

Cryogravure plasma

T. Tillocher¹

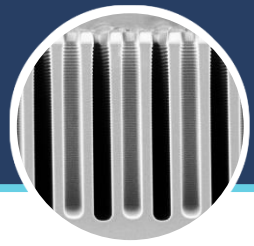
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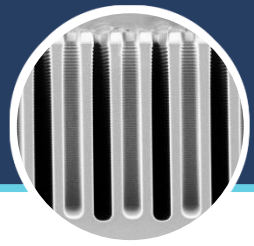
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Outline

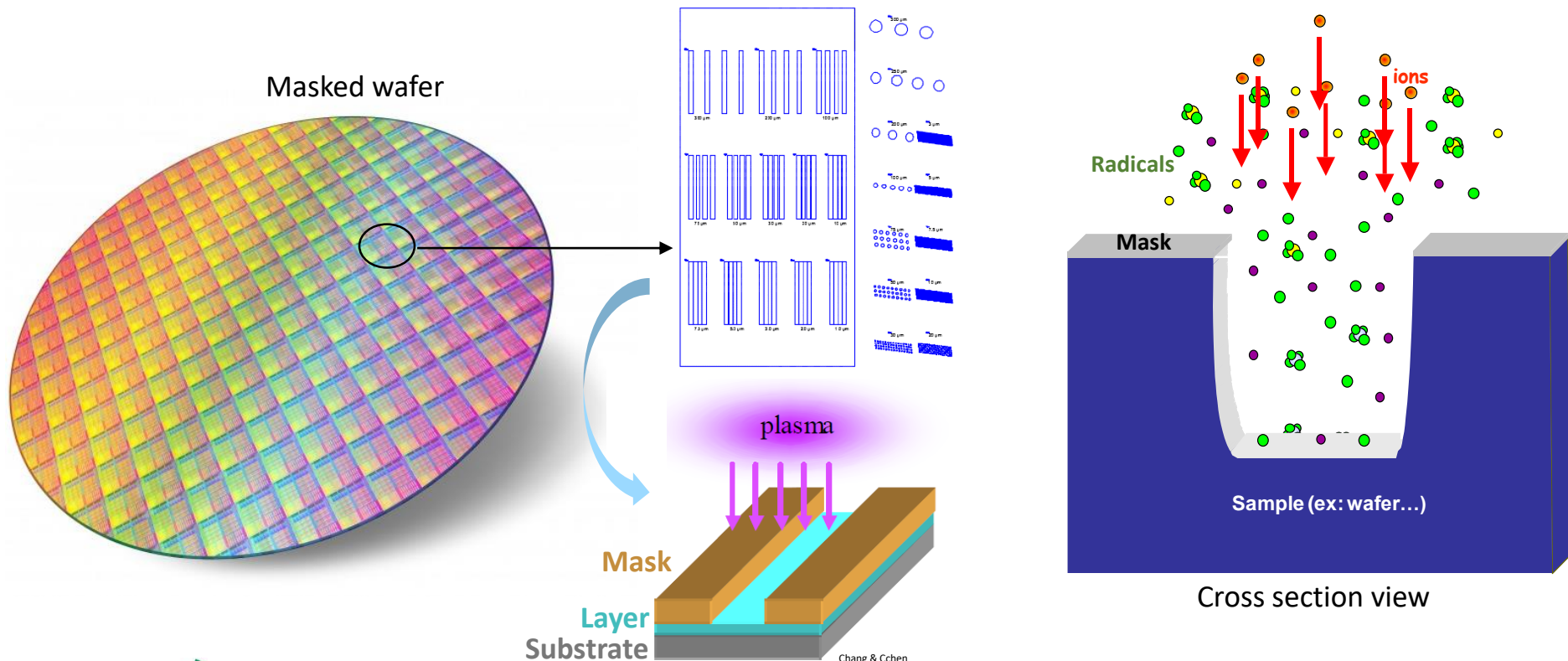
- 1) Basics of plasma etching
- 2) Deep cryoetching of silicon
- 3) Cryogenic etching process



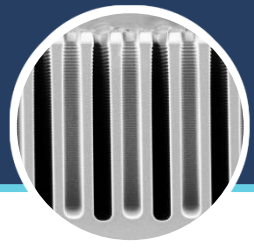
1) Basics of plasma etching

Quick definition

Plasma etching consists in using a plasma to **transfer** a **mask pattern** in a layer or the substrate itself
=> **physico-chemical interaction** between the plasma and material to remove the material



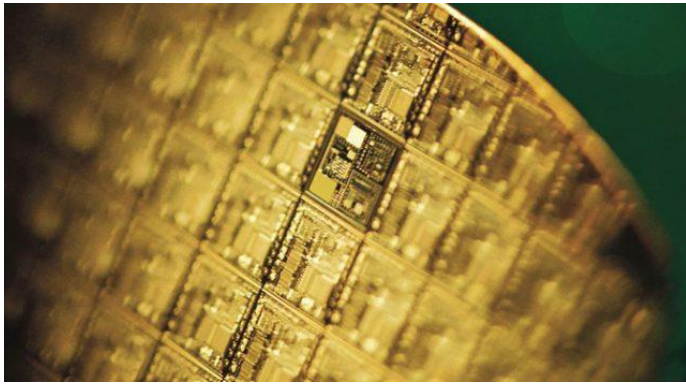
Chang & Cchen



1) Basics of plasma etching

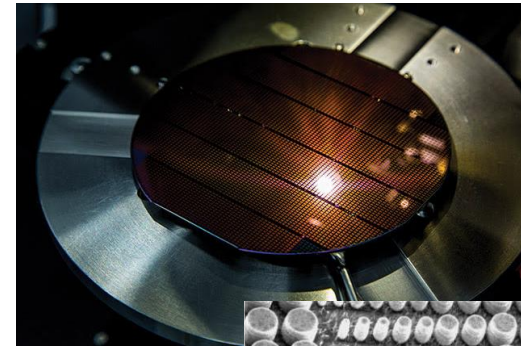
Fields of application (very general)

Microelectronics



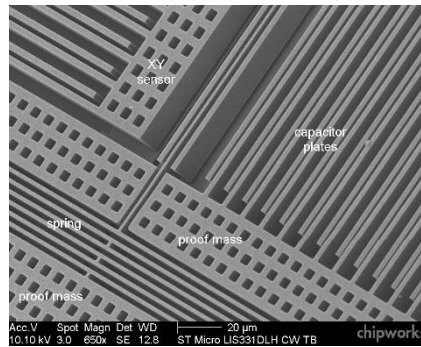
<https://www.extremetech.com>

Optoelectronics



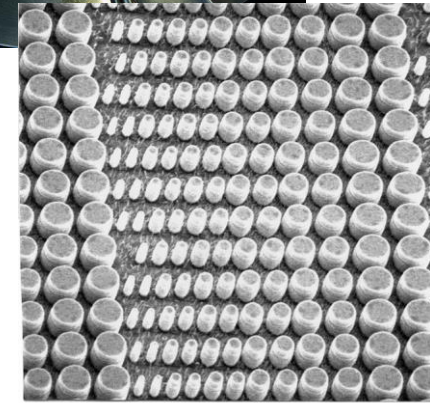
<https://www.photonics.com>

Microsystems – MEMS

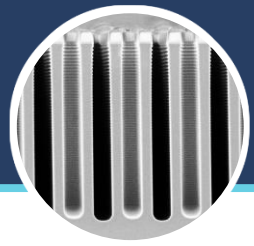


Acc: 10.10 kV Spot: 3.0 Magn: 650x Det: SE WD: 12.8 20 µm
ST Micro LIS31DLH CW TB chipworks

<https://www.memsjournal.com>



10 µm



1) Basics of plasma etching

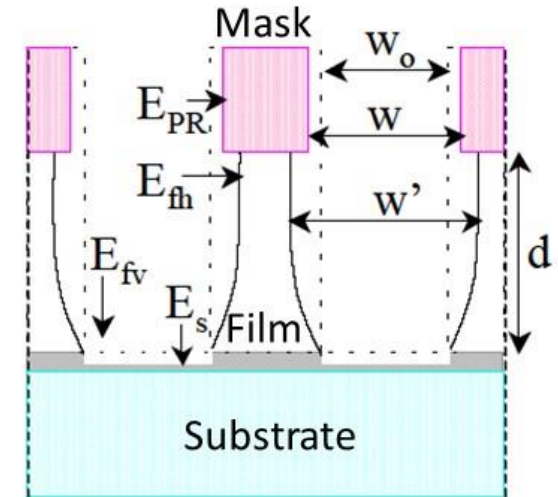
Criteria and profile shape

Parameters characterizing an etched profile => To be optimized

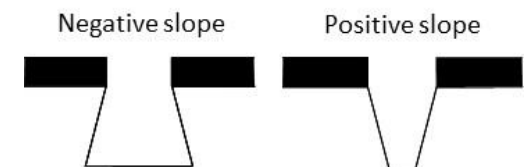
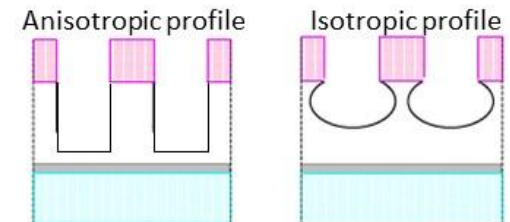
- The **depth** (d), the **critical dimension** (CD, w)
 - The **etch rate**: ER ($\mu\text{m}\cdot\text{min}^{-1}$ or $\text{nm}/\text{min}^{-1}$)
- => Etch rate for all materials exposed to the plasma (layer, mask...)

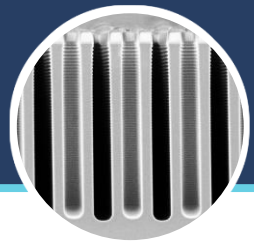
- The **selectivity** S (**critical for choice of mask**)
- => ratio of the etch rate of one material to that of another
- => usually $S = \text{ER}(\text{layer})/\text{ER}(\text{mask})$

- **Aspect ratio**: $\text{AR} = d/w$
- **Anisotropy**: is the etch directive?
- **Slope** of profile: is it tapered?



Chang & Cchen





1) Basics of plasma etching

Etching mechanisms in low pressure plasmas

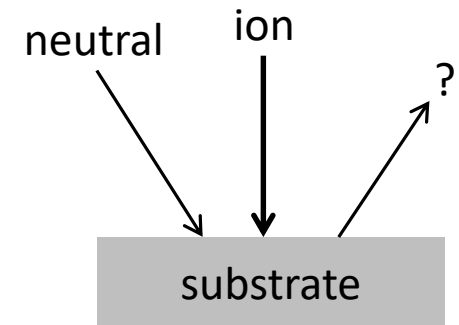
- In a plasma, both **radicals** and **ions** are involved in the etch process

① Chemical etching (spontaneous):

Adsorption, chemical reaction between radicals and the surface and desorption of etch product

② Physical etching:

Ion bombardment at the surface

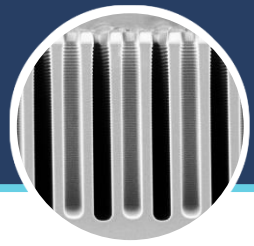


- In an etch process, these 2 mechanisms happen at the same time and more...

③ « Ion energy driven etching » : combination of mechanisms 1 and 2

④ « Ion-enhanced inhibitor etching » :

Same as before + passivation layer at the sidewalls



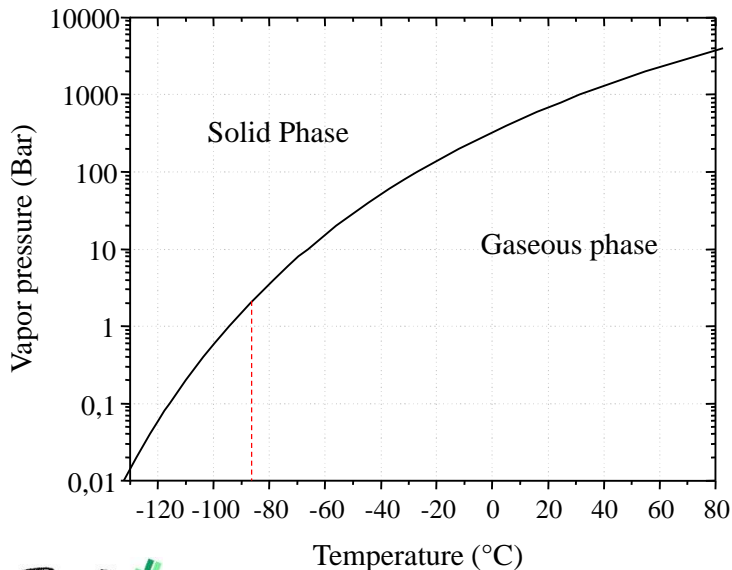
1) Basics of plasma etching

Chemical etching

The chemical etching of a material is effective if two conditions are met simultaneously

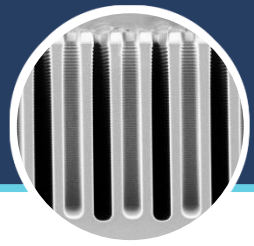
- ① Formation of a **thermodynamically stable** reaction product
- ② The etch product must be **volatile**

- Example of **silicon etching** by F-based plasma: main etch product = SiF_4 => Volatility?



