

January 28th, 2025



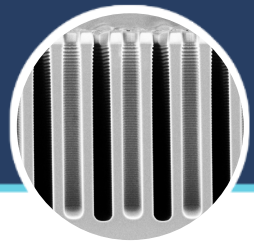
Renewed interest in cryogenic etching processes: what are the advantages of cooling the substrate?

Rémi DUSSART

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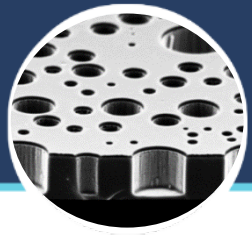
Groupe de Recherches sur l'Energétique des Milieux Ionisés
14 rue d'Issoudun 45067 Orléans Cedex 2 France



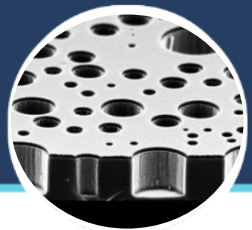


Outline

- ✓ Introduction, history and principle of cryoetching
- ✓ Deep Cryo-Etching of Silicon
- ✓ Passivation layer formation by SiF_4/O_2 plasma
- ✓ Passivation steps using CF_4 plasma instead of C_4F_8
- ✓ Cryo Atomic Layer Etching
- ✓ What makes cryogenic etching popular again in the industry ?
- ✓ What are the advantages of cooling the substrate ?



Introduction, principle and history of cryo-etching of Silicon



Introduction to cryochemistry

- // It is counterintuitive that **chemical reactions can be accelerated at low temperature**, by freezing. ^[1]
- // However, this phenomenon has been highlighted by the “cryo-chemistry” community since the 50^{ies}. ^[2]

Example ^[3]: in the following reaction,



The reaction rate is given by :

$$\text{Rate} = k'[A]^a[B]^b$$

k' is the rate coefficient and follows an Arrhenius law

$$k' = A' \exp(-E_a/RT)$$

A' = Arrhenius pre-exponential factor
 E_a : activation energy

By decreasing T , k' should decrease as well.

A' (frequency factor) should also decrease as T decreases



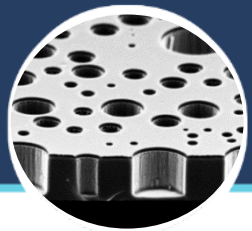
The rate should decrease...

BUT...

^[1] 2021: An, L.-Y. et al. Advances in Cryochemistry: Mechanisms, Reactions and Applications. Molecules 26 750

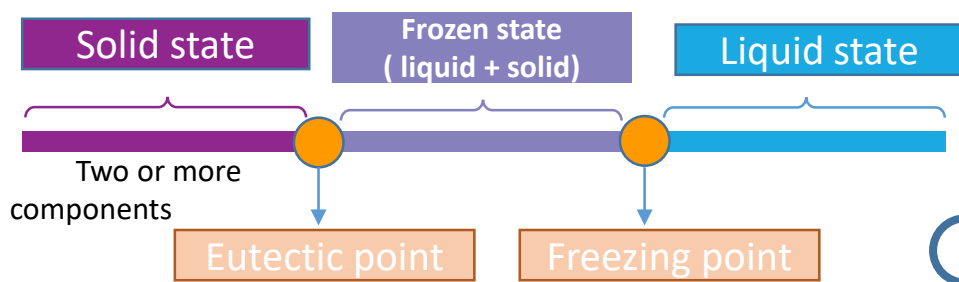
^[2] 1962: H. A. McGee and W. J. Martin Cryochemistry, Cryogenics, vol2 (5) 257

^[3] 2023: Jiaxin Lv et al. Freeze-accelerated reactions on environmental relevant processes Cell reports Physical Science 4 101456

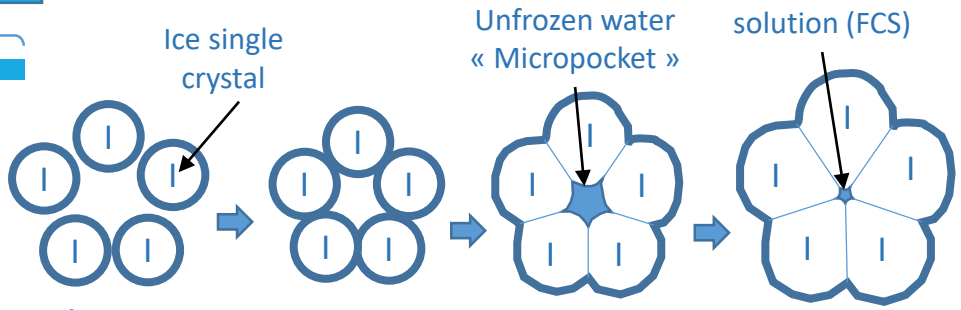


Introduction to cryochemistry (cont'd)

// Example in a frozen solution or impure solid, there is a gradual transition from solid to liquid



Adapted from [1, 2]



// Freezing begins when T is below the freezing point

// In the frozen state, the substance is in equilibrium between its solid and liquid phases.

// Unfrozen regions behave as « micropockets » and provide special microenvironments for chemical reactions.

// If all solutes are rejected from ice crystals to the unfrozen solution, the concentrations of solutes A and B in the micropockets, $[A]_{mp}$ and $[B]_{mp}$ can be expressed as:

$$[A]_{mp} = [A] C_{mp} / C_T \quad [B]_{mp} = [B] C_{mp} / C_T \quad Rate = k' [A]_{mp}^a [B]_{mp}^b$$

⇒ Concentration effect which accelerates the reaction

[1] 1996: Norimichi Takenaka et al. Acceleration Mechanism of Chemical Reaction by Freezing J. Phys. Chem., Vol. 100, No. 32

[2] 2023: Jiaxin Lv et al. Freeze-accelerated reactions on environmental relevant processes Cell reports Physical Science 4 101456



Brief history of the cryogenic etching process

Tachi's team proposed to cool the substrate down to a temperature between **-100** and **-130°C** while running a **microwave SF₆ plasma** to etch silicon anisotropically.

1988 : S. Tachi *et Al.* Appl. Phys. Lett., 52(8), 616(1988)

The idea was to **freeze chemical reactions** on vertical sidewalls of the sample and favor ion-assisted reactions at the feature bottom.

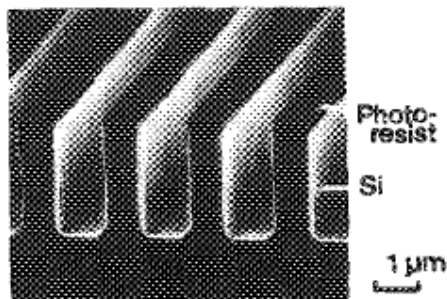
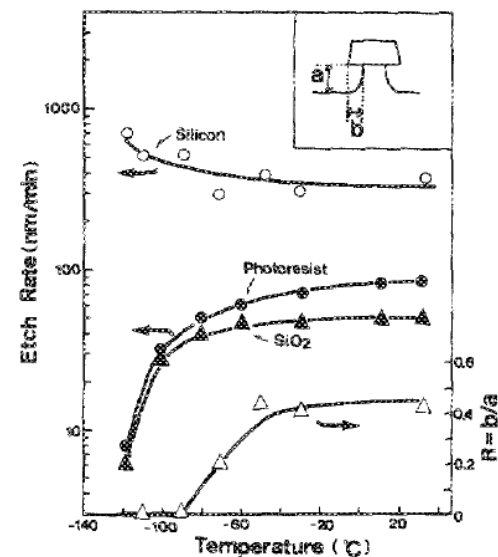


FIG. 3. Silicon profile etched at -- with the use of SF₆ gas plasma.

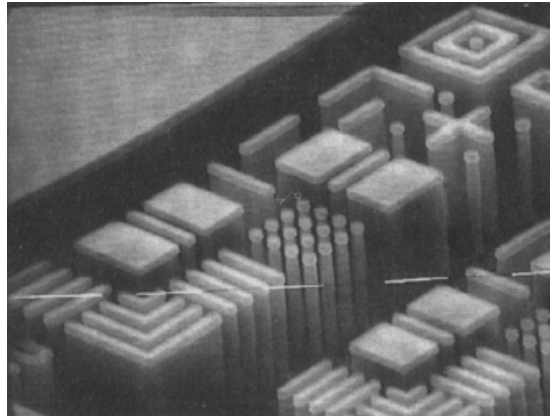


performed with high selectivities of 30 for organic resist films. High etch rates of 500 and 1000 nm/min by reactive ion etching and microwave plasma etching, respectively, were achieved with a SF₆ gas plasma at low wafer temperatures from -130 to -100°C. It is concluded that



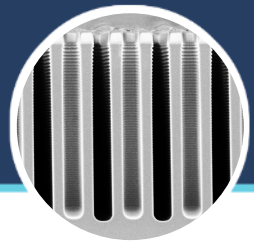
Brief history of the cryogenic process (cont'd)

- 1995 : J. W. Bartha et Al. Microelectron. J., 43, 453(1995)



plasma source. In contrast to the current understanding of low temperature etching, we did not observe a "freezing" of the lateral etching reaction, but obtained isotropic etch profiles, even at temperatures below -120°C . Anisotropic etch profiles are obtained by an addition of O_2 . We therefore propose a sidewall passivation

For the first time, a mechanism based on sidewall passivation was suggested in cryogenic etching instead of a mechanism based on a low reaction probability of the radicals on very cold silicon surfaces.



Principle of cryoetching of Silicon

SF₆/O₂ plasma

✓ Chemical etching (selective)

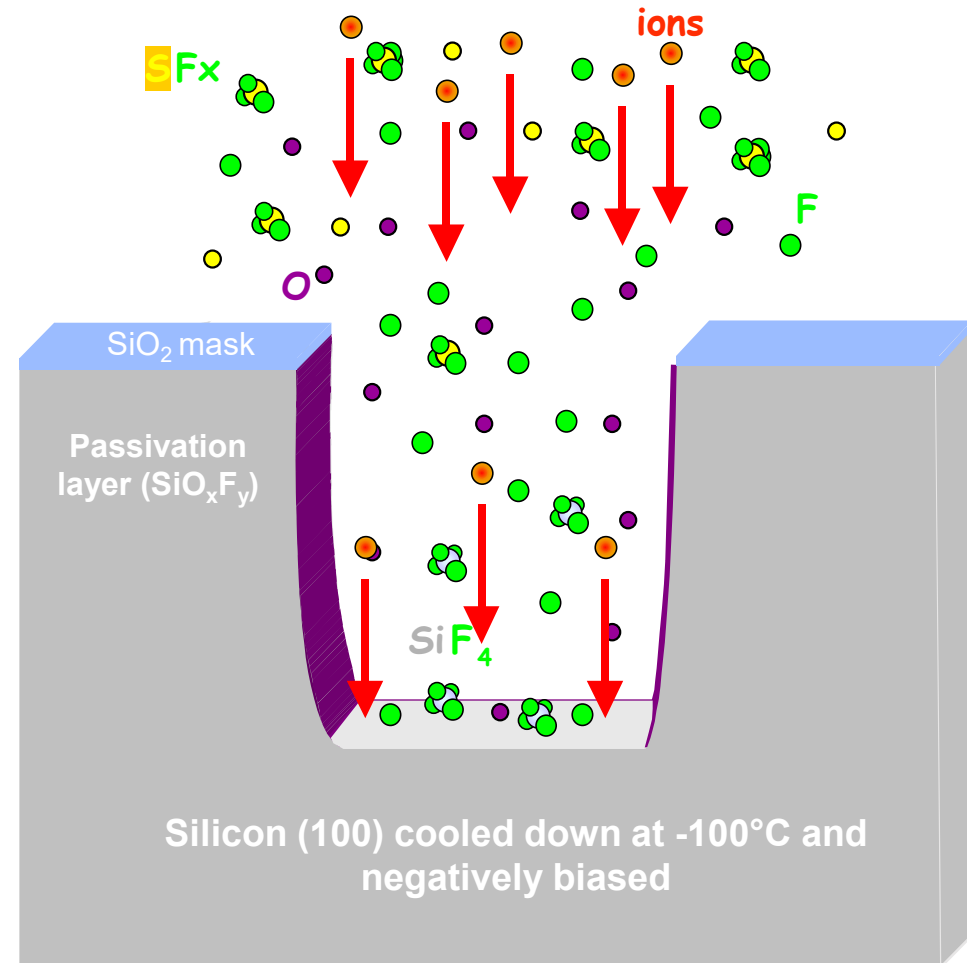
SiF₄ : main etching product

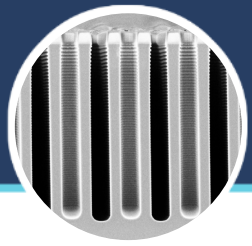
✓ Passivation layer (SiO_xF_y)

Only forms at very low temperature

✓ Fragile passivation layer, easily removed by ion bombardment

Simultaneous mechanisms





Typical reactor used for cryoetching

Parameter range:

Pressure : 3 – 10 Pa

Power : 500 – 3000 W

Bias : 0 – 100V

SF₆ : 50 – 1000 sccm

SiF₄ : 0 – 100 sccm

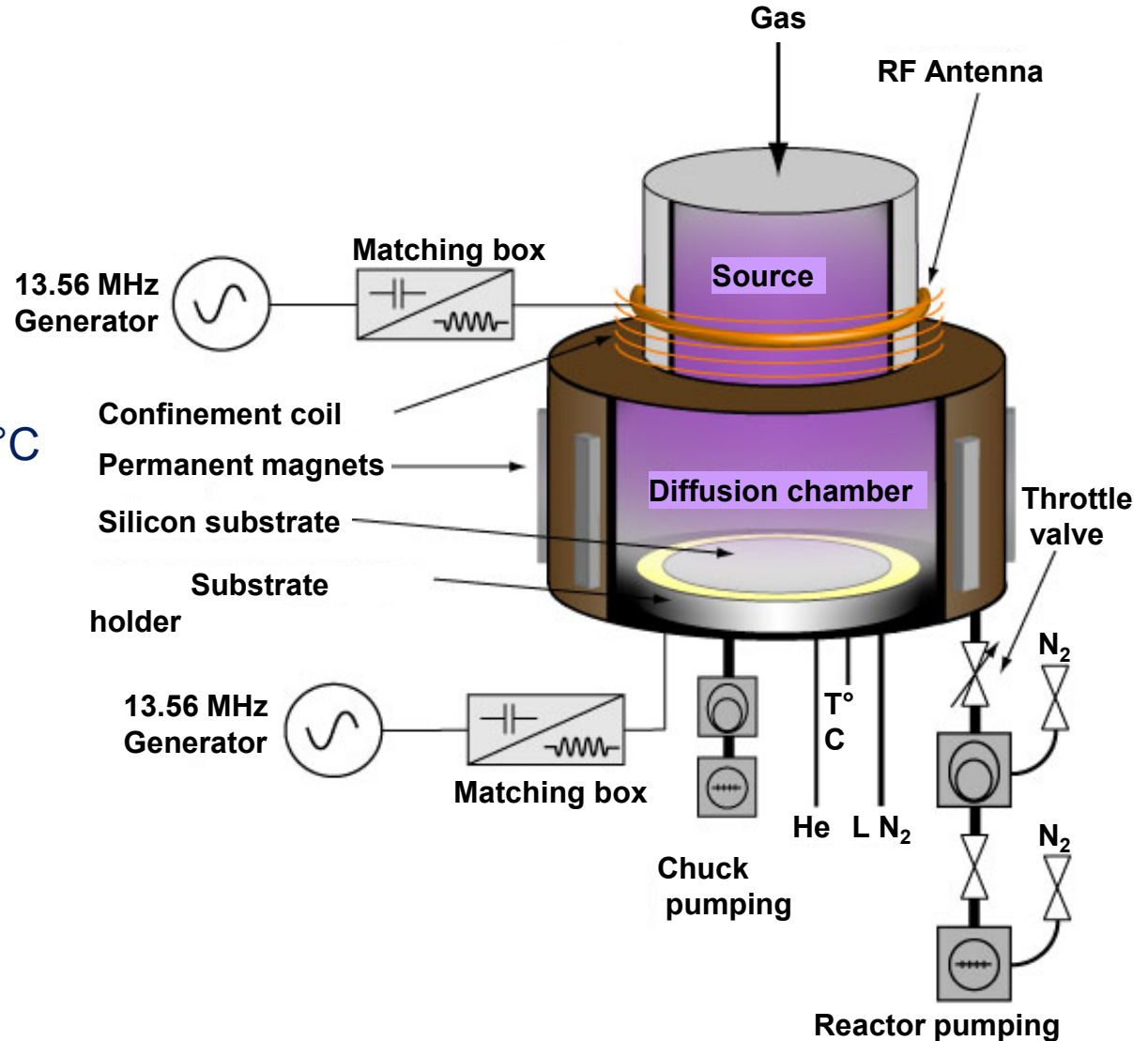
O₂ : 0 – 70 sccm

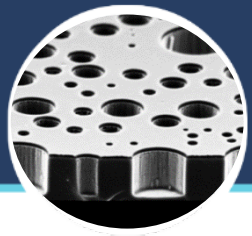
Temperature : -150°C → 20°C

Diagnostics:

In-situ ellipsometry

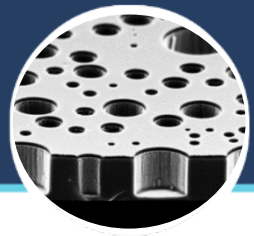
Mass spectrometry





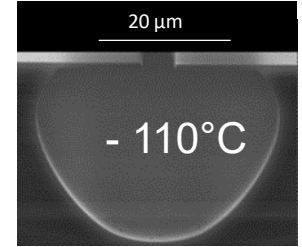
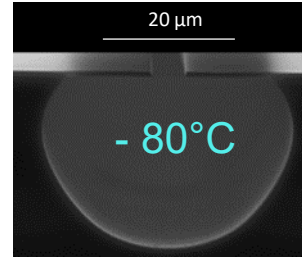
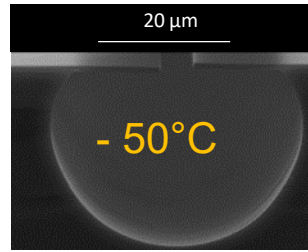
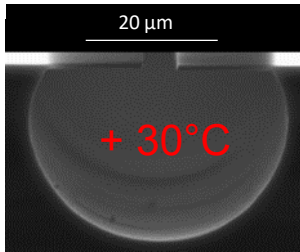
Deep Cryo-Etching of Silicon

Passivation layer characterization in
 SF_6/O_2 plasma

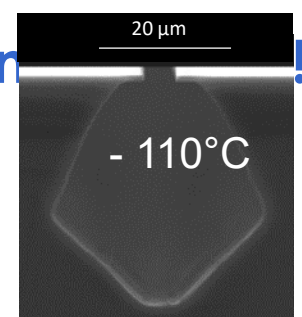
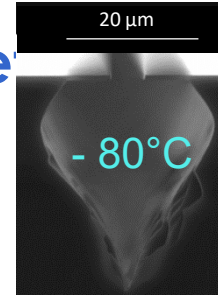
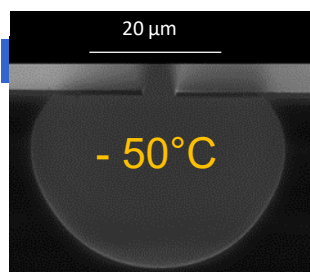
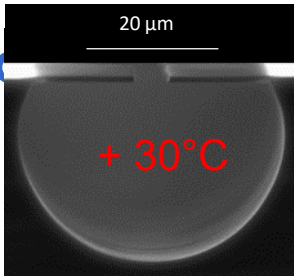


Role of oxygen, temperature and ion bombardment

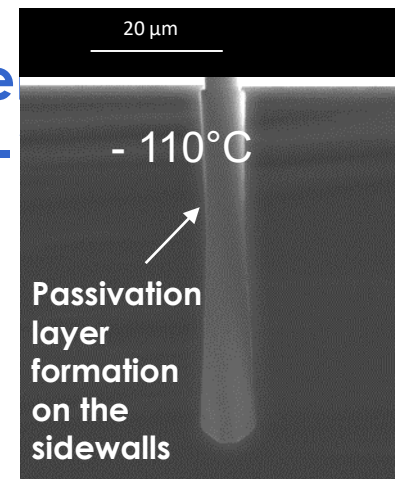
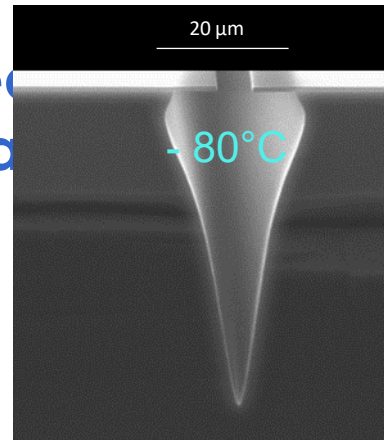
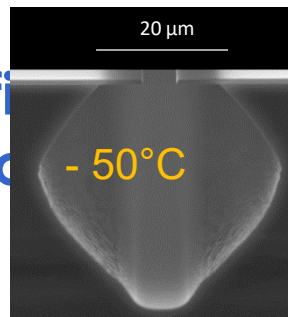
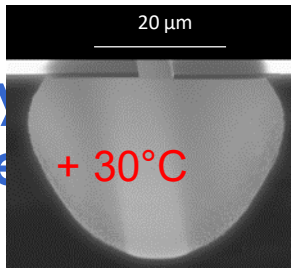
SF₆ plasma without bias



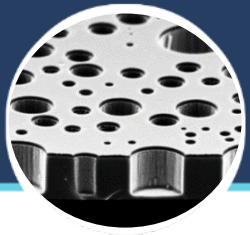
Isotropic SF₆/O₂ plasma without bias



SF₆/O₂ plasma with bias



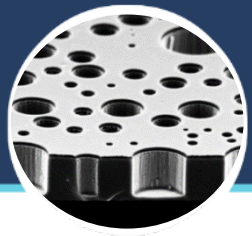
Anisotropic etching at -110°C with oxygen and bias



⇒ What is the **composition of the passivation layer** ?
What is the **role of the etched by-products** ?

⇒ What are the **main mechanisms involved in the formation of the passivation layer** ?
Why is it necessary to cool the substrate ?

⇒ How to enhance the **robustness of the passivation layer** in the cryogenic process ?

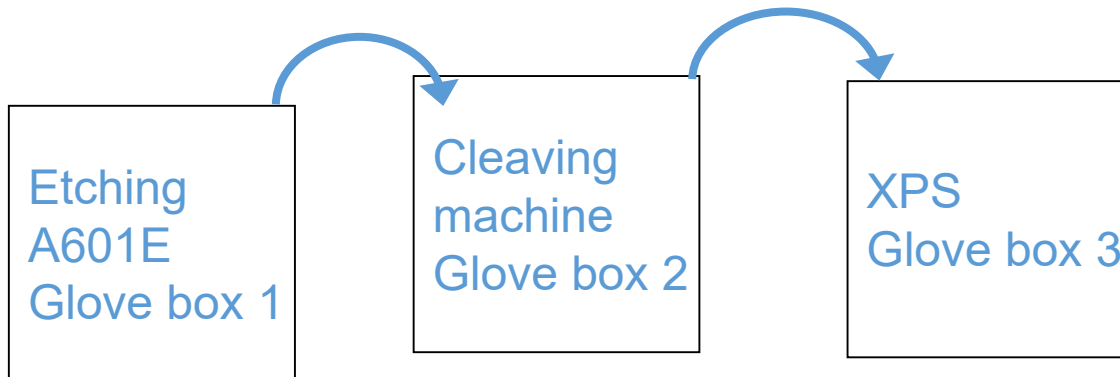


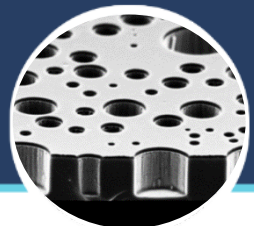
Ex-Situ XPS analysis

Objective of this experiment : analyze the passivation layer after etching, but without leaving the sample being oxidized by the ambient air.

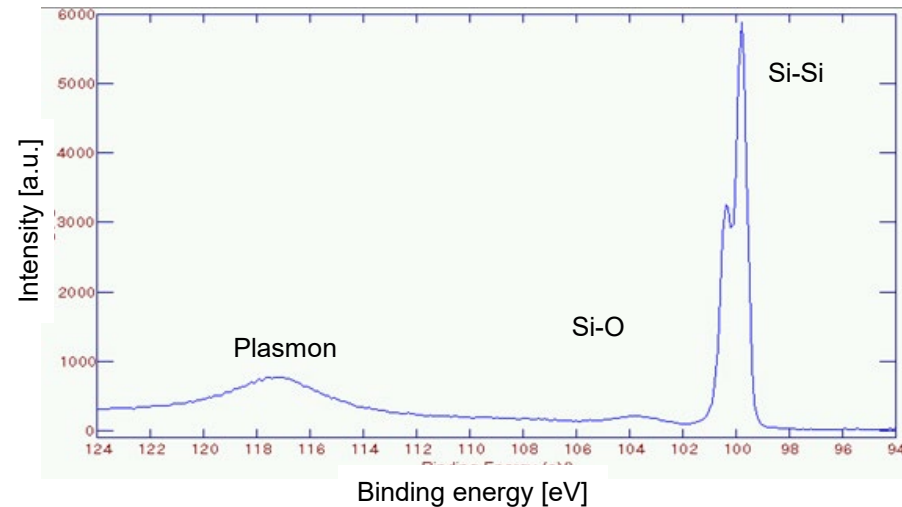
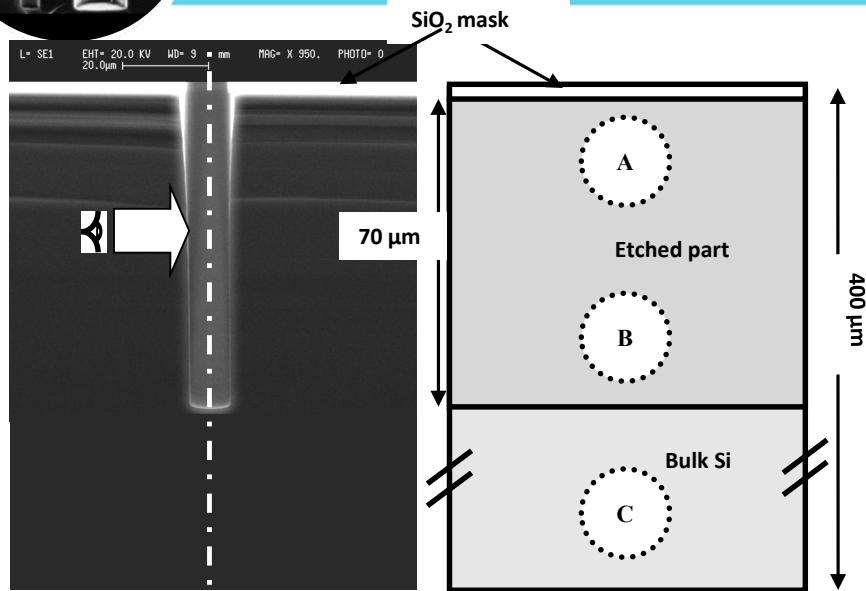
Method :

1. After etching, the sample is removed from the reactor in the glove box 1 full of pure N₂ gas
2. Transportation of the wafer (under pure N₂) toward the glove box 2 where cleavage is performed. (Residual O₂ rate controlled <0.1 %).
3. After cleavage, transportation of the trench to be analyzed in another lab in a 3rd glove box for XPS.





Ex-Situ XPS analysis (cont'd)



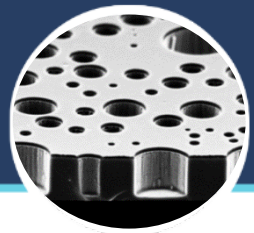
✓ Nearly no oxidation (SiO_2) on the passivated surfaces.

✓ Low contamination

⇒ The passivation layer is removed when the wafer is warmed back up to ambient temperature

Lines	Peak Center [eV]	Ratio (± 0.01)		
		A	B	C
F / Si-Si	F 1s- 688	0.02	0.02	0.01
O / Si-Si	O 1s- 533.5	0.13	0.15	0.14
C / Si-Si	C 1s- 285.3	0.11	0.15	0.11
Si-O / Si-Si	Si 2p- 103.7	0.03	0.03	0.03

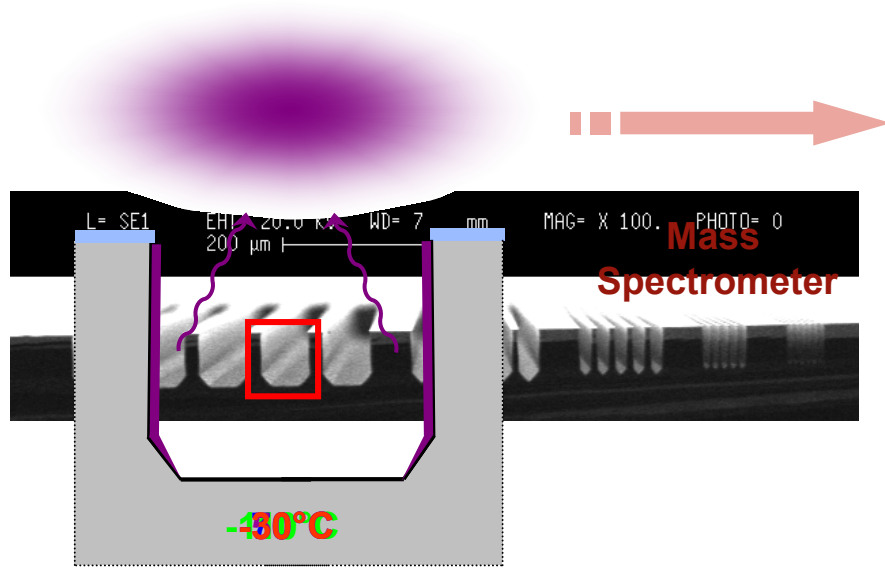
(1)R. Dussart *et al* J. Micromech. Microeng., 14, 190-196 (2004)



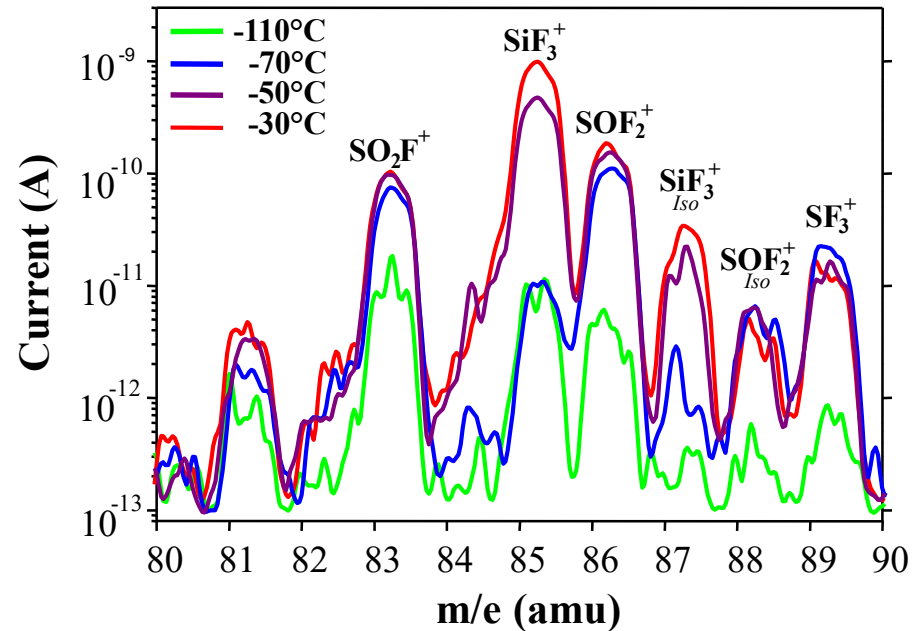
Desorbed species - Mass spectrometry analysis

Passivation layer characterization by mass spectrometry

analyze the desorbed species coming from trench sidewalls when the wafer is warmed back to ambient temperature.



