<u>Non-PFC Plasma Chemistries for</u> <u>Patterning Low-k Dielectric Materials</u>

(Task Number: 425.038)

PIs:

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Graduate Students:

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- Screen candidate etch chemistries through the use of thermodynamic analysis, including Gibbs free energy minimization
- Identify non-PFC gases that can be used (due to facility limitations, alternative locations are being explored)
- Reduce amount of PFC etchants through additive gases such as H₂
- Assess the feasibility of non-PFC chemistries in patterning low-k dielectric thin films

Composition of Low-k Dielectrics^[1]



- Previously explored trend of fluorine, carbon, and pore incorporation into low-k dielectrics
- Extension of porosity (k = 1) into the film, thereby realizing a lower dielectric constant

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

US EPA's PFC emission model* shows average PFC emissions from semiconductor manufacturing for the evolution of complex devices^[3]



• 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

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US EPA's PFC emission model* shows average PFC emissions from semiconductor manufacturing for the evolution of complex devices^[3]



• 1996—Engineering Research Center (ERC) established with goal of combating trend of increased PFC usage

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US EPA's PFC emission model* shows average PFC emissions from semiconductor manufacturing for the evolution of complex devices^[3]



• 2001—EU and EPA release reports analyzing pathways to reduce overall PFC emissions in Europe and United States, introducing stricter environmental policies

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US EPA's PFC emission model* shows average PFC emissions from semiconductor manufacturing for the evolution of complex devices^[3]



• 2011—Task 425.038 introduced to investigate the potential reduction of PFC usage

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Global Warming Potential

Chemistries	Atmospheric conc. in 2015 (ppt)	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	403x10 ⁶	379x10 ⁶	358x10 ⁶ *	-	-	variable	1	[12]
CH_4	7.22x10 ⁵	7x10 ⁵	1.7x10 ⁵ *	-	-	12.2	21	[12]
N ₂ O	326x10 ⁵	275x10 ³	311x10 ³ *	-	-	120	310	[12]
$CHClF_2$	-	-	105x10 ³ *	-	-	12.1	1400	[12]
CF ₄	76	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	21	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]
C_6F_6	-	-	-	-	-		<1	[16]
CF ₃ I	-	-	-	-	-	2 days	1	[10]

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

Target of Carbon-doped SiO₂ Etch

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

Intel specified				
nents	Range(%)			
	20%			
	40%			
	15-40%			
sity	20-25%			
kness	100nm			
ocus on:				
Trench etch (later via)				
Selectivity to PR				
Sidewall damage				
	sity kness son: Trench etch (Selectivity to Sidewall dan			

		Carbon	C	Comp	ositio	n	
	Target	doping level	Si (%)	0 (%)	C (%)	H (%)	Unit
	1	Low	15.4	23.1	15.4	46.1	SiO _{1.5} CH ₃
	2	\uparrow	20	20	20	40	SiOCH ₂
7	3	\downarrow	12.5	12.5	25	50	SiO(CH ₂) ₂
	4	High	18.2	27.2	36.4	18.2	SiO _{1.5} C ₂ H

• SEM of C-doped SiO₂ pre-metal dielectric layer^[4]



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[4] S. Rimal, et al., Evaluation of Plasma Damage to Low-k dielectric Trench Structures, ECS Solid State Letters, 3, N1-4 (2014)

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

Data for C-doped Silica is Limited

C-doped silica		$\Delta_{\mathbf{f}} \mathbf{H}$ (kJ/mol)	$\Delta_{\mathbf{f}} \mathbf{S}$ (J/mol)	$\Delta_{\mathbf{f}}\mathbf{G}$ (kJ/mol)
SiO ₂ ^[4]		-910.87	-182.53	-856.11
SiO _{1.5} CH ₃ ^[4,5] (15.4%)	0 H ⁰ H ⁰ H ⁰ O			
SiOCH ₂ ^[4,5] (20%)	$\begin{array}{c} O \\ H_2C \\ O \\ Si \\ CH_2 \\ O \\ Si \\ CH_2 \\ O \\ $	No dat	ta is av	ailable
SiO(CH ₂) ₂ ^[4,5] (25%)	0,0 H ₂ C,Si,CH ₂ CH ₂			
SiO _{1.5} C ₂ H ^[4,5,6] (36.4%)				