- At low pressure and room temperature of the substrate, SiF_4 is in **gas phase**

- Same trend even at low temperature



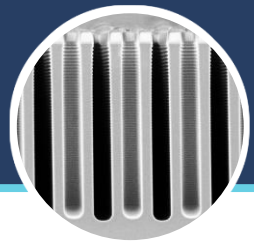
1) Basics of plasma etching

Chemical etching

- Standard plasma etch chemistries

Material	Radicals	Gases	Main Etch products
Si, Ge	F, Cl, Br	SF ₆ , Cl ₂ , HBr...	SiF ₄ , SiCl ₄ , SiBr ₄
SiO ₂	F, F + C	CHF ₃ , CF ₄ , C ₄ F ₈ ...	SiF ₄ , CO ₂ ...
Si ₃ N ₄	F, F + C	SF ₆ , CF ₄ , CHF ₃ ...	SiF ₄ , N ₂ ...
Al	Cl, (Br)	Cl ₂ , (HBr)	AlCl ₃ , (AlBr ₃)
Cu	Cl (T > 210°C)	Cl ₂	Cu ₃ Cl ₃
C, polymers	O	O ₂	CO ₂
W, Ta, Ti, Mo, Nb	F, Cl	SF ₆ , Cl ₂ ...	XF ₄ , XCl ₄
GaAs, III-V	Cl	Cl ₂ , BCl ₃ ...	GaCl ₃ , AsF ₃

⇒ No very effective chemistry for some materials (e.g. Au, Pt)

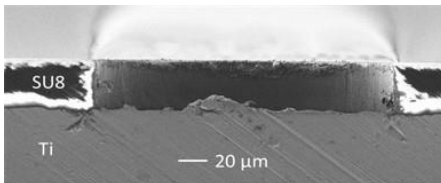


1) Basics of plasma etching

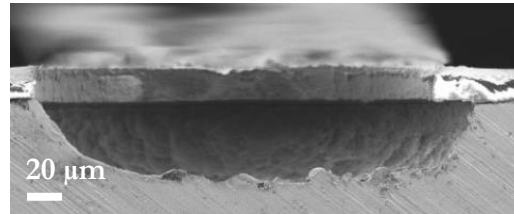
Chemical etching

- Effect of volatility of etch product: example of **titanium etched by SF₆ plasma**

Room temperature – 30 min

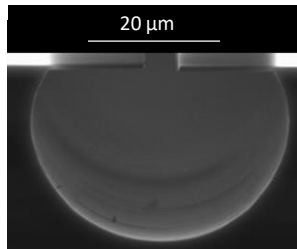


Heated surface- 30 min

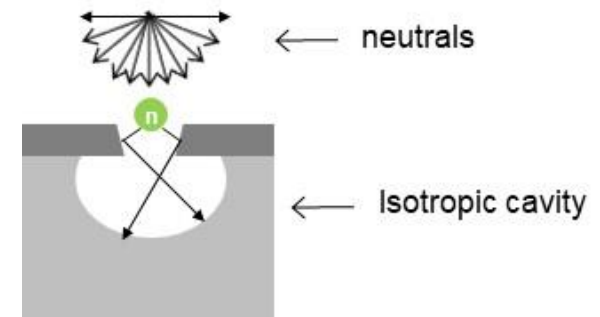


*Bulk Ti sample
SU8 mask*

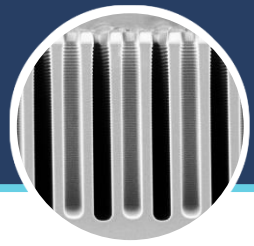
- The main etch product TiF₄ is not volatile at room temperature => heating necessary
- Silicon etching by SF₆ plasma: SiF₄ is volatile over a wide range of temperature



*Bulk Si sample
SiO₂ mask
Room temperature*



=> Pure chemical etching leads to isotropic profiles



1) Basics of plasma etching

“Physical” etching / role of ion bombardment

Ion-surface interaction: principle

- A plasma contains **positive ions** which obviously participate in the etching

⇒ The interaction depends on their energy

- Effects of ion bombardment on **adsorption**:

⇒ **Adsorption activated by induced desorption**

⇒ Adsorption induced by surface damage

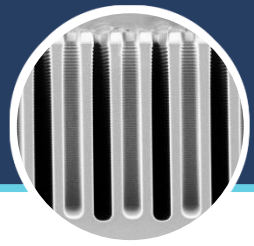
⇒ Incorporation into the material

} Less significant role

- Effects of ion bombardment on **desorption**:

⇒ **Induced desorption**

⇒ Sputtering (physical etching) => requires ion energy > sputtering threshold



1) Basics of plasma etching

“Physical” etching / role of ion bombardment

- Under etching conditions, the **energy of the ions** is generally from a few eV to a few hundreds of eV (for the strongest materials)

- Positive ions acquire their energy during the **acceleration in the RF sheath**:

$$E_i = q \cdot (V_p + V_{dc})$$

With Avec V_p the plasma potential and V_{dc} the dc bias

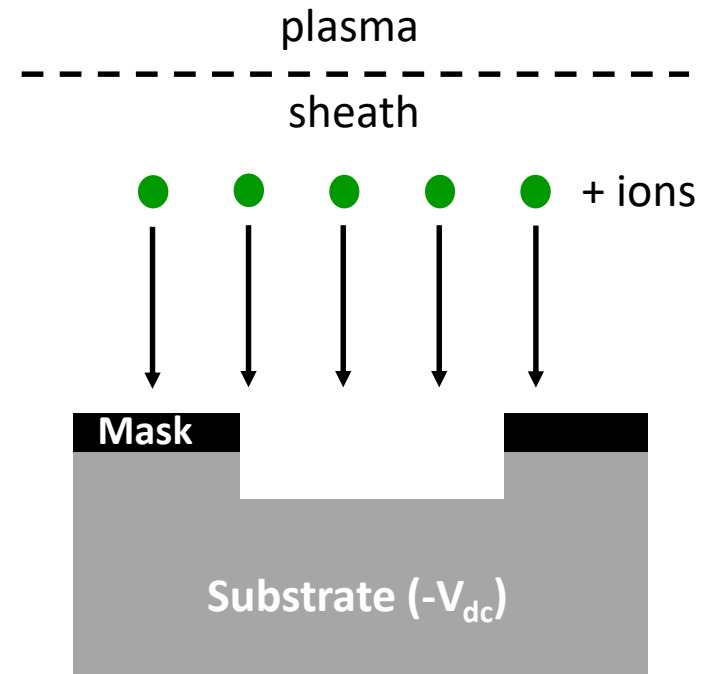
=> Approximation of monoenergetic ions

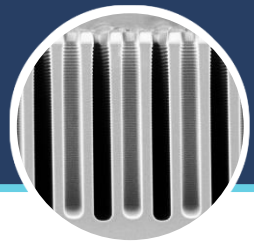
=> In reality, there is an IEDF

- Promotes **anisotropic etching** (normal incidence of ions)

- Mask can be sputtered

⇒ Risk of roughness (micromasking)

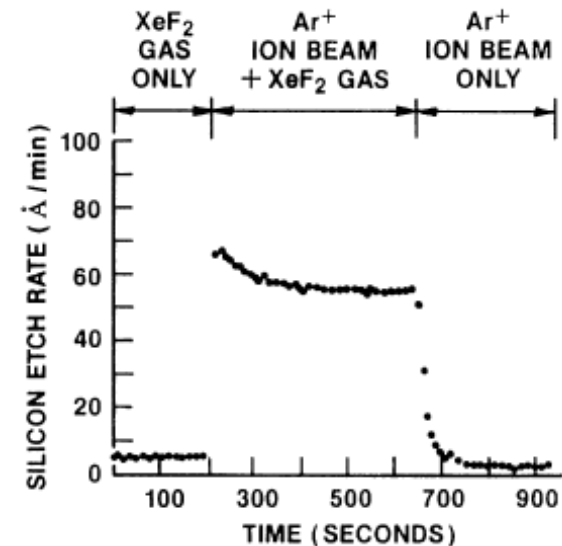
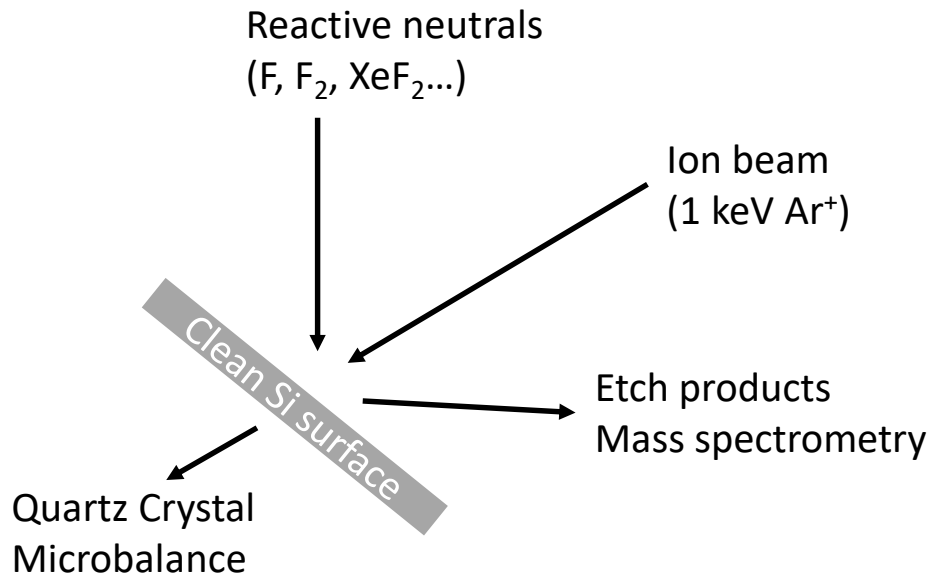




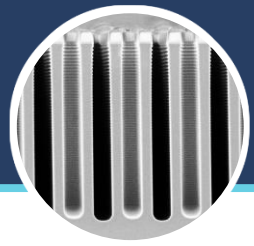
1) Basics of plasma etching

Ion energy-driven etching / Reactive Ion Etching (RIE)

- The combination of chemical etching and sputtering leads to **higher etch rates**
- Demonstrated by Coburn and Winters under specific conditions (1979):



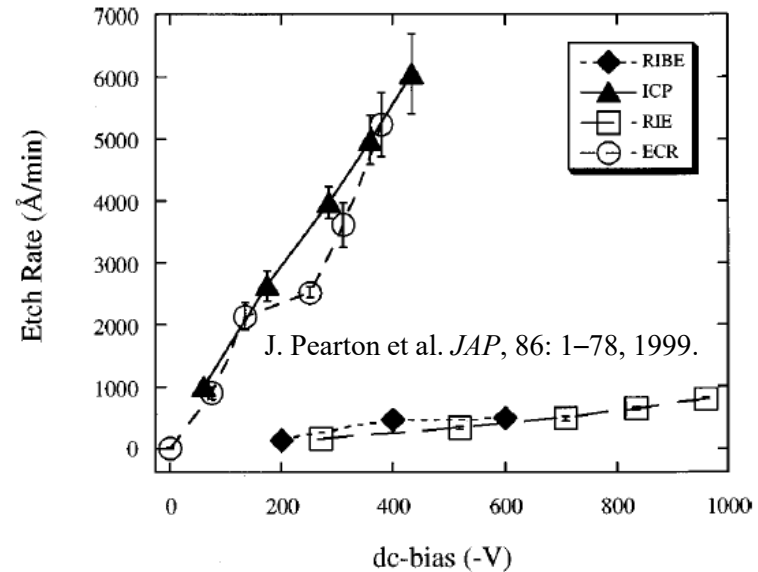
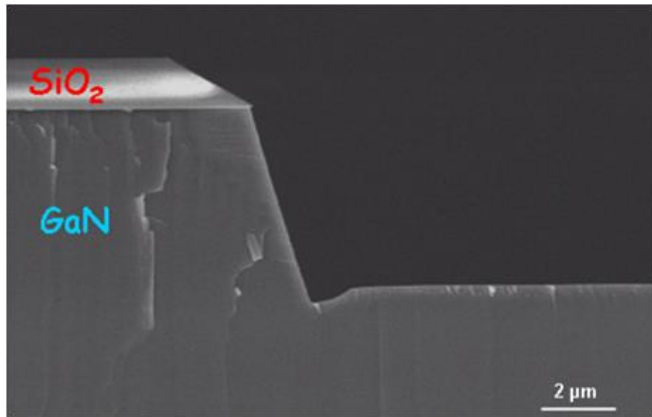
Synergy : $V_g(\text{chemical etching} + \text{sputtering}) > V_g(\text{chemical etching}) + V_g(\text{sputtering})$



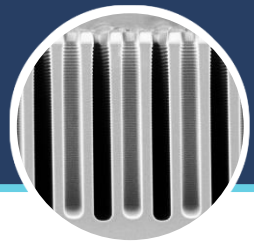
1) Basics of plasma etching

Ion energy-driven etching / Reactive Ion Etching (RIE)

- Case of GaN etching: critical role of ion bombardment (Cl_2/Ar plasma)



- Fluorine plasma chemistry not efficient \Rightarrow **GaF₃ not volatile** in standard etching conditions
- **Chlorine-based chemistry** typically used for III-V etching
 - \Rightarrow GaCl₃ boiling point: 200°C at P_{atm}, NCl₃ boiling point: 79°C at P_{atm}
- GaN is effectively etched only under ion bombardment



1) Basics of plasma etching

Ion-enhanced inhibitor etching / Deep Reactive Ion Etching (DRIE)

