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**
- **Nicholas Altieri, PhD student, Chemical and Biomolecular Engineering, UCLA**

Other Researchers:

- **Taeseung Kim, postdoc, Chemical and Biomolecular Engineering, UCLA**
- **Michael Paine, undergraduate, Chemical and Biomolecular Engineering, UCLA**

Objectives

- Assess the feasibility of non-PFC chemistries in patterning etch-resistant materials (complex materials and structures)

- Identify non-PFC alternatives for the etching of carbon doped silica

- Examine the use of bond and group additivity methods to determine thermodynamic properties of carbon doped silica

- Screen the candidates of chemistries by comparing the pressure of primary etch product in the volatility diagram

ESH Metrics and Impact

1. **Reduction in the use of PFC gases by focusing on non-PFC chemistries such as CF_3I and NF_3 in the etch of carbon doped silica**

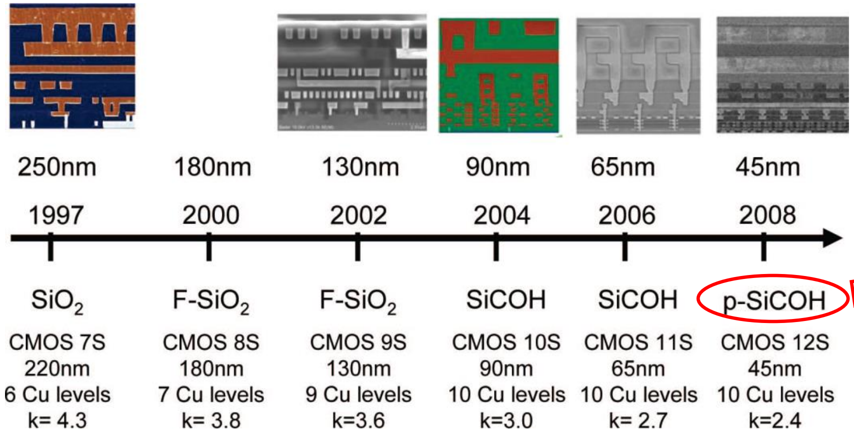
2. **Reduction in emission of PFC gases to environment**

3. **Reduction in the use of chemicals by tailoring the chemistries to the specific materials to be removed**

→ ***It is recognized that these are not yet quantitative, but as the project evolves, more quantitative measures will be provided***

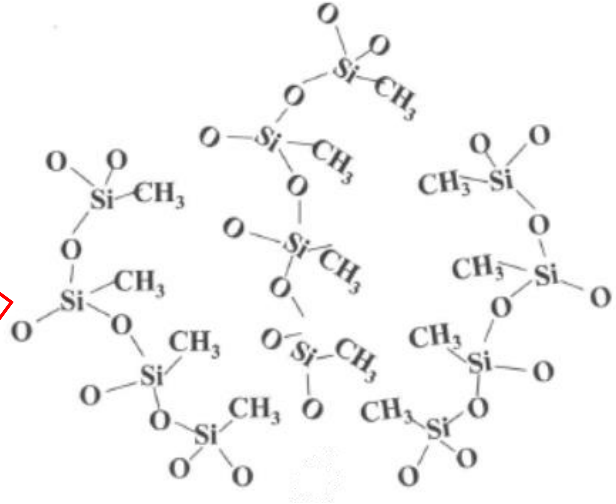
Composition of Low-k Dielectrics[1]

Dielectric materials used in IBM CMOS microprocessors as feature sizes decrease.[2]



250nm	180nm	130nm	90nm	65nm	45nm
1997	2000	2002	2004	2006	2008
SiO_2	F-SiO_2	F-SiO_2	SiCOH	SiCOH	p-SiCOH
CMOS 7S 220nm 6 Cu levels k= 4.3	CMOS 8S 180nm 7 Cu levels k= 3.8	CMOS 9S 130nm 9 Cu levels k=3.6	CMOS 10S 90nm 10 Cu levels k=3.0	CMOS 11S 65nm 10 Cu levels k= 2.7	CMOS 12S 45nm 10 Cu levels k=2.4

Porous carbon-doped silica, a promising low-k dielectric.



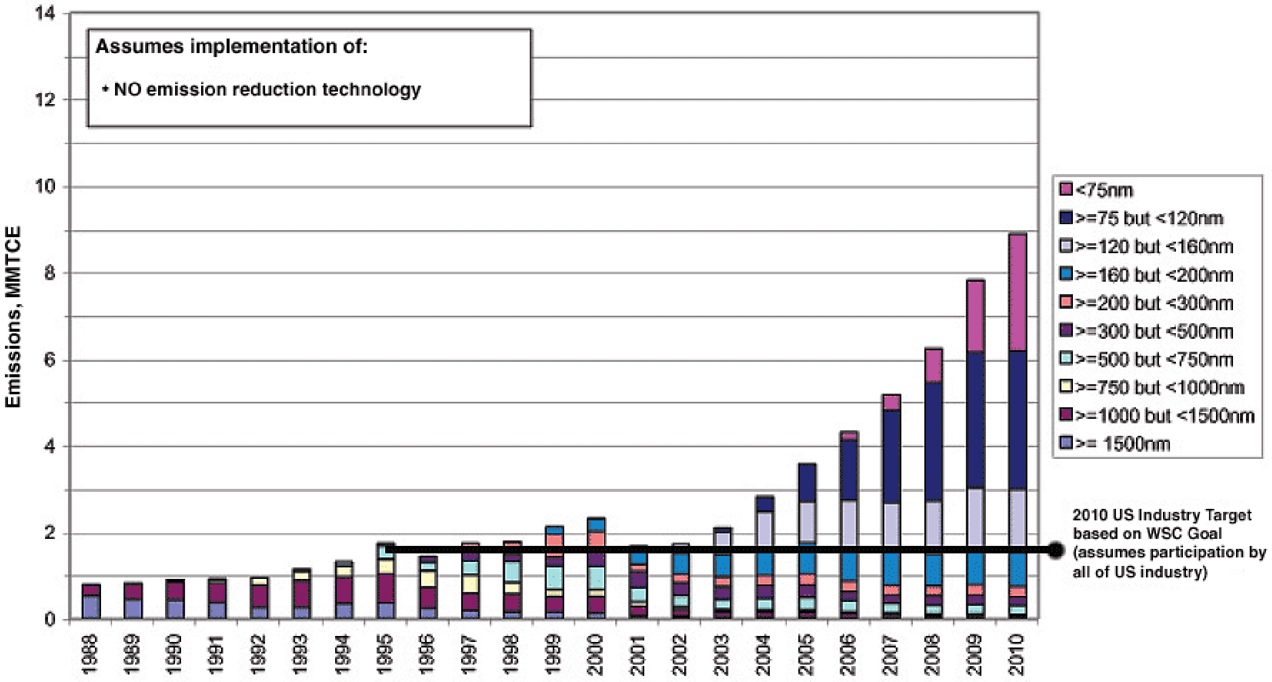
- Introduction of –CH_3 groups lowers the dielectric constant by replacing Si-O bonds with less polarizable Si-C and C-H bonds
- Porosity incorporates air (k = 1) into the film, thereby realizing a lower dielectric constant

[1] Vasarla, 2012 [2] W. Volksen, 2010

PFC Usage in BEOL

US EPA’s PFC emission model shows an average PFC emissions from semiconductor manufacturing for the evolution of complex devices[17]

MMTCE= Million metric tons of carbon



- Perfluorocarbon gases are used in BEOL for two major plasma processes: wafer patterning of thin films, especially dielectric films, and the in-situ cleaning of PECVD chambers

[17] PFC reduction/Climate partnership for the semiconductor industry, US EPA (U.S. Environmental Protection Agency), (2008); [18] W. Worth, PFC Technical Update Session, SEMICON/WEST(1995)

Global Warming Potential

Chemistries	Atmospheric conc. in 2005 (ppt)	Con. since 1994* & 1998 (ppt)	Annual emission in late 1990s (Gg)	Radiative efficiency (W/m^2/ppb)	Lifetime (year)	Global Warming Potential	Ref.
CO_2	278x10^6	358x10^6*	-	-	variable	1	[12]
CH_4	7x10^5	1721x10^3*	-	-	12.2	21	[12]
N_2O	275x10^3	311x10^3*	-	-	120	310	[12]
$CHClF_2$	-	105x10^3*	-	-	12.1	1400	[12]
CF_4	74	-	~15	0.1	50,000	6500	[13]
CCl_2F_2	-	503x10^3*	-	-	102	7100	[12]
C_2F_6	2.9	3.4	~2	0.26	10,000	9200	[13]
CHF_3	18	22	~7	0.19	270	11700	[12]
SF_6	5.6	7.1	~6	0.52	3,200	23900	[13]
NH_3	-	-	0.054	-	2 hrs	0	[14]
NF_3	<0.1	-	~2.3	0.21	740	16800	[13]
C_2F_4	-	-	-	-	1.9 days	<1	[15]
CF_3I	-	-	-	-	2 days	1	[10]
C_6F_6	-	-	-	-	-	<1	[16]

→ GWP is a simplified index based upon radiative properties that estimates the potential impacts of gases on global warming

Target of Carbon-doped SiO_2 Etch

*Material Metrics as Specified by Intel (Dr. Suri)

Intel specified metrics:

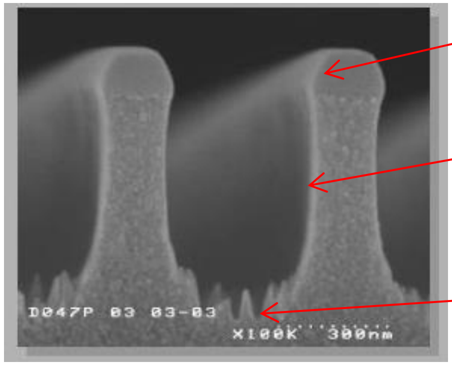
Elements	Range(%)
Si	20%
O	40%
C	15-40%
Porosity	20-25%
Thickness	100nm

Focus on:

1	Trench etch (later via)
2	Selectivity to PR
3	Sidewall damage

Target	Carbon doping level	Composition				Unit
		Si (%)	O (%)	C (%)	H (%)	
1	Low	15.4	23.1	15.4	46.1	$SiO_{1.5}CH_3$
2	↕	20	20	20	40	$SiOCH_2$
3		12.5	12.5	25	50	$SiO(CH_2)_2$
4	High	18.2	27.2	36.4	18.2	$SiO_{1.5}C_2H$

- SEM of C-doped SiO_2 etch by CF_4/Ar [a]



SiO_2 hard mask faceting

Bowed profiles

Rough bottom

[a] Etching of high k dielectrics, Plasma Technology for Advanced Devices, 2006, http://clarycon.blogspot.com/2006/12/etching-of-high-k-dielectrics.html

Systematic Approach - Thermodynamic

- 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
  - Gibbs free energy minimization

Effect of Doping

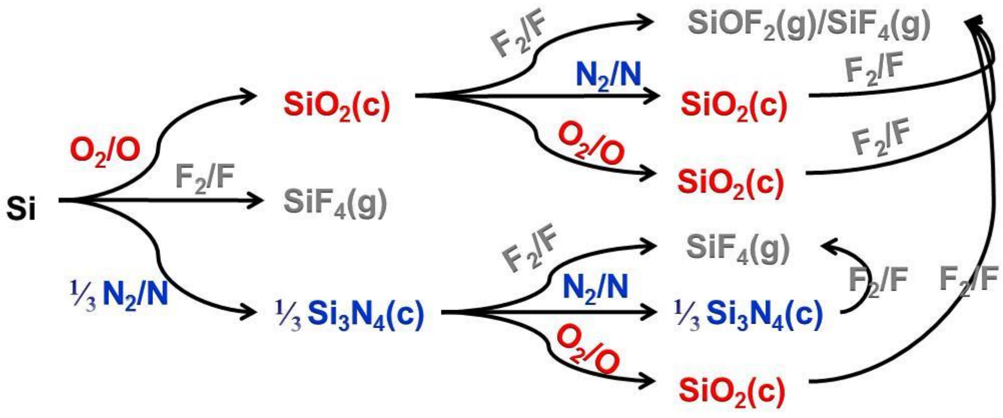
Gas phase

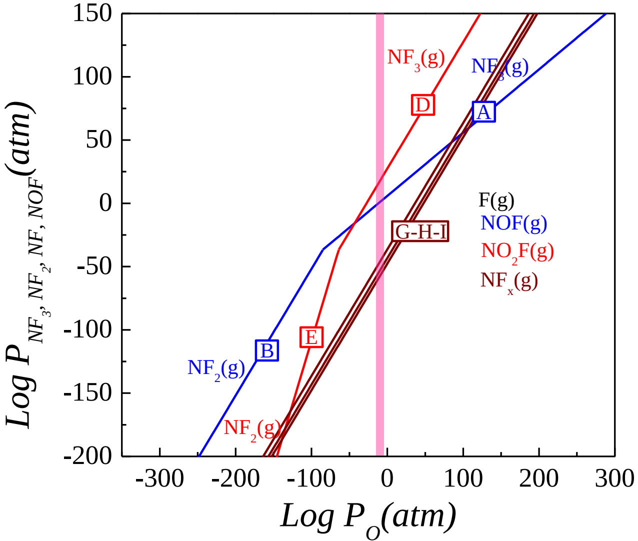
	NF_3 with Oxygen-300K	G(eV)	log(K)
A	$O(g) + NF_3(g) \rightarrow NOF(g) + 2F(g)$	-0.70	11.7
B	$O(g) + NF_2(g) \rightarrow NOF(g) + F(g)$	-2.87	48.1
C	$O(g) + NF(g) \rightarrow NOF(g)$	-5.45	91.5
D	$2O(g) + NF_3(g) \rightarrow NO_2F(g) + 2F(g)$	-3.26	54.8
E	$2O(g) + NF_2(g) \rightarrow NO_2F(g) + F(g)$	-5.43	91.3
F	$2O(g) + NF(g) \rightarrow NO_2F(g)$	-8.01	134.6

	NF_3-300K	G(eV)	log(K)
G	$NF_3(g) \rightarrow NF_2(g) + F(g)$	2.17	-36.4
H	$NF_2(g) \rightarrow NF(g) + F(g)$	2.58	-43.3
I	$NF(g) \rightarrow N(g) + F(g)$	2.84	-47.7

Surface

	Si_3N_4-O_2-F-300K	G(eV)	log(K)
1	$Si(c) + \frac{1}{2}O_2\ (g) \rightarrow SiO(g)$	-1.32	22.0
2	$SiO_2\,(c) \rightarrow SiO(g) + \frac{1}{2}O_2\ (g)$	7.56	-126.5
3	$\frac{1}{2}Si_2N_2O(c) \rightarrow Si(c) + \frac{1}{2}N_2\ (g) + \frac{1}{4}O_2\ (g)$	4.47	75.0
4	$\frac{1}{2}Si_2N_2O(c) + \frac{3}{4}O_2\ (g) \rightarrow SiO_2(c) + \frac{1}{2}N_2\ (g)$	-4.41	74.0
5	$\frac{1}{2}Si_2N_2O(c) + \frac{1}{4}O_2\ (g) \rightarrow SiO(g) + \frac{1}{2}N_2\ (g)$	3.15	-52.5
15	$SiO_2(c) + 4F(g) \rightarrow SiF_4(g) + O_2\ (g)$	-10.00	168.0
16	$SiO_2(c) + 2F(g) \rightarrow SiF_2(g) + O_2\ (g)$	1.39	-23.0
21	$\frac{1}{3}Si_3N_4(c) + \frac{1}{2}O_2\ (g) \rightarrow SiO(g) + \frac{2}{3}N_2(g)$	0.90	-15.0
22	$\frac{1}{3}Si_3N_4(c) + \frac{1}{4}O_2\ (g) \rightarrow \frac{1}{2}Si_2N_2O(c) + \frac{1}{6}N_2\ (g)$	-2.25	37.7