Heating (from -110°C to -30°C)



SO_xF_y (SO_2F^+ , SOF_2^+) desorption from -110°C to -70°C
 SF_x (SF_3^+) desorption between -110°C and -70°C
 SiF_4 (SiF_3^+) desorption between -70°C and -30°C

✓ SiF_4 forms during the sample warming

X. Mellhaoui et al., J. Appl. Phys., 98, 104901 (2005)



In-situ X-Ray Photoelectron Spectroscopy

OPTIMIST Platform (IMN, Nantes)



Christophe Cardinaud, Aurélie Girard

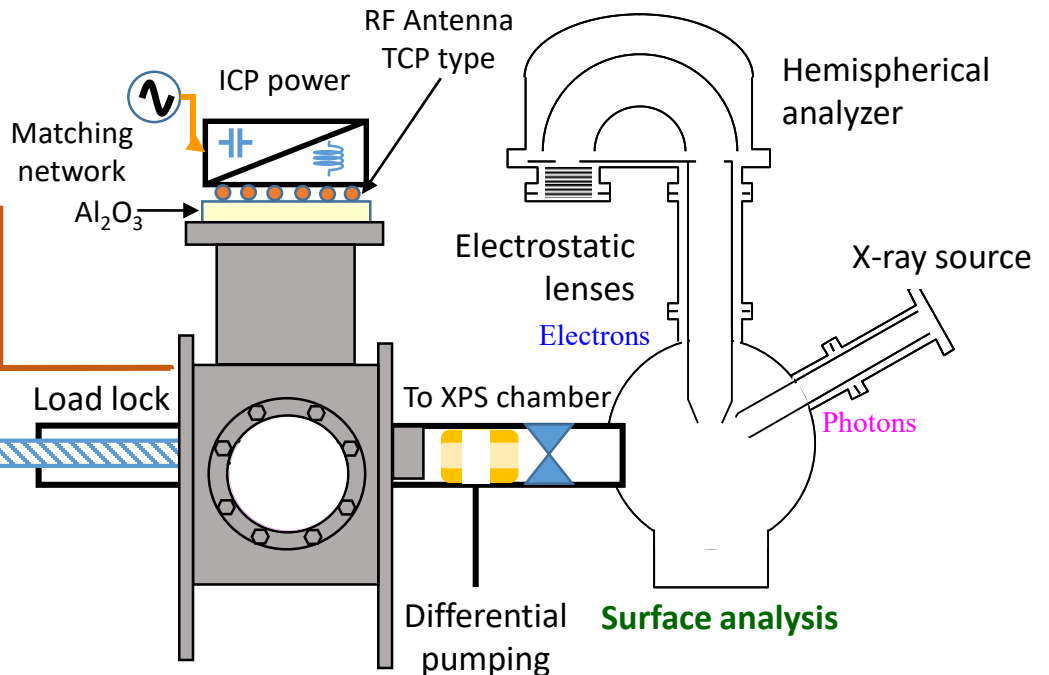


SF₆/O₂ in
overpassivating
regime

4 gas feed

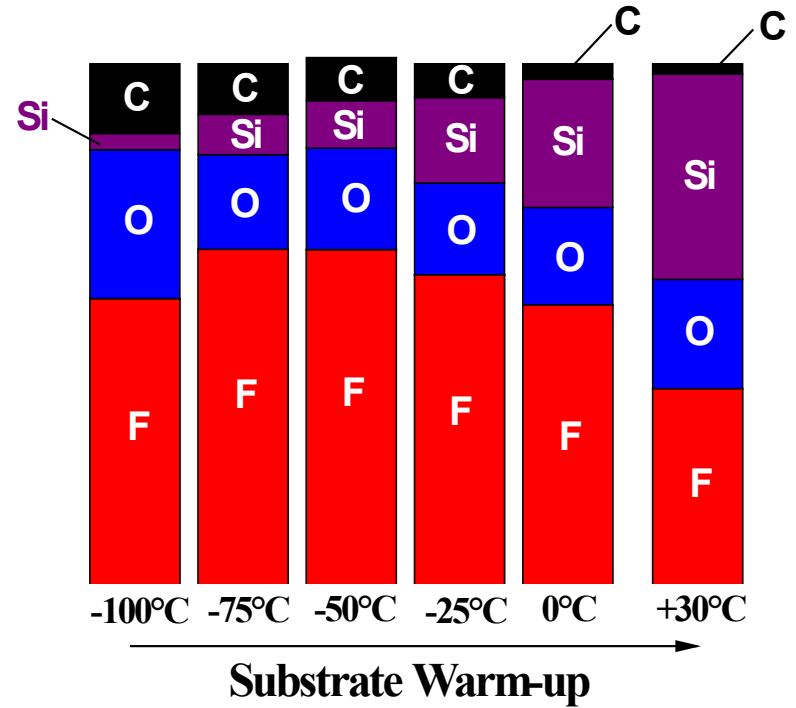
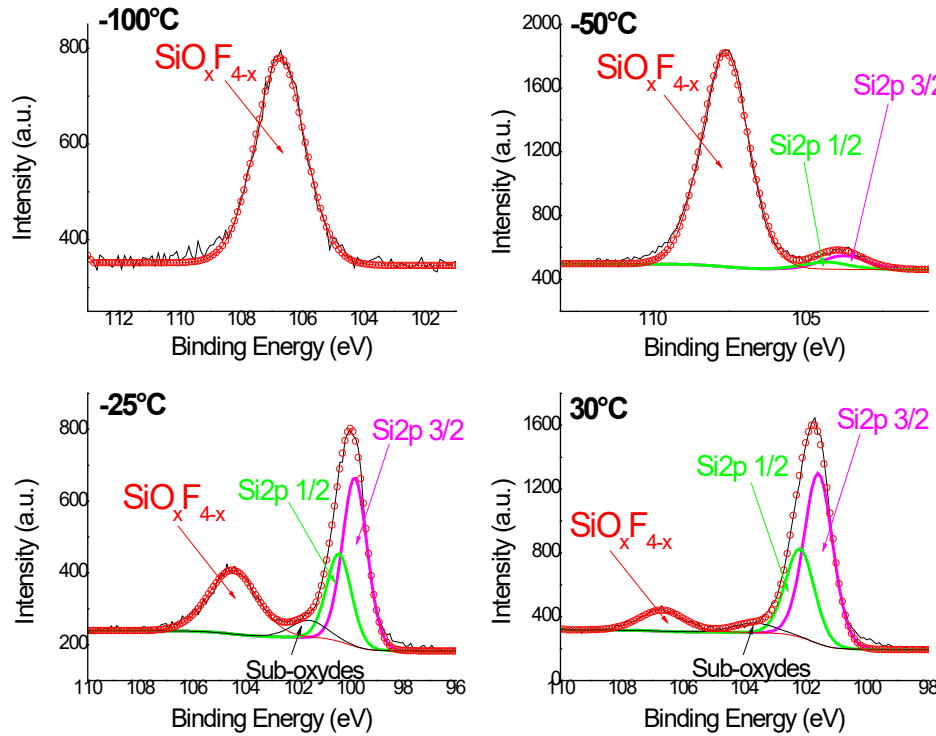
T controller

Sample rod
-180°C < T < +1100°C



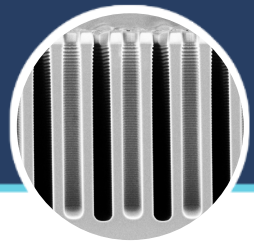


In-situ X-Ray Photoelectron Spectroscopy



- > **Decrease of $\text{SiO}_x\text{F}_{4-x}$ contribution with temperature**
- > **Appearance and increase of Si matrix contribution**

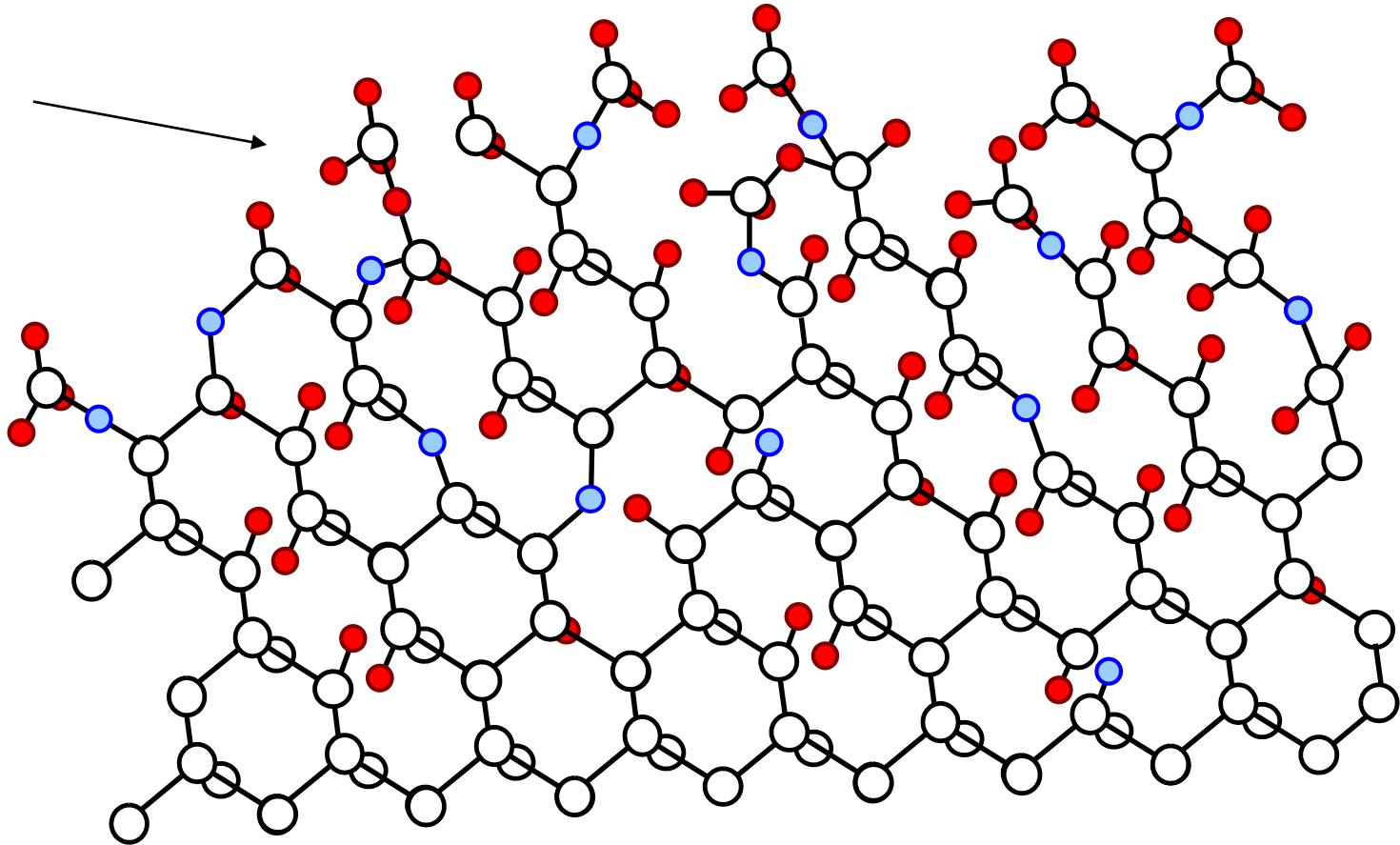
- > $[\text{O}]_{\text{at}}$ remains almost **constant** from -75°C
- > $[\text{F}]_{\text{at}}$ **decreases** from -75°C



Silicon matrix after SF₆/O₂ overpassivating plasma

SiF₄ volatile groups

SiOF₃



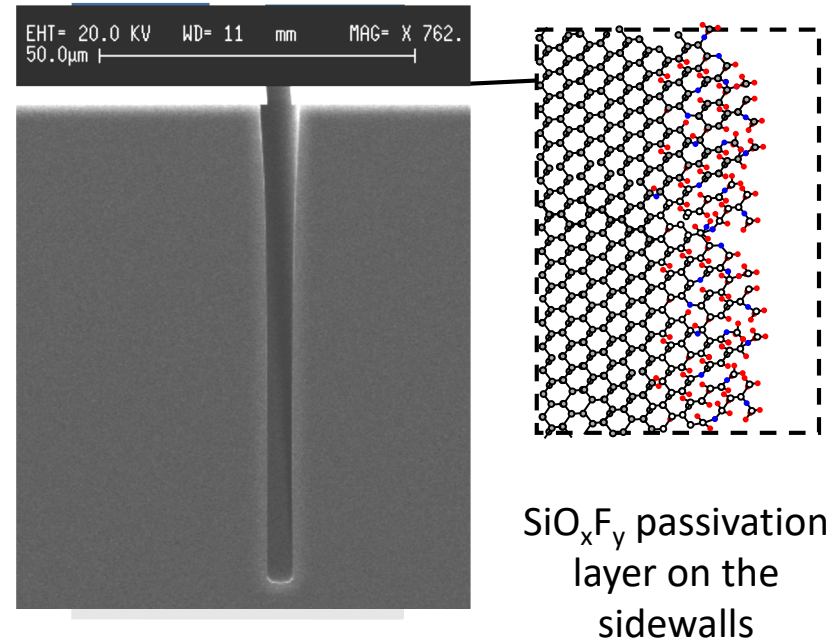
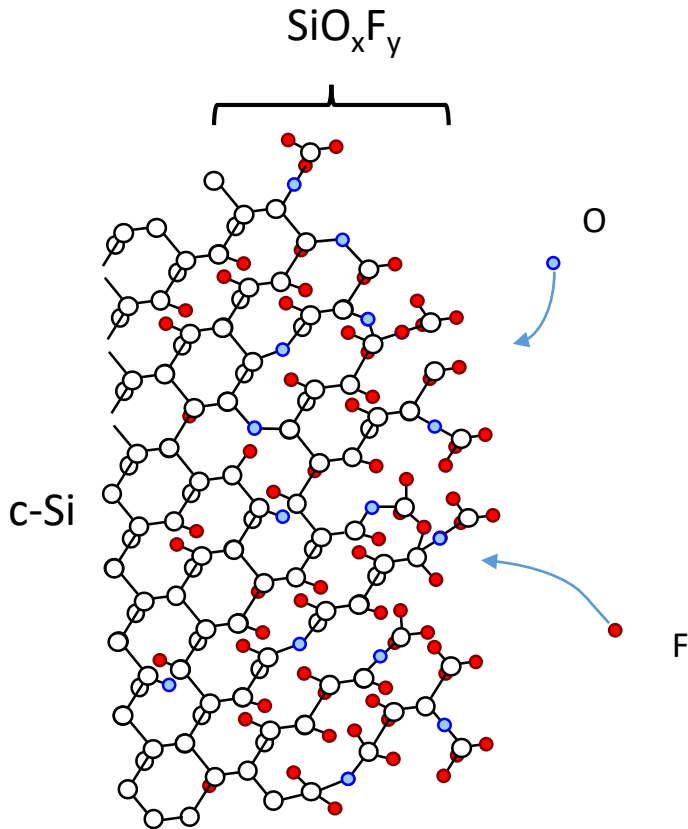
-100°C