Bosch process

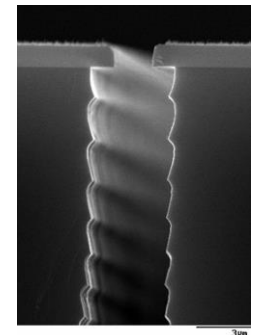
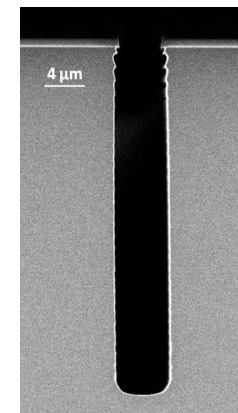
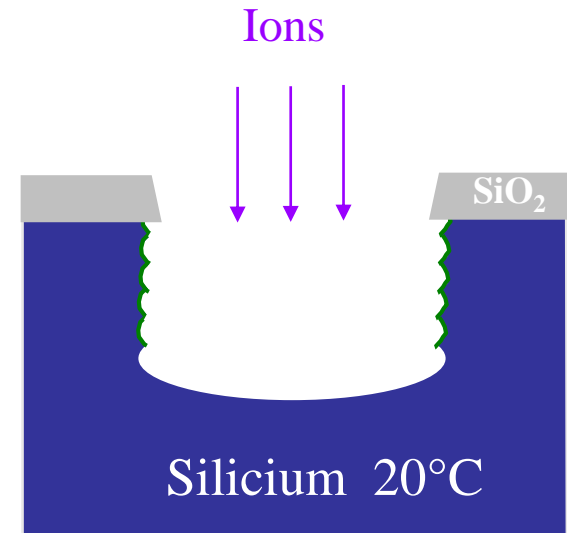
- ✓ Isotropic etching step: SF_6 plasma
- ✓ Passivation step: C_4F_8 plasma
 - ➔ Passivation layer = Fluoropolymer (C_xF_y)
- ✓ The **polymer** at the etch front is **sputtered** by the ion bombardment in the etch step

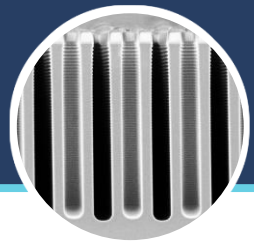
Advantages

- Process at **room temperature**
- **Robust** process

Drawbacks

- « **Scalloping** » at the sidewalls
- Decrease of etch rate because of passivation steps
- Removal of polymer by O_2 plasma





1) Basics of plasma etching

Cryogenic etching

Plasma etching with **substrate temperature $< -90^{\circ}\text{C}$**

=> Use of **liquid nitrogen**

=> specific design of the substrate holder

- First question: **Can we etch at such low temperature?**

=> **YES**, if the etch product is still volatile

=> Case of silicon compounds etched with F-based plasmas



- Second question: **Why etching at such low temperature?**

=> Etching of high aspect ratio profiles (deep silicon etching)

=> Part 2

=> Specific mechanisms at cryo temperature (passivation)

=> Part 2

=> Low damage etching and low contamination of reactor

=> Part 3



2) Deep cryoetching of silicon

Brief history of cryoetching

At **room temperature**, high etch rate with SF_6 plasmas, but anisotropic etching was not possible without adding another gas (CF_4/O_2 , SF_6/CHF_3 , ...)

Tachi's team proposed to cool the substrate down to a temperature between **-100** and **-130°C** while running a **microwave SF_6 plasma**.

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.

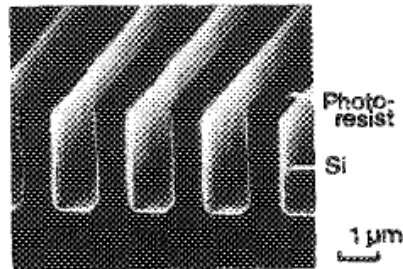
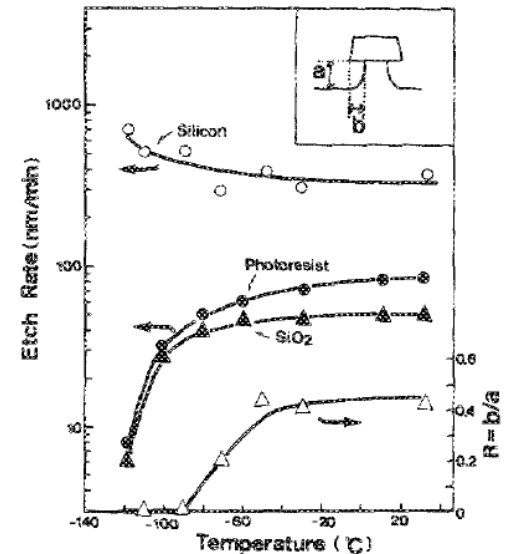


FIG. 3. Silicon profile etched at ... with the use of SF_6 gas plasma.



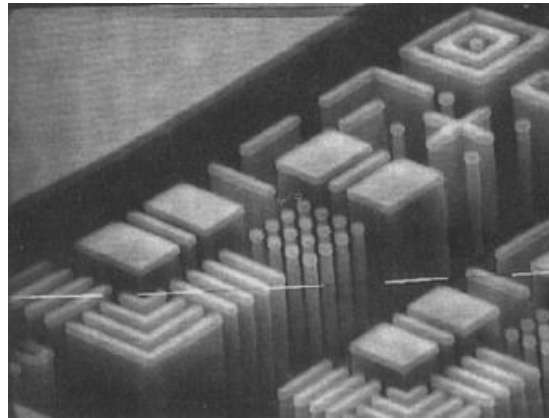
performed with high selectivities of 30 for organic resist films. High etch rates of 500 and 1000 nm/min by reactive ion etching and microwave plasma etching, respectively, were achieved with a SF_6 gas plasma at low wafer temperatures from -130 to -100 °C. It is concluded that



2) Deep cryoetching of silicon

Brief history of cryoetching

- **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.

- **1997** : Beginning of cryogenic etching related activities at GREMI (initiated with STMicr.Tours)



2) Deep cryoetching of silicon

Principle of the standard cryogenic process

Monocyclic SF_6/O_2 plasma

✓ **Chemical etching** (selective)

SiF_4 : main etch product

✓ **Passivation layer** (SiO_xF_y)

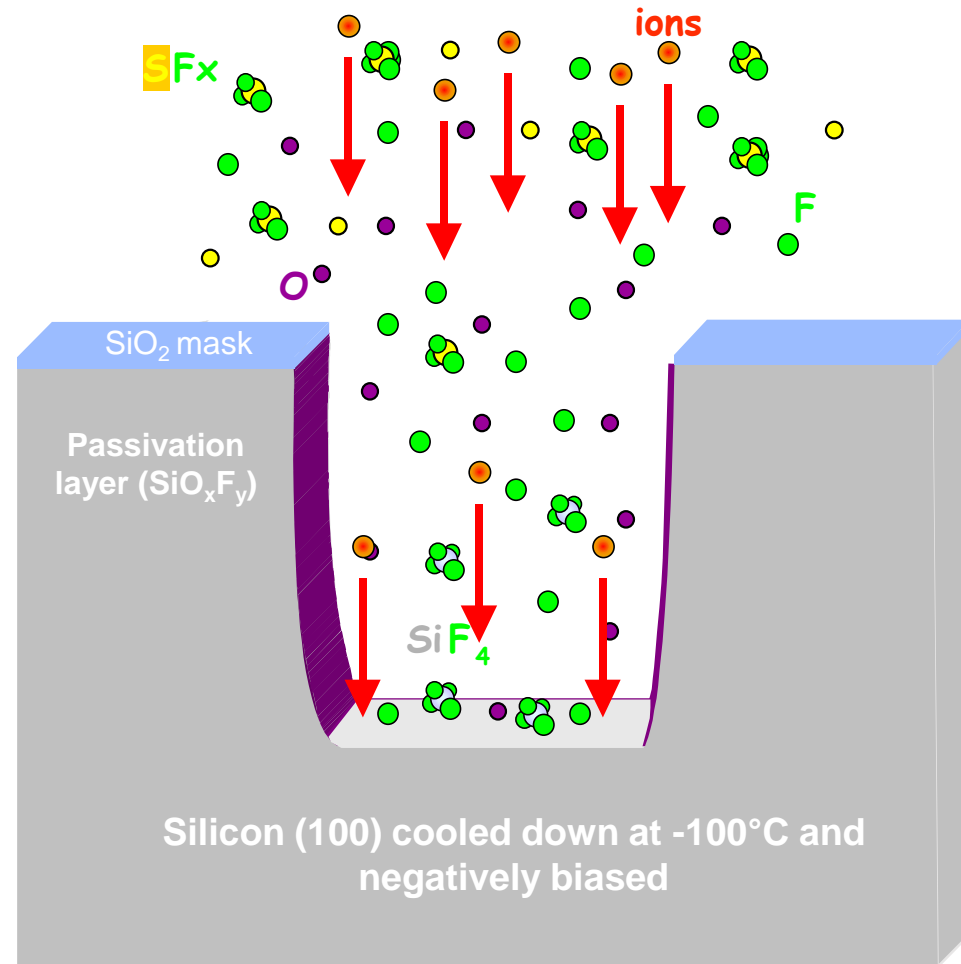
⇒ Forms only at very low temperature

⇒ Fragile passivation layer, easily removed by ion bombardment

Simultaneous mechanisms

Etching of high aspect ratio features

⇒ *Depth* >> *CD*

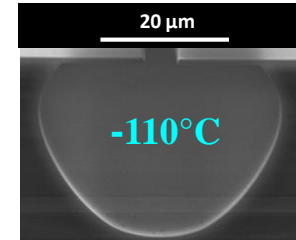
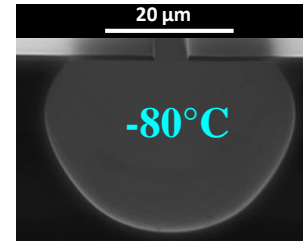
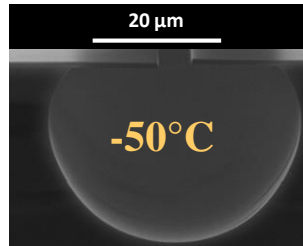
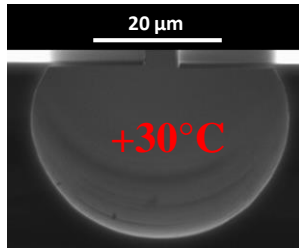




2) Deep cryoetching of silicon

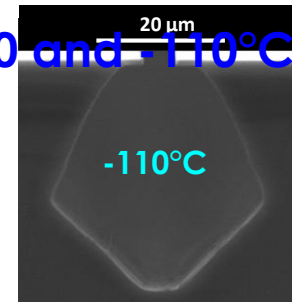
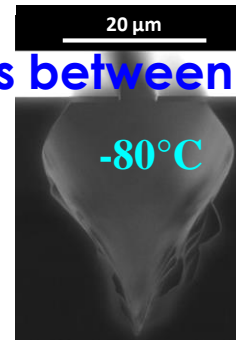
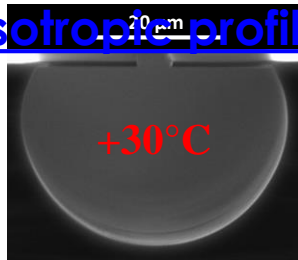
Low temperature + oxygen + bias = anisotropic structures

SF₆
without
bias

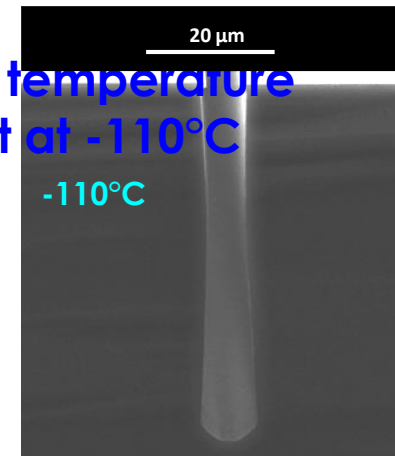
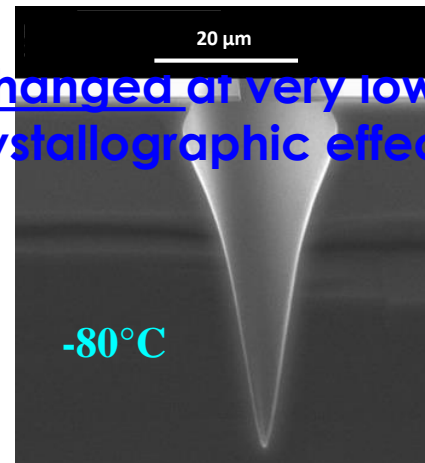
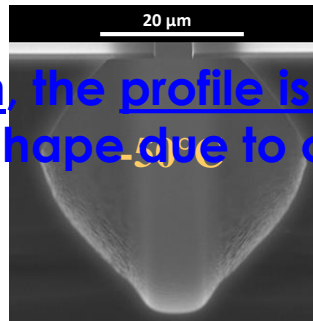
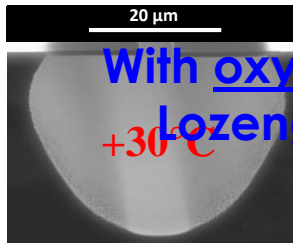