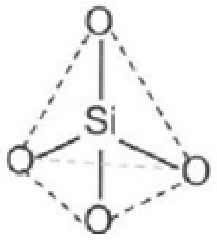
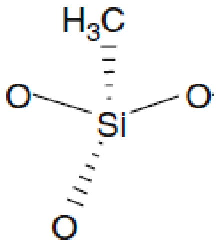
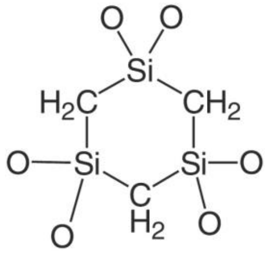
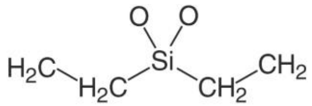
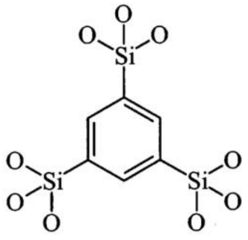




- Doping changes the etching characteristics

[4] NIST-JANAF Thermochemical (2013)

Data for C-doped Silica is Limited

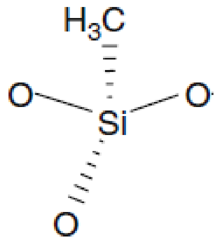
C-doped Silica	SiO_2[4]	$SiO_{1.5}CH_3$[4,5] (15.4%)	$SiOCH_2$[4,5] (20%)	$SiO(CH_2)_2$[4,5] (25%)	$SiO_{1.5}C_2H$[4,5,6] (36.4%)
Molecular Structure					
$\Delta_f H$ (kJ/mol)	-910.87				
$\Delta_f S$ (J/mol)	-182.53		No data is available		
$\Delta_f G$ (kJ/mol)	-856.11				

- The thermodynamic data of C-doped silica is not available in NIST, HSC chemistry and Perry's handbook

[4] NIST-JANAF Thermochemical (2013)

Bond and Group Additivity Method

- The bond additivity and group additivity methods proposed by Benson and Buss[3], is used to determine the energy of formation for C-doped silica
- Test case of one unit $SiO_{1.5}CH_3$ (15.4%)



$$\Delta_f S_{carbon-doped\ SiO_2} = S^\circ_{carbon-doped\ SiO_2} - (nS^\circ_{Si} + xS^\circ_{O_2} + yS^\circ_{C} + zS^\circ_{H_2})$$

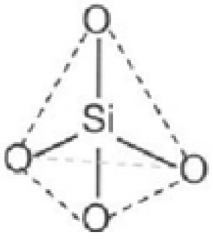
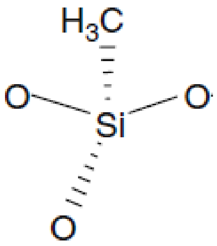
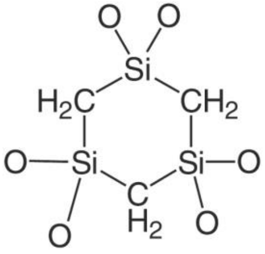
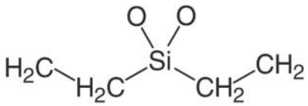
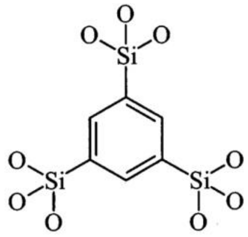
$$\Delta_f G_{carbon-doped\ SiO_2} = \Delta_f H_{carbon-doped\ SiO_2} - T \times \Delta_f S_{carbon-doped\ SiO_2}$$

Group / Bond	No. in $SiO_{1.5}CH_3$	Enthalpy[5] (kJ/mol)	Entropy[5] (J/mol*K)
SiO_2[6]	3/4	-910.9	-
CH_4[6]	3/4	-50.6	-
Si-C	1	-25.1	57.9
Si-O	3	-	-5.2
C-H	3	-	54.0
Total	-	-746.2	204.3

T=300K	SiO_2[4]	$SiO_{1.5}CH_3$
$\Delta_f H$(kJ/mol)	-910.9	-746.2
$\Delta_f S$(J/mol)	-182.5	-324.8
$\Delta_f G$(kJ/mol)	-856.1	-648.8

[3] S. W. Benson, ACS Symposium series 677, (1988) [4] NIST-JANAF Thermochemical (2013)

Compare ΔG for C-doped Silica

C-doped Silica	SiO_2[4]	$SiO_{1.5}CH_3$[4,5] (15.4%)	$SiOCH_2$[4,5] (20%)	$SiO(CH_2)_2$[4,5] (25%)	$SiO_{1.5}C_2H$[4,5,6] (36.4%)
Molecular Structure					
$\Delta_f H$ (kJ/mol)	-910.87	-746.20	-517.40	-538.00	-662.70
$\Delta_f S$ (J/mol)	-182.53	-324.77	-44.88	-141.84	-328.86
$\Delta_f G$ (kJ/mol)	-856.11	-648.80	-503.90	-495.50	-564.10

- Compounds with fewer Si-O bonds may be more readily etched

[4] NIST-JANAF Thermochemical (2013)

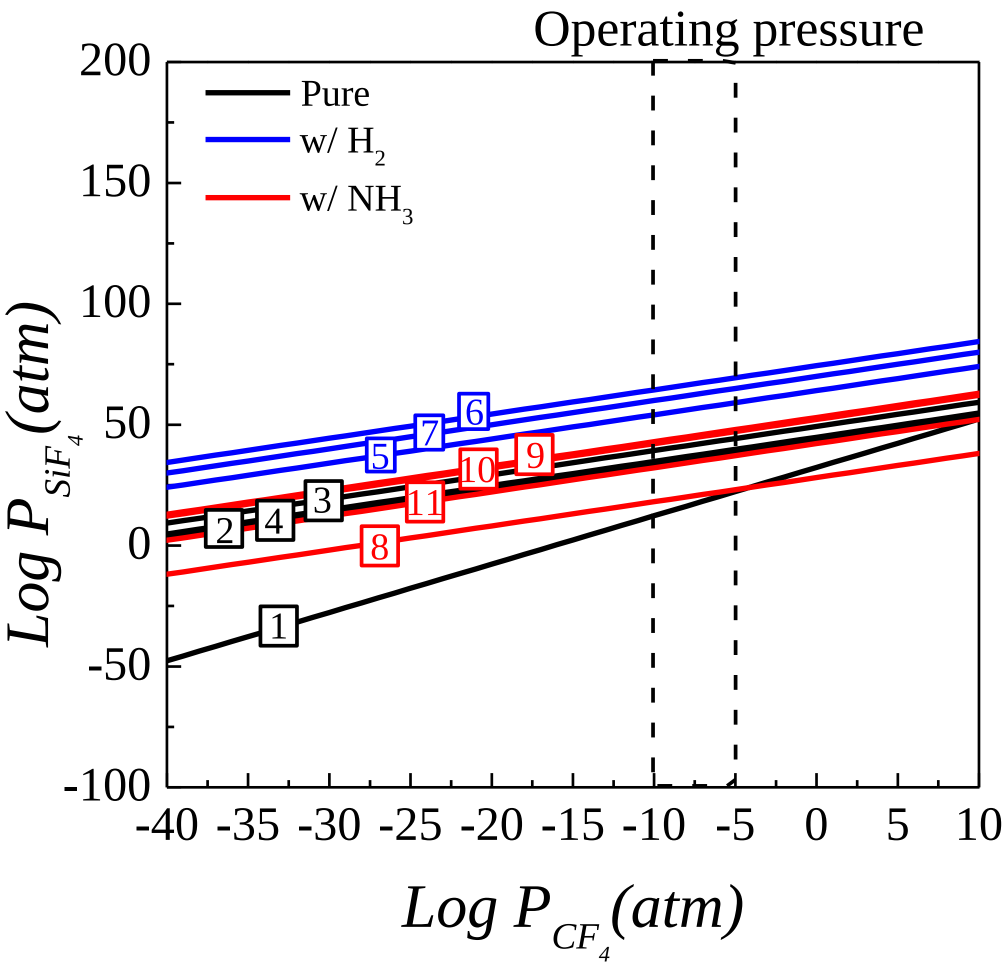
Selection of Chemistry

- Comparison of non-PFC and PFC in C-doped silica etch
- Consider the additives such as H_2 and NH_3 to facilitate the formation of volatile C-containing compounds from highly-doped silica (>15%C)

Reaction	**ΔG (eV)**
SiO_2	
$SiO_2(c) + \mathbf{2CF_4(g)} \rightarrow SiF_4(g) + 2COF_2(g)$	-1.92
$SiO_2(c) + \mathbf{2CF_4(g)} + \mathbf{H_2(g)} \rightarrow SiF_4(g) + 2COF(g) + 2HF(g)$	1.51
$SiO_2(c) + \mathbf{2CF_4(g)} + \mathbf{NH_3(g)} \rightarrow SiF_4(g) + COF_2(g) + HCN(g) + HOF(g) + HF(g)$	3.03
$SiO(CH_2)_2(c)$ (25%C-doped silica)	
$SiO(CH_2)_2(c) + \mathbf{CF_4(g)} \rightarrow SiF_4(g) + CO(g) + C_2H_4(g)$	-2.67
$SiO(CH_2)_2(c) + \mathbf{CF_4(g)} + 2\mathbf{H_2}(g) \rightarrow SiF_4(g) + CO(g) + 2CH_4(g)$	-4.43
$SiO(CH_2)_2(c) + \mathbf{CF_4(g)} + \mathbf{2/3NH_3(g)} \rightarrow SiF_4(g) + CO(g) + 2/3HCN(g) + 4/3CH_4(g)$	-3.10

[5] NIST-JANAF Thermochemical (2013)

Etching with CF_4

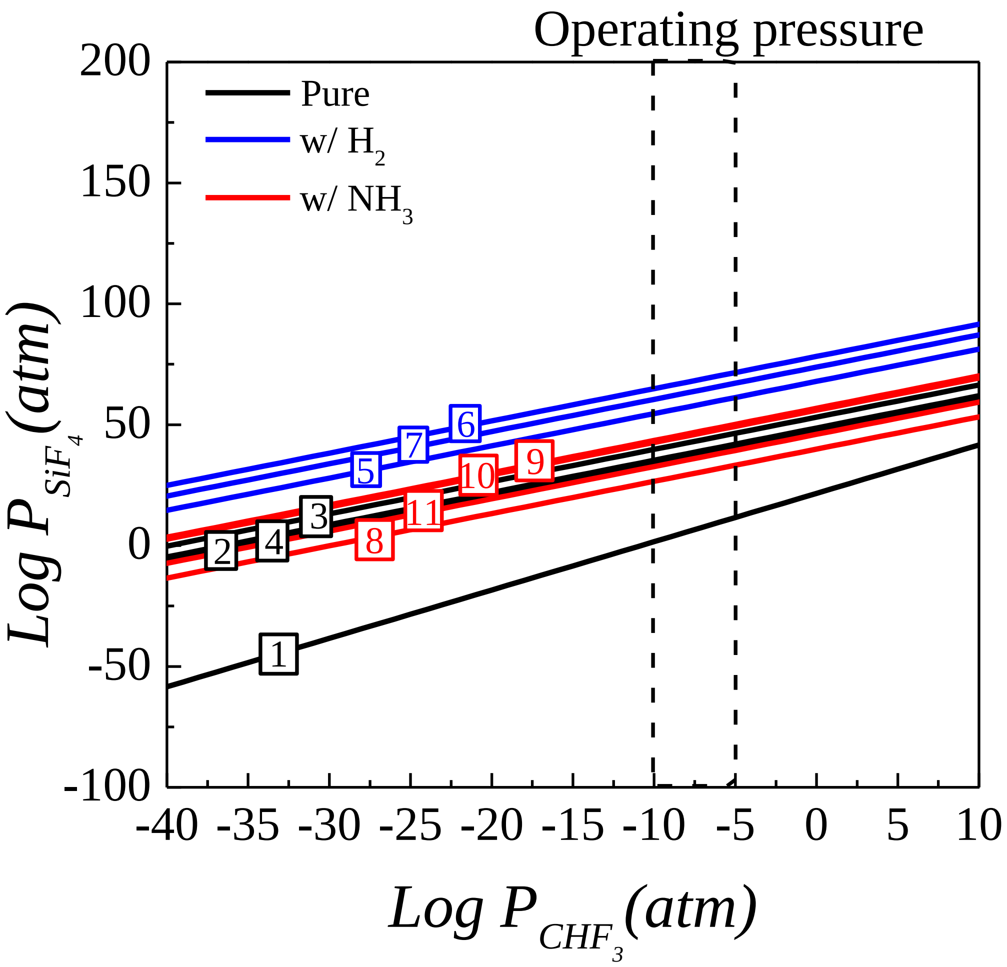


	Reaction	G (eV) [4,5,6]
[1]	**SiO_2**(c) + $2CF_4$(g) → SiF_4(g) + $2COF_2$(g)	-1.92
[2]	2**$SiO_{1.5}CH_3$**(c) + $2CF_4$(g) → $2SiF_4$(g) + COH_2(g) + CH_4(g) + 2CO(g)	-5.24
[3]	2**$SiOCH_2$**(c) + $2CF_4$(g) → $2SiF_4$(g) + C_2H_4(g) + 2CO(g)	-5.87
[4]	**$SiO(CH_2)_2$**(c) + CF_4(g) → SiF_4(g) + CO(g) + C_2H_4(g)	-2.67
[5]	**$SiOCH_2$**(c) + CF_4(g) + **H_2**(g) → SiF_4(g) + CO(g) + CH_4(g)	-3.82
[6]	**$SiO(CH_2)_2$**(c) + CF_4(g) + 2**H_2**(g) → SiF_4(g) + CO(g) + $2CH_4$(g)	-4.43
[7]	2**$SiO_{1.5}C_2H$**(c) + $2CF_4$(g) + 5**H_2**(g) → $2SiF_4$(g) + 3CO(g) + $3CH_4$(g)	-8.33
[8]	6**$SiO_{1.5}CH_3$**(c) + $6CF_4$(g) + **NH_3**(g) → $6SiF_4$(g) + HCN(g) + $6COH_2$(g) + $2CH_4$(g) + 3CO(g)	-12.88
[9]	3**$SiOCH_2$**(c) + $3CF_4$(g) + **NH_3**(g) → $3SiF_4$(g) + 3CO(g) + HCN(g) + $2CH_4$(g)	-9.46
[10]	3**$SiO(CH_2)_2$**(c) + $3CF_4$(g) + 2**NH_3**(g) → $3SiF_4$(g) + 3CO(g) + 2HCN(g) + $4CH_4$(g)	-9.31
[11]	6**$SiO_{1.5}C_2H$**(c) + $6CF_4$(g) + 5**NH_3**(g) → $6SiF_4$(g) + 9CO(g) + 5HCN(g) + $4CH_4$(g)	-15.07

