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Limits and interest of a low surface temperature for plasma etching processes

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The advent of silicon deep etching at cryogenic temperatures has allowed obtaining microelectromechanical systems (MEMS) and through-silicon via (TSV) with high aspect ratio, controllable sidewall profiles, and high selectivity [1] [2]. Recently, low-temperature cooling of the sample has proved to be of great interest in atomic layer etching processes [3] [4] [5]. Behind these technological advances lie several physicochemical mechanisms and phenomena occurring on the uppermost atomic layers of the cooled surface. For example, formation of a dense but limited silicon oxyfluoride passivation layer at the feature sidewall is recognised as the key of an efficient Si deep etching process in SF₆ (or SiF₄) / O₂ chemistry.

Nonetheless, the physicochemical mechanisms in cryogenic conditions, *i.e.*, the species concerned and their surface mobility, residence times, reactivity, and adsorption rates, are still far from being fully identified and understood. Moreover, little attention is usually paid to the cooling step of the sample or to the singularities and atomic-scale mechanisms at the lower temperature limits. Therefore, to advance our knowledge on some of these issues, we performed a detailed X-ray Photoelectron Spectroscopy (XPS) study of the chemistry of the uppermost layers of Si samples (with native oxide) cooled under the residual vacuum of the etching device or within the XPS chamber. One of the main advantages of our facility [7] is that the sample can be transferred in vacuum at low temperature from the etching device to the XPS analysis chamber (quasi *in situ* condition).

When cooled down under the high vacuum level of the etching device (10⁻⁶ mbar), the Si surface sees its chemical state practically unchanged from room temperature (RT) to -120 °C. However, for lower temperatures, water physisorption becomes the major phenomenon. XPS signals from the Si substrate and the adventitious carbon contamination are buried progressively under this water layer (Fig. 1 and Fig. 2). The thickness of the H₂O layer as well as its concentration on the Si surface increase exponentially as temperature decreases, following an inverse Arrhenius law for the latter (Fig. 3). Physisorption of fluorine is also detected from -100 °C, although less markedly; from -120°C these species seem to be incorporated within the growing H₂O layer (Fig.4). Both molecules desorb when the sample is returned to RT, which attests the physical nature of the adsorption/desorption mechanism.

These results demonstrate the impact of the residual gases present in the plasma reactors and are powerful to establish the limits in the ranges of operation in cryogenic etching and deposition. By re-examining some of the data presented previously (at PlaCEP2022 and PSEM2022), the presentation will also address the possible role of water physisorption during the modification step in an atomic layer etching processes.

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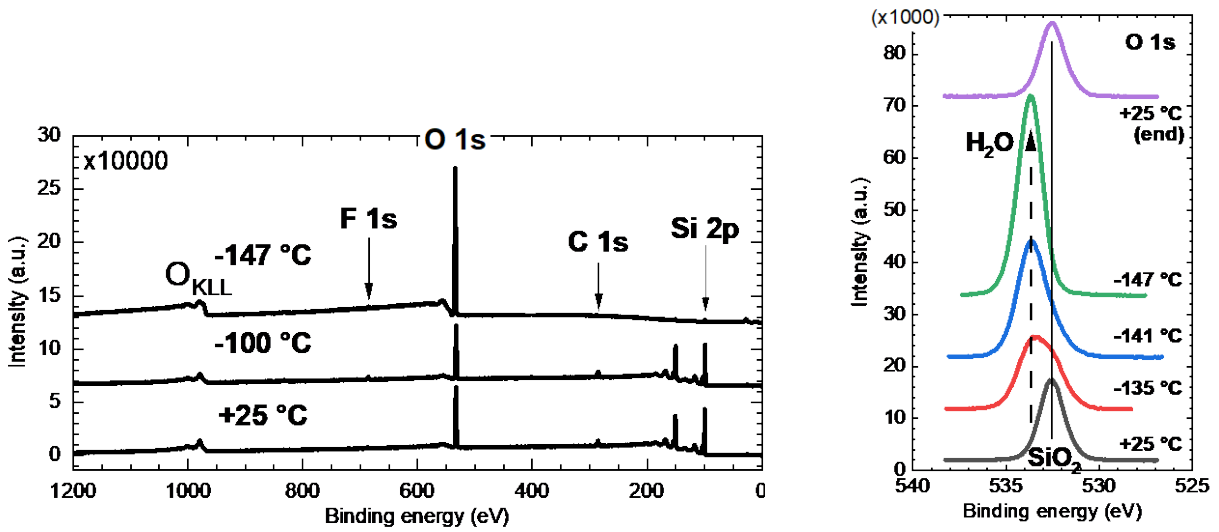