Isotropic profile for all temperatures between +30 and -110°C

SF₆/O₂
without
bias



SF₆/O₂
with bias



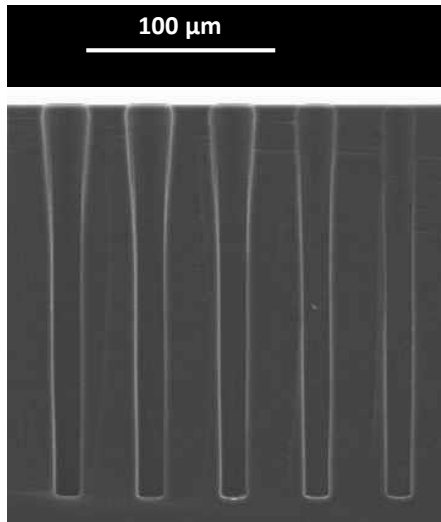
With oxygen, the profile is changed at very low temperature
Lozenge shape due to crystallographic effect at -110°C

Anisotropic etching at -110°C, with oxygen and bias

2) Deep cryoetching of silicon



Performances of the cryogenic process



Example of profiles :

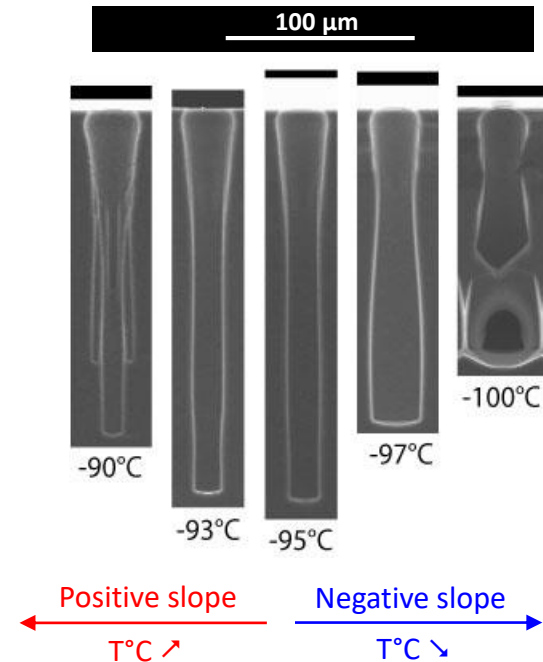
Holes (TSV)
CD = 14 μm

30 minutes

Depth = 210 μm

AR = 15

ER (30') = 7 μm/min



Advantages

- › Monocyclic process
 - ⇒ High etch rates, smooth sidewalls
- › Desorption of the passivation layer at room temperature
 - ⇒ clean process

Drawbacks

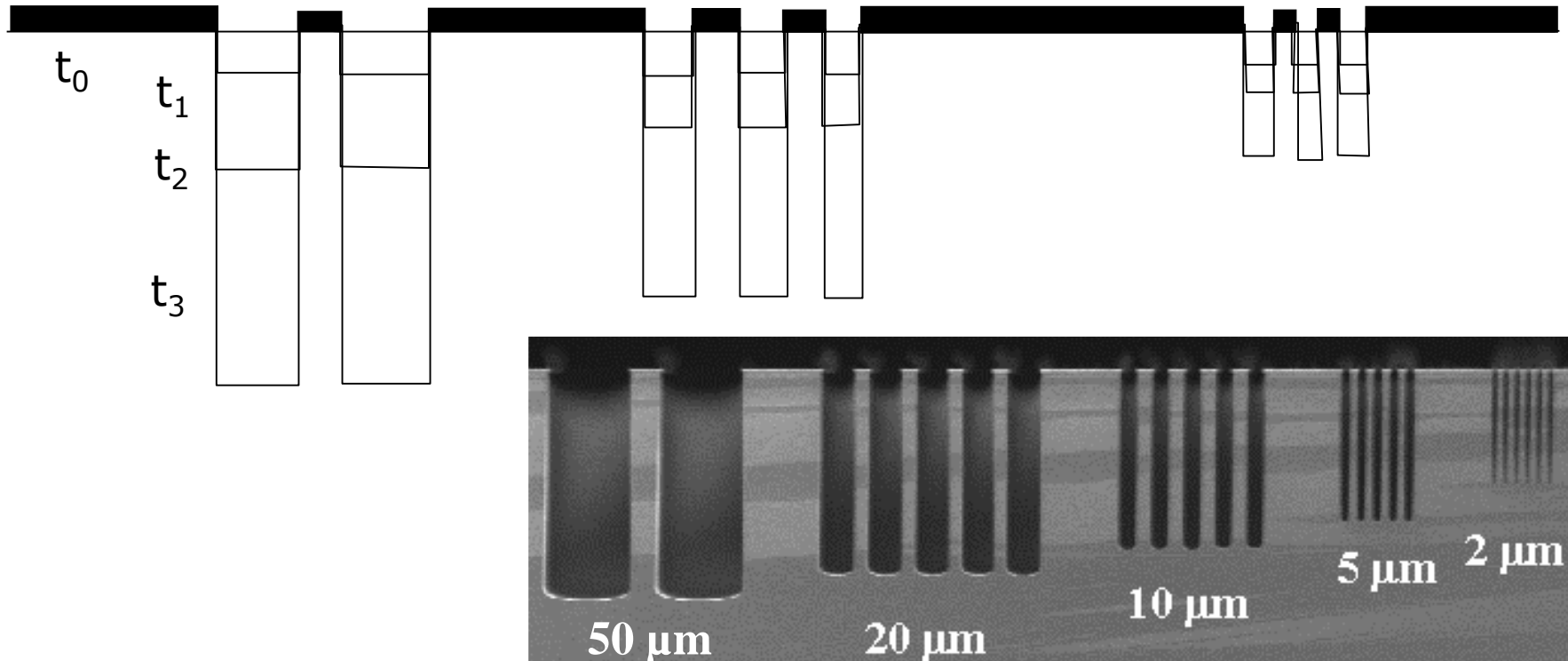
- › Very sensitive to temperature and O₂ flow variations
- › Requires liquid nitrogen
- › ARDE is quite pronounced



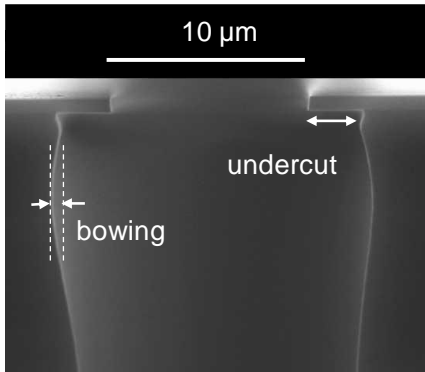
2) Deep cryoetching of silicon

Effet ARDE (Aspect Ratio Dependent Etching)

- ARDE is also called « RIE Lag » (Reactive Ion Etching Lag)
- The etch rate of features with increasing aspect ratio (narrower or deeper) decreases because of the decrease of both ion and neutral fluxes



2) Deep cryoetching of silicon



Undercut and bowing

Undercut:

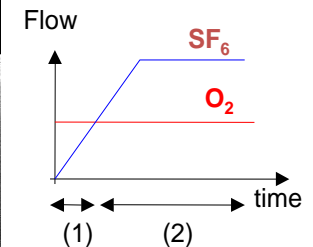
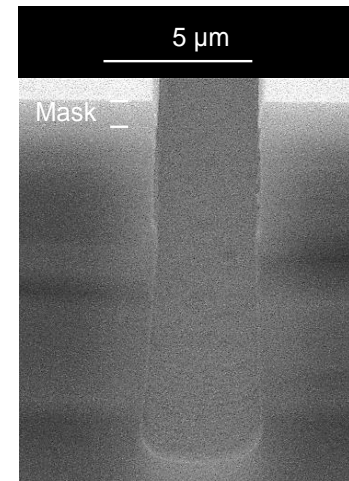
- caused by spontaneous reactions of fluorine radicals.
- keeps growing linearly during process

Possible to decrease significantly decrease this defect

Local bowing increases with:

- process time
- self-bias (ions are partially responsible for its formation)
- the mask side slope (straight and vertical mask side slopes are preferred)

Bowing appearance can be controlled by adjusting the balance between ion flux and F/O relative density ratio



- (1): overpassivation regime
(2): etching regime (standard mode)

Undercut = 0.46 μm
ER = 4.7 μm.min

M. Boufnichel et al. 2002 J. Vac. Sci. Technol. B 20 1508

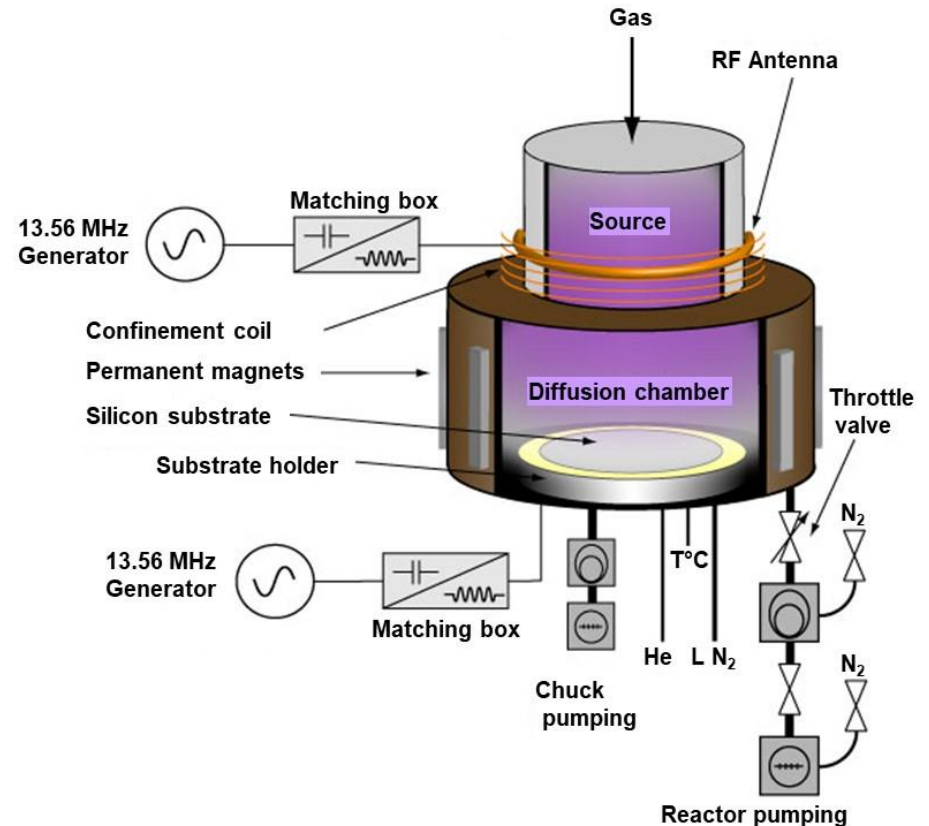
M. Boufnichel et al. 2003 J. Vac. Sci. Technol. B 21 267

M. Boufnichel et al. 2005 Microelectron. Eng. 77 327



2) Deep cryoetching of silicon

ICP reactor with cryogenic cooling capability



13,56 MHz ICP source

13,56 MHz RF substrate holder (synchronized)

Substrate temperature : -130°C → 30°C

Cooling with liquid nitrogen



2) Deep cryoetching of silicon

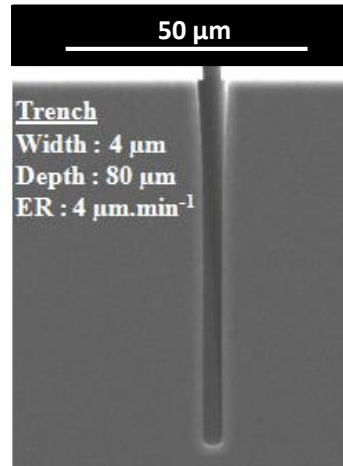
Applications

Deep Etching is intensively in MEMS and microelectronic industries

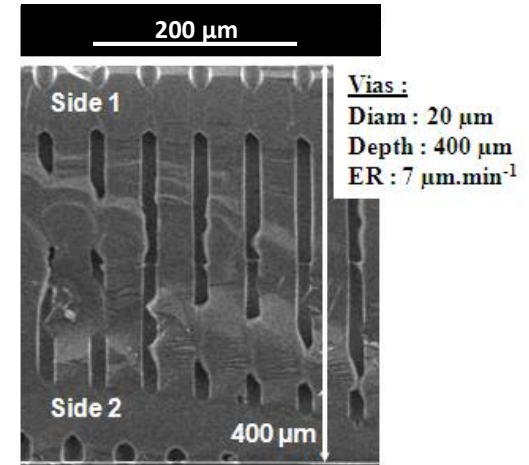
Key parameters for profiles are:

- › Etch rate, throughput (units per hour)
- › Sidewall roughness
- › Reduction/Elimination of defects
- › Reproducibility