- H_2 addition increases the etch product pressure – most effective with 25% C-doped silica
- NH_3 addition has little effect – most effective with 20% C-doped silica

[4] NIST-JANAF Thermochemical (2013)

Etching with CHF_3

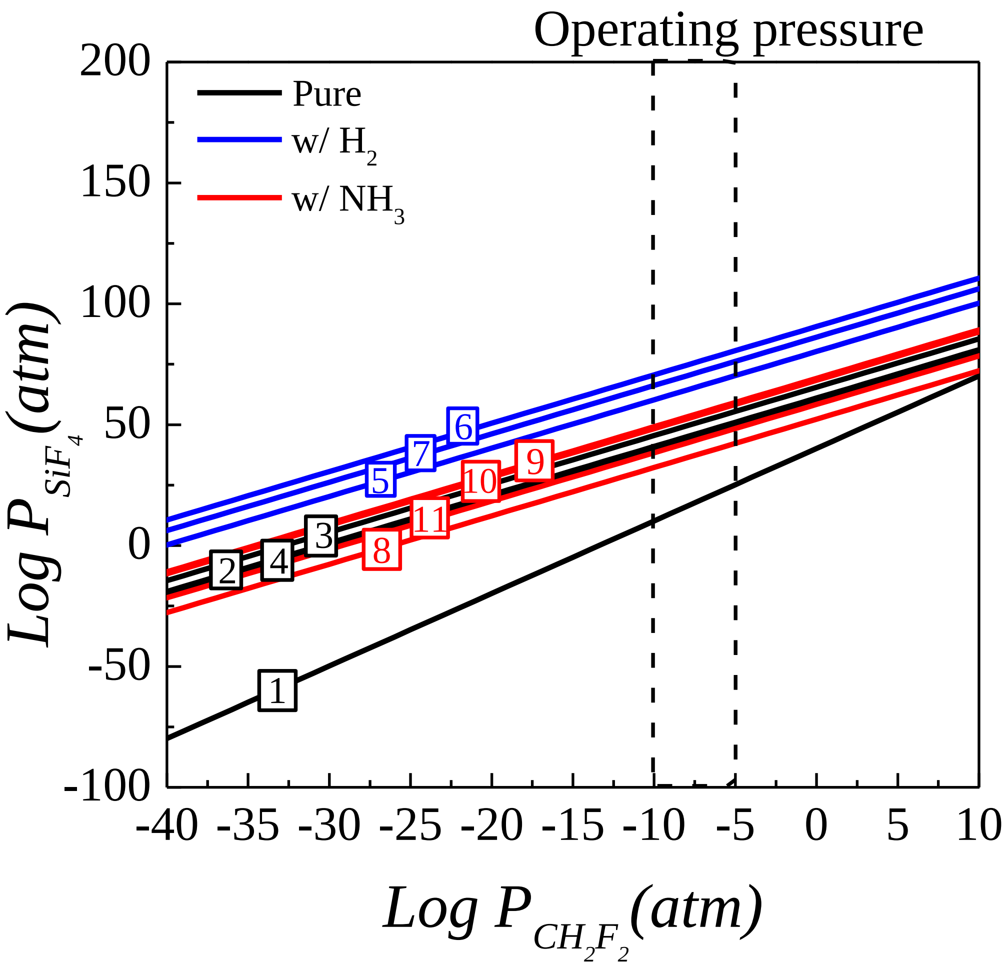


	Reaction	G (eV) [4,5,6]
[1]	$\mathbf{SiO_2}(c) + 2CHF_3(g) \rightarrow COF_2(g) + SiF_4(g) + COH_2(g)$	-1.29
[2]	$6\mathbf{SiO_{1.5}CH3}(c) + 8CHF_3(g) \rightarrow 6SiF_4(g) + 3COH_2(g) + 5CH_4(g) + 6CO(g)$	-17.09
[3]	$6\mathbf{SiOCH_2}(c) + 8CHF_3(g) \rightarrow 6SiF_4(g) + 6CO(g) + 3C_2H_4(g) + 2CH_4(g)$	-18.97
[4]	$3\mathbf{SiO(CH_2)_2}(c) + 4CHF_3(g) \rightarrow 3SiF_4(g) + 3CO(g) + CH_4(g) + 3C_2H_4(g)$	-8.68
[5]	$3\mathbf{SiOCH_2}(c) + 4CHF_3(g) + 3\mathbf{H_2}(g) \rightarrow 3SiF_4(g) + 3CO + 4CH_4(g)$	-12.13
[6]	$3\mathbf{SiO(CH_2)_2}(c) + 4CHF_3(g) + 6\mathbf{H_2}(g) \rightarrow 3SiF_4(g) + 3CO(g) + 7CH_4(g)$	-13.97
[7]	$6\mathbf{SiO_{1.5}C_2H}(c) + 8CHF_3(g) + 15\mathbf{H_2}(g) \rightarrow 6SiF_4(g) + 9CO(g) + 11CH_4(g)$	-26.37
[8]	$6\mathbf{SiO_{1.5}CH_3}(c) + 8CHF_3(g) + \mathbf{NH_3}(g) \rightarrow 6SiF_4(g) + HCN(g) + 6COH_2(g) + 4CH_4(g) + 3CO(g)$	-14.25
[9]	$3\mathbf{SiOCH_2}(c) + 4CHF_3(g) + \mathbf{NH_3}(g) \rightarrow 3SiF_4(g) + 3CO(g) + HCN(g) + 3CH_4(g)$	-10.14
[10]	$3\mathbf{SiO(CH_2)_2}(c) + 4CHF_3(g) + 2\mathbf{NH_3}(g) \rightarrow 3SiF_4(g) + 3CO(g) + 2HCN(g) + 5CH_4(g)$	-10.00
[11]	$6\mathbf{SiO_{1.5}C_2H}(c) + 8CHF_3(g) + 5\mathbf{NH_3}(g) \rightarrow 6SiF_4(g) + 9CO(g) + 5HCN(g) + 6CH_4(g)$	-16.43

- H_2 addition increases the etch product pressure – most effective with 25% C-doped silica
- NH_3 addition has little effect – most effective with 20% C-doped silica

[4] NIST-JANAF Thermochemical (2013)

Etching with CH_2F_2

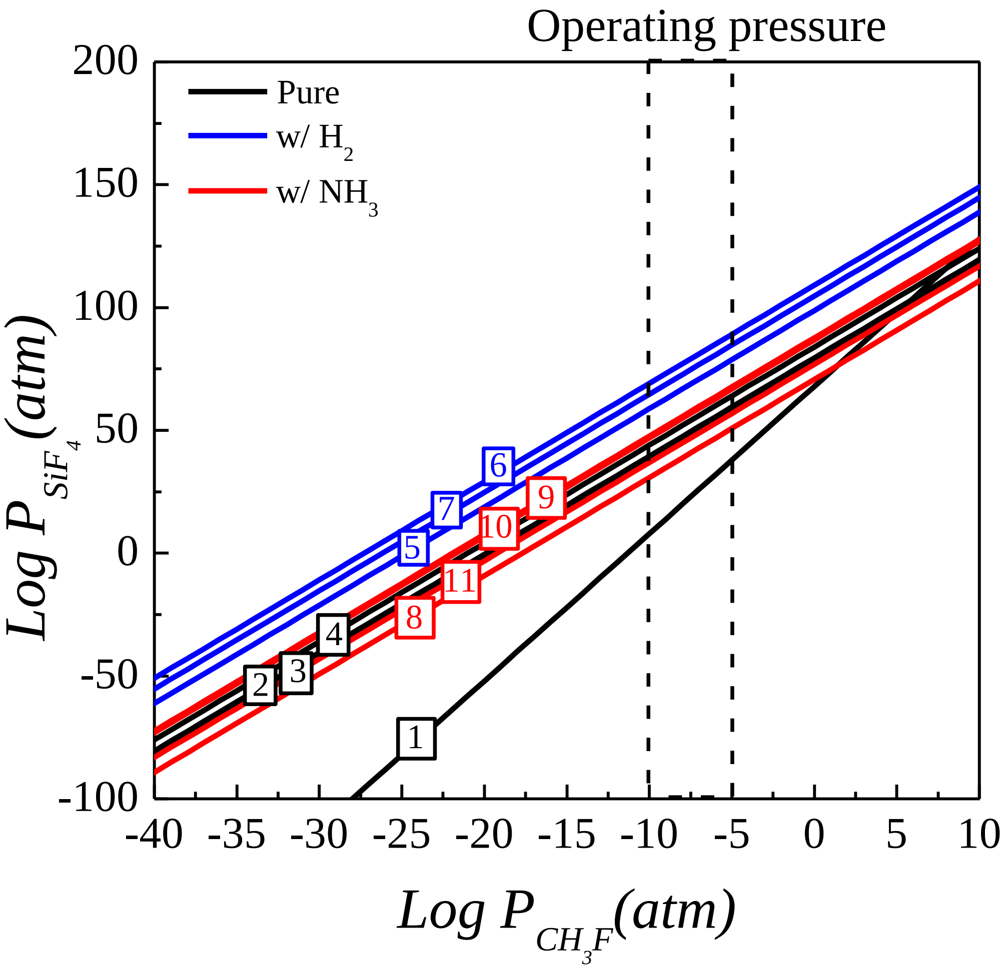


	Reaction	G (eV) [4,5,6]
[1]	$\mathbf{SiO_2}(c) + 3CH_2F_2(g) \rightarrow COF_2(g) + SiF_4(g) + COH_2(g) + CH_4(g)$	-2.39
[2]	$2\mathbf{SiO_{1.5}CH_3}(c) + 4CH_2F_2(g) \rightarrow 2SiF_4(g) + COH_2(g) + 2CO(g) + 3CH_4(g)$	-7.17
[3]	$2\mathbf{SiOCH_2}(c) + 4CH_2F_2(g) \rightarrow 2SiF_4(g) + 2CO(g) + C_2H_4(g) + 2CH_4(g)$	-7.80
[4]	$\mathbf{SiO(CH_2)_2}(c) + 2CH_2F_2(g) \rightarrow SiF_4(g) + CO(g) + C_2H_4(g) + CH_4(g)$	-3.63
[5]	$\mathbf{SiOCH_2}(c) + 2CH_2F_2(g) + \mathbf{H_2}(g) \rightarrow SiF_4(g) + CO(g) + 2CH_4(g)$	-4.78
[6]	$\mathbf{SiO(CH_2)_2}(c) + 2CH_2F_2(g) + 2\mathbf{H_2}(g) \rightarrow SiF_4(g) + CO(g) + 3CH_4(g)$	-5.39
[7]	$2\mathbf{SiO_{1.5}C_2H}(c) + 4CH_2F_2(g) + 5\mathbf{H_2}(g) \rightarrow 2SiF_4(g) + 3CO(g) + 5CH_4(g)$	-10.26
[8]	$6\mathbf{SiO_{1.5}CH_3}(c) + 12CH_2F_2(g) + \mathbf{NH_3}(g) \rightarrow 6SiF_4(g) + HCN(g) + 6COH_2(g) + 8CH_4(g) + 3CO(g)$	-18.67
[9]	$3\mathbf{SiOCH_2}(c) + 6CH_2F_2(g) + \mathbf{NH_3}(g) \rightarrow 3SiF_4(g) + 3CO(g) + HCN(g) + 5CH_4(g)$	-12.35
[10]	$3\mathbf{SiO(CH_2)_2}(c) + 6CH_2F_2(g) + 2\mathbf{NH_3}(g) \rightarrow 3SiF_4(g) + 3CO(g) + 2HCN(g) + 7CH_4(g)$	-12.21
[11]	$6\mathbf{SiO_{1.5}C_2H}(c) + 12CH_2F_2(g) + 5\mathbf{NH_3}(g) \rightarrow 6SiF_4(g) + 9CO(g) + 5HCN(g) + 10CH_4(g)$	-20.86

- H_2 addition increases the etch product pressure – most effective with 25% C-doped silica
- NH_3 addition has little effect – most effective with 20% C-doped silica

[4] NIST-JANAF Thermochemical (2013)