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[5] NIST-JANAF Thermochemical (2013) [6] S. W. Benson, ACS Symposium series 677, (1988)

Data for C-doped Silica is Needed

C-doped silica		$\Delta_{\mathbf{f}}\mathbf{H}$ (kJ/mol)	$\Delta_{\mathbf{f}} \mathbf{S}$ (J/mol)	∆ _f G (kJ/mol)		Group Bond
	0					SiO ₂ [6]
SiO ₂ ^[4]	Si	-910 87	-182 53	-856 11		CH ₄ ^[6]
	0,000	-710.07	-102.55	-050.11		Si-C
CIT [4 2]	H ₃ C					Si-O
$SIO_{1.5}CH_3^{[+,5]}$ (15.4%)	0- <u>si</u> 0					C-H
(13.170)	0////0					Total
SiOCH ₂ ^[4,5] (20%)	0 0 H_2C CH_2 H_2C CH_2 O Si CH_2 Si O Si Si O	No da		ailabla		Δ
		NU Ua	a 15 av	allable		Z
SiO(CH ₂) ₂ ^[4,5] (25%)	0,0 H ₂ C,SiCH ₂ CH ₂				Δ_{j}	Δ $f S_{C-SiO}$
SiO _{1.5} C ₂ H ^[4,5,6] (36.4%)					Δ_j	$_{c}G_{C-SiO}$ The <u>met</u>

Gro Bo	oup / ond	No. in SiO _{1.5} CH ₃	Enth (kJ/	Enthalpy ^[5] (kJ/mol)		py ^[5] l*K)
SiC	$D_2^{[6]}$	3/4	-91().866	-	
CH	$H_4^{[6]}$	3/4	-50	.618	-	
Si	-C	1	-2	-25.1) 1
Si	-0	3		-		9
С	-H	3		-		€7
Тс	otal	746.2		46.2	204.	25
	T=	=300K	SiO ₂ ^[4]	SiO	1.5CH3	
	$\Delta_{\rm f} {\rm H}$	(kJ/mol) -	910.866		-746.2	
	$\Delta_{\rm f} S$	(J/mol)	-182.53	-	324.77	
	$\Delta_{\rm f} G$	(kJ/mol) -	856.106		-648.8	

$$\Delta_f S_{C-SiO_2} = S_{C-SiO_2}^{\circ} - (nS_{Si}^{\circ} + xS_{O_2}^{\circ} + yS_C^{\circ} + zS_{H_2}^{\circ})$$

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

The <u>bond additivity and group additivity</u> <u>methods^[6] is used to determine the energy of</u> formation for C-doped silica

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[5] NIST-JANAF Thermochemical (2013) [6] S. W. Benson, ACS Symposium series 677, (1988)