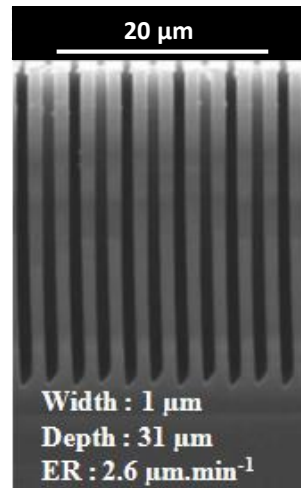
Deep Trench Insulation



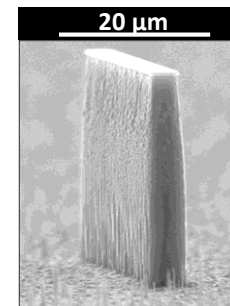
TSV for interconnects



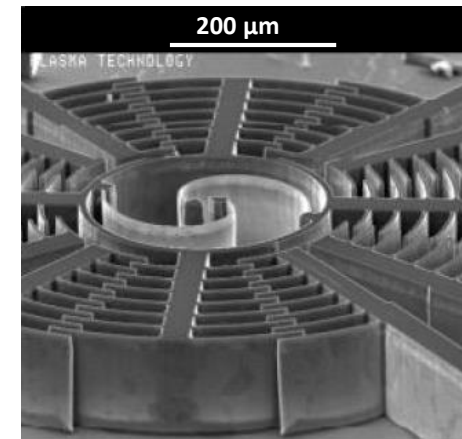
3D capacitors



MOEMS



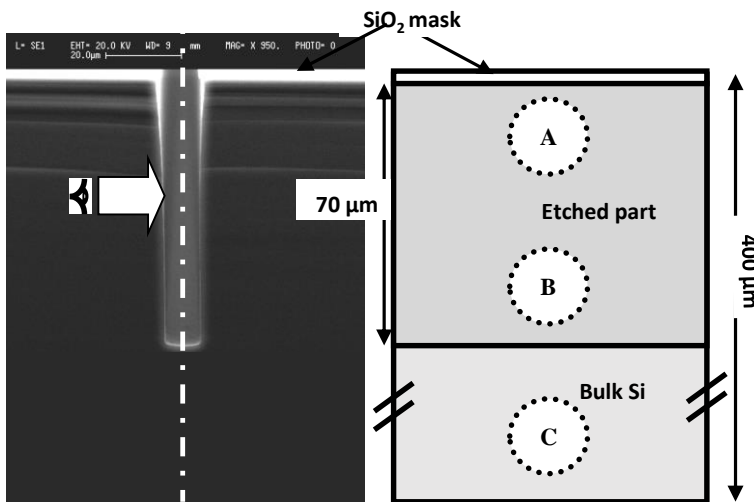
MEMS



2) Deep cryoetching of silicon

Passivation mechanism

- SF_6/O_2 plasma interacting with a cooled silicon wafer
- The passivation layer is composed of silicon, oxygen and fluorine ($\sim SiOF_3$, shown by XPS analyses)
 - ⇒ It is formed at the surface from SiF_x and O species at the surface (not in gas phase)
 - ⇒ The passivation is more fluorinated as the temperature decreases
 - ⇒ The physisorbed layer desorbs at room temperature, which releases SiF_4 (desorption mass spectrometry)
- The passivation layer is almost entirely removed when the substrate is warmed back up to room temperature



Ex-situ XPS

Lines	Center [eV]	Ratio ($\pm 0,01$)		
		A	B	C
F / Si-Si	F 1s- 688	0,02	0,02	0,01
O / SiSi	O 1s- 533,5	0,13	0,15	0,14
C / SiSi	C 1s- 285,3	0,11	0,15	0,11
Si-O / SiSi	Si 2p- 103,7	0,03	0,03	0,03

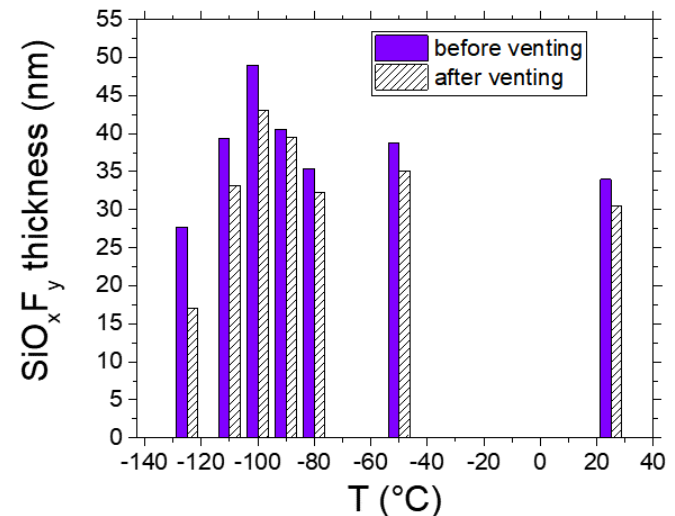
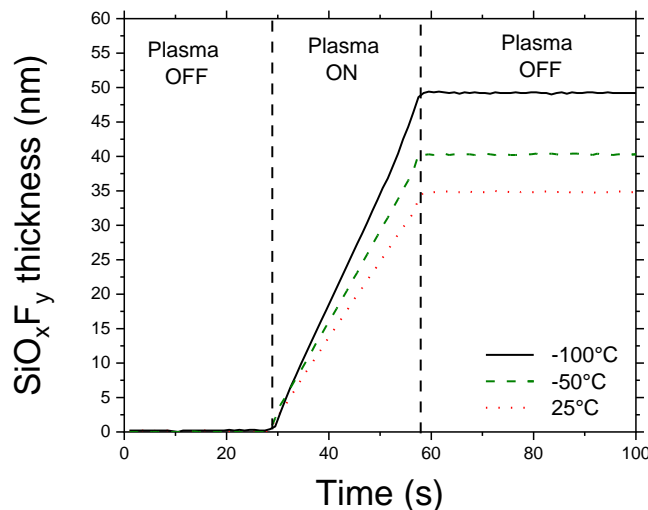
R. Dussart *et al* J. Micromech. Microeng., 14, 190-196 (2004)

2) Deep cryoetching of silicon

Passivation mechanism

- A SiF_4/O_2 plasma can be used to grow a SiO_xF_y passivation layer on flat surfaces
 - ⇒ monitoring of growth by in-situ ellipsometry for different substrate temperatures
- Thicker layer by decreasing the temperature from 20 to -100°C

O_2 flow = SiF_4 flow = 50 sccm, $P_{\text{source}} = 1000$ W, $P_{\text{bias}} = 0$ W, Pressure = 3 Pa, process duration: 30 s



- During venting/warming of the sample, a part of the layer desorbs.
- Below -100°C , the deposited layer thickness decreases

G. Antoun et al. JJAP, 58, SEEB03 (2019)

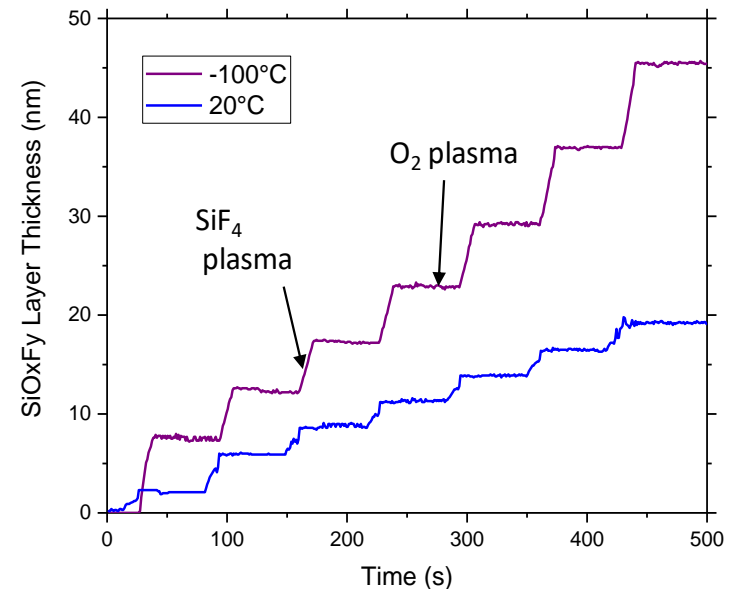
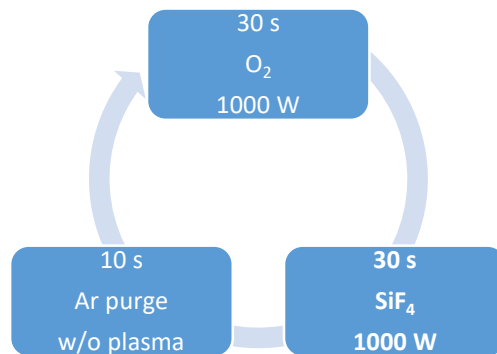
2) Deep cryoetching of silicon

Passivation mechanism

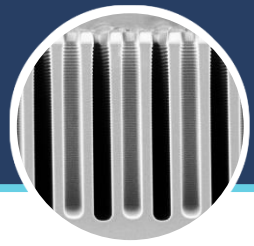
- Both SiF_x species and O atoms are required to form the SiO_xF_y layer
- Decomposition of their role...

R. Dussart et al., J. Phys. D: Appl. Phys. 47 123001 (2014)

- Alternation of O_2 plasma and SiF_4 plasma



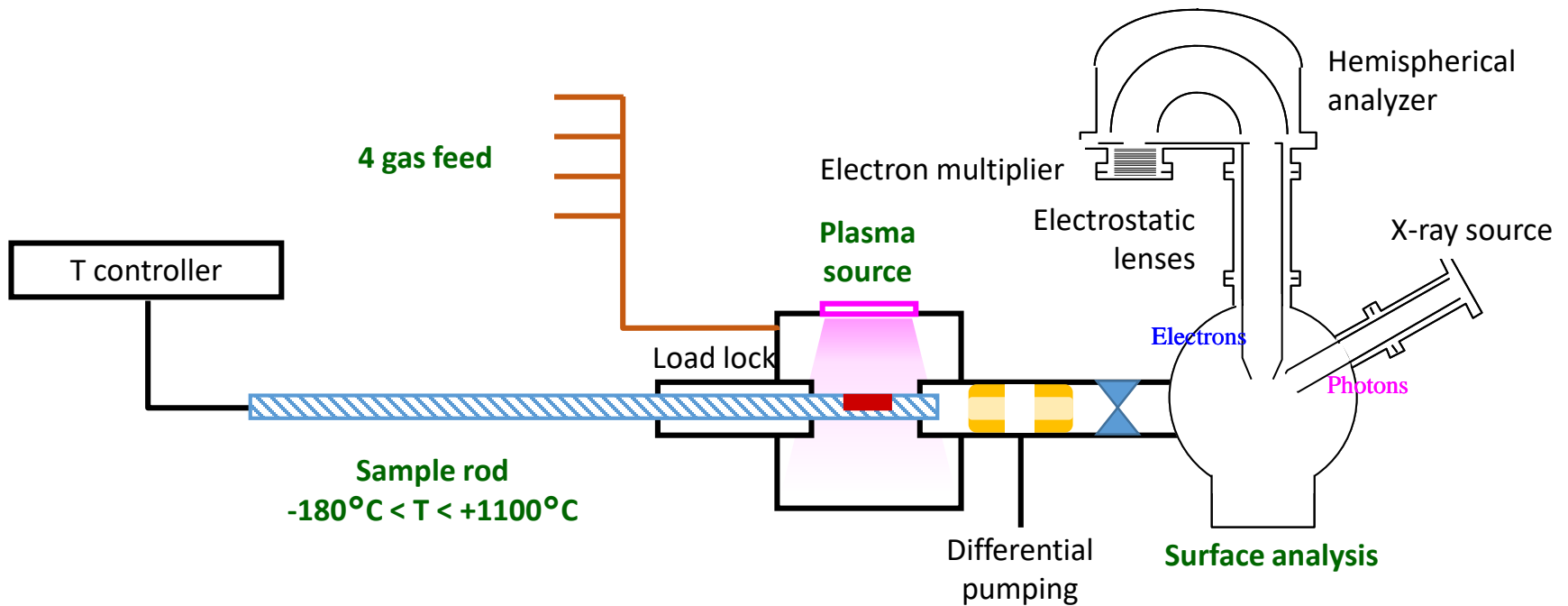
- Growth during the SiF_4 plasma
- Thicker SiO_xF_y layer at low temperature
 - ⇒ Physisorption and accumulation of SiF_x at the surface favored at low temperature
 - ⇒ Higher surface residence time
 - ⇒ Oxidation of the SiF_x during the oxygen plasma (not detected by ellipsometry)



2) Deep cryoetching of silicon

Passivation mechanism

OPTIMIST Platform (IMN, Nantes) => Quasi in-situ XPS



2) Deep cryoetching of silicon

Passivation mechanism

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

a-Si sample ; 30 s ; SiF_4/O_2 : 25 % ; 3.0 Pa ; 200 W ICP power ; no bias

- At -40°C, 17.1% [F] ; 23.6% [O]