+30°C

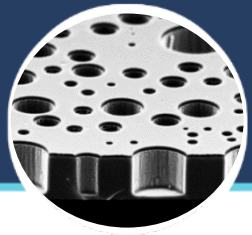


Summary on the SiO_xF_y Passivation layer in SF_6/O_2 plasma

// Thin SiO_xF_y formation by SF_6/O_2 plasma at low temperature



// In SF_6/O_2 plasma, fluorine radicals will form SiF_x sites at the silicon surface and oxygen will react with SiF_x sites to form a thin SiO_xF_y layer.
⇒ quite fragile passivation layer.



Passivation layer formation by SiF_4/O_2 plasma



Passivation layer construction with SiF₄

Two cavity test experiments to study the passivation layer formation

Principle : - we start with an isotropic etching (**Initial profile**)
- we try to form the passivation layer with SiF₄ and O₂

INI : Isotropic etching

- 10 min
- SF₆: 200 sccm
- Source: 1000 W
- Bias: 0 V
- -83°C

END : Additional etching

- 3 min
- SF₆: 200 sccm
- Source: 1000 W
- Bias: 37 V
- -83°C

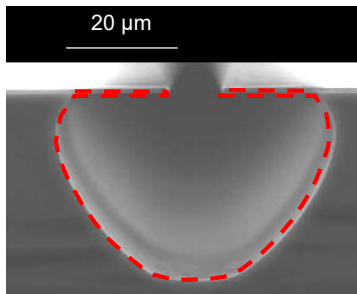
Isotropic etching continues

Passivation

- 1 min
- SiF₄/O₂ 65%
- Source: 1000 W
- Bias: 37 V
- -83°C

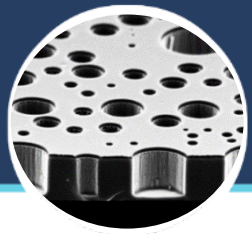
END

Efficient passivation layer



Reference profile

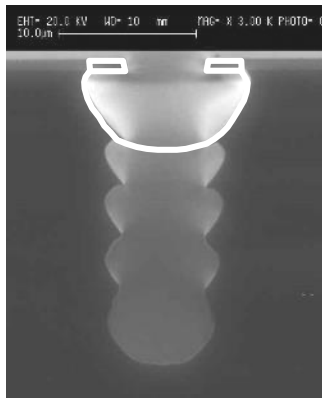
SiF₄/O₂ plasma can be used to form an efficient passivation layer.



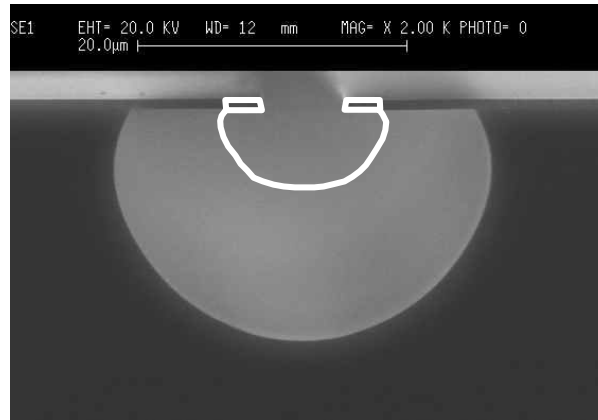
STiGer process

Alternation of isotropic SF_6 etching steps and SiF_4/O_2 passivation steps
4 alternances 1min SF_6 etching - SiF_4/O_2 deposition + a final 1 min etch

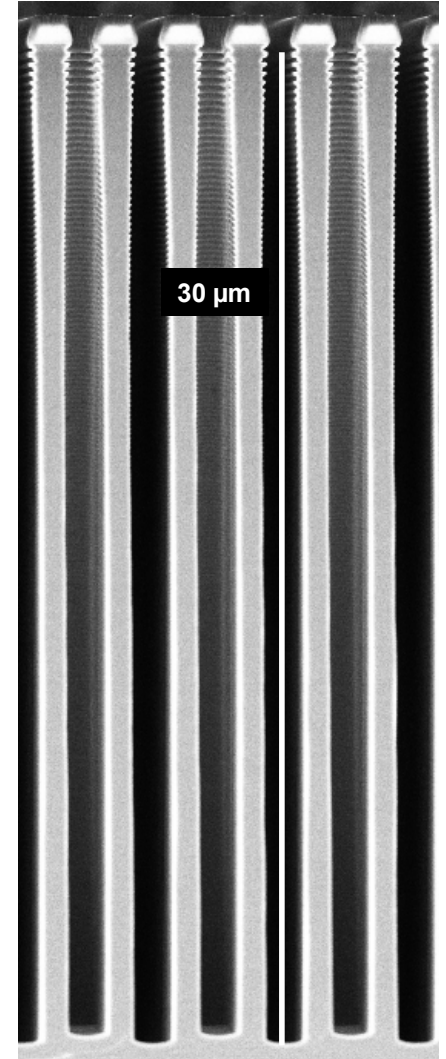
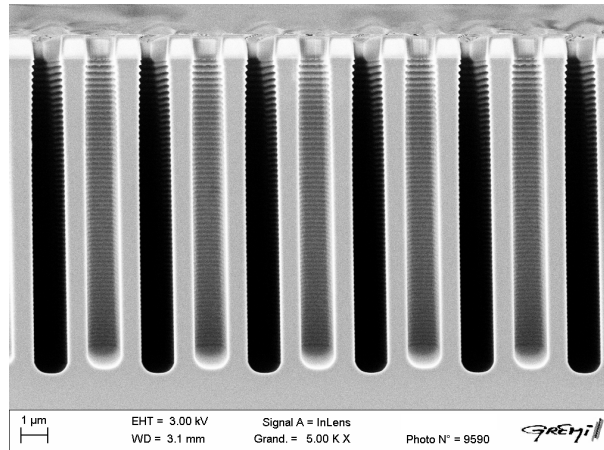
-83°C

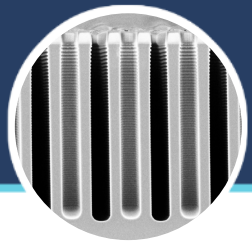


Same experiment at 0°C



Anisotropic microstructures can be obtained by alternating SF_6 etching and SiF_4/O_2 passivation steps, but it only works at cryogenic temperature





In-situ X-Ray Photoelectron Spectroscopy



OPTIMIST Platform

// $\text{SiF}_4 / \text{O}_2$ plasma experimental conditions:
a-Si sample ; 30 s ; $\text{SiF}_4 / \text{O}_2$: 25 % ; 3.0 Pa ;
200 W ICP power ; no bias.

// SiO_xF_y layer growth at 3 different temperatures : -40, -65 and -100°C

// At -40°C, 17.1% [F] ; 23.6% [O]

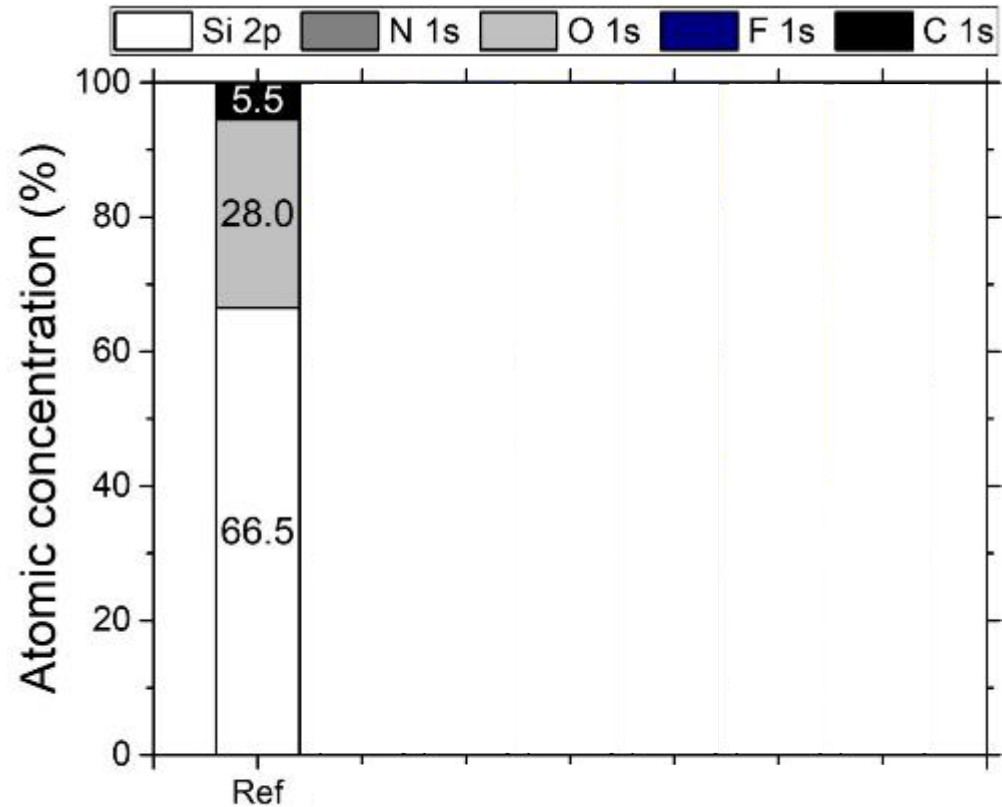
After heating: no significant change

// At -65°C, 20.0% [F] ; 22.8% [O]

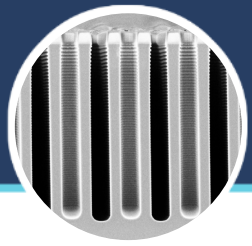
After heating: a little decrease of [F]

// At -100°C, 52.3% [F] ; 18.0% [O] ;
15% [N] (stoichiometry of $\sim\text{SiOF}_3$)

After heating: a large part of Fluorine based species has desorbed



G. Antoun *et al* 2022 *ECS J. Solid State Sci. Technol.* **11** 013013



Role of SiF_x physisorption at low temperature

// 2014 : Molecular dynamics by Stefan Tinck from PLASMANT laboratory (Antwerpen)

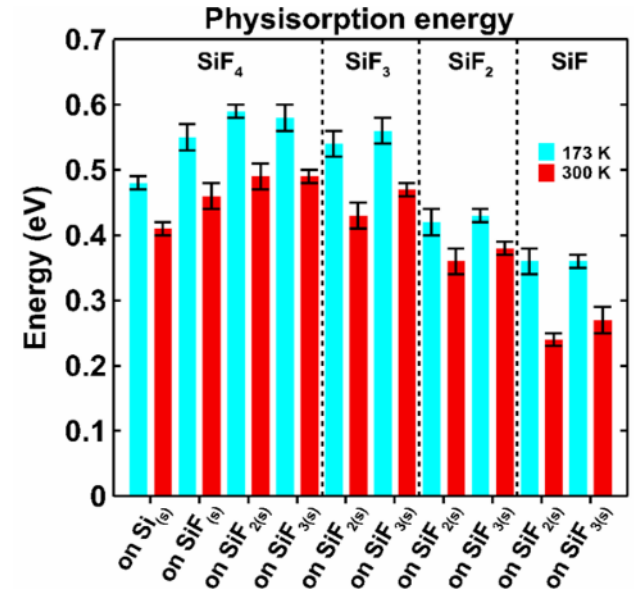
S. Tinck et al. J. Phys. Chem. C 2014, 118, 30315–30324

Fluorine – silicon surface reactions at cryogenic temperature

Calculated **probabilities for immediate sticking** upon impact of various impinging species on different surfaces ⁽¹⁾

Sticking : creation of a chemical bond (within 12.5 ps in this work).

Impinging species	on Si		on SiF		on SiF ₂		on SiF ₃	
	300 K	173 K	300 K	173 K	300 K	173 K	300 K	173 K
F	0,98	0,98	0,92	0,93	0,59	0,61	0,23	0,25
Si	1	1	1	1	0,41	0,40	0,20	0,19
Si _F	0,88	0,89	0,49	0,50	0	0	0	0
SiF ₂	0,51	0,50	0,18	0,19	0	0	0	0
SiF ₃	0,37	0,37	0,06	0,06	0	0	0	0
SiF ₄	0	0	0	0	0	0	0	0
F ₂	1	1	1	1	0,77	0,77	0,3	0,31



$$k = Ae^{-\frac{E_a}{k_B T}}$$

Rate constant for thermal desorption

E_a increases by lowering T, due to smaller oscillation amplitudes between adsorbent and surface.