Figure 1: XPS survey spectra recorded for the Si sample at 25°C, -100°C and -147°C, showing the burying of the Si 2p and C1s signals.

Figure 2: Evolution with temperature of the O1s spectra showing the growth of a H₂O component;

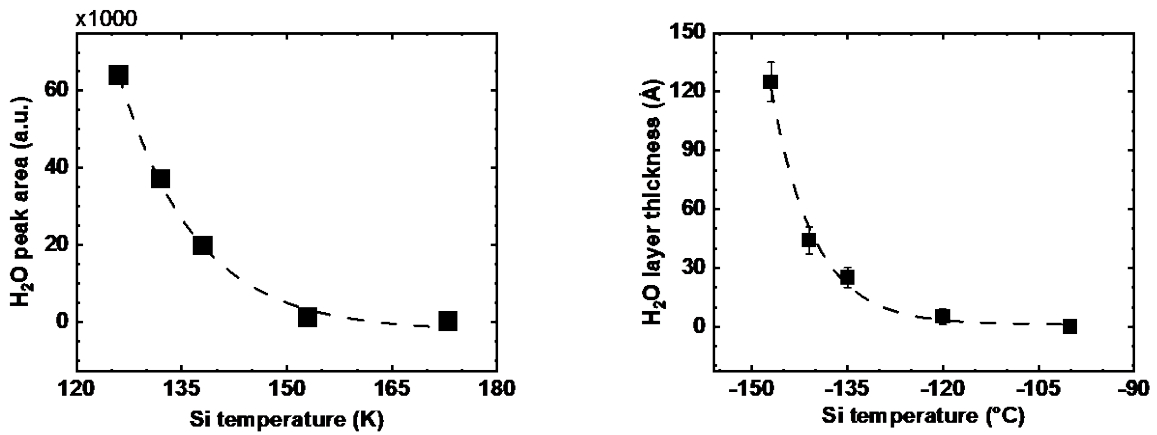


Figure 3: Intensity of the O1s (H₂O) component (right) and evolution of the H₂O layer thickness (left) versus sample temperature.

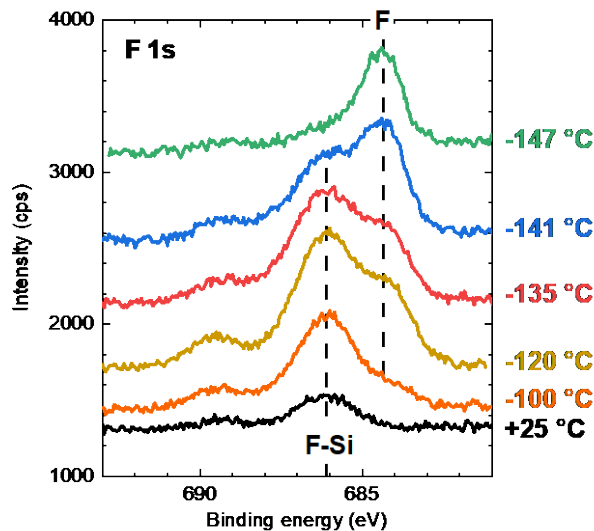


Figure 4: Evolution with temperature of the F1s spectra.