=> After heating: no significant change

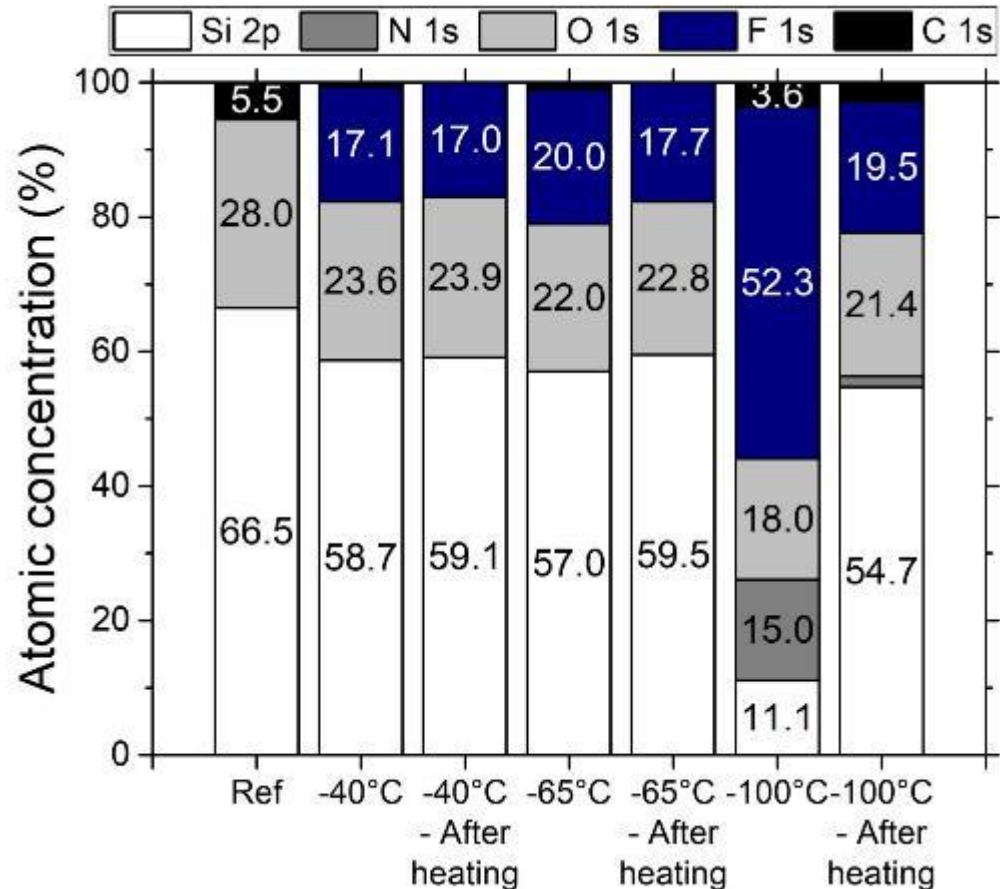
- At -65°C, 20.0% [F] ; 22.8% [O]

=> 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 F-based species has desorbed ($\sim\text{SiO}_2$)

Optical index n of the order of 1.2 , which indicates that the remaining layer is quite porous





2) Deep cryoetching of silicon

Passivation mechanism: summary

- Passivation at **cryogenic temperature**: **higher surface residence** time favoring reaction between SiF_x and O
- SiO_xF_y passivation layer obtained by $\text{SiF}_4 / \text{O}_2$ plasma
 - => grows **more efficiently at low temperature** with an optimum at -100°C
 - => partial **desorption of the SiO_xF_y layer (physisorbed species)** when the wafer is warmed back to room T
 - => the remaining layer after venting is mainly composed of **porous SiO_2 (chemisorbed species)**
 - => **Fluorine content** in the SiO_xF_y layer **increases at low temperature**
- SiF_4/O_2 plasma can reinforce the passivation layer => **Time-multiplexed cryoetching**
- Enhanced physisorption at cryogenic temperature
 - => **Low-damage cryogenic etching of low-k materials**
 - => **Cryogenic Atomic Layer Etching**

2) Deep cryoetching of silicon

STiGer process

Alternated cryogenic process

- 1 SF₆ plasma step
- 2 SiF₄/O₂ passivation step

A few seconds to a few 10s μs for each step

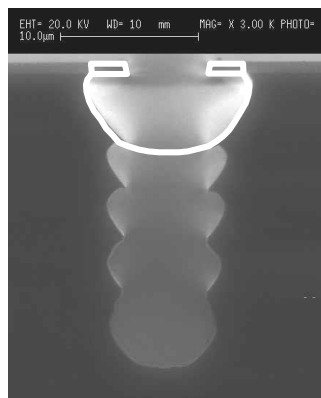
➔ Anisotropic profiles

Role of substrate temperature

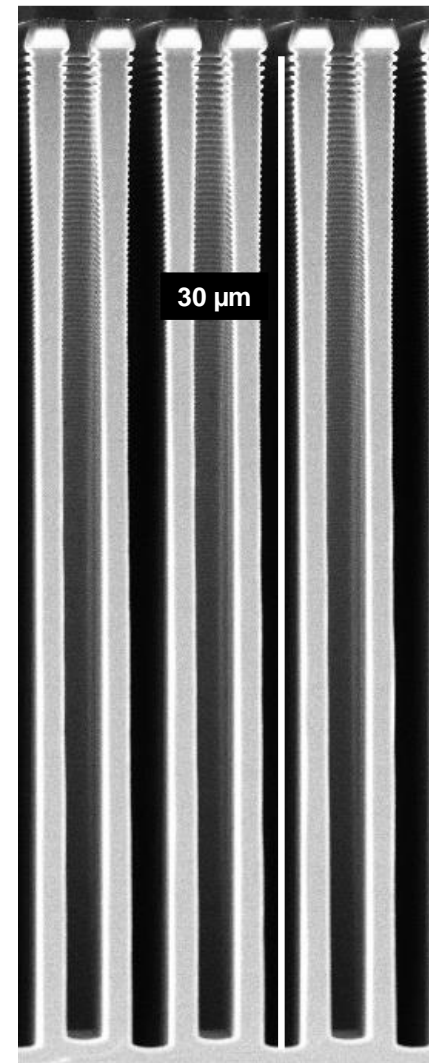
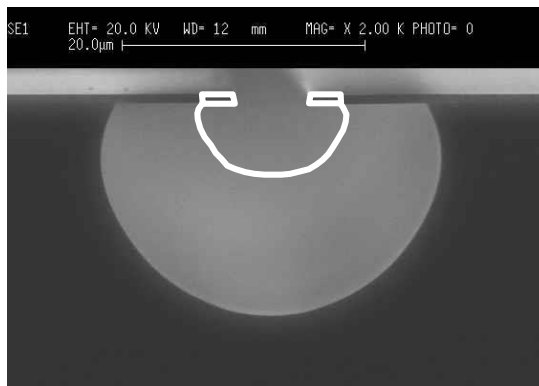
➔ 4 alternances 1min SF₆ etching - SiF₄/O₂ deposition + a final 1 min etch

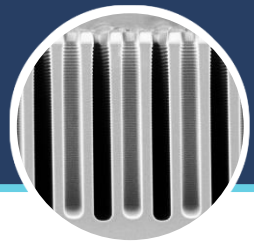
Anisotropic microstructures can be etched provided the substrate is cooled at cryogenic temperature

-83°C



Same experiment at 0°C





3) Cryogenic plasma processes

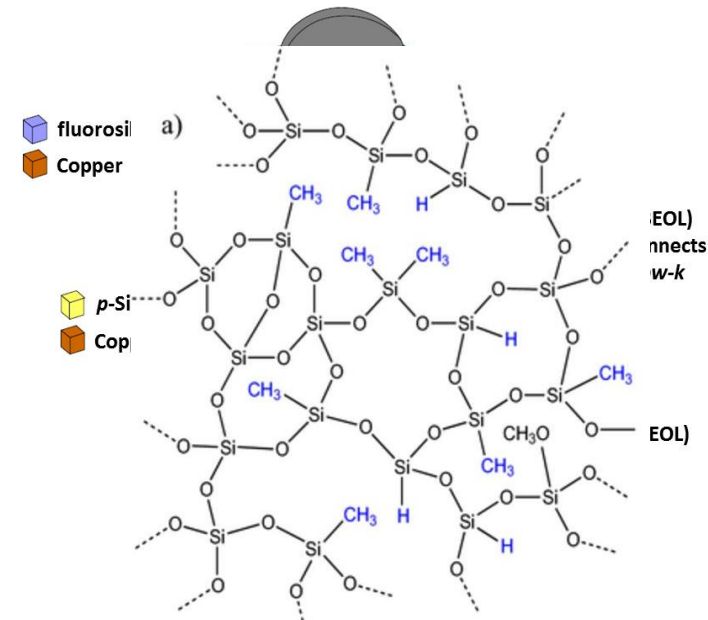
Cryoetching of low-k materials

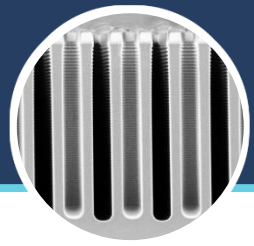


- **Porous Organosilicate glasses** (OSG) are **low-k** materials used for advanced interconnects
- Miniaturization of integrated circuits \Rightarrow Increase of switching time (RC-like)
- \Rightarrow Materials with lower and lower dielectric constant

- Introduction of **porosity** (p-SiOCH)
 - \Rightarrow plasma-sensitive materials
 - \Rightarrow very sensitive to radicals, ions and UV photons
 - \Rightarrow Depletion of methyl groups (increase ok both k-value + leakage current)
 - \Rightarrow "Plasma Induced Damage"

Reduction of PID by cryogenic etching





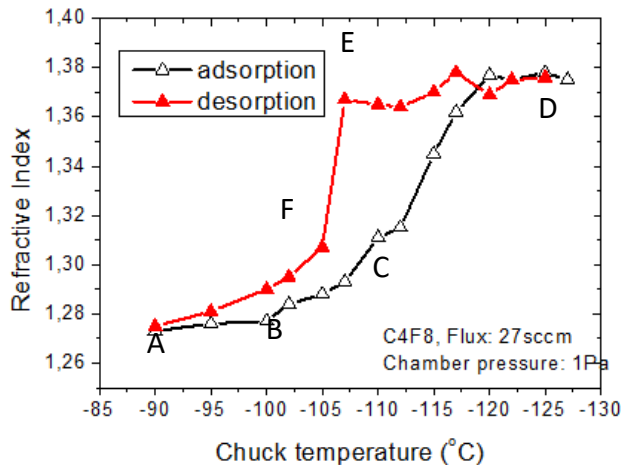
3) Cryogenic plasma processes

Cryoetching of low-k materials

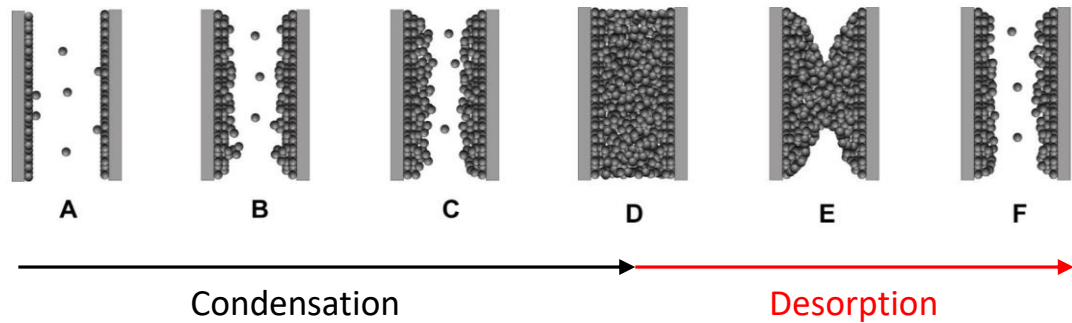
Capillary condensation of a fluorocarbon gas (example of C_4F_8 below)



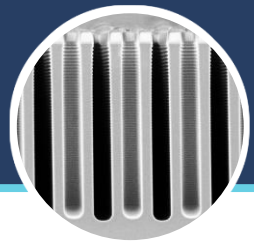
- Goal : etching of a **densified** material to prevent radical diffusion



Monitoring of RI with in-situ ellipsometry



- Hysteresis between adsorption/condensation and desorption
- Full condensation at -120°C for C_4F_8
- Other chemicals with a Higher Boiling Point Organic (HBPO) can be used
=> Full condensation at -50°C



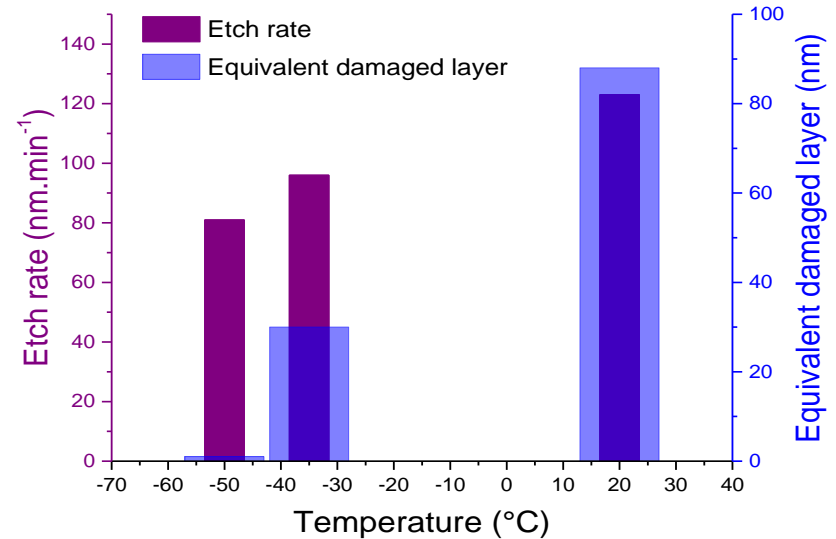
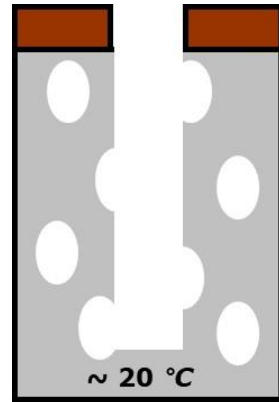
3) Cryogenic plasma processes