T. Lill et al. JVSTA 41 023005 (2023)

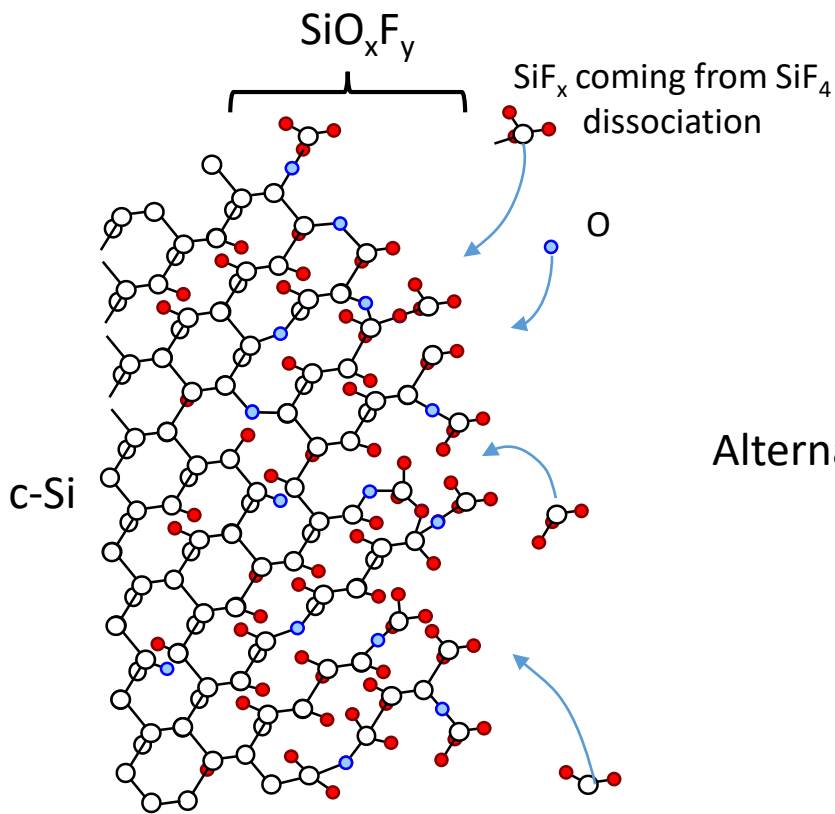
⇒ Probabilities for Immediate Sticking do not depend on temperature from 173 to 300 K !

⇒ Long thermal desorption time at low temperature as compared to room temperature ⇒ longer residence time of the species at the surface



Conclusion on SiF₄/O₂ plasma at low T

// It is possible to reinforce the passivation layer by using SiF₄/O₂ plasma steps

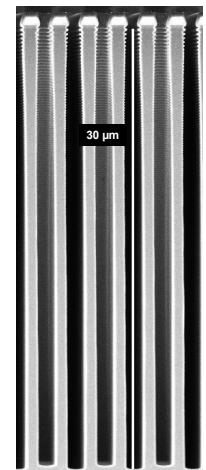
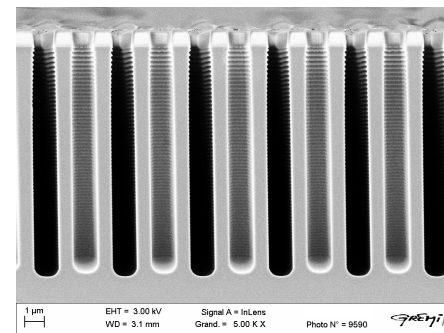


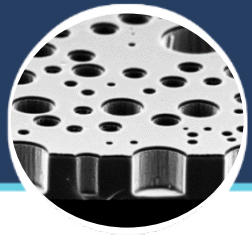
// **SiF_x radicals** coming from SiF₄ dissociation deposit on the sidewalls with a much longer residence time. They react with oxygen atoms to create a thicker SiO_xF_y layer.

⇒ **more robust** passivation layer.

⇒ STiGer process

Alternation of SF₆ plasma – SiF₄/O₂ plasma





Passivation steps using CF_4 plasma instead of C_4F_8



C_xF_y plasma for passivation

// C_4F_8 is the main gas used for passivation steps at room temperature in the so-called Bosch process to cover the sidewalls with a CF_x protective layer

// Low F/C ratio gases are highly polymerizing

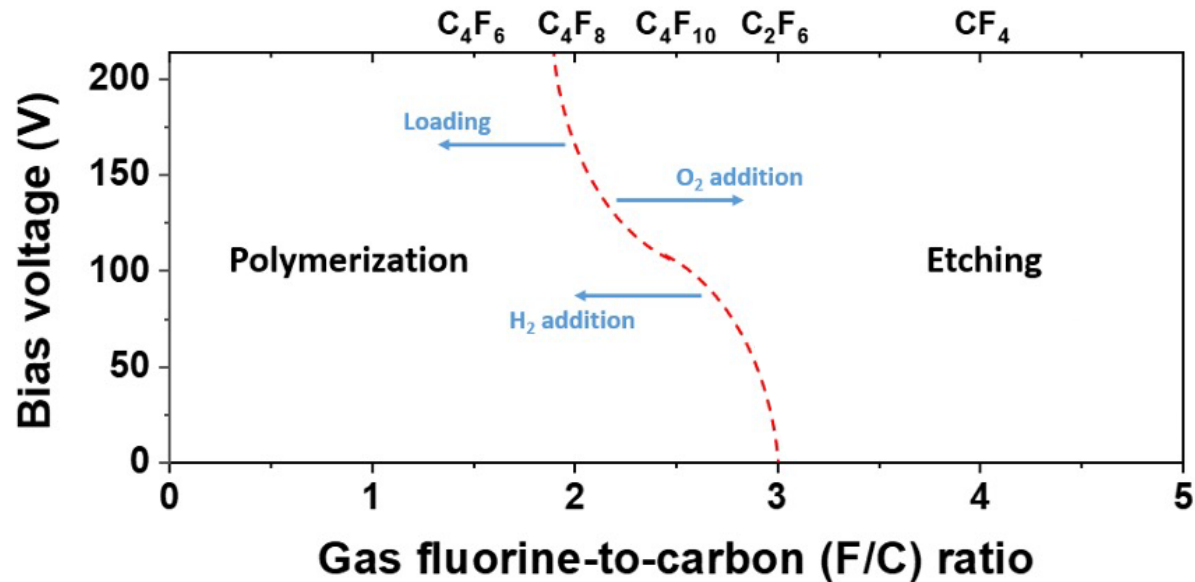
// Deposition regime unless a high bias voltage is applied

// But, CF_x deposits everywhere even on reactor walls, leading to process drifts.

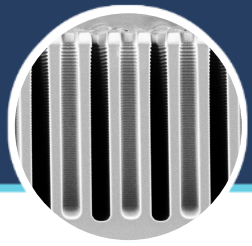
// Cleaning steps are needed

// The boundary line can be shifted to the right by decreasing the substrate temperature

// Is it possible to use CF_4 instead of C_4F_8 at low temperature ?



Adapted from J. W. Coburn and H. F. Winters, Plasma etching—A discussion of mechanisms, J Vac Sci Technol 16, 391 (1979)



CF₄ plasma at cryogenic temperature

J. Nos et al. Appl. Phys. Lett. 126, 031602 (2025)

Etching step: 3 s

300 sccm SF₆, 3 Pa, 1500 Ws, -135 Vb

x200

Passivation step: 2 s

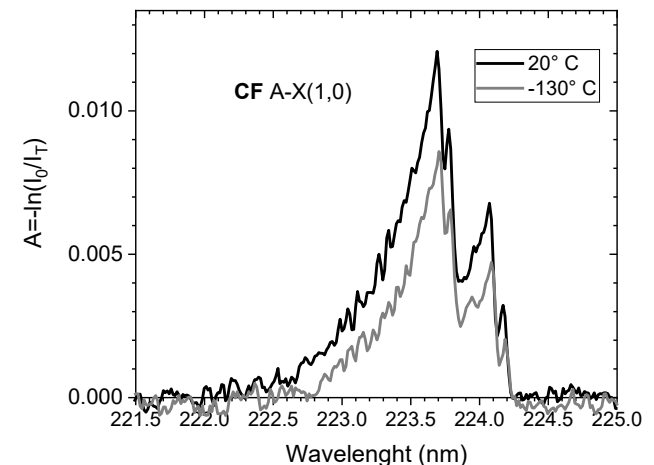
20 sccm CF₄, 1 Pa, 1500 Ws, -65 Vb

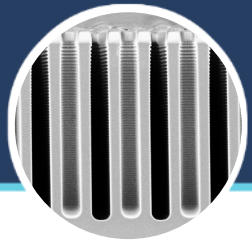
=> Strong CF_x deposition at -100°C

=> Enhanced passivation at -100°C

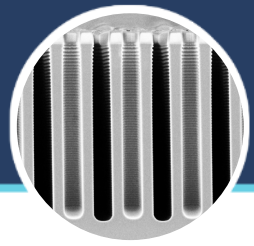
- // CF concentration drops at low wafer temperature, which shows that CF sticks more efficiently at low temperature. This is not the case for CF₂.
- // CF₄ is a good candidate to passivate the trench sidewalls without depositing on the reactor walls.

UV absorption experiments with G. Cunge and M. Kogelschatz (LTM)

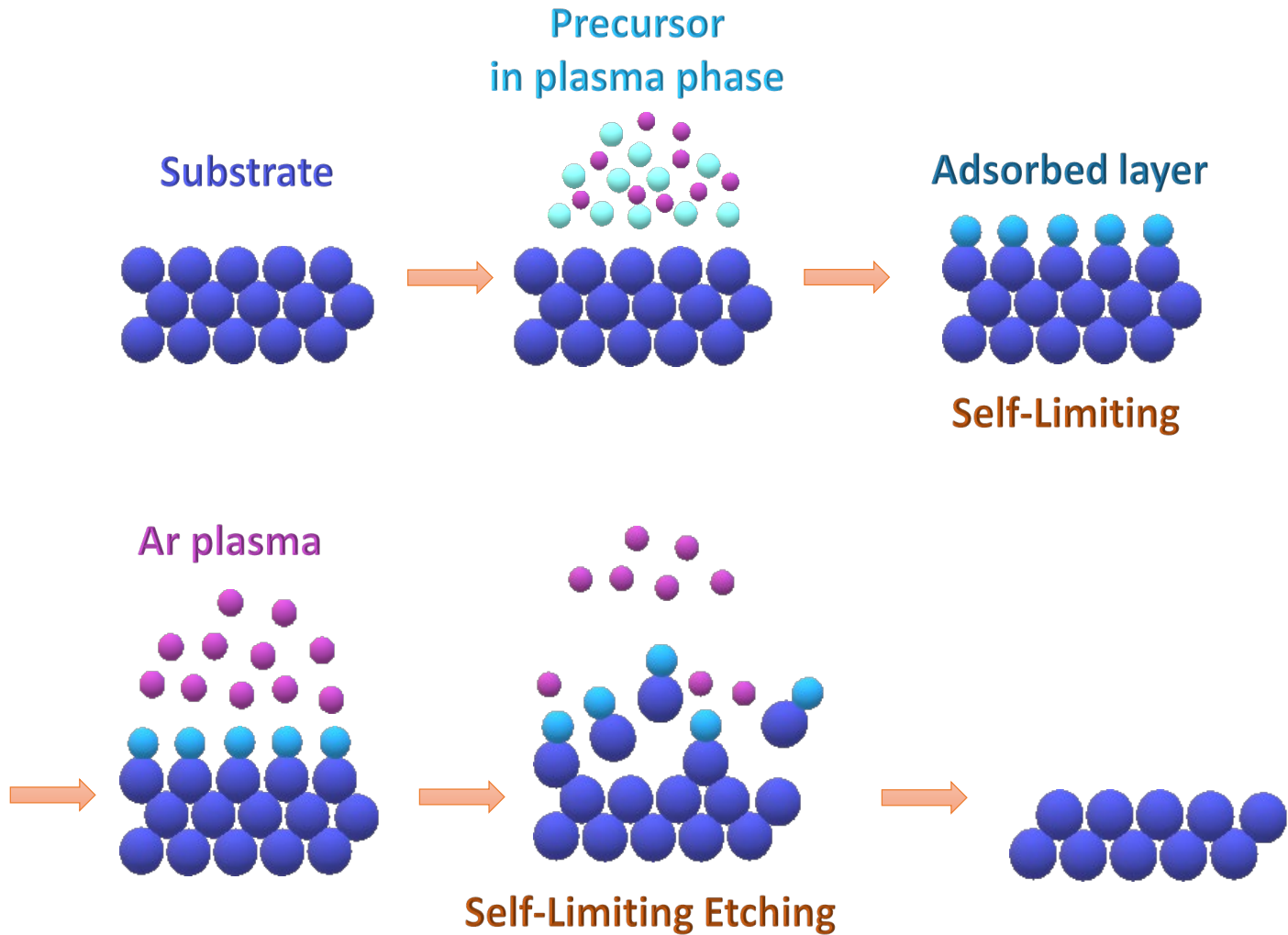




Cryo-Atomic Layer Etching



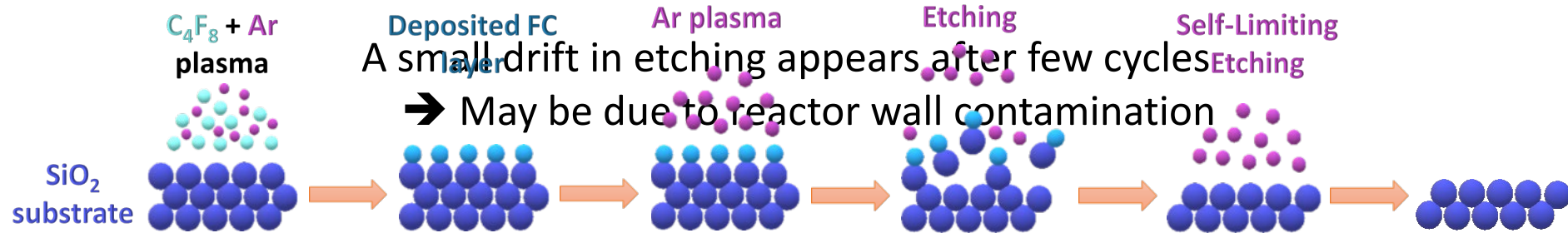
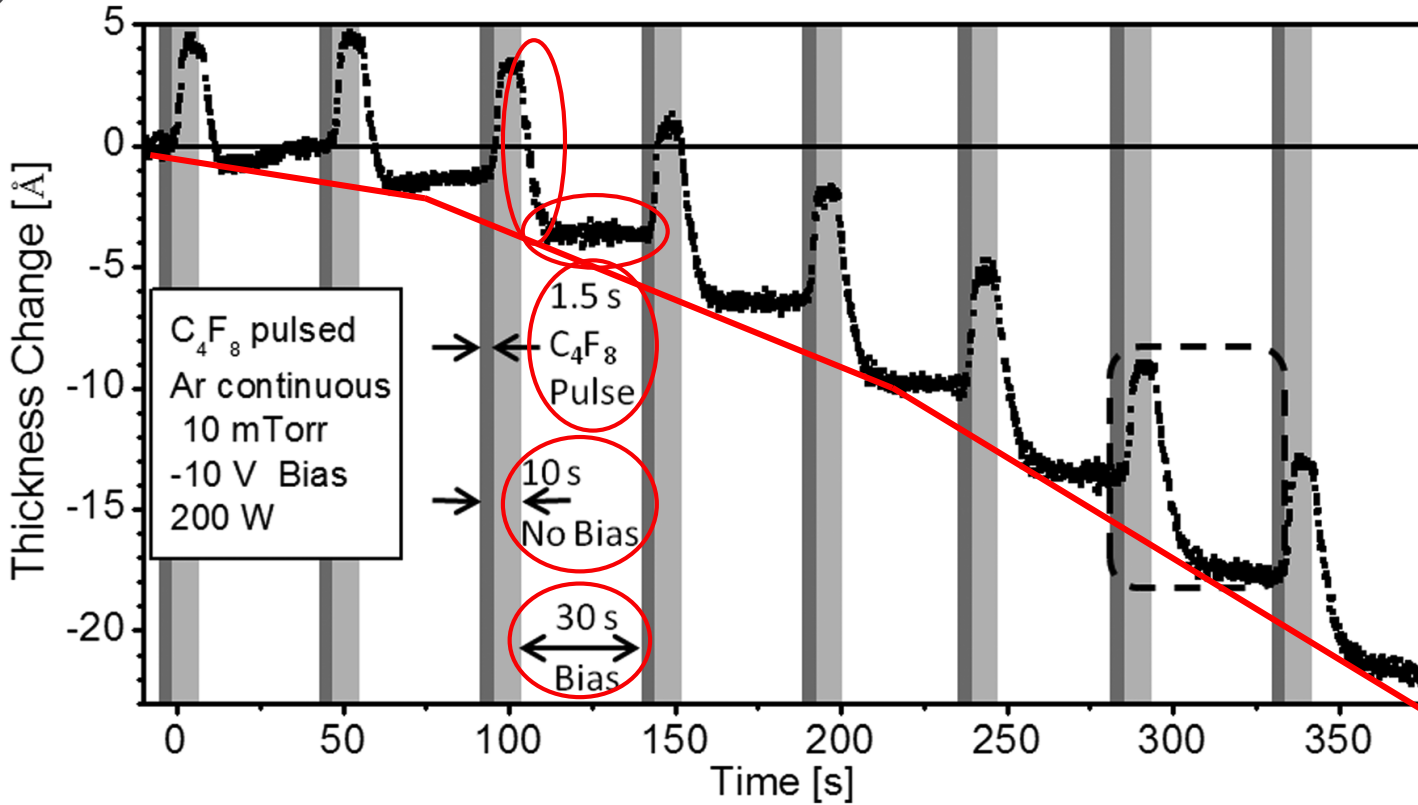
Principle of Atomic Layer Etching