Etching with CH_3F

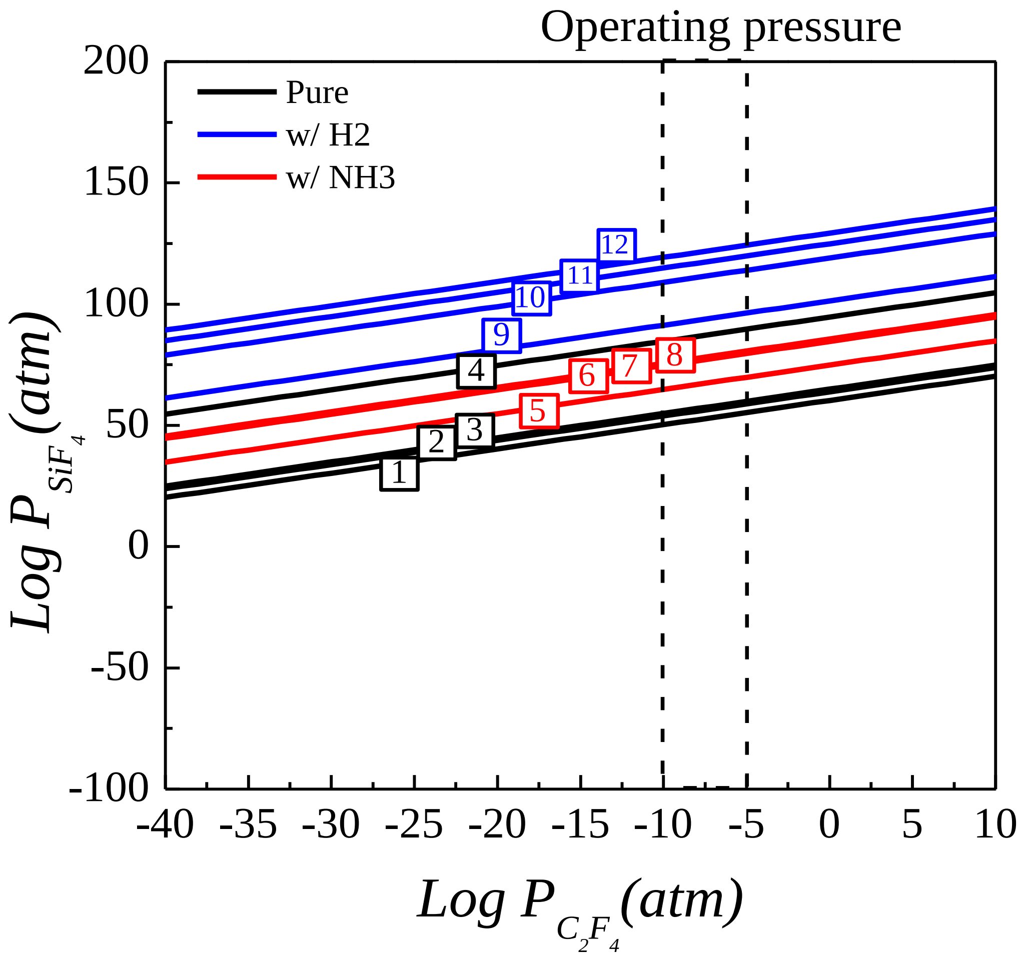


	Reaction	G (eV) [4,5,6]
[1]	$\mathbf{SiO_2}(c) + 6CH_3F(g) \rightarrow COF_2(g) + SiF_4(g) + 4CH4(g) + COH2(g)$	-4.04
[2]	$2\mathbf{SiO_{1.5}CH_3}(c) + 8CH_3F(g) \rightarrow 2SiF_4(g) + 2CO(g) + COH_2(g) + 7CH_4(g)$	-9.37
[3]	$2\mathbf{SiOCH_2}(c) + 8CH_3F(g) \rightarrow 2SiF_4(g) + 2CO(g) + C_2H_4(g) + 6CH_4(g)$	-10.00
[4]	$\mathbf{SiO(CH_2)_2}(c) + 4CH_3F(g) \rightarrow SiF_4(g) + CO(g) + C_2H_4(g) + 3CH_4(g)$	-4.73
[5]	$\mathbf{SiOCH_2}(C) + 4CH_3F(g) + \mathbf{H_2}(g) \rightarrow SiF_4(g) + CO(g) + 4CH_4(g)$	-5.88
[6]	$\mathbf{SiO(CH_2)_2}(c) + 4CH_3F(g) + 2\mathbf{H_2}(g) \rightarrow SiF_4(g) + CO(g) + 5CH_4(g)$	-6.50
[7]	$2\mathbf{SiO_{1.5}C_2H}(c) + 8CH_3F(g) + 5\mathbf{H_2}(g) \rightarrow 2SiF_4(g) + 3CO(g) + 9CH_4(g)$	-12.47
[8]	$6\mathbf{SiO_{1.5}CH_3}(c) + 24CH_3F(g) + \mathbf{NH_3}(g) \rightarrow 6SiF_4(g) + HCN(g) + 6COH_2(g) + 20CH_4(g) + 3CO(g)$	-25.28
[9]	$3\mathbf{SiOCH_2}(c) + 12CH_3F(g) + \mathbf{NH_3}(g) \rightarrow 3SiF_4(g) + 3CO(g) + HCN(g) + 11CH_4(g)$	-15.66
[10]	$3\mathbf{SiO(CH_2)_2}(c) + 12CH_3F(g) + 2\mathbf{NH_3}(g) \rightarrow 3SiF_4(g) + 3CO(g) + 2HCN(g) + 13CH_4(g)$	-15.51
[11]	$6\mathbf{SiO_{1.5}C_2H}(c) + 24CH_3F(g) + 5\mathbf{NH_3}(g) \rightarrow 6SiF_4(g) + 9CO(g) + 5HCN(g) + 22CH_4(g)$	-27.46

- H_2 addition increases the etch product pressure – most effective with 25% C-doped silica
- NH_3 addition has little effect – most effective with 20% C-doped silica

[4] NIST-JANAF Thermochemical (2013)

Etching with C_2F_4

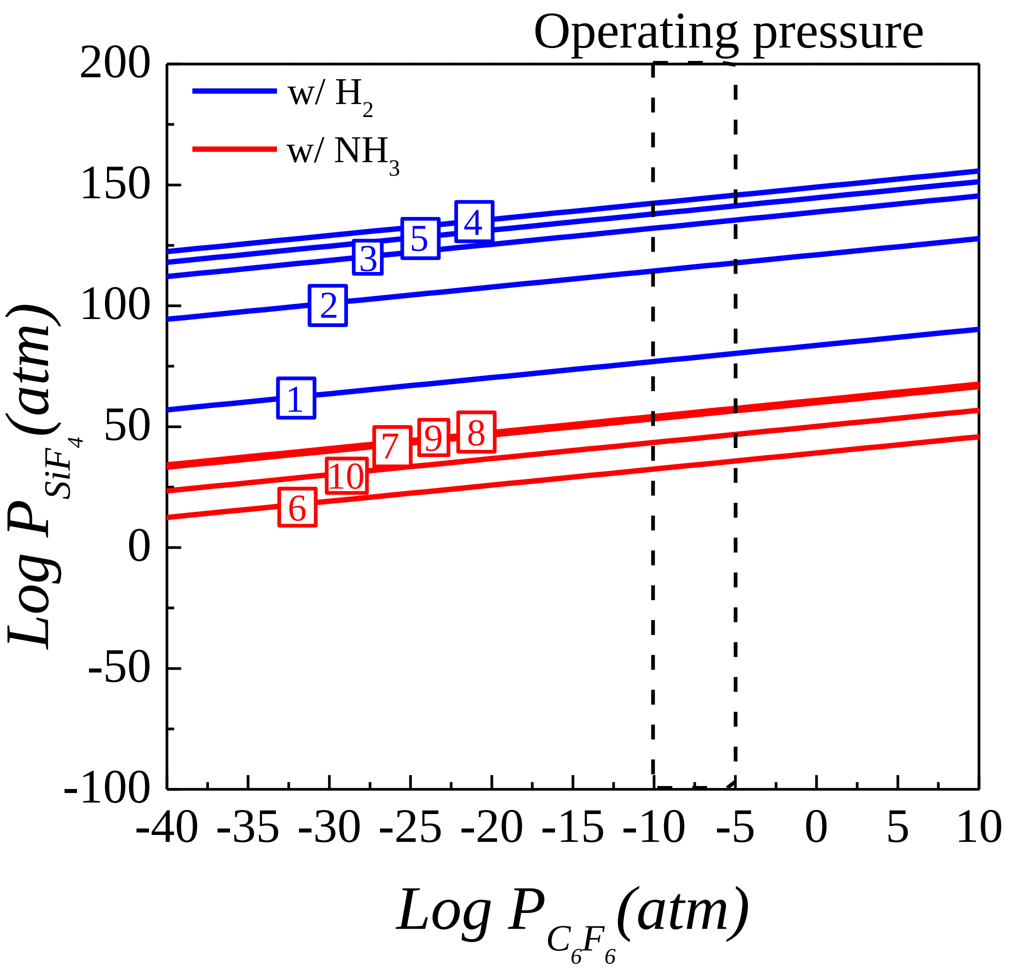


	Reaction	G (eV) [4,5,6]
[1]	2SiO(CH2)2(c) + 2C2F4(g) → 2SiF4(g) + 2CO(g) + C2H4(g) + 2C2H2(g)	-7.18
[2]	SiO2(c) + C2F4(g) → SiF4(g) + 2CO(g)	-3.80
[3]	SiOCH2(c) + C2F4(g) → SiF4(g) + CO(g) + C2H2(g)	-3.87
[4]	4SiO1.5CH3(c) + 4C2F4(g) → 4SiF4(g) + 6CO(g) + 3CH4(g)	-22.55
[5]	2SiO1.5C2H(c) + 2C2F4(g) + 3NH3(g) → 2SiF4(g) + 3CO(g) + 2CH4(g) + 3HCN(g)	-8.91
[6]	2SiO1.5CH3(c) + 2C2F4(g) + NH3(g) → 2SiF4(g) + HCN(g) + 3CO(g) + 2CH4(g)	-10.08
[7]	3SiO(CH2)2(c) + 3C2F4(g) + 4NH3(g) → 3SiF4(g) + 4HCN(g) + 3CO(g) + 5CH4(g)	-15.15
[8]	SiOCH2(c) + C2F4(g) + NH3(g) → SiF4(g) + HCN(g) + CO(g) + CH4(g)	-5.10
[9]	2SiO1.5CH3(c) + 2C2F4(g) + 3H2(g) → 2SiF4(g) + 3CO(g) + 3CH4(g)	-12.06
[10]	SiOCH2(c) + C2F4(g) + 3H2(g) → SiF4(g) + CO(g) + 2CH4(g)	-7.08
[11]	2SiO1.5C2H(c) + 2C2F4(g) + 9H2(g) → 2SiF4(g) + 3CO(g) + 5CH4(g)	-14.87
[12]	SiO(CH2)2(c) + C2F4(g) + 4H2(g)→ SiF4(g) + CO(g) + 3CH4(g)	-7.70

- All chemistries are most effective with 36.4% C-doped silica

[4] NIST-JANAF Thermochemical (2013)

Etching with C_6F_6

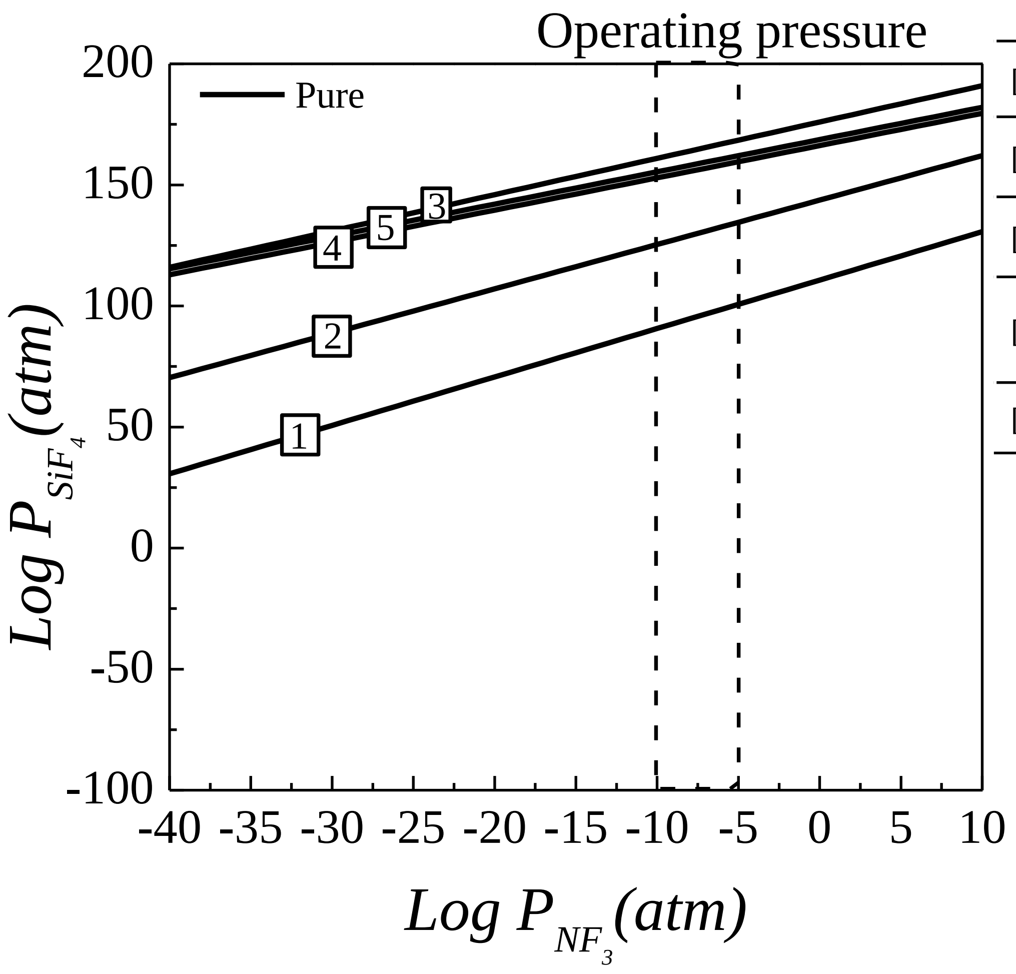


	Reaction	G (eV) [4,5,6]
[1]	$3\mathbf{SiO_2}(c) + 2C_6F_6(g) + 12\mathbf{H_2}(g) \rightarrow 3SiF_4(g) + 6CO(g) + 6CH_4(g)$	-14.94
[2]	$6\mathbf{SiO_{1.5}CH_3}(c) + 4C_6F_6(g) + 33\mathbf{H_2}(g) \rightarrow 6SiF_4(g) + 9CO(g) + 21CH_4(g)$	-43.25
[3]	$3\mathbf{SiOCH_2}(c) + 2C_6F_6(g) + 21\mathbf{H_2}(g) \rightarrow 3SiF_4(g) + 3CO(g) + 12CH_4(g)$	-24.78
[4]	$3\mathbf{SiO(CH_2)_2}(c) + 2C_6F_6(g) + 24\mathbf{H_2}(g) \rightarrow 3SiF_4(g) + 3CO(g) + 15CH_4(g)$	-26.63
[5]	$6\mathbf{SiO_{1.5}C_2H}(c) + 4C_6F_6(g) + 51\mathbf{H_2}(g) \rightarrow 6SiF_4(g) + 9CO(g) + 27CH_4(g)$	-51.68
[6]	$3\mathbf{SiO_2}(c) + 2C_6F_6(g) + 4\mathbf{NH_3}(g) \rightarrow 3SiF_4(g) + 6CO(g) + 4HCN(g) + 2CH_4(g)$	-6.99
[7]	$6\mathbf{SiO_{1.5}CH_3}(c) + 4C_6F_6(g) + 11\mathbf{NH_3}(g) \rightarrow 6SiF_4(g) + 9CO(g) + 11HCN(g) + 10CH_4(g)$	-21.40
[8]	$3\mathbf{SiOCH_2}(c) + 2C_6F_6(g) + 7\mathbf{NH_3}(g) \rightarrow 3SiF_4(g) + 3CO(g) + 7HCN(g) + 5CH_4(g)$	-10.88
[9]	$3\mathbf{SiO(CH_2)_2}(c) + 2C_6F_6(g) + 8\mathbf{NH_3}(g) \rightarrow 3SiF_4(g) + 3CO(g) + 8HCN(g) + 7CH_4(g)$	-10.73
[10]	$6\mathbf{SiO_{1.5}C_2H}(c) + 4C_6F_6(g) + 17\mathbf{NH_3}(g) \rightarrow 6SiF_4(g) + 9CO(g) + 17HCN(g) + 10CH_4(g)$	-17.91

- H_2 addition increases the etch product pressure – most effective with 25% C-doped silica
- NH_3 increases the etch product pressure – most effective with 15.4% C-doped silica

[4] NIST-JANAF Thermochemical (2013)