Cryoetching of low-k materials

Process based on capillary condensation step (case of HBPO here)

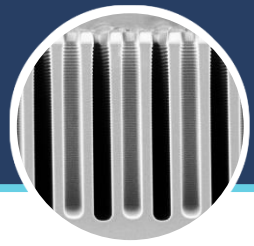


- 1 Cooling at -50°C
- 2 Pore Condensation of HBPO (flow of SF_6/HBPO , 3 Pa)
- 3 SF_6 or SF_6/HBPO plasma (500W, 3 Pa, 1 min)
- 4 Heating/Unloading
- 5 Annealing at 400°C under N_2 (10 min)



- Low etch rate in the absence of ion bombardment => **Anisotropic etching**
- Strong reduction of the damage layer as a function of temperature, until it reaches almost 0 at -50°C

⇒ **Almost no PID at -50°C**



3) Cryogenic plasma processes

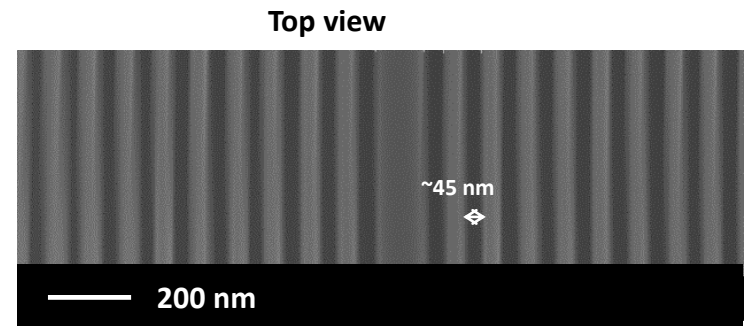
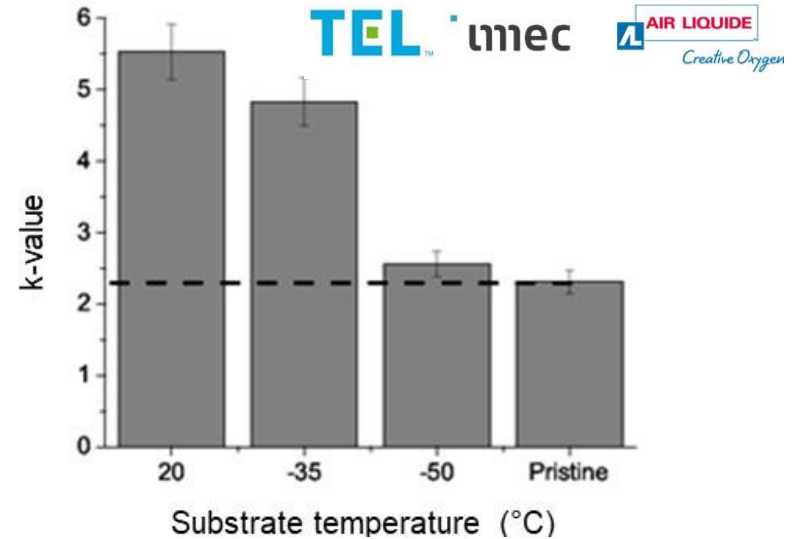
Cryoetching of low-k materials

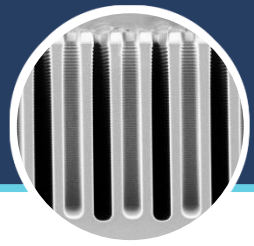
Measurement of k-value (IMEC)

- The k-value is very close to its initial value after etching at -50°C
- Confirmation of the strong reduction of PID

Etching of patterned samples

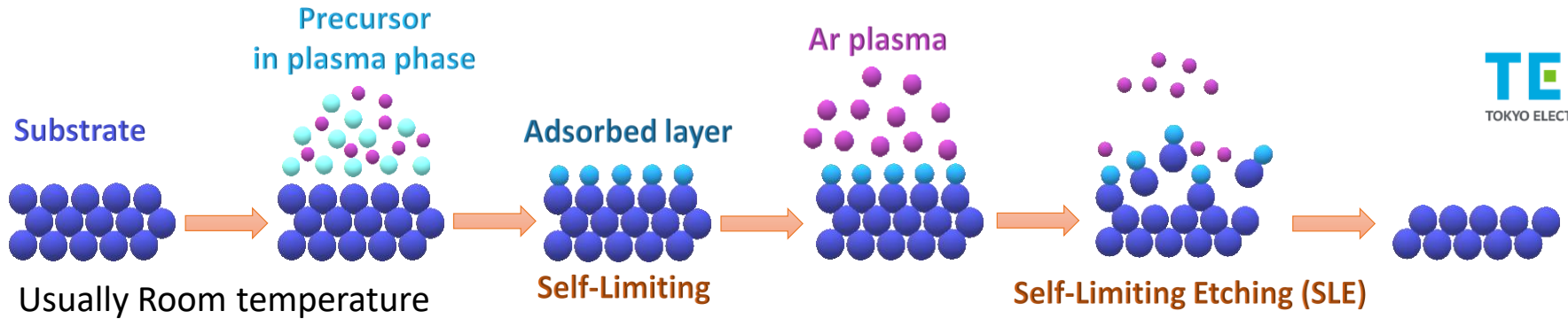
- ⇒ Trenches of CD = 45 nm
- ⇒ Depth = 67 nm within 1 min 30
 - ⇒ $44,7 \text{ nm}\cdot\text{min}^{-1}$
- ⇒ **Anisotropic profiles**, no defects
- ⇒ **No distortion of the original pattern**





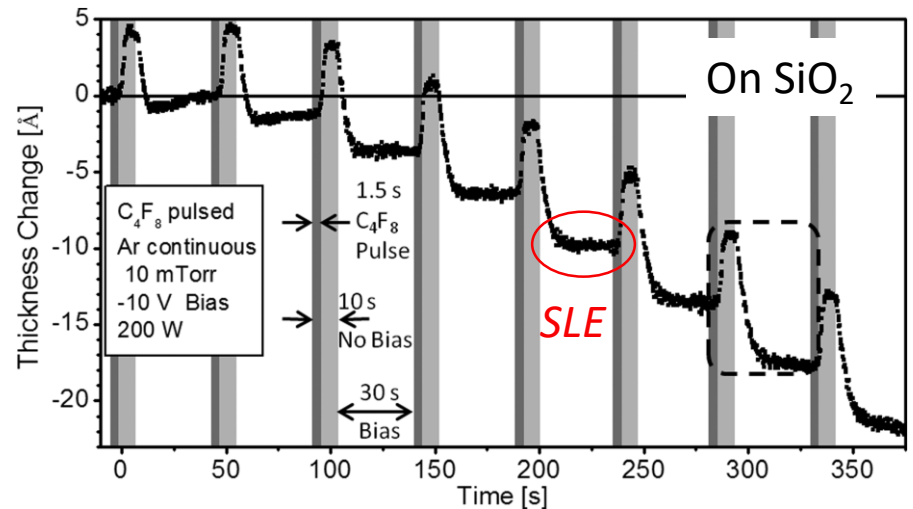
3) Cryogenic plasma processes

Cryogenic Atomic Layer Etching (CryoALE) of SiO₂



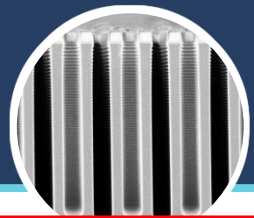
Why ALE ?

- Atomic precision (scale reduction of Integrated Circuits)
- Etching of thin layers with no damage to underlying layers
- « Infinite » selectivity

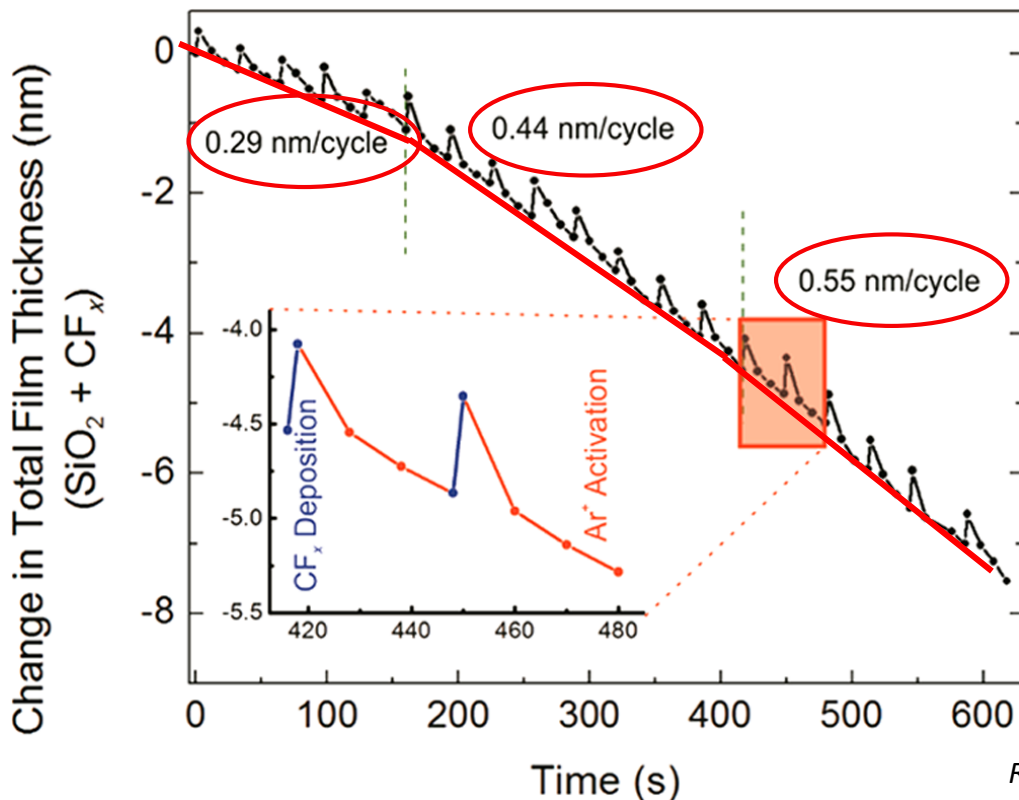
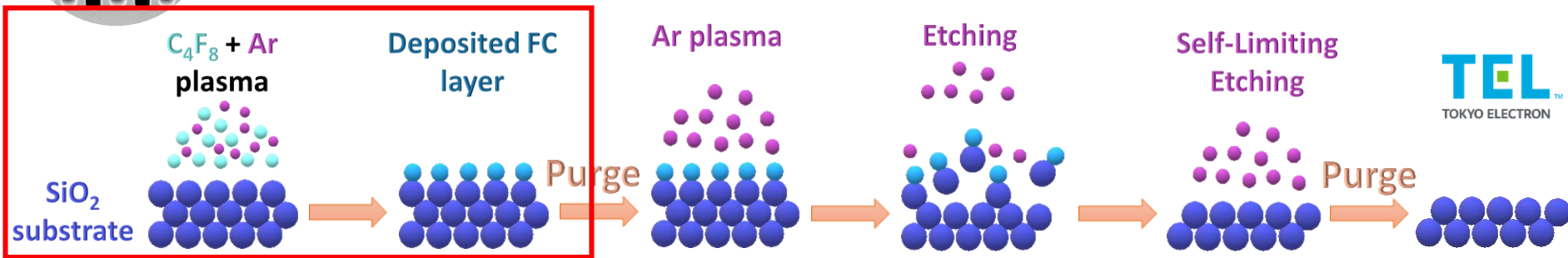


atomiclimits.com

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



3) Cryogenic plasma processes

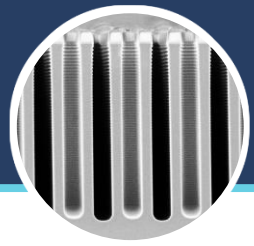


ALE of SiO₂ at room temperature

“SiO₂ etching continues past the removal of the CF_x film, which clearly indicates the presence of a secondary supply of F from the chamber walls”

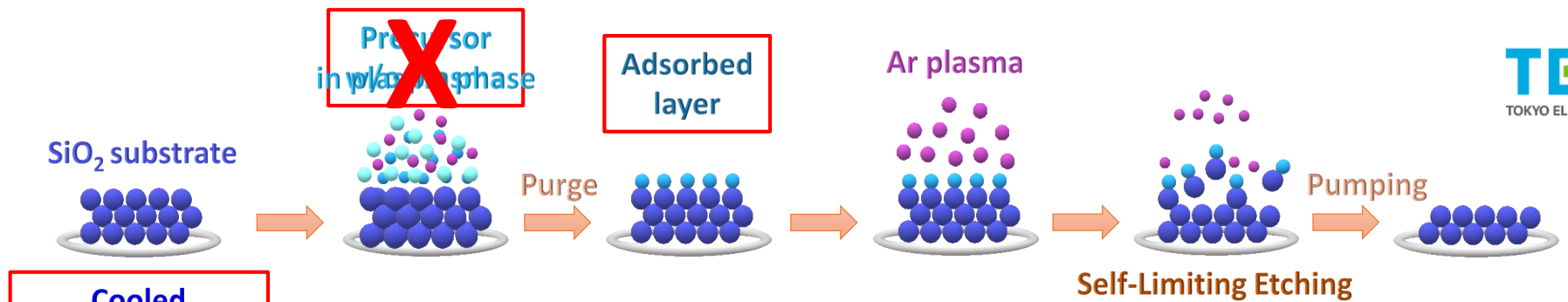
Observation of a **drift** after few cycles due to **reactor wall contamination**