Atomic Layer Etching for SiO₂

T=10°C

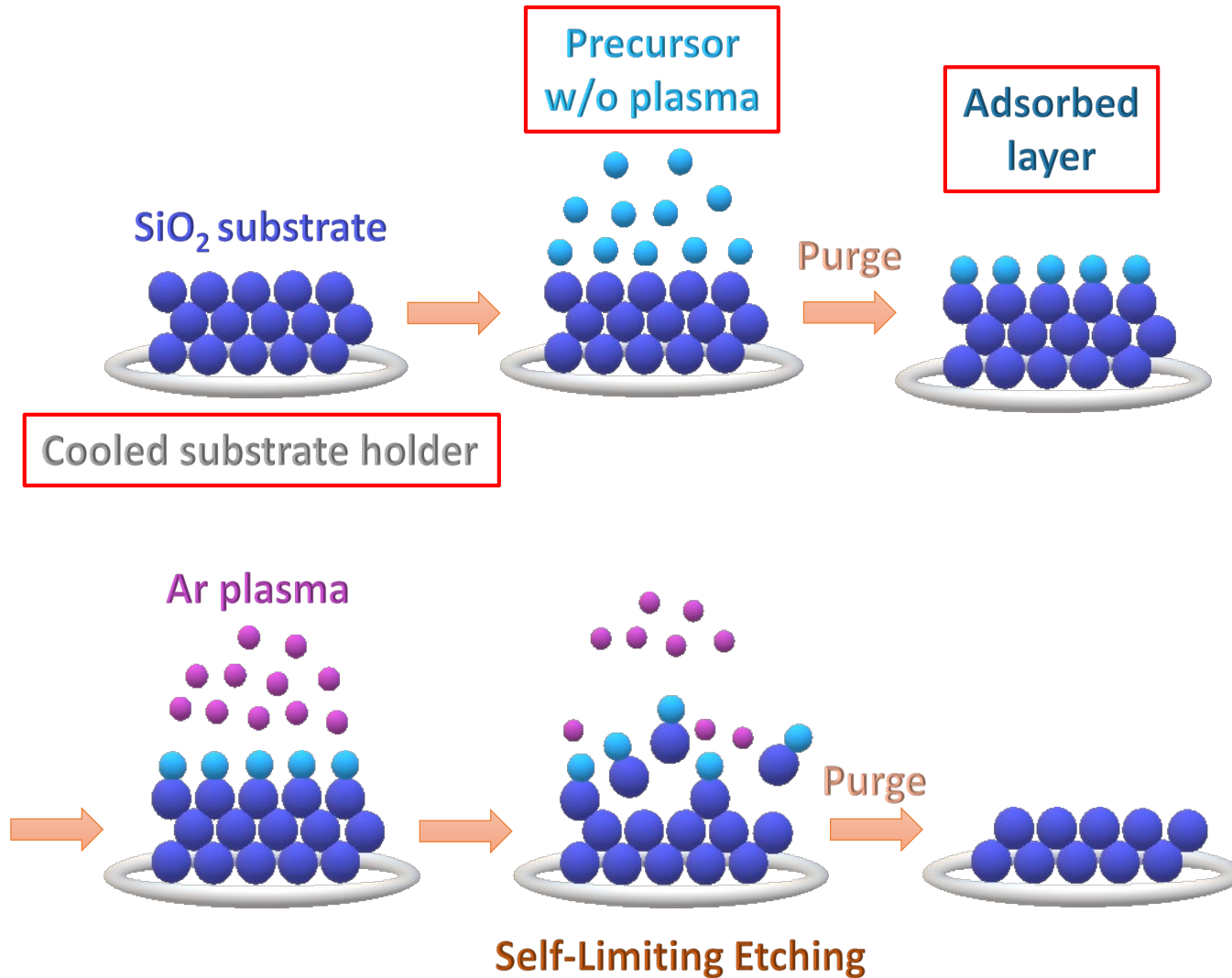


D. Metzler *et al.*, J. Vac. Sci. Technol. Vac. Surf. Films **32**, 020603 (2014)



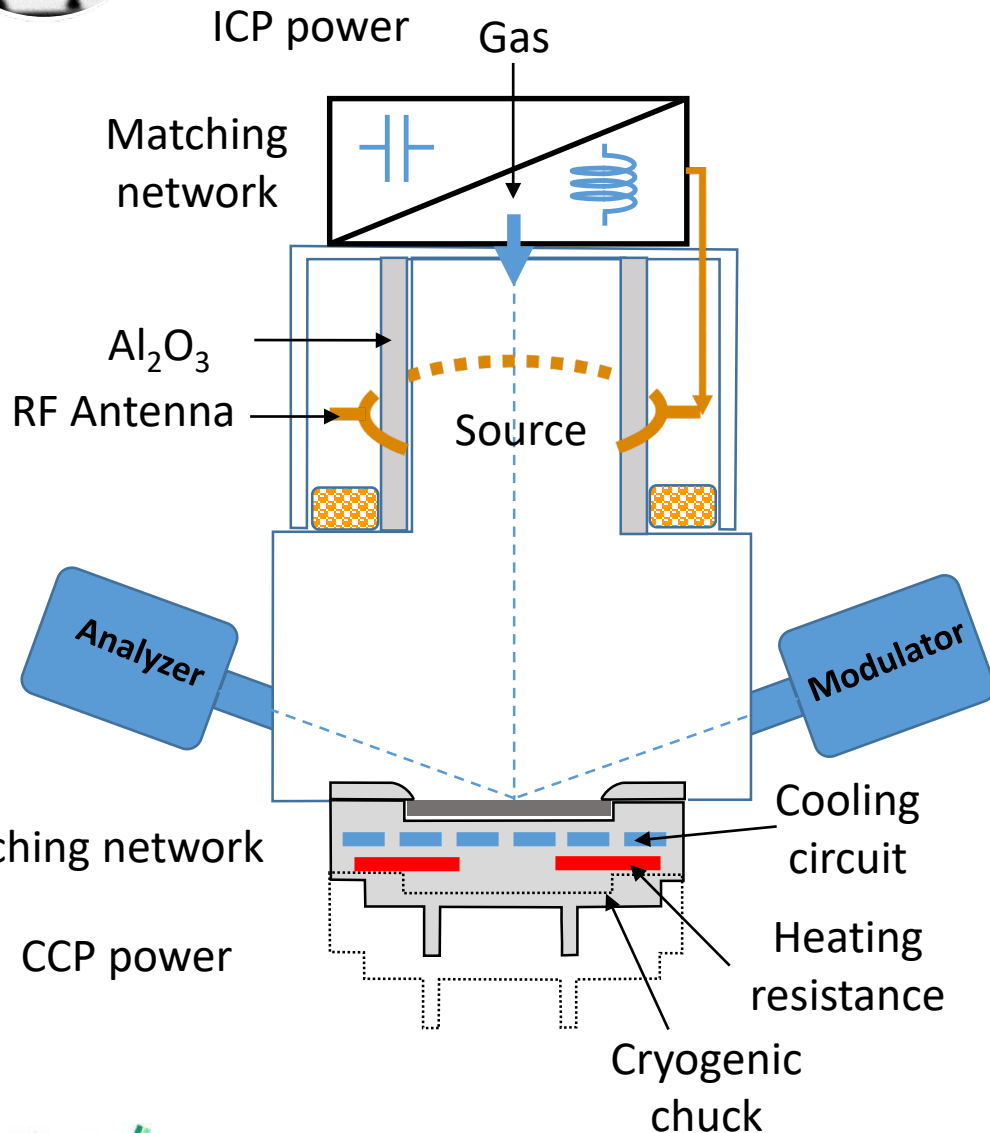


Principle of Cryo-Atomic Layer Etching for SiO₂





Inductively Coupled Plasma (ICP) reactor

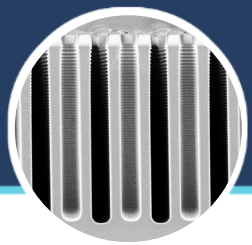


Parameter range:

- Fast ALD valves for C₄F₈ gas injection
- Pressure : 1 – 10 Pa
- Power : 500 – 3000 W
- Bias : 0 – 100 V
- C₄F₈ : 0 – 14 sccm
- Ar : 0 – 280 sccm
- Temperature : -150 – 30 °C

Diagnostic:

- In-situ ellipsometry
- Mass spectrometry



Principle of Cryo-Atomic Layer Etching for SiO₂

T < -80°C

~~Precursor in vapor phase~~

Adsorbed layer

Ar plasma

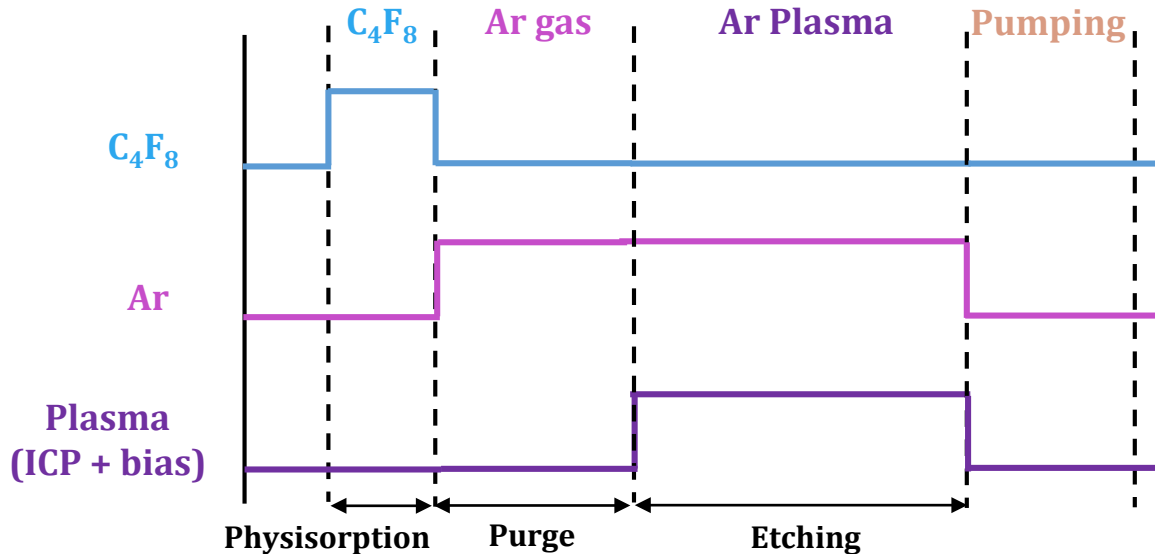
Self-Limiting Etching

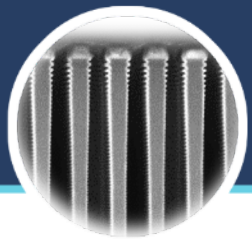
SiO₂ substrate

Purge

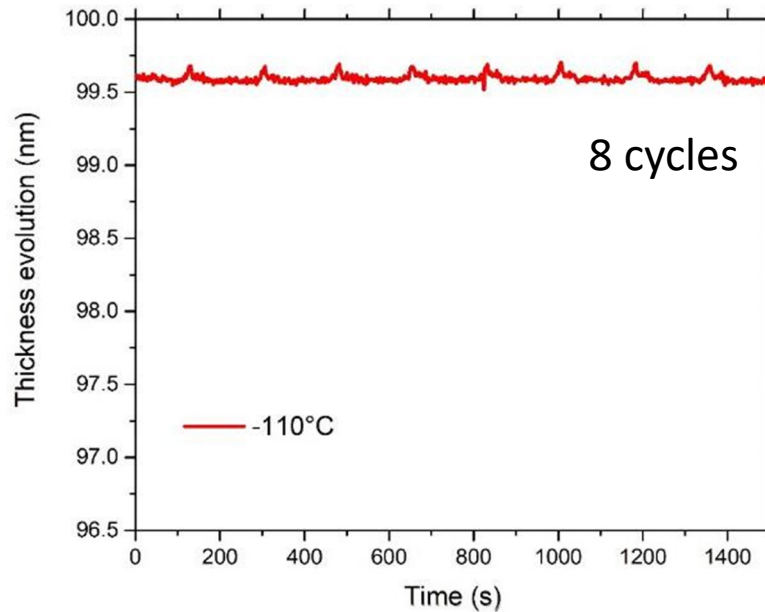
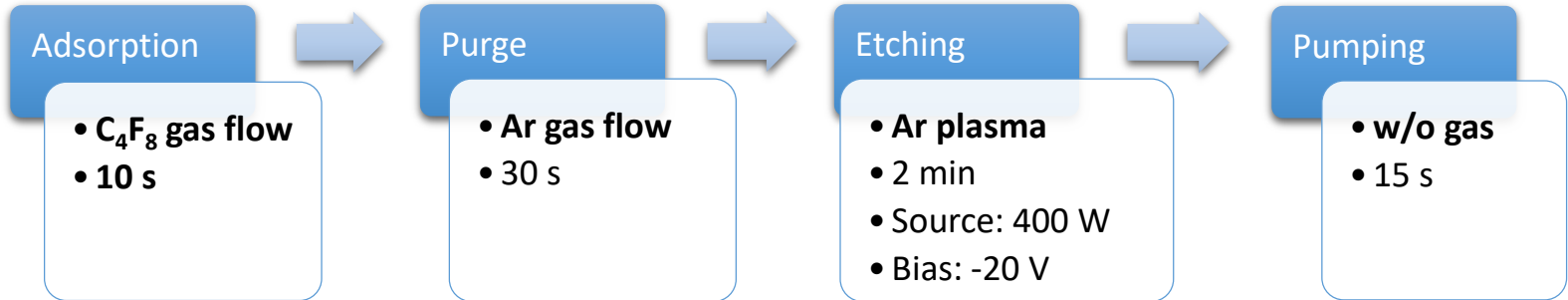
Purge