Etching with NF_3



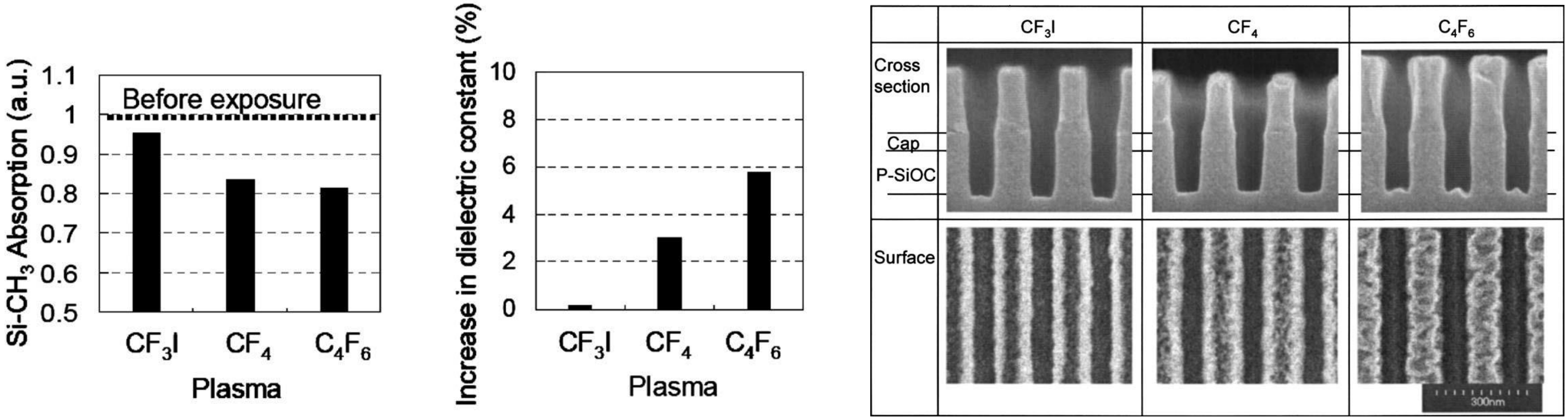
	Reaction	G (eV) [4,5,6]
[1]	$\mathbf{SiO_2}$(c) + 2NF_3(g) → SiF_4(g) + 2NOF(g)	-6.59
[2]	6$\mathbf{SiO_{1.5}CH_3}$(c) + 11NF_3(g) → 6SiF4(g) + 9NOF(g) + 2HCN(g) + 4CH4(g)	-51.34
[3]	2$\mathbf{SiOCH_2}$(c) + 3NF_3(g) → 2SiF_4(g) + HF(g) + HCN(g) + COH_2(g) + N_2O(g)	-20.95
[4]	3$\mathbf{SiO(CH_2)_2}$(c) + 4NF_3(g) → 3SiF_4(g) + 2HCN(g) + CO(g) + COH_2(g) + N_2O(g) + 2CH_4(g)	-29.69
[5]	6$\mathbf{SiO_{1.5}C_2H}$(c) + 8NF_3(g) → 6SiF_4(g) + 6HCN(g) + N_2O(g) + 2CO_2(g) + 4CO(g)	-60.27

- $NF_{3\ \mathrm{is}}$ most effective with 20% C-doped silica

Low Damage Etching with CF_3I[8]

Etchant	Etch Rate (nm/min)	GWP
CF_3I	250	1
CF_4	200	6500
C_4F_6	410	290

- CF_3I produces less UV radiation
- I atoms scavenge F radicals to form IF_x
- Less damage to doped carbon keeps dielectric constant low



(a) (b) (c)

(a) Decrease in absorption corresponding to Si-CH_3 bond. (b) Increase in dielectric constant after etching. (c) Etch profiles of porous SiOCH.

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[8] Eiichi Soda, et al. J. Vac. Sci. Tech. A 26, 875 (2008) – from Semiconductor Leading Edge Technologies, Inc., Japan & Tohoku University, Japan

Environmental Impact of CF_3I

Amount of fluorocarbons detected in exhaust in sccm. Feed gases are all 40 sccm except CF_4 (15 sccm) – CHF_3 (25 sccm).[9]

Etchant	**CF_4 (6300)**	**CHF_3 (12100)**	**CF_3I (1)**	**C_2F_6 (12500)**	**C_3F_8 (6950)**	**C_2F_4**
CF_4	29	0.6	-	2.3	0.6	0.6
CHF_3	4.5	23	-	0.9	0.2	1.6
CF_3I	1.2	-	26	3.4	0.3	1.9
CF_4-CHF_3	11	15	-	1.1	2.9	1.8

- Recombination in plasma forms high-GWP gases (CF_4, C_2F_6)
  - CF_3I has less than 1/3 total impact on global warming compared to other fluorocarbons
- CF_3I photolyzes within days in atmosphere to eventually form CO_2, HF, and HI. [10]
  - This is responsible for GWP_{100} values ≈1.
  - When released from sea level, CF_3I has small effect on ozone (ozone depletion potential = 0.018). [11]

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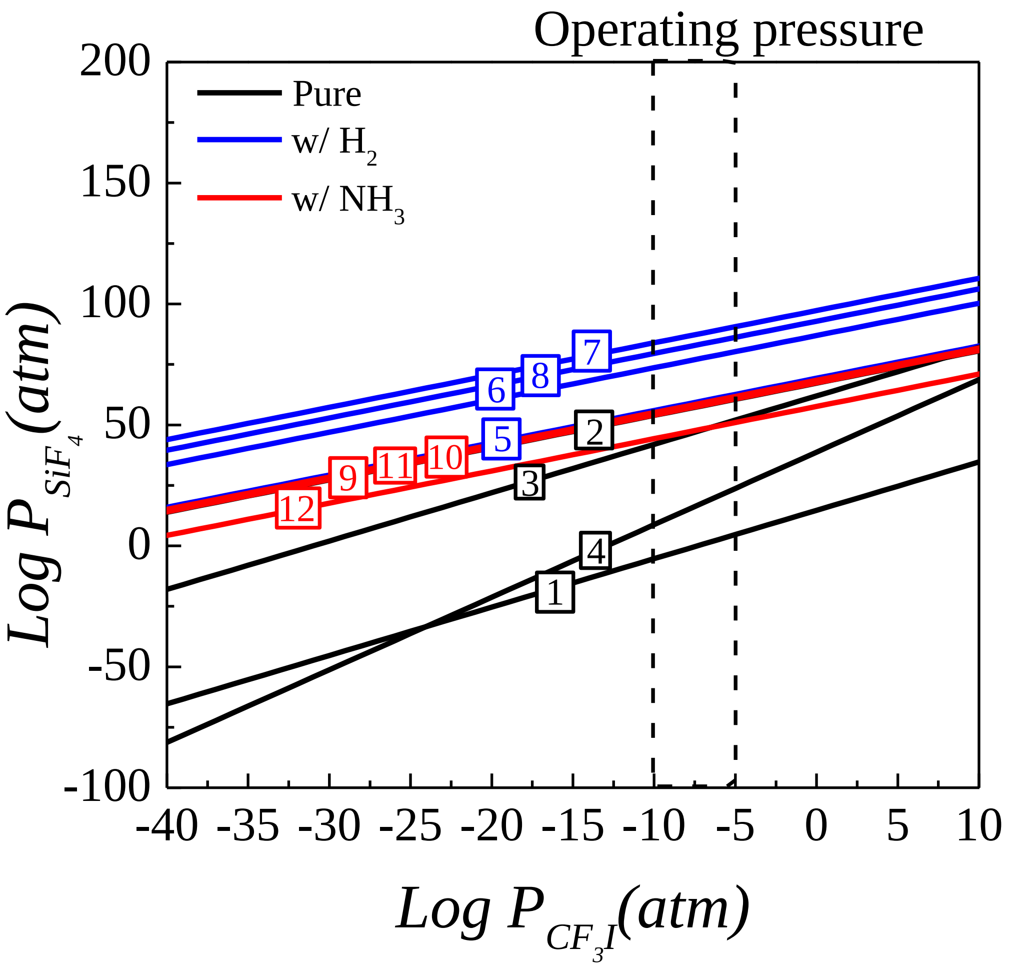
[9] F. Fracassi, R. d'Agostino, J. Vac. Sci. Tech. B 16, 1867 (1998).

Etching with CF_3I

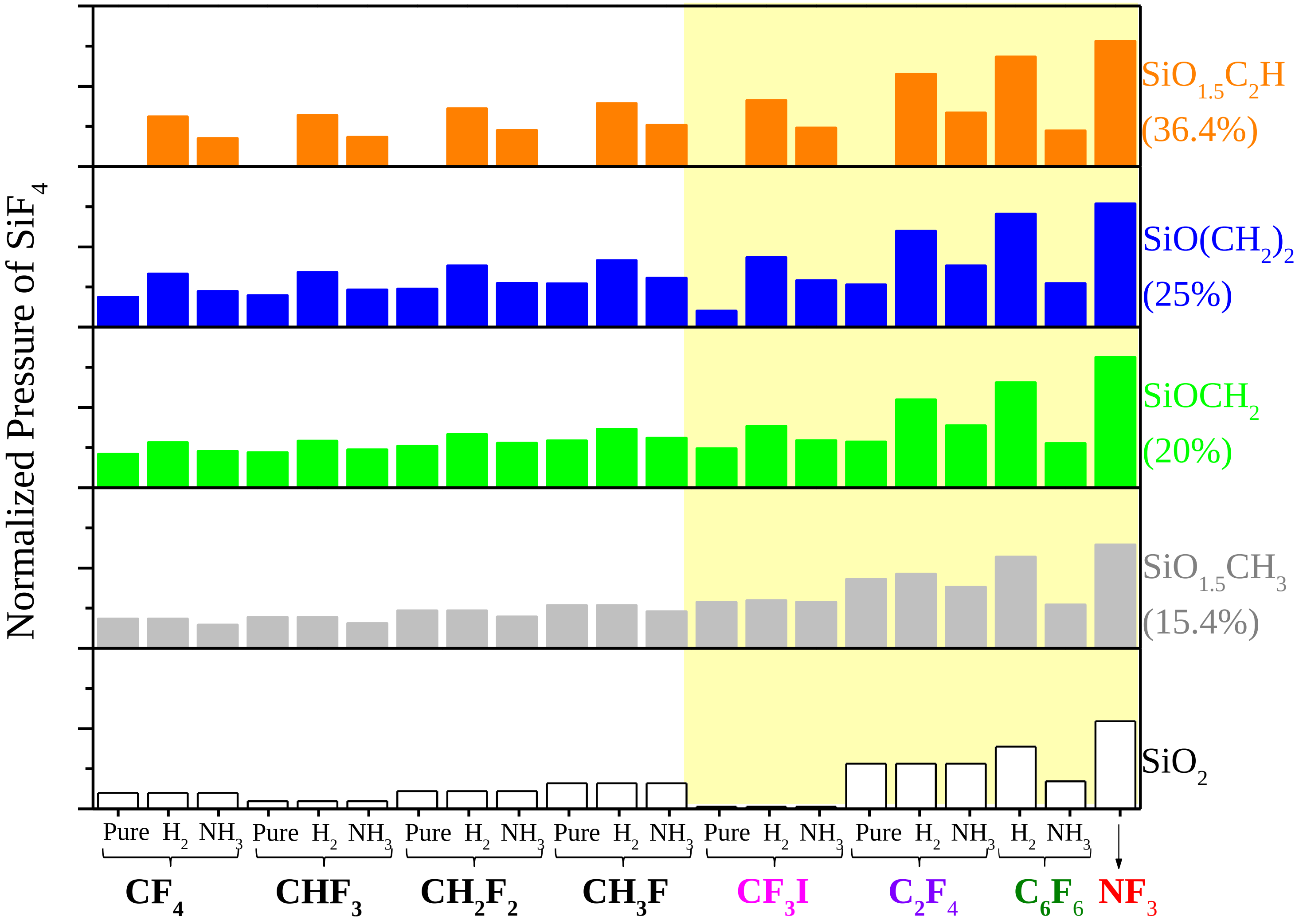


	Reaction	G (eV) [4,5,6]
[1]	$\mathbf{SiO_2}(c) + 2CF_3I(g) \rightarrow SiF_4(g) + 2CO(g) + 2IF(g)$	-0.87
[2]	$6\mathbf{SiO_{1.5}CH_3}(c) + 8CF_3I(g) \rightarrow 6SiF_4(g) + 4I_2(g) + 9CO(g) + C_2H_6(g) + 3CH_4(g)$	-24.02
[3]	$\mathbf{SiOCH_2}(c) + 2CF_3I(g) \rightarrow SiF4(g) + C_2H_2F_2(g) + CO(g) + I_2(g)$	-3.69
[4]	$\mathbf{SiO(CH_2)_2}(c) + 3CF_3I(g) \rightarrow SiF_4(g) + 2C_2H_2F_2(g) + CO(g) + IF(g) + I_2(g)$	-2.31
[5]	$6\mathbf{SiO_{1.5}CH_3}(c) + 8CF_3I(g) + \mathbf{H_2}(g) \rightarrow 6SiF_4(g) + 4I_2(g) + 9CO(g) + 5CH_4(g)$	-24.75
[6]	$3\mathbf{SiOCH_2}(c) + 4CF_3I(g) + 5\mathbf{H_2}(g) \rightarrow 3SiF_4(g) + 3CO(g) + 4CH_4(g) + 2I_2(g)$	-15.53
[7]	$3\mathbf{SiO(CH_2)_2}(c) + 4CF_3I(g) + 8\mathbf{H_2}(g) \rightarrow 3SiF_4(g) + 2I_2(g) + 3CO(g) + 7CH_4(g)$	-17.37
[8]	$6\mathbf{SiO_{1.5}C_2H}(c) + 8CF_3I(g) + 19\mathbf{H_2}(g) \rightarrow 6SiF_4(g) + 4I_2(g) + 9CO(g) + 11CH_4(g)$	-33.17
[9]	$18\mathbf{SiO_{1.5}CH_3}(c) + 24CF_3I(g) + \mathbf{NH_3}(g) \rightarrow 18SiF_4(g) + 12I_2(g) + HCN(g) + 27CO(g) + 14CH_4(g)$	-72.25
[10]	$9\mathbf{SiOCH_2}(c) + 12CF_3I(g) + 5\mathbf{NH_3}(g) \rightarrow 9SiF_4(g) + 9CO(g) + 5HCN(g) + 7CH_4(g) + 6I_2(g)$	-36.66
[11]	$9\mathbf{SiO(CH_2)_2}(c) + 12CF_3I(g) + 8\mathbf{NH_3}(g) \rightarrow 9SiF_4(g) + 6I_2(g) + 9CO(g) + 8HCN(g) + 13CH_4(g)$	-36.23
[12]	$18\mathbf{SiO_{1.5}C_2H}(c) + 24CF_3I(g) + 19\mathbf{NH_3}(g) \rightarrow 18SiF_4(g) + 12I_2(g) + 27CO(g) + 19HCN(g) + 14CH_4(g)$	-61.77

- H_2 addition increases etch product pressure – most effective with 25% C –doped silica
- CF_3I w/ and w/o NH_3 – most effective with 20% C-doped silica

[5] NIST-JANAF Thermochemical (2013)

Comparison of Etch Chemistries



The y-axis represents the normalized partial pressure of SiF_4, one of the primary etch products. The normalization is with respect to the partial pressure of SiF_4 generated in CF_4 etching SiO_2 where all the thermodynamic data are from NIST JANAF Thermodynamic Table, 2013

[4] NIST-JANAF Thermochemical (2013)

Systematic Approach – min{G_{tot}}

Case Study (literature report): $Cr(s) + Cl_2(g) + O_2(g) \rightarrow \boxed{CrCl_2O_2(g),\ CrO_2(s),\ Cr_2O_3(s)\ldots}$