Data for C-doped Silica is Needed

	∆ _f H (kJ/mol)	Δ _f S (J/mol)	∆ _f G (kJ/mol)		Group / Bond	No. in SiO _{1.5} CH ₃	Enthalpy ^[5] (kJ/mol)	Entropy ^[5] (J/mol*K)
0					SiO ₂ ^[6]	3/4	-910.866	-
	010.97	192.52	956 11		CH ₄ ^[6]	3/4	-50.618	-
	-910.07	-162.33	-050.11		Si-C	1	-25.1	57.91
H₂C					Si-O	3	-	-5.19
0 0	746.20	324 77	648.80		С-Н	3	-	53.97
ō.,,,,,,	-740.20	-324.77	-048.80		Total	-	-746.2	204.25
0 0 Si					T	=300K S	iO ₂ ^[4] SiO	1.5CH3
H ₂ C CH ₂	-517.40	-44.88	-503.90		$\Delta_{\rm f} { m H}$	(kJ/mol) -91	10.866	-746.2
O - Si Si - O $/ C / H_2 O$					$\Delta_{\rm f}$	S(J/mol) -1	182.53 -	-324.77
0					$\Delta_{\rm f} { m G}$	(kJ/mol) -85	56.106	-648.8
	-538.00	-141.84	-495 50					
- H ₂ C CH ₂	-330.00	-1-1.0-		Δ_f	S_{C-SiO_2} =	$=S_{C-SiO_2}^{\circ}-0$	$(nS_{Si}^{\circ} + xS_{O}^{\circ})$	$y_2 + yS_C^\circ + zS_{H_2}^\circ$
0, 0 Si 0				Δ_f	G_{C-SiO_2}	$=\Delta_f H_{C-SiC}$	$D_2 - T \times \Delta_f S$	S_{C-SiO_2}
	-662.70	-328.86	-564.10					
				•	The <u>k</u>	ond additiv	ity and grou	i <u>p additivity</u>
0 0		l		I	<u>meth</u>	ous ^{res} is used	to determine	ie the energy of
	$\begin{array}{c} & & & \\$	$\begin{array}{ c c } & & & & & & & \\ & & & & & \\ \hline & & & & &$	$\begin{array}{c c} \Delta_{\mathbf{f}}\mathbf{H} & \Delta_{\mathbf{f}}\mathbf{S} \\ (\mathbf{J}/\mathbf{mol}) & (\mathbf{J}/\mathbf{mol}) \\ \hline & & & & \\ \hline \hline \end{array} \end{array} \end{array} $	$\Delta_{r}H$ (kJ/mol) $\Delta_{r}S$ (J/mol) $\Delta_{r}G$ (kJ/mol) $\widehat{\Box}$ -910.87-182.53-856.11 $\widehat{\Box}$ -910.87-182.53-856.11 $\widehat{\Box}$ -746.20-324.77-648.80 $\widehat{\Box}$ -517.40-44.88-503.90 $\widehat{\Box}$ -517.40-44.88-503.90 $\widehat{\Box}$ -538.00-141.84-495.50 $\widehat{\Box}$ $\widehat{\Box}$ -662.70-328.86-564.10	$\begin{array}{ c c c c c } & \Delta_{r}H & \Delta_{r}S & \Delta_{r}G & \Delta_$	$\begin{array}{c c c c c c c } & & & & & & & & & & & & & & & & & & &$	$\begin{array}{c c c c c c c c c } & \Delta_{\mathbf{r}}\mathbf{H} & \Delta_{\mathbf{r}}\mathbf{S} & \Delta_{\mathbf{r}}\mathbf{G} & \mathbf{S} \\ \hline \mathbf{K}\mathbf{J/mol} & \mathbf{K}\mathbf{J/mol} & \mathbf{K}\mathbf{J}\mathbf{M}\mathbf{M}\mathbf{M} & \mathbf{K}\mathbf{J}\mathbf{M}\mathbf{M}\mathbf{M} \\ \hline \mathbf{K}\mathbf{K}\mathbf{J/mol} & \mathbf{K}\mathbf{K}\mathbf{J}\mathbf{M}\mathbf{M}\mathbf{M} & \mathbf{K}\mathbf{K}\mathbf{J}\mathbf{M}\mathbf{M}\mathbf{M} \\ \hline \mathbf{K}\mathbf{K}\mathbf{J}\mathbf{M}\mathbf{M}\mathbf{M} & \mathbf{K}\mathbf{K}\mathbf{J}\mathbf{M}\mathbf{M}\mathbf{M} & \mathbf{K}\mathbf{K}\mathbf{J}\mathbf{M}\mathbf{M} \\ \hline \mathbf{K}\mathbf{K}\mathbf{J}\mathbf{M}\mathbf{M} & \mathbf{K}\mathbf{K}\mathbf{J}\mathbf{M}\mathbf{M} & \mathbf{K}\mathbf{K}\mathbf{J}\mathbf{M}\mathbf{M} \\ \hline \mathbf{K}\mathbf{K}\mathbf{J}\mathbf{K}\mathbf{M}\mathbf{M} & \mathbf{K}\mathbf{K}\mathbf{J}\mathbf{K}\mathbf{M}\mathbf{M} & \mathbf{K}\mathbf{K}\mathbf{K}\mathbf{M}\mathbf{M} \\ \hline \mathbf{K}\mathbf{K}\mathbf{K}\mathbf{K}\mathbf{K}\mathbf{K}\mathbf{K}\mathbf{K}\mathbf{K}\mathbf{K}$	$\begin{array}{c c c c c c c c } & \Delta_{\rm f} {\rm H} & \Delta_{\rm f} {\rm S} & \Delta_{\rm f} {\rm G} & {\rm SiO}_1 {\rm G} & {\rm SiO}_1 {\rm G} {\rm SiO}_1 {\rm G} {\rm G} & {\rm SiO}_1 {\rm G} {\rm G} {\rm G} & {\rm Gi} {\rm $