Cooled substrate holder





Proof of principle

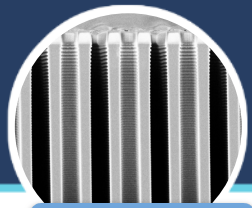


EPC < 0.5 nm/cycle

No etching at -110 °C (and higher temperatures)

G.Antoun et al, Appl. Phys. Lett. 115, 153109 (2019)

Cryo-ALE of SiO₂ based on C₄F₈ physisorption



Adsorption

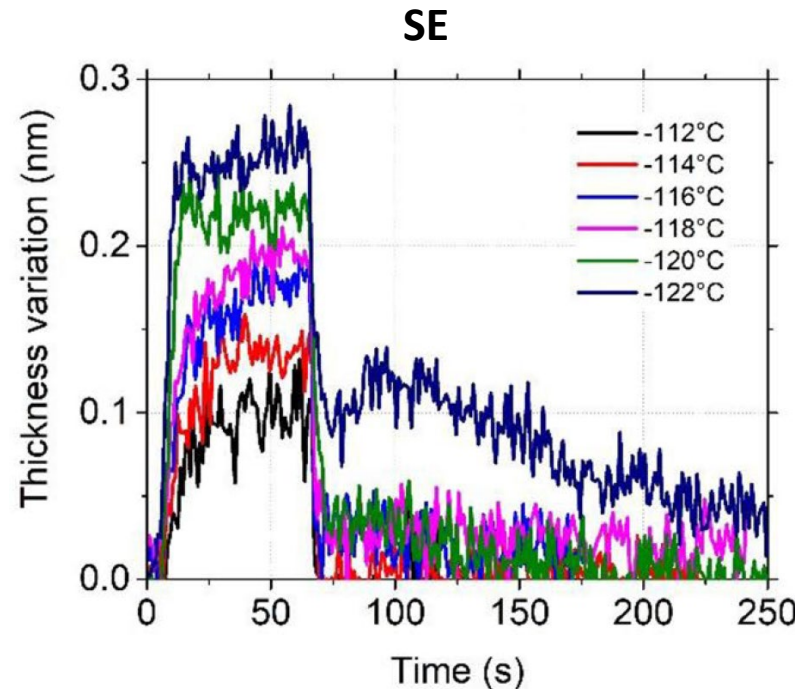
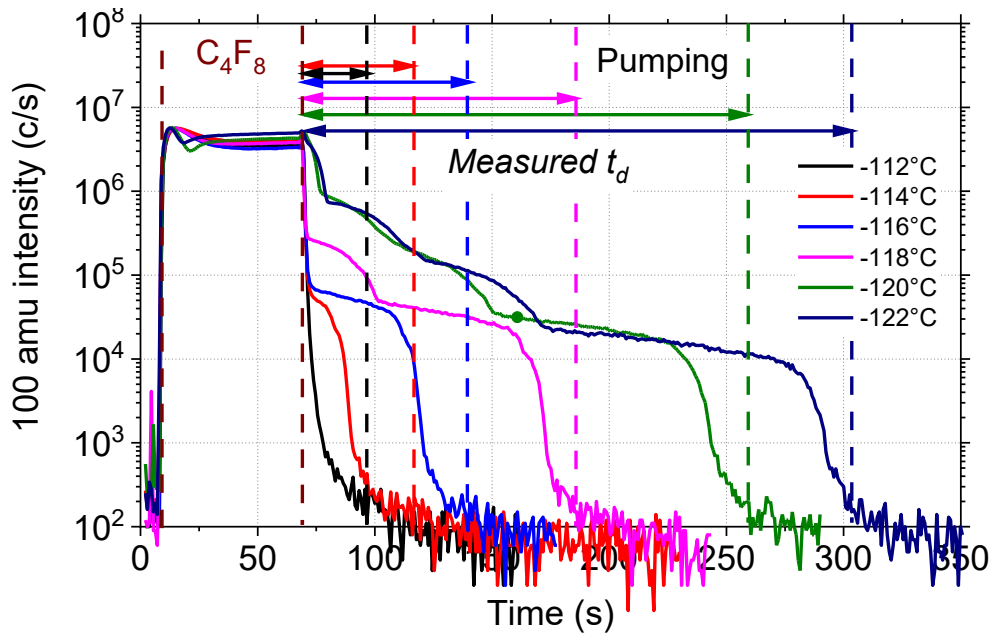
- C₄F₈ gas flow
- 1 min
- 3.0 Pa



Pumping

- w/o gas
- X min

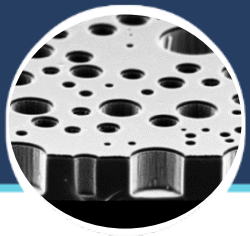
- **100 amu** peak corresponding to C₂F₄⁺ signal was monitored in Multiple Ion Detection (MID) mode by mass spectrometry
- Adsorption of C₄F₈ on SiO₂ was monitored by ellipsometry



At constant pressure, by decreasing temperature:

- Several desorption rates are observed

- Desorption rate \searrow
- Residence time on the substrate surface \nearrow



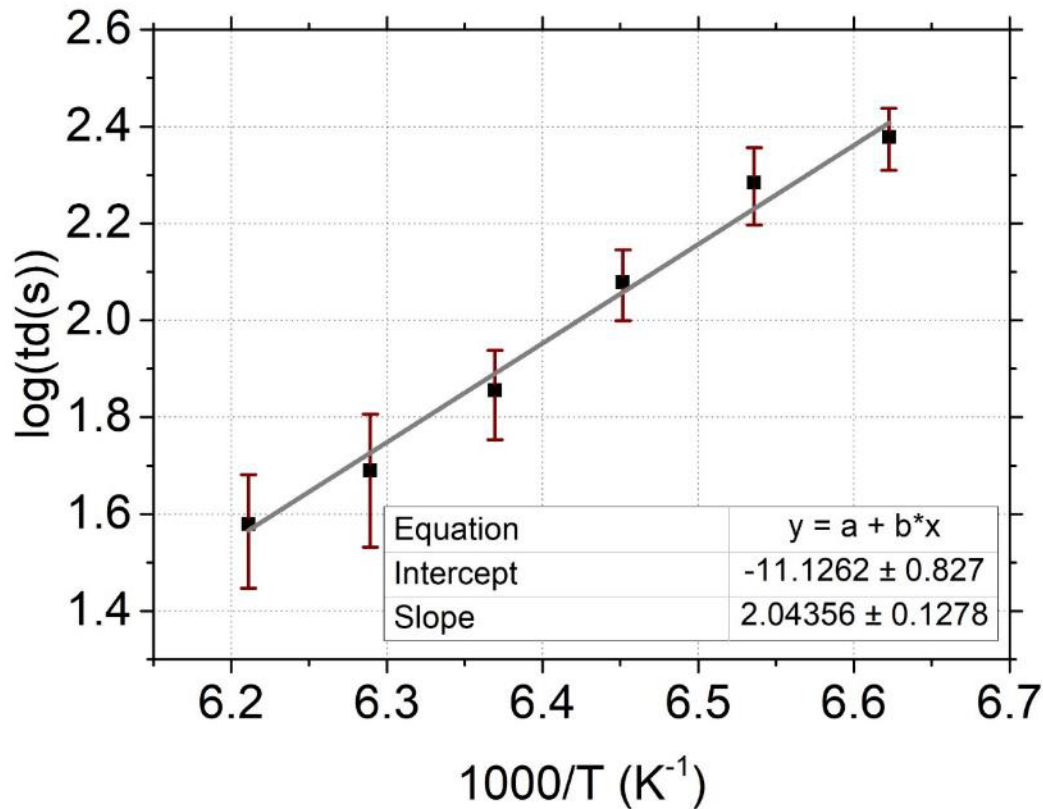
Desorption rate

$$t_d = t_d^0 \exp^{E_d/k_B T}$$

t_d : residence time (s)

t_d^0 : attempt time of the particle for desorption (s)

E_d : desorption energy (eV)

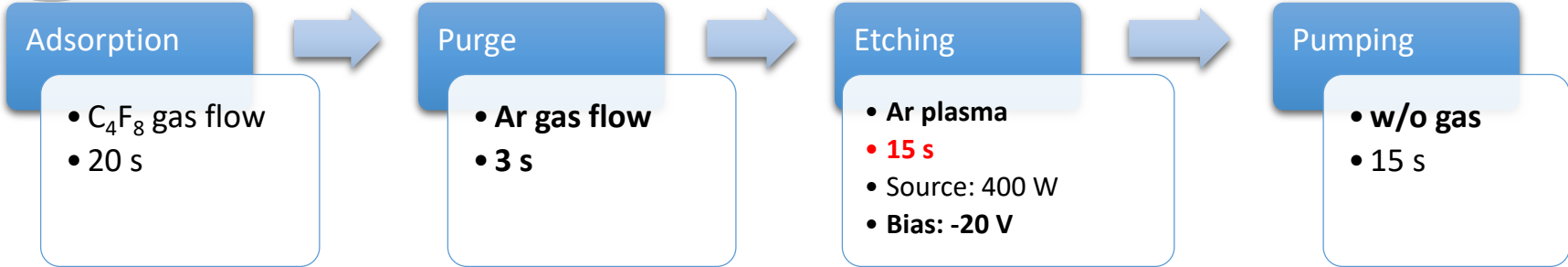


$$t_d^0 = 1.0 \times 10^{-11} \text{ s}$$

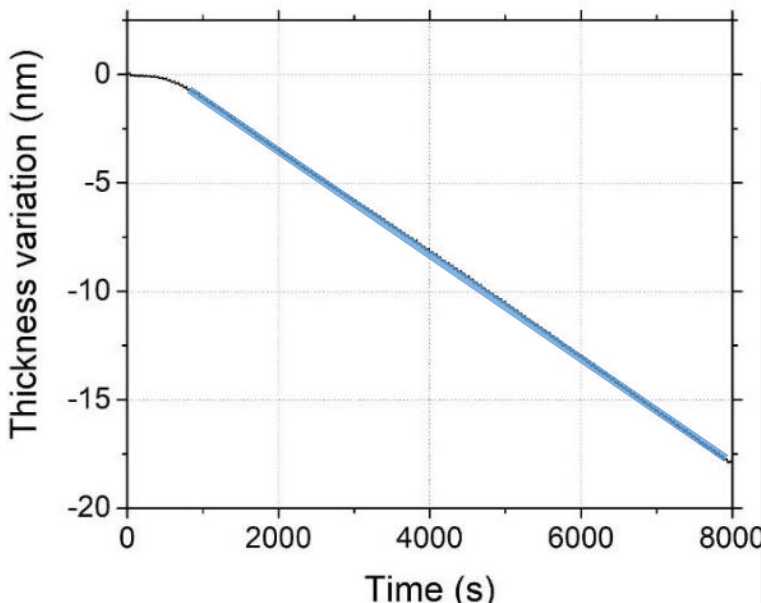
$$E_d = 0.404 \text{ eV.}$$



Cryo-ALE of SiO₂ based on C₄F₈ physisorption at higher temperature



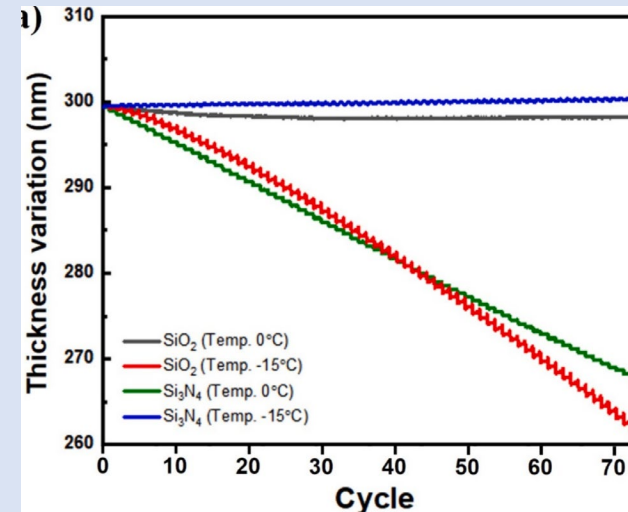
150 cycles at -90°C



- No drift observed due to reactor wall contamination
- ≈ 18 nm etched in 150 cycles

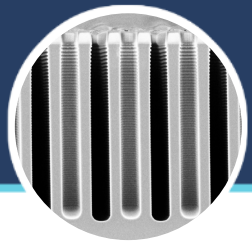
Sungkyunkwan University (South Korea)

Same type of experiments with C₆F₆ which adsorbs at higher temperature. High etch selectivity obtained between SiO₂ and Si₃N₄.



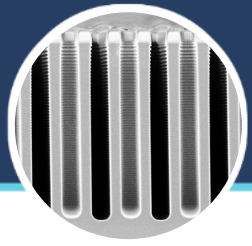
G. Antoun et al, Scientific Reports | (2021) 11:357

D I Sung et al Applied Surface Science 670 (2024) 160574

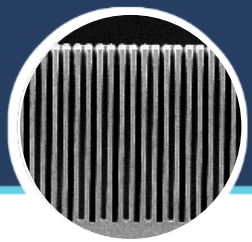


Conclusion on physisorption based cryo ALE

- // **Cryo Atomic Layer Etching of SiO_2** was shown by **physisorbing C_4F_8** at the surface at **-120°C** followed by Ar plasma.
- // A **threshold temperature** has to be reached to enable cryo-ALE.
- // **Residence time of physisorbed species** can be determined by mass spectrometry.
- // **Ar plasma** has to be ignited before the end of the residence time.
- // Reactor wall **contamination** is significantly **reduced**. **The etch per cycle** remains quite **regular**, even after many cycles.
- // **Self-Limiting Etching** is achieved.

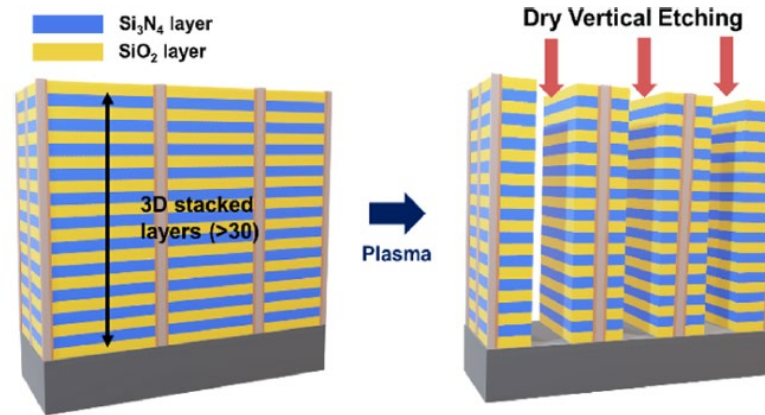


What makes cryogenic etching popular again in the industry ?