1. Linear constraint of atomic mass conservation

$$\begin{array}{r}Cr\ balance\\ Cl\ balance\\ O\ balance\end{array}\begin{pmatrix} n_{Cr,Cr} & n_{Cr,Cl_2} & n_{Cr,O_2} & n_{Cr,CrCl_2O_2} & n_{Cr,CrO_2} & n_{Cr,Cr_2O_3} \\ n_{Cl,Cr} & n_{Cl,Cl_2} & n_{Cl,O_2} & n_{Cl,CrCl_2O_2} & n_{Cl,CrO_2} & n_{Cl,Cr_2O_3} \\ n_{O,Cr} & n_{O,Cl_2} & n_{O,O_2} & n_{O,CrCl_2O_2} & n_{O,CrO_2} & n_{O,Cr_2O_3} \end{pmatrix}\begin{pmatrix} x_1 \\ x_2 \\ x_3 \end{pmatrix}=\begin{pmatrix} n_{Cr,in} \\ n_{Cl,in} \\ n_{O,in} \end{pmatrix}$$

⇩

2. Feed: 1 mol Cr(s), 130 mol $Cl_2(g)$, 5 mol $O_2(g)$

$$\begin{array}{r}Cr\ balance\\ Cl\ balance\\ O\ balance\end{array}\begin{pmatrix} 1 & 0 & 0 & 1 & 1 & 2 \\ 0 & 2 & 0 & 2 & 0 & 0 \\ 0 & 0 & 2 & 2 & 2 & 3 \end{pmatrix}\begin{pmatrix} x_1 \\ x_2 \\ x_3 \end{pmatrix}=\begin{pmatrix} 1 \\ 260 \\ 10 \end{pmatrix}$$

⇩

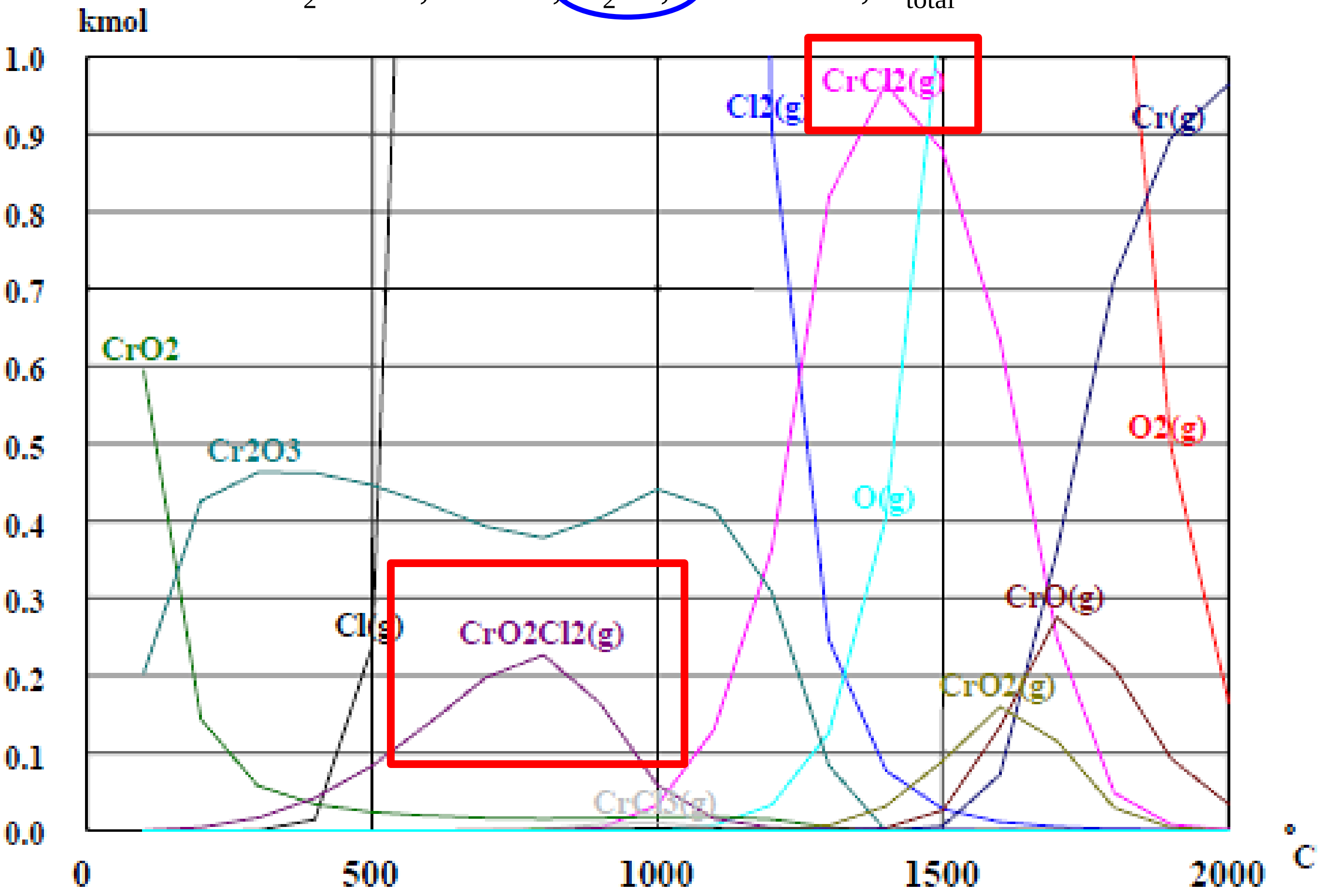
3. Minimize total Gibbs free energy function

$$\min_{n_j}\left\{\frac{G_{tot}}{RT}=\sum_j\frac{n_j\mu_j}{RT}=\sum_j n_j\left(\frac{\Delta G_j^{0}}{RT}+\ln\left[\left(\frac{P}{P_0}\right)\frac{n_j}{\sum_j n_j}\right]\right)\right\}$$

[17] B. Wu, “Thermodynamic study of photomask plasma etching”, *Proc. SPIE* 5567 (2004)

min{G_{tot}} for Cr Etch [17]

Feed: Cl_2 = 130, He=50, O_2=5, Cr=1 kmol; P_{total} = 0.01mb

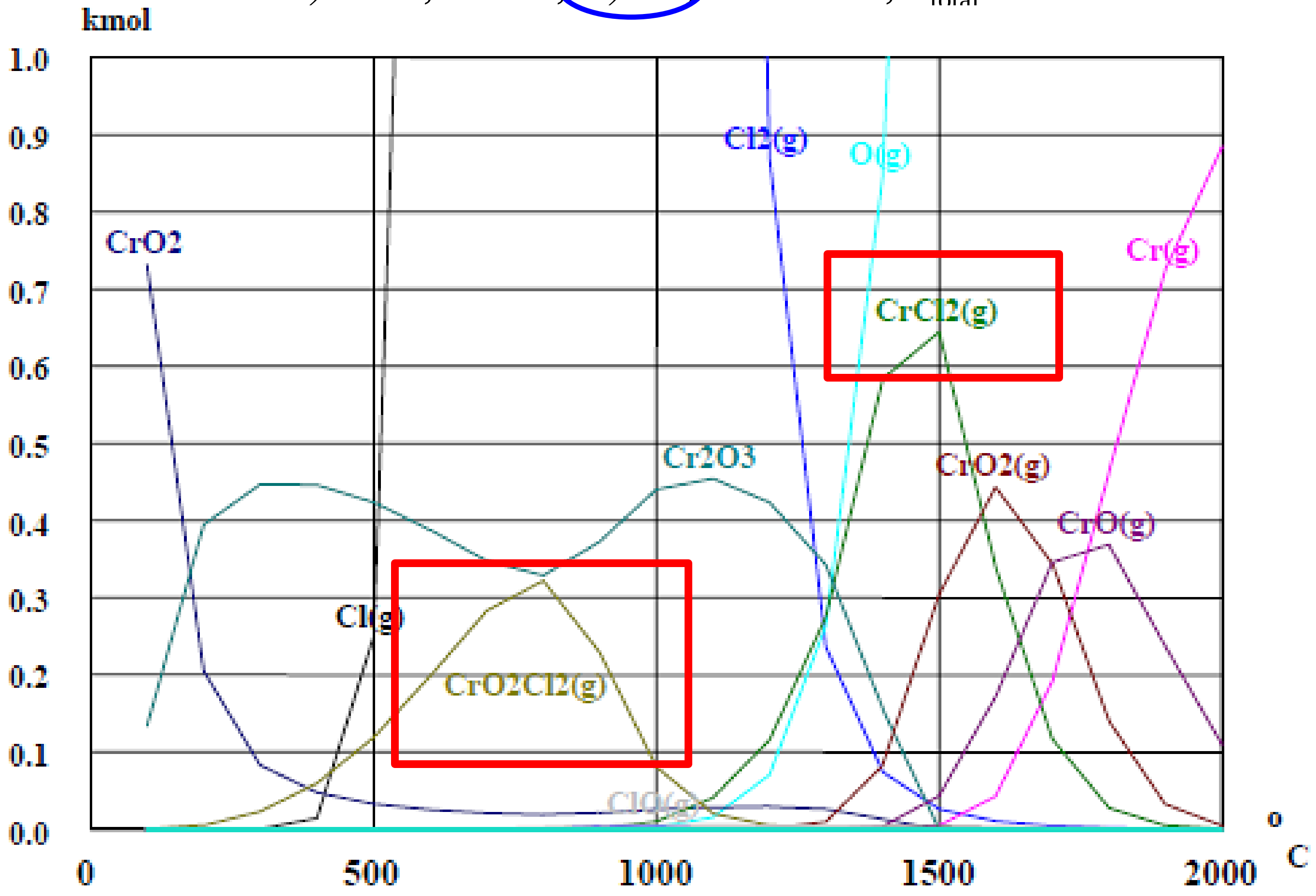


- HSC commercial software calculates equilibrium distribution

[17] B. Wu, "Thermodynamic study of photomask plasma etching", *Proc. SPIE* 5567 (2004)

min$\{G_{tot}\}$ for Cr Etch [17]

Feed: Cl_2 = 130, He=50, O_2=20, Cr=1 kmol; P_{total} = 0.01mb

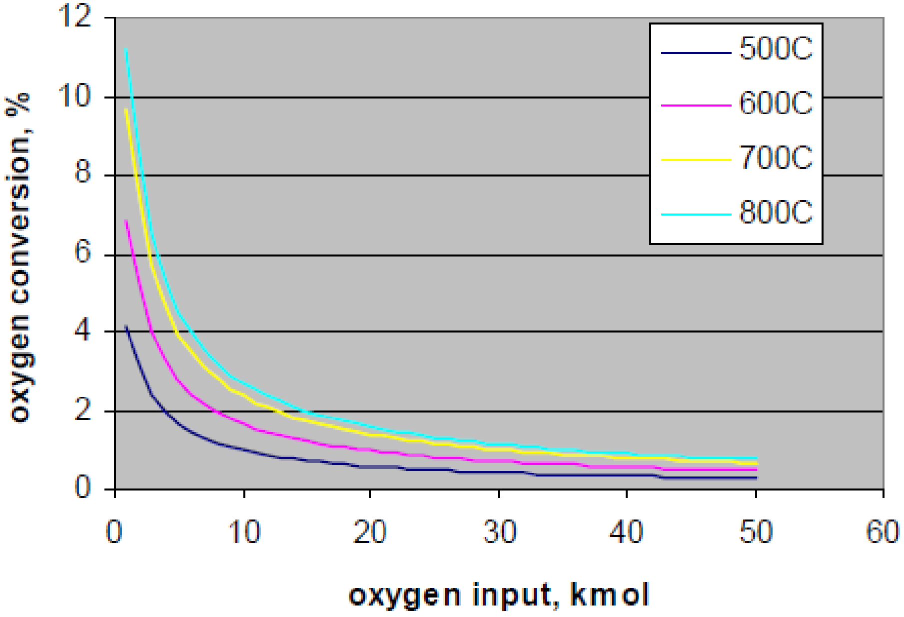


- In general, changing O_2 content causes a shift in equilibrium

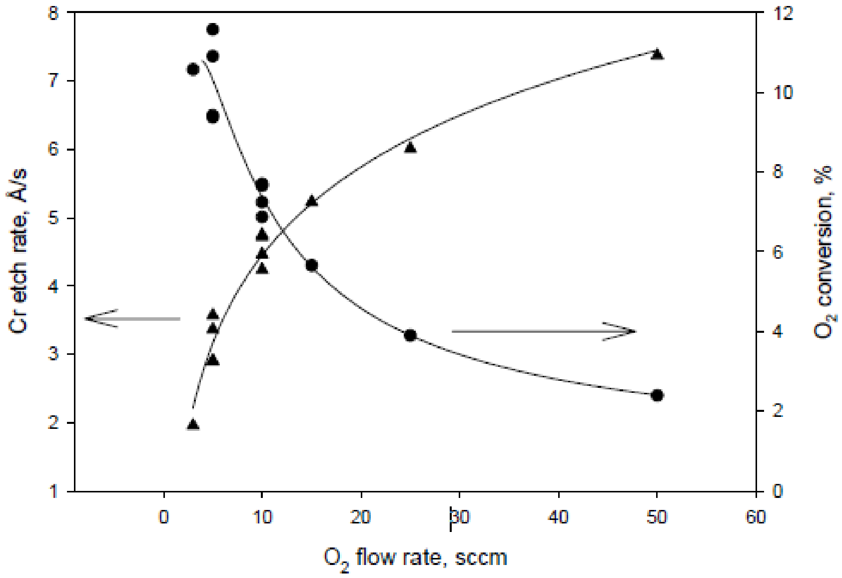
[17] B. Wu, "Thermodynamic study of photomask plasma etching", *Proc. SPIE* 5567 (2004)

min{G_{tot}} for Cr Etch [17,18]

O_2 conversion (generation of $CrCl_2O_2$(g))
vs. O_2 input between 500ºC-800ºC



Comparison of Cr etch rate,
O_2 conversion, and O_2 flow rate



- The authors concluded “oxygen equilibrium conversion was strongly affected by oxygen flow rate and may limit etch rate”