R.J. Gasvoda et al, ACS Appl. Mater. Interfaces 9, 31067 (2017)

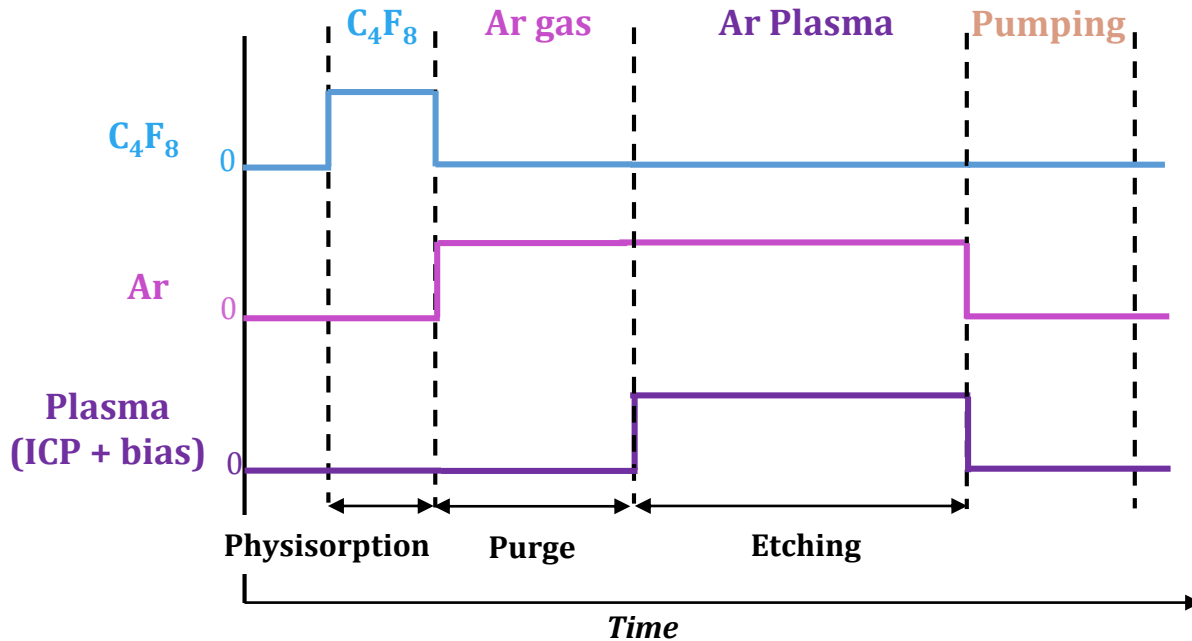
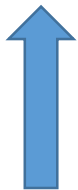


3) Cryogenic plasma processes

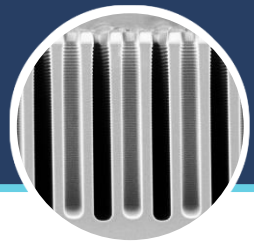
Cryogenic Atomic Layer Etching (CryoALE) of SiO₂



Cooled substrate holder

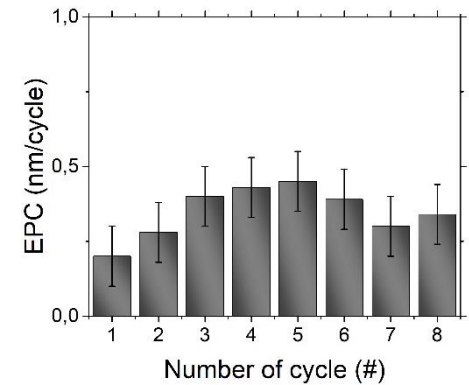
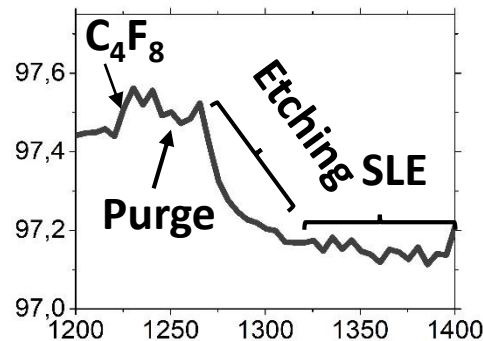
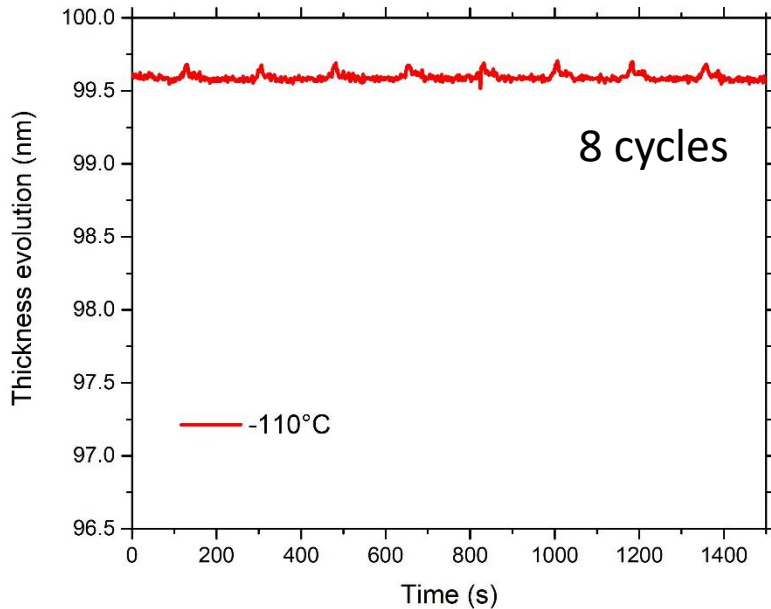
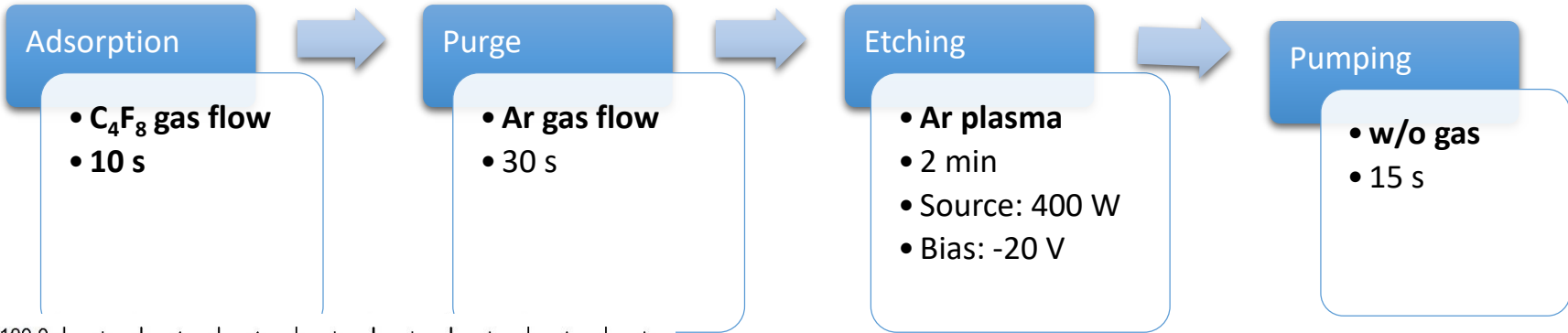


G. Antoun *et al.*,
Appl. Phys. Lett. 115
(2019) 153109



3) Cryogenic plasma processes

Cryogenic Atomic Layer Etching (CryoALE) of SiO₂

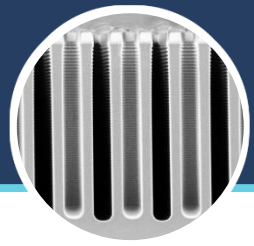


- Self-Limiting Etch was reached

- Regular EPC < 0.5 nm/cycle (~ one monolayer)

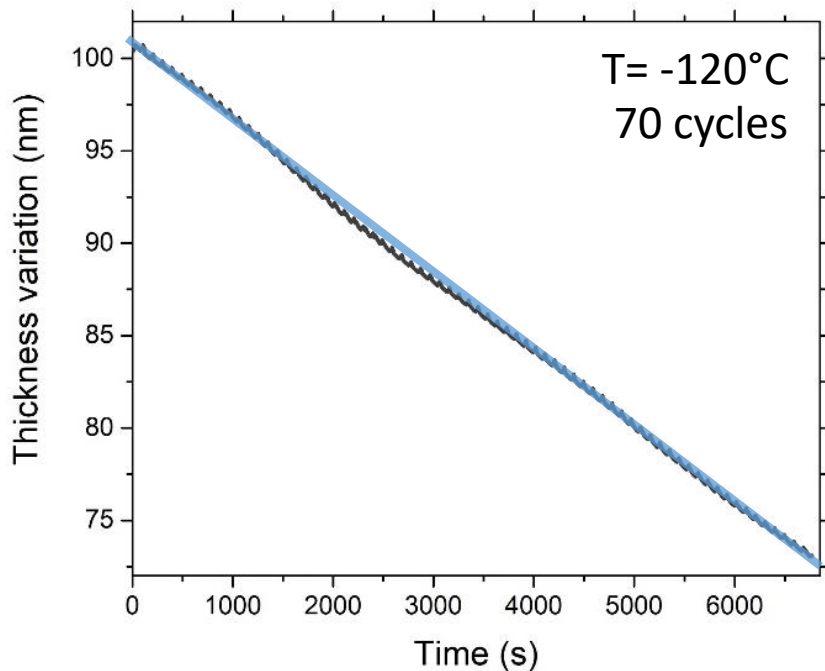
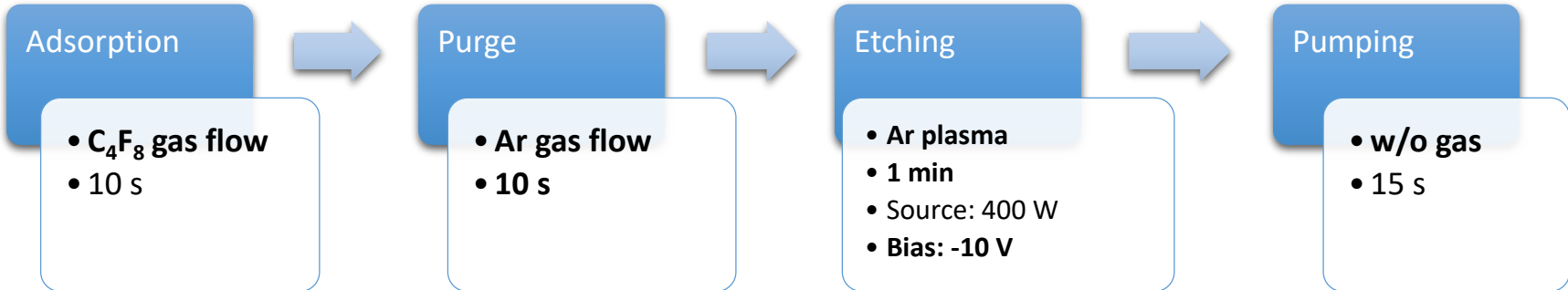
• No etching at -110 °C (and higher temperatures)

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



3) Cryogenic plasma processes

Cryogenic Atomic Layer Etching (CryoALE) of SiO₂

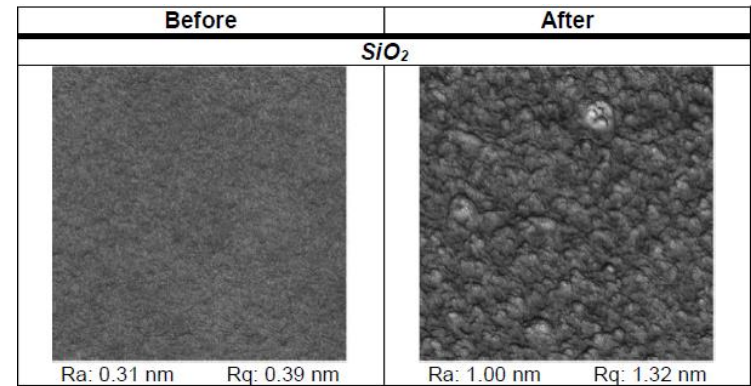


Regular Etching along cycles

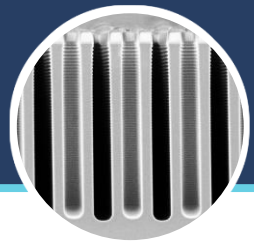
- 27 nm etched in 70 cycles
- EPC ≈ 0.4 nm/cycle

No drift, no wall contamination

-11 nm



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



To summarize...

Benefit of cryogenic cooling of substrates for plasma etching

- ⇒ Increased surface residence time of species => passivation, capillary condensation, physisorption...
- ⇒ Less plasma induced damage
- ⇒ Less contamination of chamber walls (layers deposited only on cooled surfaces)

Renewed interest of cryogenic industries in the industry... but little communication

- ⇒ Alternative designs of cooling technologies (without liquid Nitrogen)
- ⇒ Main argument = less damaging (lower radical diffusion)
- ⇒ example: Flash Memories (vias in 3D NAND)

Thank you !

The authors would like to thank



for their valuable **financial** and **scientific** supports

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