SiO₂ and Si₃N₄ deep etching

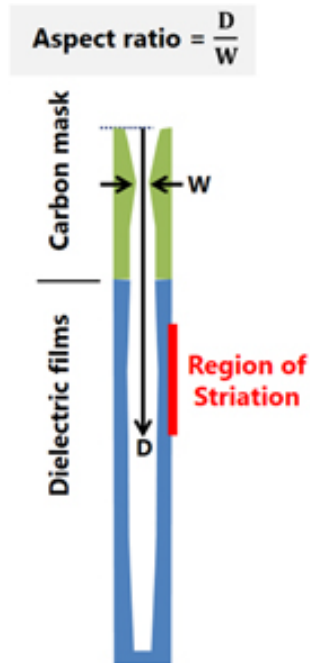
- // Need to etch very high aspect ratio holes on Si₃N₄ and SiO₂ for 3D NAND technology.

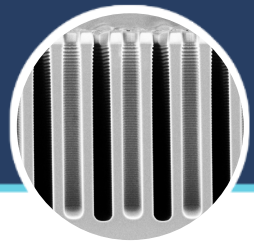


From H.I. Lee et al. ACS Sustainable Chem. Eng. 9, 4948, 2021

- // Usually, CF-based plasmas (C₄F₈, CHF₃...) are used to etch SiO₂ and Si₃N₄ at room temperature.
- // However, some deposition occurs on the carbon mask which leads to striation on the dielectric film
- // This aperture reduction increases the ARDE effect.

Mitsuhiro Omura et al 2019
Jpn. J. Appl. Phys. 58 SEEB02





Why using cryoetching ?

// Increase of etch rate of SiO_2 at low T in CHF_3 plasma.

Conditions :

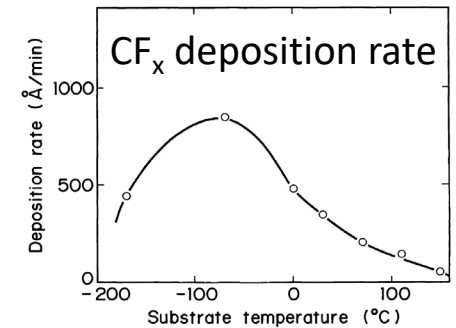
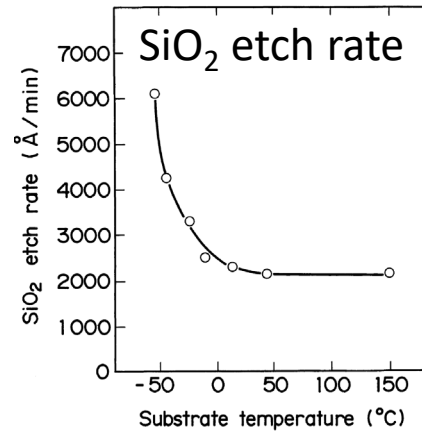
CHF_3 gas

Magnetron RIE system

$P = 40$ mTorr

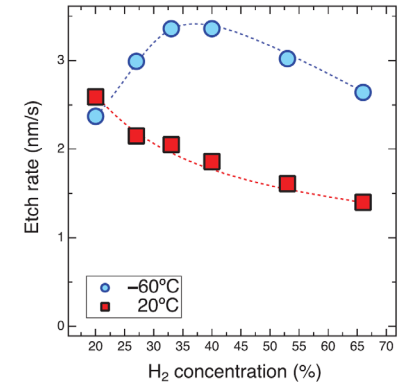
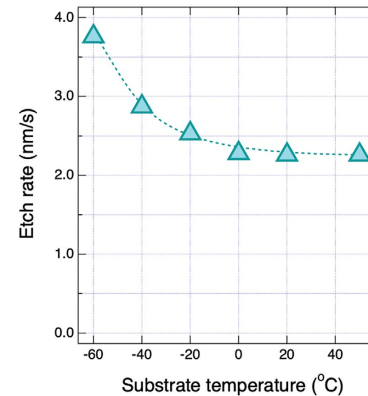
RF power density : 1.4 W/cm^2

T. Ohiwa, et al. Jpn. J. Appl. Phys.31. 405-410(1992)

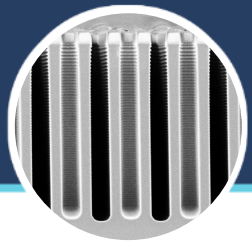


// CF_4/H_2 plasma cryoetching : etch rate increases by adding H_2

// They showed by in-situ FTIR that HF was forming at the surface at low temperature



S-N. Hsiao et al. Small Methods 2400090 (2024)

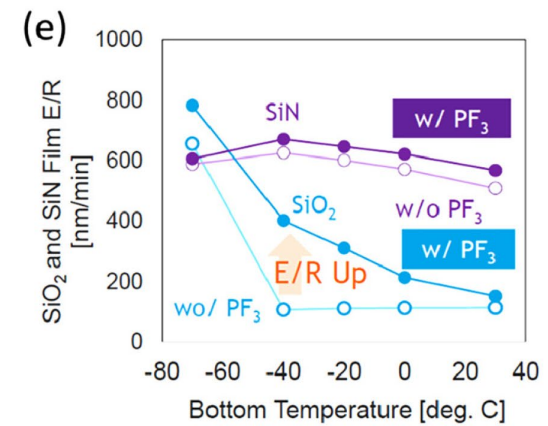
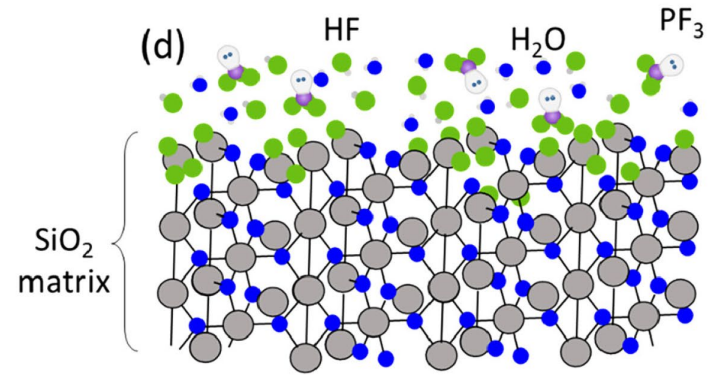
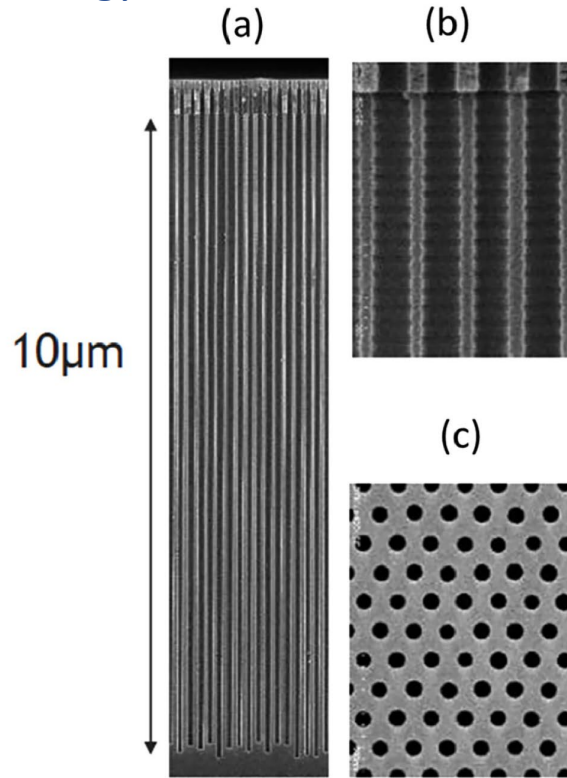


High aspect ratio holes in Si₃N₄/SiO₂ stacked layers

// Pure HF and H₂O plasma mixture can be used to etch SiO₂ and Si₃N₄ at low temperature (below -40°C) with high energy ions.



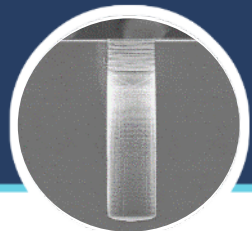
84% Reduction of Global Warming Potential



Y. Kihara et al. 2023 IEEE Symposium on VLSI Technology and Circuits (VLSI Technology and Circuits), Kyoto, Japan, 1-2 (2023).

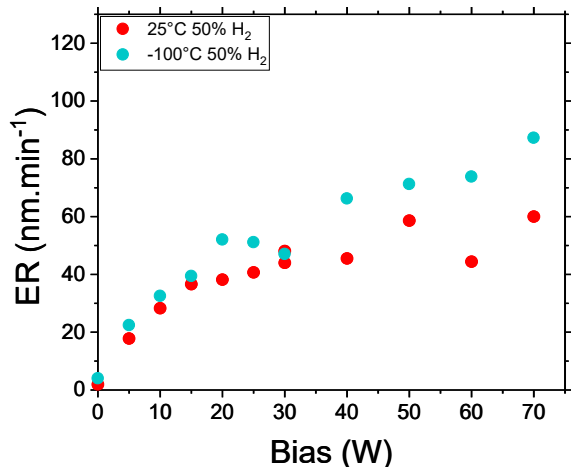
G. S. Oehrlein et al., "Future of plasma etching for microelectronics: Challenges and opportunities" JVST. B 42, 041501 (2024)



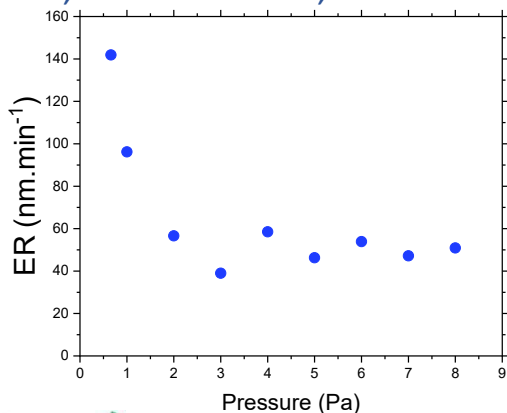


SiO₂ cryoetching in SF₆/H₂ plasma

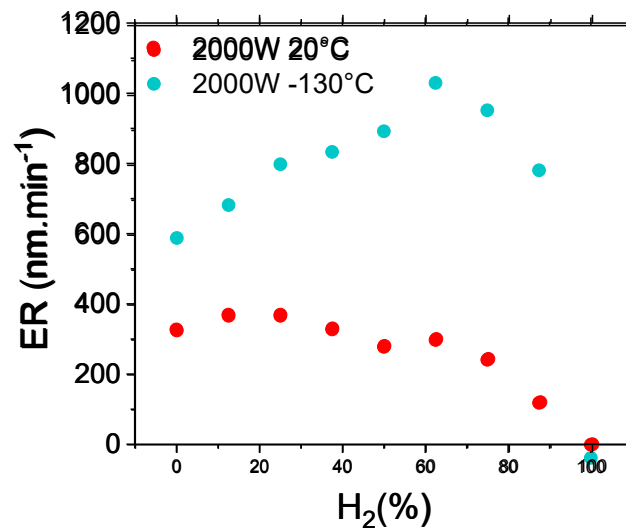
// SiO₂ cryoetching versus bias power
(1,6 Pa, 1000 W source)



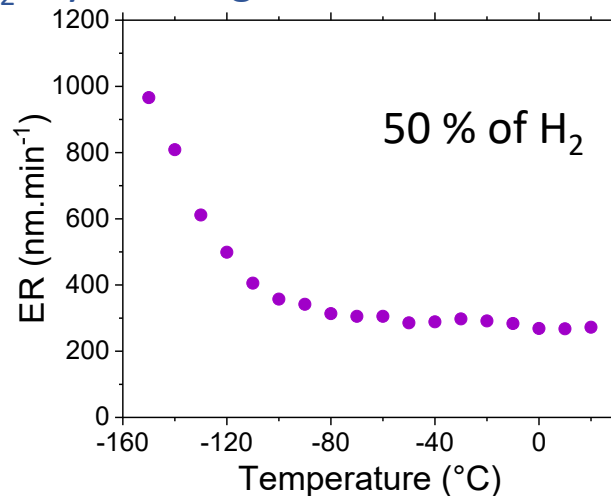
// SiO₂ cryoetching versus pressure
(-100°C, 1000W source, 150 W bias)

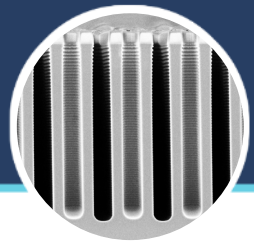


// SiO₂ cryoetching versus H₂ % (0,6 Pa, 150 W bias)



// SiO₂ cryoetching versus T (0,6 Pa, 150 W bias)





What are the advantages of cooling the substrate ?

- // In terms of physical and chemical mechanisms, cooling the substrate can :
 - // Increase the **residence time** of physisorbed species
 - // **Promote chemical reactions** at the surface
 - // **Modify the stoichiometry** of the deposited layer
 - // Create a mix between **physisorbed** and **chemisorbed** species at the surface
 - // Limit surface diffusion
- // In terms of process, cooling the substrate can:
 - // **Avoid contamination** of the reactor walls
 - // Increase the **etch rate**
 - // Increase the **selectivity**
 - // **Protect porous material** during etching



PlaCEP workshop (Plasma Cryogenic Etching Processes)

- 1st edition in 2022 : Orléans (France)
- 2nd edition in 2024 : Leuven (Belgium)
- 3rd edition in 2025 : *New Taipei (Taiwan)*

The **3rd PlaCEP Workshop** will take place at **Ming Chi University of Technology** in **New Taipei City, Taiwan** in **June 25-28, 2025**

organized within the International Plasma Technology Joint Conference 2025 (IPTJC-2025)

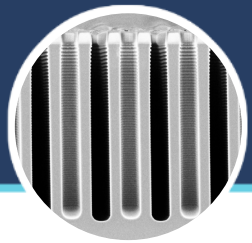
Cryogenic etching processes of silicon, dielectrics, cryo-ALE, modeling and simulation



IPTJC2025
The International Plasma Technology Joint Conference 2025

June 25 to 28, 2025 Ming Chi University of Technology, New Taipei, Taiwan





Acknowledgment

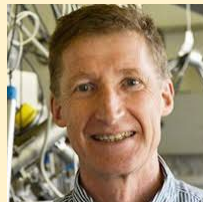
Larry
Overzet
(UT Dallas)



Rim Ettouri



Christophe Cardinaud



Aurélie Girard



IMN, Nantes

Gaelle Antoun



Emilie
Despiau-Pujo



Gilles Cunge

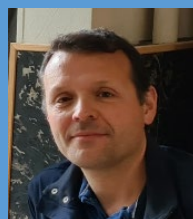


Martin Kogelschatz



LTM Grenoble

Thomas
Tillocher



Philippe
Lefauchaux



Loic Becerra



Sylvain
Iséni



Arnaud Stolz



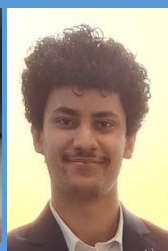
Jack Nos



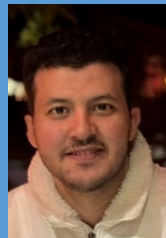
Lamiae
Hamraoui



Madjid
Adjabi



Ayoub
Rhallali

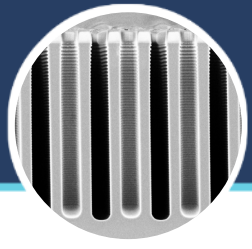


Elane
Kouadou



Baptiste
Disson





Thank you !