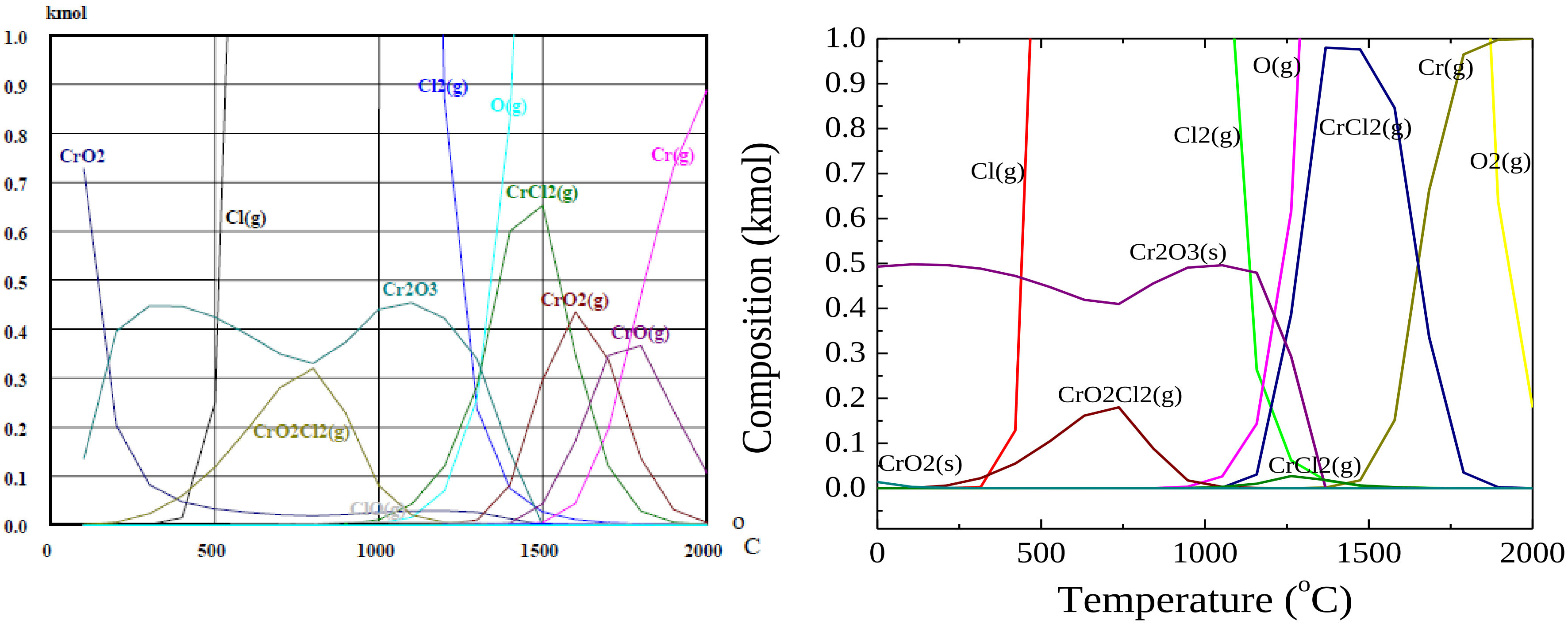
[17] B. Wu, “Thermodynamic study of photomask plasma etching”, Proc. SPIE 5567 (2004)

[18] B. Wu, “An investigation of Cr etch kinetics,” Proc. SPIE 5256 (2003)

HSC Software (Commercial)

Cl_2 = 200, He = 50, O_2 = 20, Cr = 1 kmol

HSC Earlier version [Wu, SPIE] [HSC Current version]



- Since the software is a “black box”, it may be beneficial to independently validate the calculations

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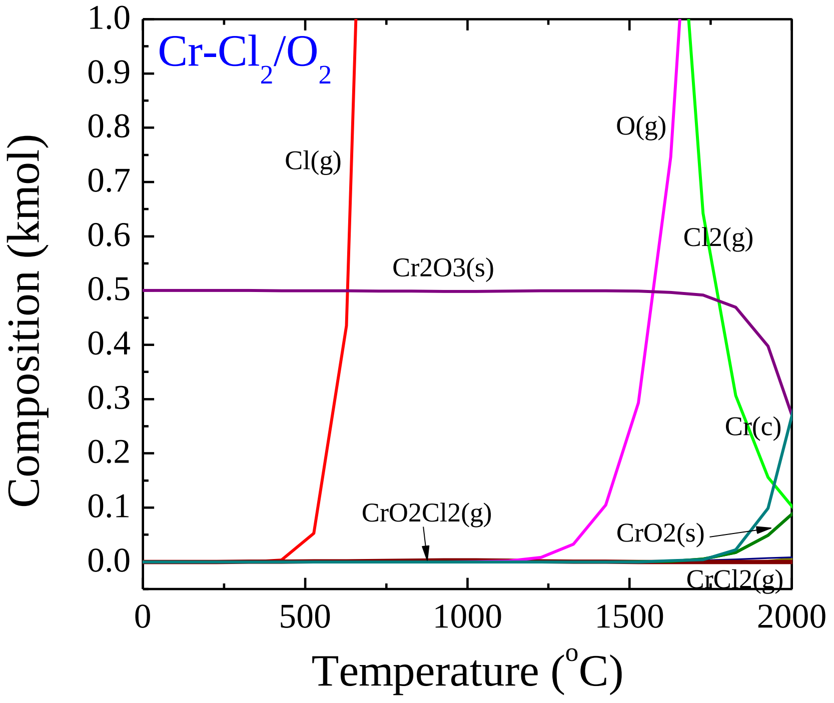
[17] B. Wu, “Thermodynamic study of photomask plasma etching”, *Proc. SPIE* 5567 (2004)

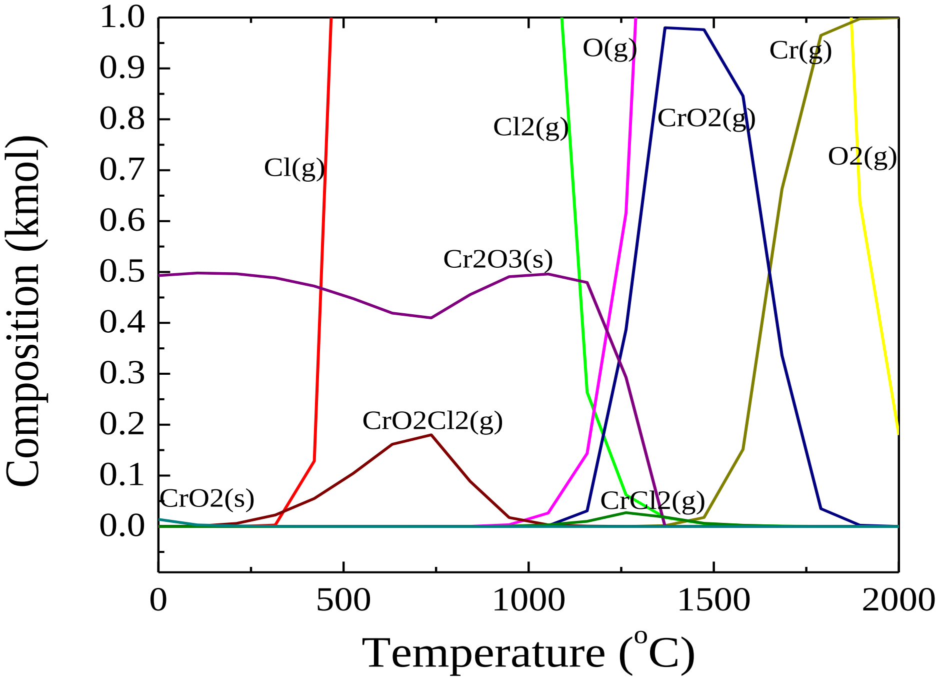
Verification of Calculation

Cr Etch: Cl_2 = 200, He = 50, O_2 = 20, Cr = 1 kmol

Matlab



[HSC Current version]



- The calculation was independently determined for Cr etch in Cl_2/O_2 as well as SiO_2 etching in CF_4

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[17] B. Wu, "Thermodynamic study of photomask plasma etching", *Proc. SPIE* 5567 (2004)

Selection of Chemistry

- Comparison of non-PFC and PFC in C-doped silica etch
- Consider the additives such as H_2 and NH_3 to facilitate the formation of volatile C-containing compounds from highly-doped silica (>15%C)

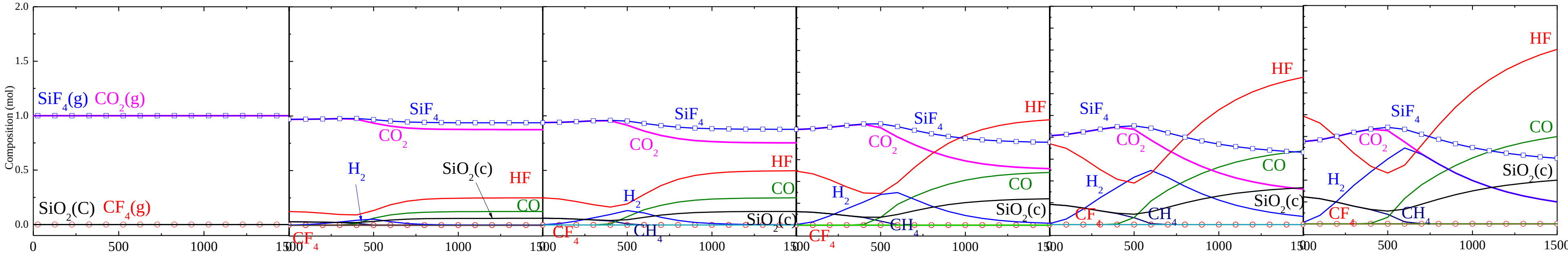
Reaction	ΔG (eV)
SiO_2	
$SiO_2(c) + \mathbf{2CF_4(g)} \rightarrow SiF_4(g) + 2COF_2(g)$	-1.92
$SiO_2(c) + \mathbf{2CF_4(g)} + \mathbf{H_2(g)} \rightarrow SiF_4(g) + 2COF(g) + 2HF(g)$	1.51
$SiO_2(c) + \mathbf{2CF_4(g)} + \mathbf{NH_3(g)} \rightarrow SiF_4(g) + COF_2(g) + HCN(g) + HOF(g) + HF(g)$	3.03
$SiO(CH_2)_2(c)$ (25%C-doped silica)	
$SiO(CH_2)_2(c) + \mathbf{CF_4(g)} \rightarrow SiF_4(g) + CO(g) + C_2H_4(g)$	-2.67
$SiO(CH_2)_2(c) + \mathbf{CF_4(g)} + 2\mathbf{H_2}(g) \rightarrow SiF_4(g) + CO(g) + 2CH_4(g)$	-4.43
$SiO(CH_2)_2(c) + \mathbf{CF_4(g)} + \mathbf{2/3NH_3(g)} \rightarrow SiF_4(g) + CO(g) + 2/3HCN(g) + 4/3CH_4(g)$	-3.10

[5] NIST-JANAF Thermochemical (2013)

SiO_2 Etch, Effect of CF_4 and H_2

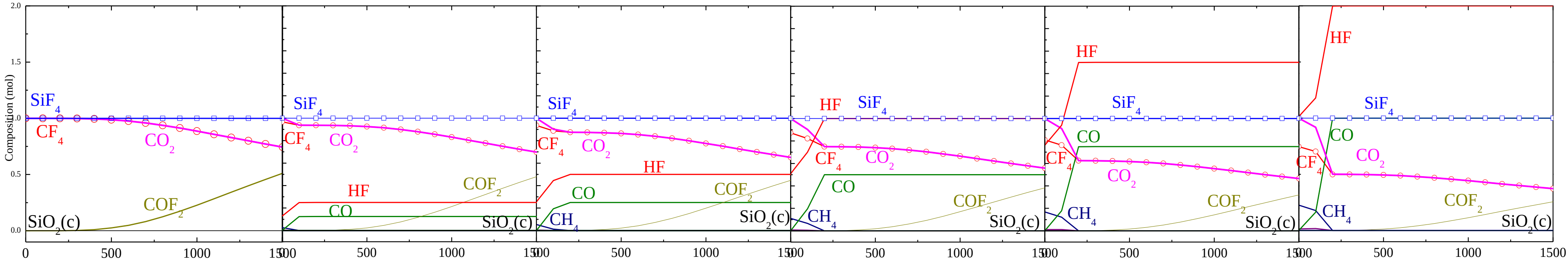
$SiO_2(c) + CF_4(g) + \mathbf{m}\, H_2(g)$

m = 0 **m = 1**



$SiO_2(c) + \mathbf{2}CF_4(g) + \mathbf{n}\, H_2(g)$

n = 0 **n= 1**



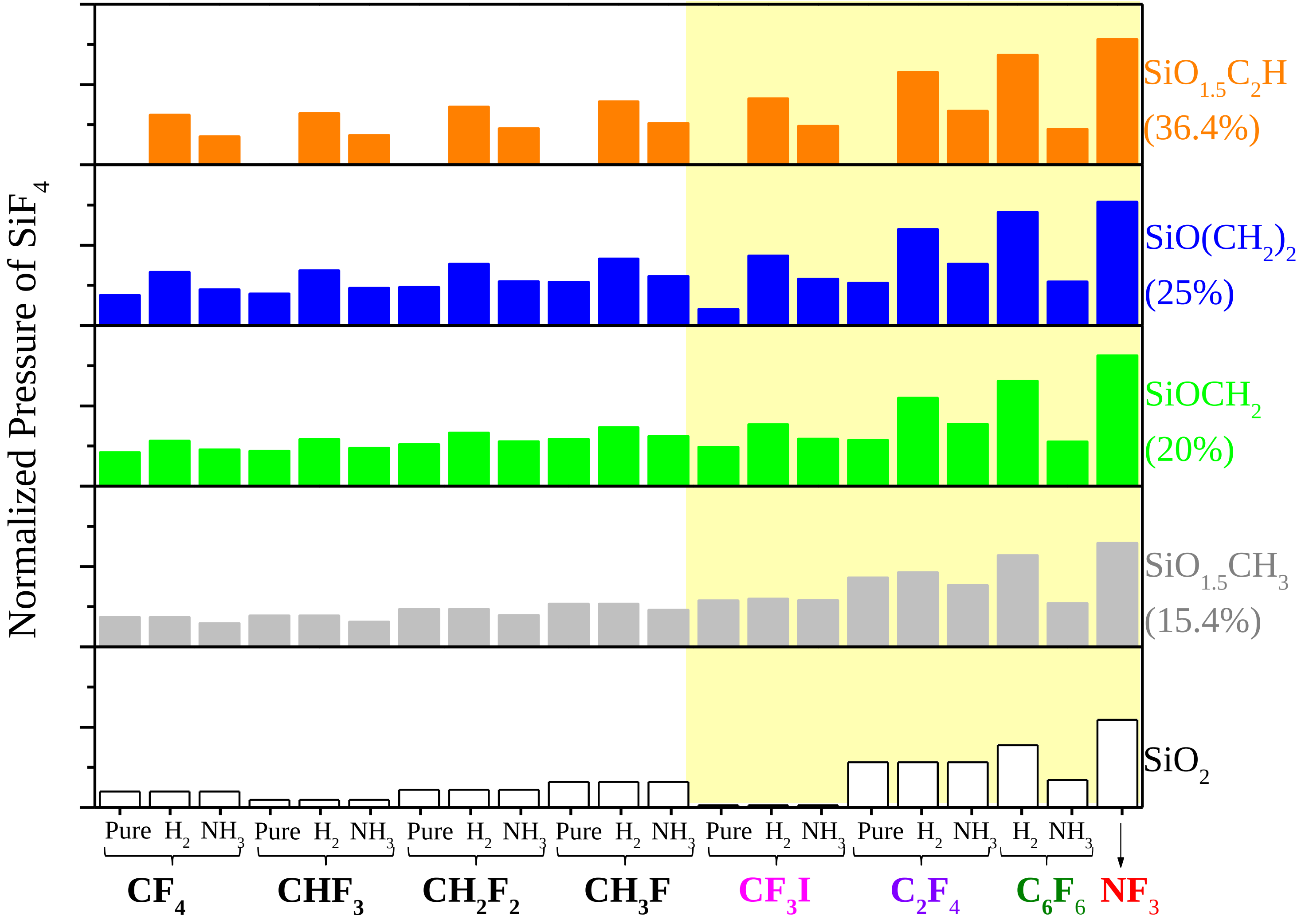
- The additional of hydrogen resulted in the formation of HF