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[5] NIST-JANAF Thermochemical (2013) [6] S. W. Benson, ACS Symposium series 677, (1988)

Selection of Chemistry

	Reaction	ΔG (eV)
SiO ₂		
	$\operatorname{SiO}_2(c) + \mathbf{2CF_4}(g) \rightarrow \operatorname{SiF}_4(g) + 2\operatorname{COF}_2(g)$	-1.92
	$\operatorname{SiO}_2(c) + 2\operatorname{CF}_4(g) + \operatorname{H}_2(g) \rightarrow \operatorname{SiF}_4(g) + 2\operatorname{COF}(g) + 2\operatorname{HF}(g)$	1.51
SiO(CH ₂) ₂ (c) (Medium-doped silica)	
	$\operatorname{SiO}(\operatorname{CH}_2)_2(c) + \operatorname{CF}_4(g) \rightarrow \operatorname{SiF}_4(g) + \operatorname{CO}(g) + \operatorname{C}_2\operatorname{H}_4(g)$	-2.67
	$\operatorname{SiO}(\operatorname{CH}_2)_2(c) + \operatorname{CF}_4(g) + 2\operatorname{H}_2(g) \rightarrow \operatorname{SiF}_4(g) + \operatorname{CO}(g) + 2\operatorname{CH}_4(g)$	-4.43

- Comparison of non-PFC and PFC in C-doped silica etch
- Consider additives such as H₂ to facilitate the formation of volatile Ccontaining compounds from highly-doped silica (>15%C)

Gibbs Free Energy Minimization

•T=1000K, P=1atm, input: H₂O 4mole, CH₄ 1mole

No.	Component	Gibbs Energy kcal/gm-mol	Feed gm-mol	Effluent Initial Estimate
1	CH ₄	4.61		0.001
2	C ₂ H ₄	28.249		0.001
3	C ₂ H ₂	40.604		0.001
4	CO ₂	-94.61		0.993
5	CO	-47.942		1
6	O2	0		0.0001 ^a
7	H ₂	0		5.992
8	H ₂ O	-46.03	4	1
9	C ₂ H ₆	26. 1 3	1	0.001

- R, T, P, and P_o are known constants
- G_j^o is provided by the NIST-JANAF and HSC thermochemical tables
- Set G_{tot} as objective function to be minimized
- Compute finite set of n_j to reach global minimum of G_{tot}

Two criteria need to be achieved:

1: Elemental balance

Oxygen Balance	$g_1 = 2n_4 + n_5 + 2n_6 + n_7 - 4 = 0$
Hydrogen Balance	$g_2 = 4n_1 + 4n_2 + 2n_3 + 2n_7 + 2n_8 + 6n_9 - 14 = 0$
Carbon Balance	$g_3 = n_1 + 2n_2 + 2n_3 + n_4 + n_5 + 2n_9 - 2 = 0$

2: Minimization of Gibbs free energy



Gibbs Free Energy Minimization Approach

Gibbs free energy minimization $Cr(s) + Cl_2(g) + O_2(g) \rightarrow CrCl_2O_2(g), CrO_2(s), Cr_2O_3(s)...$

$$G_{tol} = \sum_{j} n_{j} \mu_{j} = \sum_{j} n_{j} \left(\Delta G_{j}^{0} + RT \ln \left[\left(\frac{P}{P_{0}} \right) \frac{n_{j}}{\sum_{j} n_{j}} \right] \right)$$
1. Linear constraint of atomic mass constraint of atomic mass conservation
Cr balance
Cr balac
Cr balance
Cr balanc

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Commercially Available Software

• Feed: $Cl_2 = 200$, He = 50, $O_2 = 20$, Cr = 1 kmol



• Refinements were made to HSC program between 2004 and 2013; however, no details were given about exact changes

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Validating the MATLAB Code



 Through incorporation of gas and condensed phase data, MATLAB code was able to reproduce HSC results

