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Cryo-ALE of Si based on SF₆ Physisorption

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Introduction:

Cryo-ALE: Atomic Layer Etching process performed at cryogenic temperatures (-70°C to -120°C)

Previous (C₄F₈ + Ar) cryo-ALE research campaign on SiO₂: Study of C₄F₈ physisorption

→ Obtention of a self-limiting etching regime at T = -120°C (≈ 1 nm/cycle)

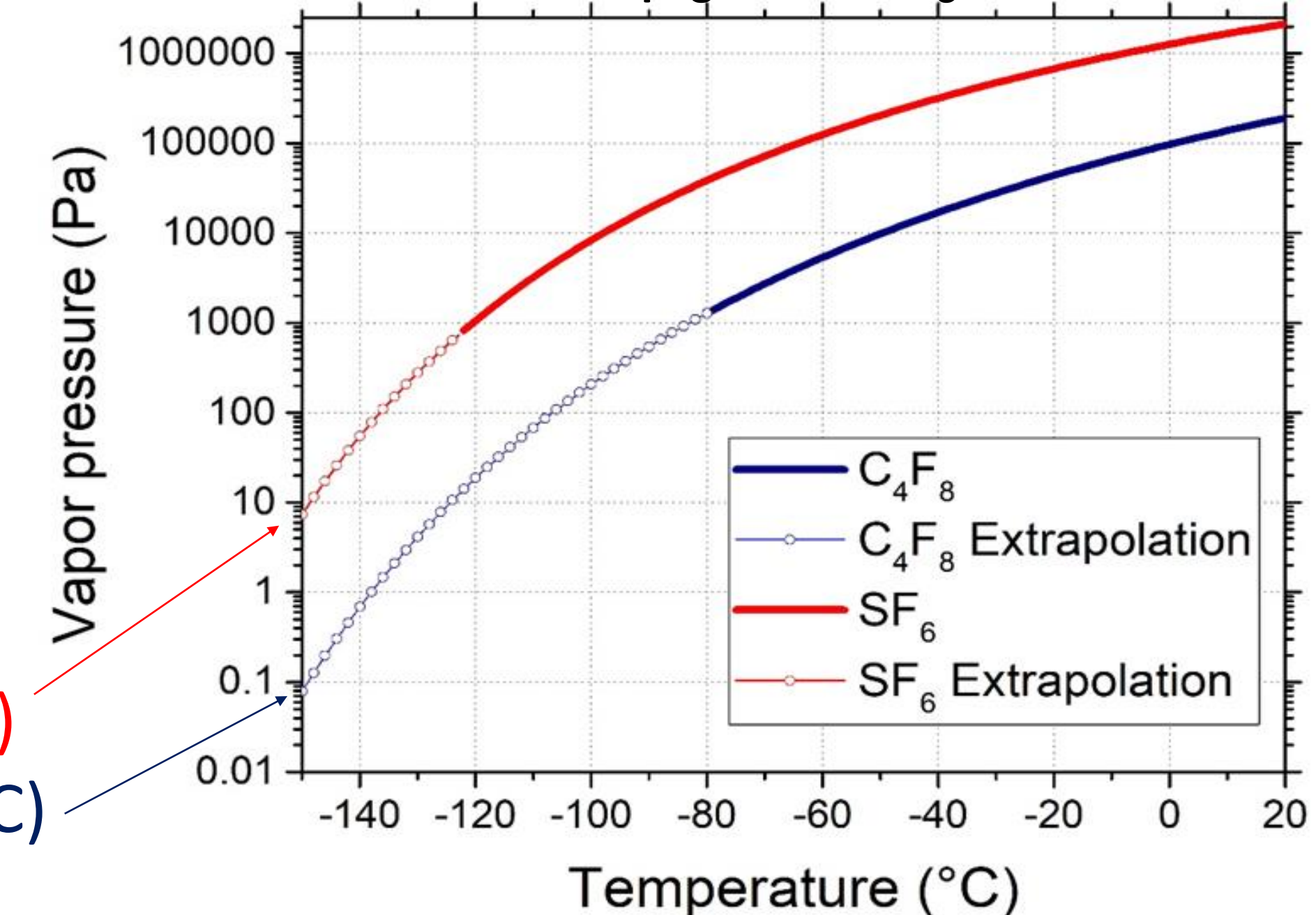
→ No particular etching selectivity of SiO₂ over Si or Si₃N₄

Objectives:

Cryo-ALE process optimization: Comparison of etching properties obtained with SF₆ and C₄F₈ physisorptions at similar process conditions

Study of SF₆ physisorption: Study of plasma-surface interactions during cryo-ALE processes based on SF₆ physisorption

Figure 1: Vapor pressure in relation to temperature for C₄F₈ and SF₆



At low pressures (1 to 3 Pa):

SF₆ vapor pressure equilibrium (≈ -150°C)

C₄F₈ vapor pressure equilibrium (≈ -130°C)

Experimental equipment:

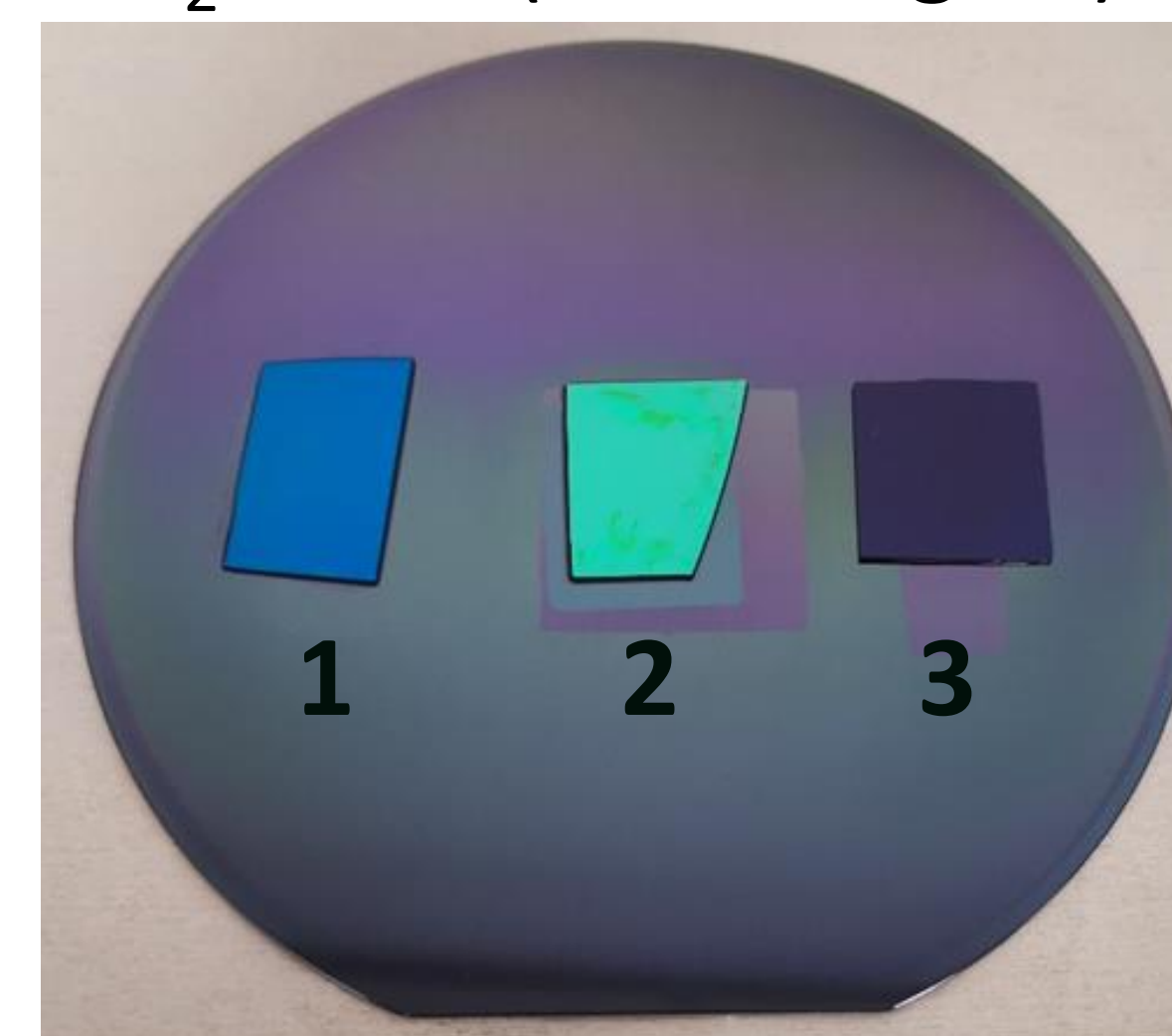
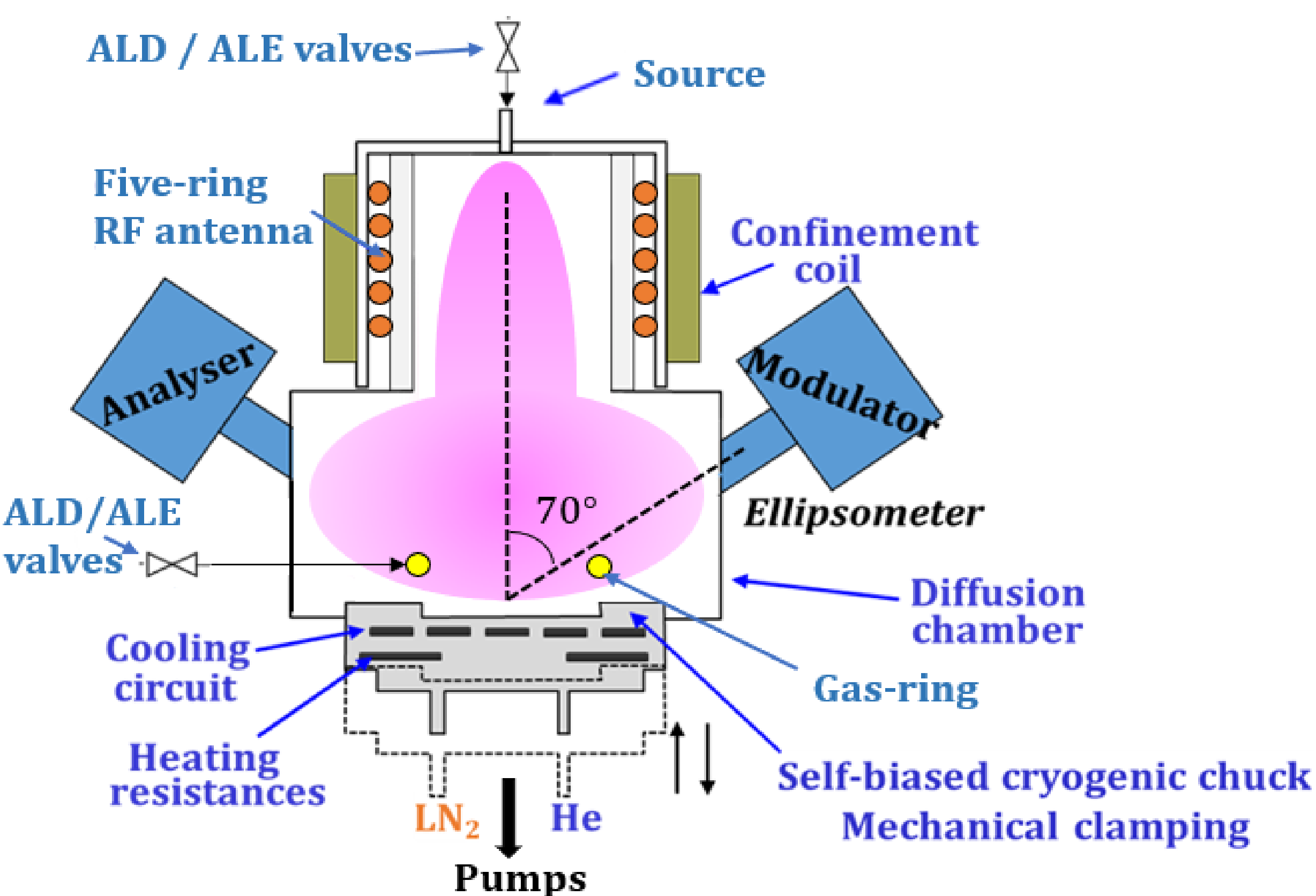
Etching equipment: Oxford Plasma Pro 100 Cobra (ICP reactor)

Characterization tools:

Ellipsometry: in-situ Horiba Jobin Yvon ellipsometer

→ Chemical composition and thickness of the etched substrate

Samples: Use of p-Si, SiO₂ and Si₃N₄ coupons (2x2 cm) glued on a 4'' or 6'' SiO₂ wafer (thermal glue)



- 1: Silicon nitride (Si₃N₄)
- 2: Polycrystalline silicon (p-Si)
- 3: Silicon dioxide (SiO₂)

Experimental Results:

(SF₆ + Ar) cryo-ALE process parameters:

- 1) SF₆ Physisorption: 10 s; 3 Pa; SF₆ (50 sccm) Gas-ring injection
- 2) 1st Purge (Ar gas): 15 s; 3,4 Pa; Ar (100 sccm) Source injection
- 3) Ar Etching: 120 s; 3,4 Pa; 400 W_S; 0 W_B; Ar (100 sccm) Source injection
- 4) 2nd Purge (Pumping): 15 s; 0 Pa

Time chart of the (SF₆ + Ar) ALE process cycle

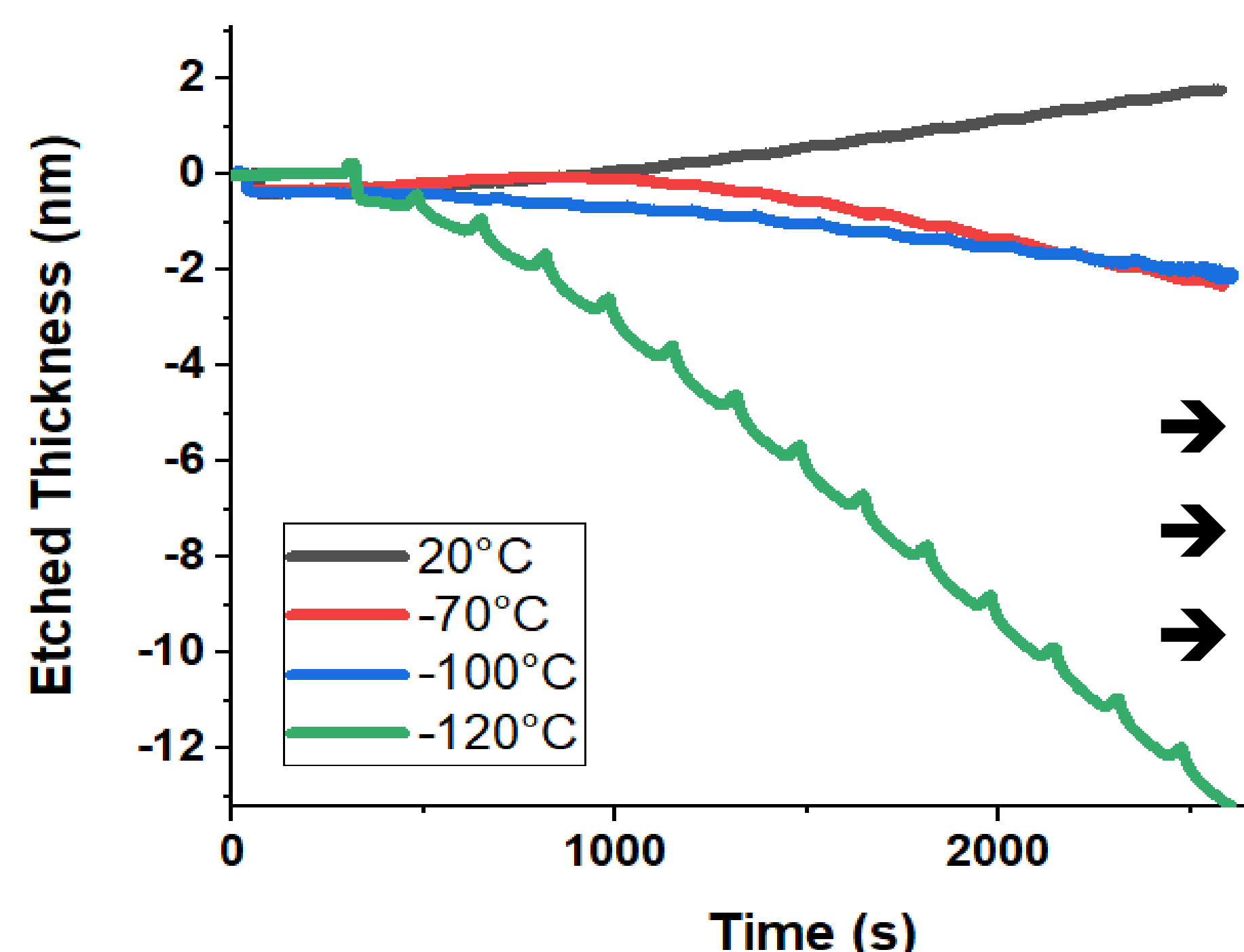
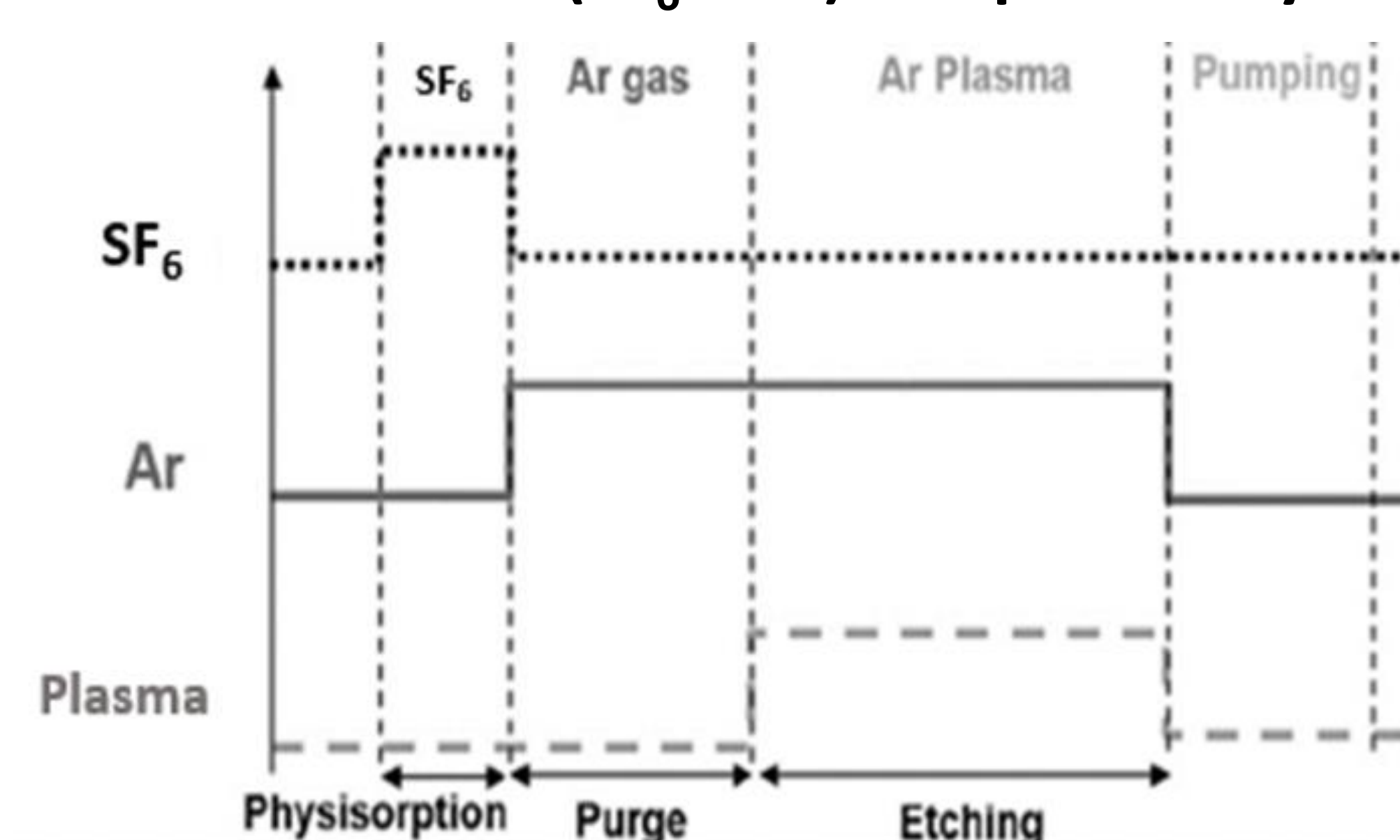


