NF ₃ (g)	-61.4

SF₆

Formation of non-volatile byproduct	G(eV)
Si(c) + SF ₆ (g) → SiF ₄ (g) + <u>S₂Si(c)</u>	-28.0
Removal of non-volatile byproduct	G(eV)
<u>S₂Si(c)</u> + 16F(g) → SiF ₄ (g) + 2SF ₆ (g)	-47.5

Removal of Si₃N₄(c) & SiO₂(c) by NF₃ & O₂		G(eV)
Removal of Si₃N₄(c)	<u>Si₃N₄(c)</u> + 7O ₂ (g) → <u>3SiO₂(c)</u> + 4NO ₂ (g)	-17.8
Removal of SiO₂(c)	<u>SiO₂(c)</u> + 5/2NF ₃ (g) → SiF ₄ (g) + 1/2NO ₂ F(g) + NOF ₃ (g) + N ₂ (g)	-6.5
Total reaction	<u>Si₃N₄(c)</u> + 7O ₂ (g) + 15/2NF ₃ → 3SiF ₄ (g) + 4NO ₂ (g) + 3/2NO ₂ F(g) + 6NOF ₃ (g) + 6N ₂ (g)	-37.1

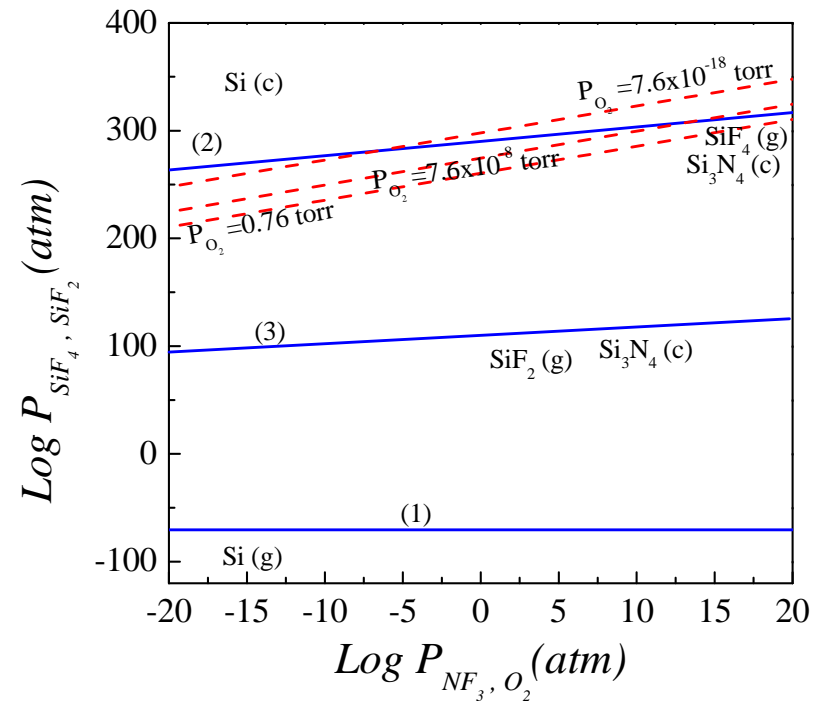
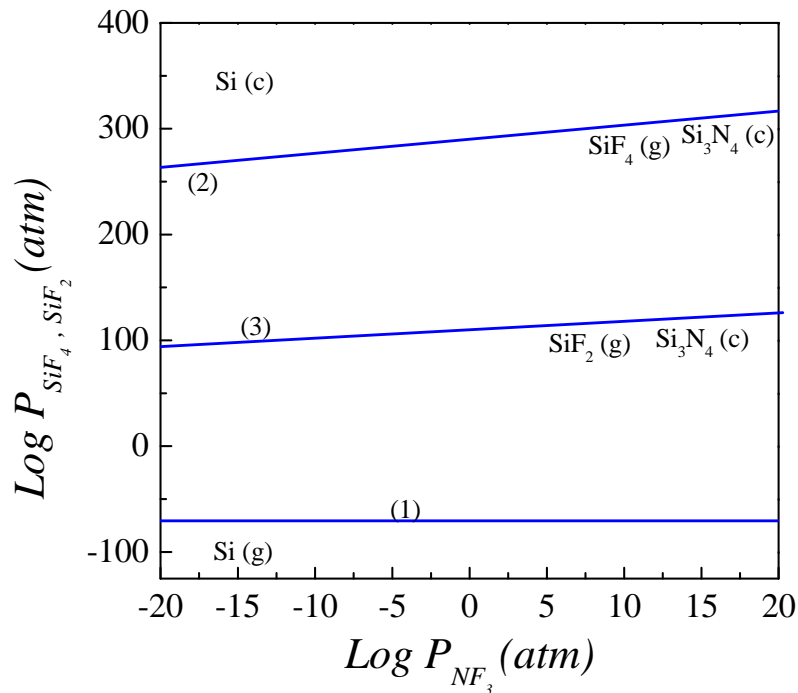
- From the analysis of ΔG, the non-volatile byproducts, Si₃N₄ could be removed by NF₃ - O₂ plasmas

Silicon Etching by $\text{NF}_3 - \text{O}_2$



	Reactions	G(eV)	log(K)
1	$\text{Si}(\text{c}) \rightarrow \text{Si}(\text{g})$	4.2	-70.5
2	$4\text{NF}_3(\text{g}) + 6\text{Si}(\text{c}) \rightarrow \text{Si}_3\text{N}_4(\text{c}) + 3\text{SiF}_4(\text{g})$	-51.8	870.2
3	$4\text{NF}_3(\text{g}) + 9\text{Si}(\text{c}) \rightarrow \text{Si}_3\text{N}_4(\text{c}) + 6\text{SiF}_2(\text{g})$	-40.1	673.97

$P_{\text{O}_2}(\text{torr})$	Reaction 4	G(eV)	log(K)
0.76	$\text{Si}_3\text{N}_4(\text{c}) + 7\text{O}_2(\text{g}) + 15/2\text{NF}_3 \rightarrow 3\text{SiF}_4(\text{g}) + 4\text{NO}_2(\text{g}) + 3/2\text{NO}_2\text{F}(\text{g}) + 6\text{NOF}_3(\text{g}) + 6\text{N}_2(\text{g})$	-37.1	623.4
7.6×10^{-8}			
7.6×10^{-18}			



Future Plans

Next Year Plans

- **Identify potential low impact gases in target applications**
- **Perform thermodynamic calculations to assess potential impact and projected effectiveness**
- **Implement target chemistries and carry out plasma etching assessment**

Long-Term Plans

- **Formulate the models to predict emission from plasma processes**
- **Assess the effectiveness of the plasma chemistries compared to that of the PFC gases**

Publications, Presentations, and Recognitions/Awards

- **Invited talk to AVS International Symposium, October 2012**
- **Presentation at the Plasma Processing Science Gordon Research Conference (GRC), July 2012**

Industrial Interactions and Technology Transfer

- **Video conference to Intel, January 31, 2012 (Karson Knutson, Doosik Kim)**
- **Conference call with Novellus, March 2012 (Ron Powell, Roey Shaviv, Juwen Gao)**
- **Student interview at IBM, February 29, 2012**
- **Student interview with Intel, March 2012**
- **Student will present at the Gordon Research Conference, July, 2012**

→ *After studying the TSV by detailed thermodynamic analysis, the next step is to carry out kinetic measurements.*

→ *Complex oxides? Magnetic materials? Noble metals?*

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