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Validating the MATLAB Code



 The most favorable reaction pathway for SiO₂ with CF₄ is to form SiF₄ and CO₂

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• SiO₂ and CF₄ was taken as a baseline condition to examine the change in product distribution

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• Hydrogen can first be incorporated by switching to a partially hydrogenated etchant (CHF₃), causing the product distribution to become significantly more complex

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• Molecular hydrogen can also be added to allow for independent control of the F/H ratio beyond the fixed composition of the etchant molecule

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Comparison of Etchant Chemistries



The y-axis represents the normalized partial pressure of SiF4, one of the primary products. The normalization is with respect to the partial pressure of SiF4 generated in CF4 etching SiO2 where all the thermodynamics data are from NIST JANAF Thermodynamics Table, 2013

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[5] NIST-JANAF Thermochemical (2013)

Process Chemistry Availability



Available gases:

- H_2
- **O**₂ Ar
- Cl₂ SF₆
- **CF**₄

STS Advanced Oxide ICP Etcher



Available gases:

- H_2
- O_2
- Ar
- SF_6
- CF₄
 - CHF₃
- C_4F_8

- CF₄ and H₂ were the only fluorocarbon chemistries available for previous studies using Ulvac NE-550
- Transition to STS Oxide etcher allowed for comparison of CF₄ and CHF₃ chemistries with additive H₂



• Previous results show agreement between etch rate studies and thermodynamic analysis, H₂ addition causes increase in etch rate

Bias Power Dependence

Plasma power = 1400W, P = 6mtorr, CF_4 flow rate = 20 sccm

Blanket C-SiO₂ thick films



• Bias power also have significant effect, particularly on medium C% SiO₂ films (in addition to studying the effects of hydrogen chemistry)

Bias Power Dependence



films was performed using x-ray photoelectron spectroscopy (XPS)



• Plasma conditions were changed, and experiments performed in a separate etcher that could accommodate both CF₄ and CHF₃ with H₂



• Both blank and patterned carbon doped films exhibit maximum etch rates upon addition of ~25% hydrogen



• Addition of hydrogen to CHF₃ shows negative effect on etch rate of blank carbon doped film



• A similar effect is seen for etchant feed composition on patterned films



• Optical emission spectroscopy (OES) measurements showing emission from atomic fluorine at 685.6nm and 703.7nm

OES of CF₄/CHF₃



• OES measurements confirm similar trend of F radical intensities with increasing H₂, correlating to changes in etch rate for blanket films

Optimized H₂ Amount in CHF₃



- CHF₃ with smaller amounts of additive H₂ was probed with OES
- Future studies will focus on etch rate measurements to determine optimized F intensity in CHF_3 with H_2



• Carbon doped silica films can be patterned successfully using Ti hard mask and combination of different chemistries

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[5] NIST-JANAF Thermochemical (2013)

References

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Industrial Interactions and <u>Technology Transfer</u>

- Conference call with Intel, June 12, 2014 (Satyarth Suri)
- Conference call with Intel, July 9, 2014 (Satyarth Suri)
- Conference call with Intel, August 14, 2014 (Satyarth Suri)
- Conference call with Intel, September 11, 2014 (Satyarth Suri)
- Conference call with Intel, October 30, 2014 (Satyarth Suri)
- Conference call with Intel, December 18, 2014 (Satyarth Suri)
- Conference call with Intel, February 19, 2015 (Satyarth Suri)

Future Plans

Next Year Plans

- Establish point of contact with industrial sponsor to study etching efficacy of NF₃ and CF₃I not currently available in facilities (exploring possibility at IM Flash Technologies)
- Utilize optical emission measurements to determine etch rate correlation with atomic fluorine intensity

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, November 2014

(J.K. Chen, N. Altieri, M. Paine, and J.P. Chang, "Non-PFC Plasma Chemistries for Patterning Low-k Dielectric Materials")

• SRC ERC EHS TeleSeminar, March 5, 2015

Publication:

- "Thermodynamic assessment and experimental verification of reactive ion etching of magnetic metal elements", June 2014
- "Viable chemical approach for patterning nanoscale magnetoresistive random access memory", January 2015
- Deliverable Report, P065582, "Non-PFC Plasma Chemistries for Patterning Complex Materials/Structures", January 2014