Comparison of reactions

	Reaction	ΔG (eV)
SiO_2		
	$SiO_2(c) + \mathbf{2CF_4(g)} \rightarrow SiF_4(g) + 2COF_2(g)$	-1.92
	$SiO_2(c) + \mathbf{2CF_4(g)} + \mathbf{H_2(g)} \rightarrow SiF_4(g) + 2COF(g) + 2HF(g)$	1.51
	$SiO_2(c) + \mathbf{2CF_4(g)} + \mathbf{NH_3(g)} \rightarrow SiF_4(g) + COF_2(g) + HCN(g) + HOF(g) + HF(g)$	3.03
HSC	$SiO_2(c) + \mathbf{2CF_4(g)} \rightarrow SiF_4(g) + CO_2(g) + CF_4(g)$	-2.31
	$SiO_2(c) + \mathbf{2CF_4(g)} + \mathbf{H_2(g)} \rightarrow SiF_4(g) + CO_2(g) + 0.75CF_4(g) + 0.25CH_4(g) + HF(g)$	-2.98
	$SiO_2(c) + \mathbf{2CF_4(g)} + \mathbf{NH_3(g)} \rightarrow SiF_4(g) + 0.625CF_4(g) + 0.5N_2(g) + 0.375CH_4(g) + 1.5HF(g) + CO_2$	-3.15
$SiO(CH_2)_2(c)$ (25%C-doped silica)		
	$SiO(CH_2)_2(c) + \mathbf{CF_4(g)} \rightarrow SiF_4(g) + CO(g) + C_2H_4(g)$	-2.67
	$SiO(CH_2)_2(c) + \mathbf{CF_4(g)} + \mathbf{2H_2}(g) \rightarrow SiF_4(g) + CO(g) + 2CH_4(g)$	-4.43
	$SiO(CH_2)_2(c) + \mathbf{CF_4(g)} + \mathbf{2/3NH_3(g)} \rightarrow SiF_4(g) + CO(g) + 2/3HCN(g) + 4/3CH_4(g)$	-3.10
HSC	$SiO(CH_2)_2(c) + \mathbf{CF_4(g)} \rightarrow SiO(CH_2)_2(c) + CF_4(g)$ **->no reaction at all**	0.00
	$SiO(CH_2)_2(c) + \mathbf{CF_4(g)} + \mathbf{2H_2}(g) \rightarrow 0.493SiF_4(g) + 0.377CF_4(g) + 0.246CO_2 + 1.36CH_4(g) + 0.507SiO(CH_2)_2(c) + 0.522HF$	-2.97
	$SiO(CH_2)_2(c) + \mathbf{CF_4(g)} + \mathbf{2/3NH_3(g)} \rightarrow 0.255SiF_4(g) + 0.697CH_4(g) + 0.685CF4(g) + 0.745SiO(CH_2)_2(c) + 0.335N_2(g) + 0.241HF(g) + 0.127CO_2(g)$	-1.41

- For SiO_2, HSC show lower ΔG due to incomplete consumption of CF_4
- For $SiO(CH_2)_2$, HSC show higher ΔG and incomplete consumption of CF_4

Comparison of Etch Chemistries



The y-axis represents the normalized partial pressure of SiF$_4$, one of the primary etch products. The normalization is with respect to the partial pressure of SiF$_4$ generated in CF$_4$ etching SiO$_2$ where all the thermodynamic data are from NIST JANAF Thermodynamic Table, 2013

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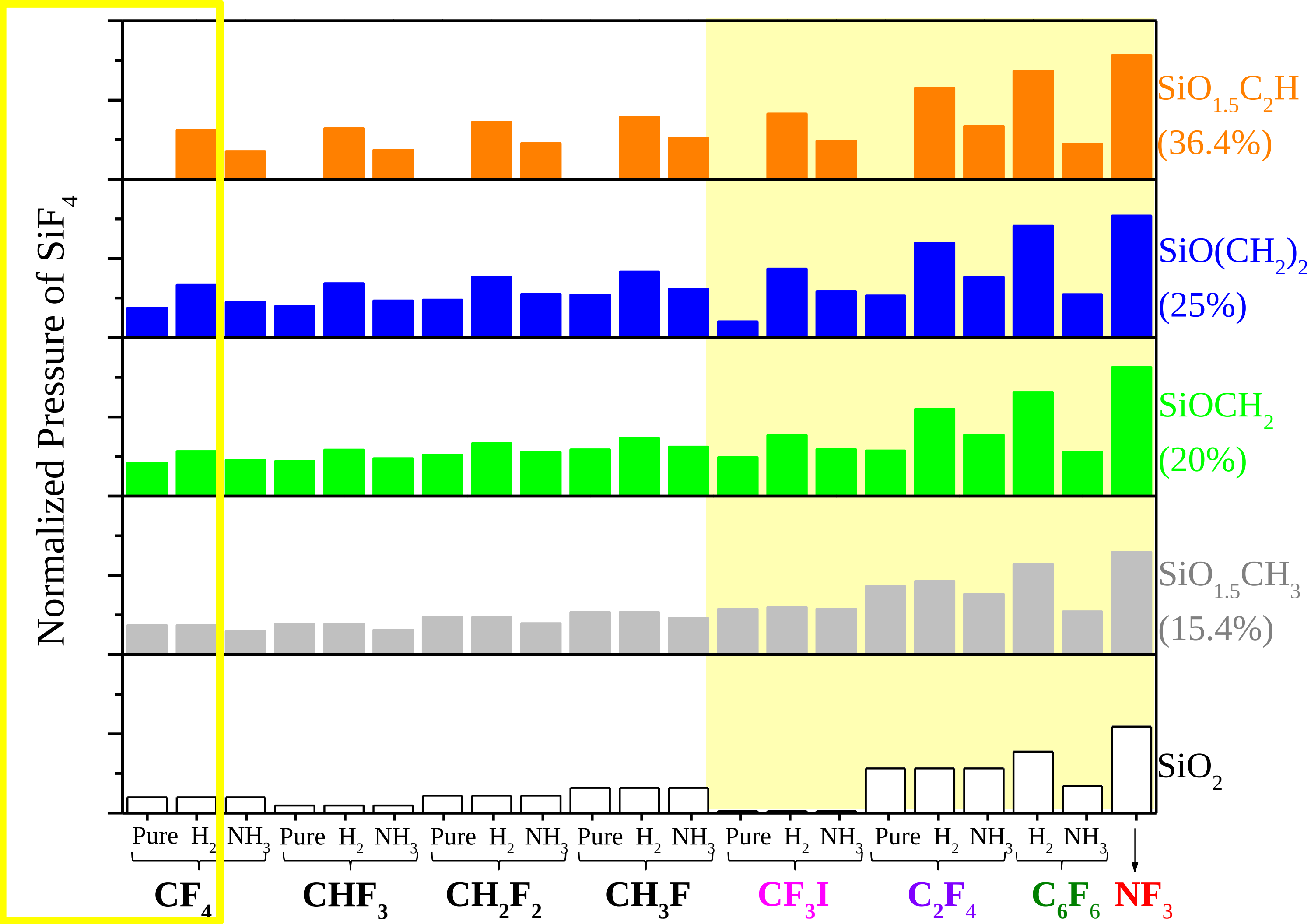
[4] NIST-JANAF Thermochemical (2013)

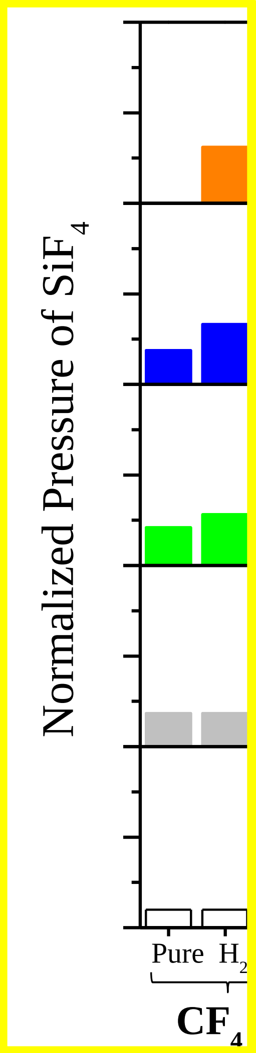
Comparison of Etch Chemistries



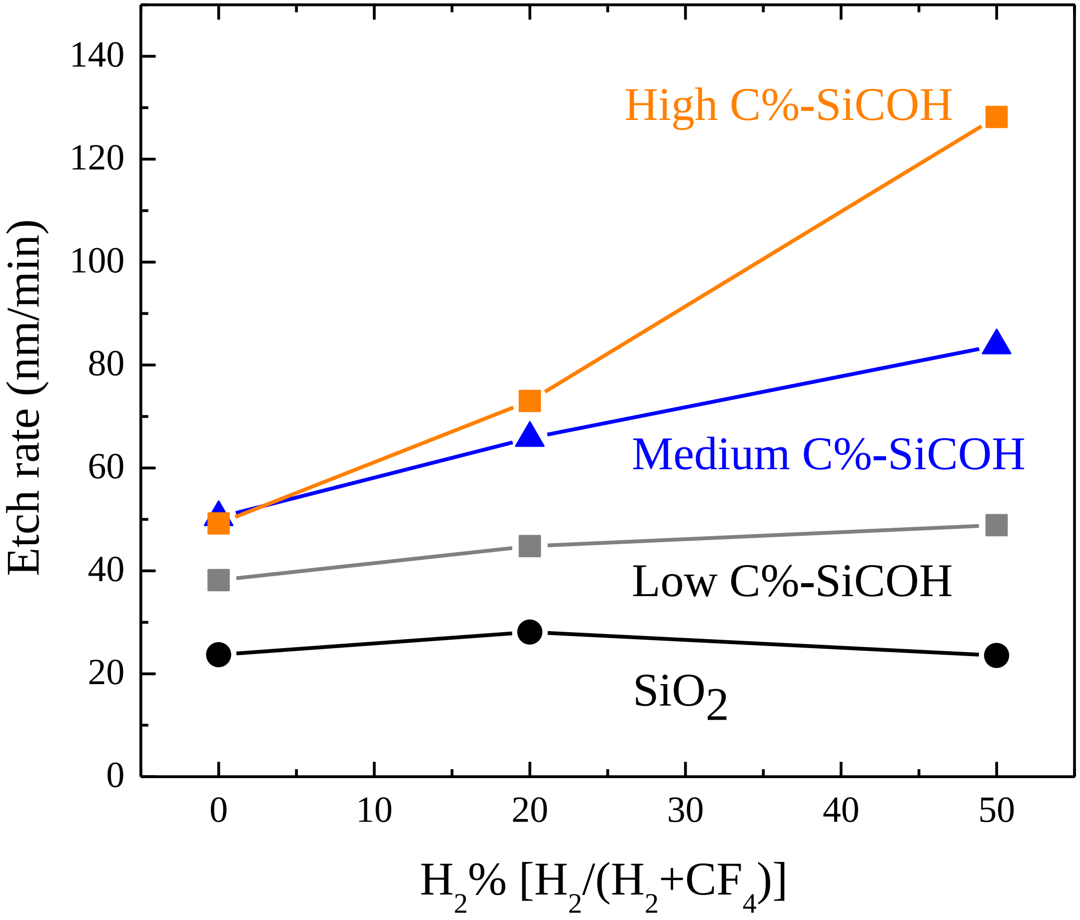
The y-axis represents the normalized partial pressure of SiF_4, one of the primary etch products. The normalization is with respect to the partial pressure of SiF_4 generated in CF_4 etching SiO_2 where all the thermodynamic data are from NIST JANAF Thermodynamic Table, 2013

[4] NIST-JANAF Thermochemical (2013)

Comparison of Etch Chemistries



Plasma power = 100W, P = 30mtorr, Bias = 20 W, CF_4 flow rate = 20 sccm



- Preliminary experimental results agree with theoretical predictions

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[4] NIST-JANAF Thermochemical (2013)

Summary

- Volatility diagrams can be used to assess general trends in potential etchant chemistries
- Thermodynamic properties of carbon doped silicon model compounds were evaluated via established bond and group additivity methods
- Addition of hydrogen via H_2 in general increases the pressure of the primary etch product
- Non-PFC alternative chemistries C_2F_4, C_6F_6, NF_3, and CF_3I are shown to be more effective in producing SiF_4 from each of the carbon doped species than fluoromethanes (e.g. CF_4, CHF_3, etc)
- With the exception of NF_3, each of the etchants examined have relatively low global warming potentials (GWPs); however, NF_3 can be effectively abated through thermal processes
- CF_3I is not as effective as NF_3, yet still viable, due to less environmental impact
- Preliminary experimental results agree with the theoretical prediction

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Industrial Interactions and Technology Transfer

- Conference call with Intel, January 10, 2013, (Satyarth Suri)
- Conference call with Intel, February 21, 2013, (Satyarth Suri)
- Visited Intel, Portland, OR, April, 3, 2013, (Bob Turkot, Satyarth Suri)
- Conference call with SRC, April 24, 2013 (Bob Haveman)
- Conference call with Intel, May 16, 2013, (Satyarth Suri)
- Conference call with Intel, June 13, 2013, (Satyarth Suri)
- Conference call with Intel, July 18, 2013, (Satyarth Suri)
- Conference call with Intel, August 29, 2013, (Satyarth Suri)
- Conference call with Intel, October 10, 2013, (Satyarth Suri)
- Conference call with Intel, November 14, 2013, (Satyarth Suri)
- Conference call with Intel, February 13, 2014, (Satyarth Suri)
- Conference call with Intel, March 20, 2014, (Satyarth Suri)

Future Plans

Next Year Plans

- Perform thermodynamic calculations to assess potential impact and projected effectiveness
- Investigate additional iodofluorocarbon etchants as well as other alternatives to PFCs
- Obtain carbon doped silica samples from Intel for experimental validation

Long-Term Plans

- Formulate the models to predict etch product from plasma processes
- Suggest viable plasma chemistries
- Experimental validation and assessment of EHS impact

Publications, Presentations, and Recognitions/Awards

Presentation:

- Contributed talk at AVS International Symposium, October 2013
  (J. K. Chen and J. P. Chang, "Selection of non-PFC Chemistries for Through-Silicon via Etch")
- Contributed talk at AIChE Annual Meeting, October 2013
  (J. K. Chen and J. P. Chang, "Selection of non-PFC Chemistries for Through-Silicon via Etch") (T. Kim, J. K. Chen and J. P. Chang, "Thermodynamic Approach to Select Viable Etch Chemistry for Magnetic Metals")
- SRC ERC EHS TeleSeminar, January 9, 2014

Publication:

- Deliverable Report, P066013, "Non-PFC Plasma Chemistries for Patterning Complex Materials and Structures", January 2013
- Deliverable Report, P065582, "Non-PFC Plasma Chemistries for Patterning Complex Materials/Structures", January 2014
- Pre-print for Review, "Thermodynamic Assessment and Experimental Verification of Reactive Ion Etching of Magnetic Metal Elements," Feb 2014