Figure 2: (SF₆ + Ar) cryo-ALE of SiO₂ at different temperatures

- Deposition regime at 20°C
- No significant etching at -70°C and -100°C
- Self-limiting quasi-ALE etching regime at -120°C

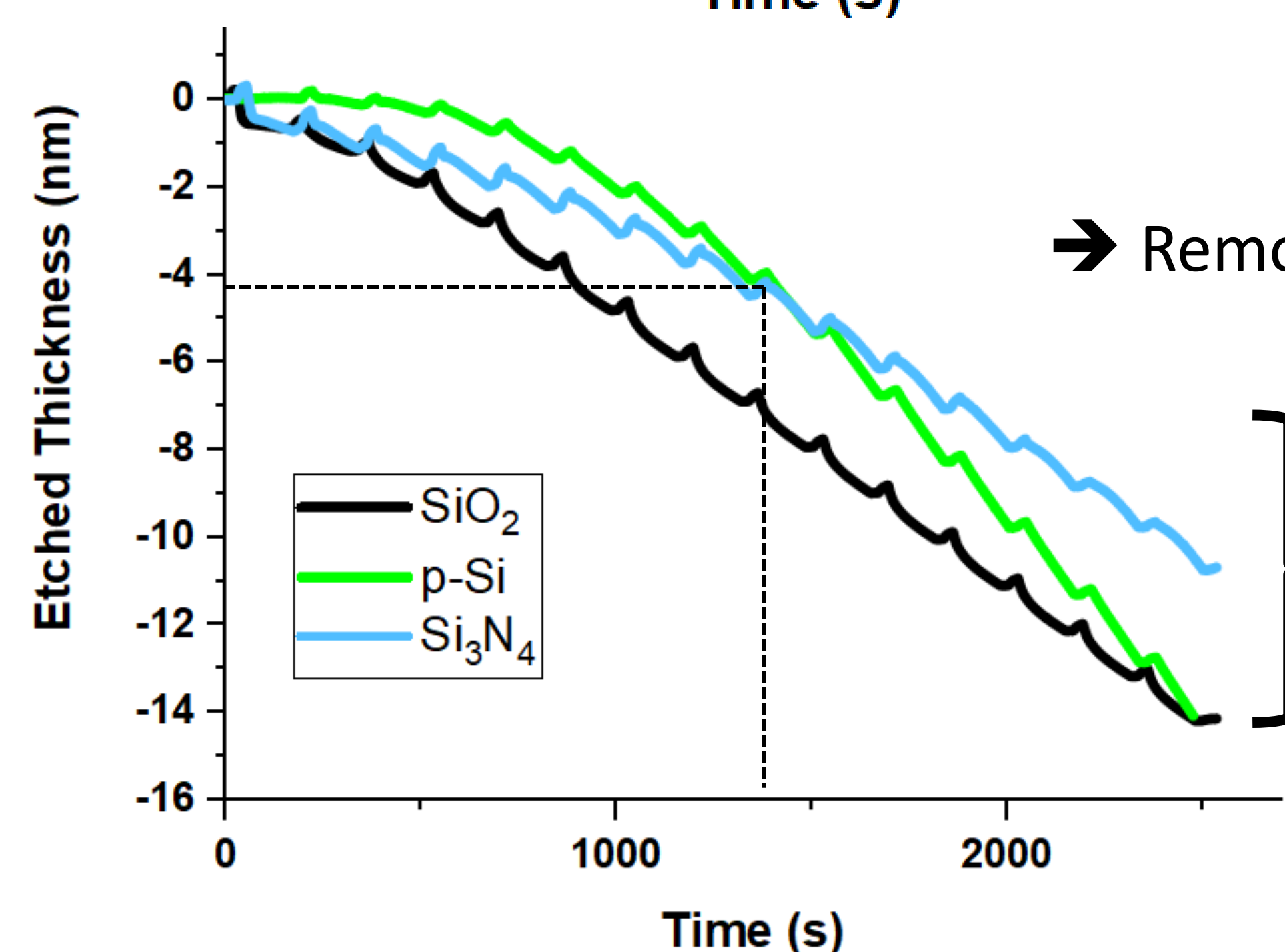


Figure 3: (SF₆ + Ar) cryo-ALE of different Si-based materials at -120°C

→ Removal of native oxide at p-Si and Si₃N₄ surface

Materials	EPC (nm/cycle)
p-Si	1,4
SiO ₂	1,1
Si ₃ N ₄	1,1

Conclusion and perspectives:

→ Observation of a quasi-ALE etching regime at T=-120°C for p-Si, SiO₂ and Si₃N₄ (≈ 6 monolayers of Si / cycle); The increased surface residence time of SF₆ at -120°C allows etching during the Ar etching step; No etching selectivity between Si, SiO₂ and Si₃N₄

→ Further characterizations (Plasma probes, XPS spectroscopy, Optical spectroscopy and Mass spectrometry) to better understand plasma-surface interactions during SF₆ physisorption at cryogenic temperatures in order to optimize the process.

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