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Plasma cryogenic etching processes: what are the mechanisms involved at very low temperature?

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In 1988, the first cryoetching process of silicon was introduced by a Japanese team to form anisotropic profiles [1]. The substrate was cooled down to a very low temperature (of the order of -100°C) in order to freeze chemical reactions on vertical sidewalls while etching the structure bottom submitted to the ion bombardment. Some years later, it was shown that a passivation mechanism was actually responsible for the etching anisotropy [2]. Standard deep cryoetching of silicon consists in using a plasma mixture of SF_6/O_2 to etch silicon and form a SiO_xF_y inhibiting film at the sidewalls. Many cryogenic etching experiments have been reported showing high aspect ratio structures and interesting behaviors by varying different parameters, but without completely explaining the physical and chemical mechanisms behind this particular process at low temperature [3-5]. The role of physisorption of SiF_4 in the cryogenic process was highlighted by Tinck *et al.* who performed molecular dynamic modelization at low temperature [6]. The SiF_4 physisorbtion was also investigated experimentally to better understand the involved mechanisms in the formation of the passivation layer [7]. A plasma of SiF_4/O_2 regularly injected during the process can help in enforcing the passivation layer: this is the so-called StiGer process [8]. An example of deep etching of submicron trenches in silicon is given in Fig. 1.

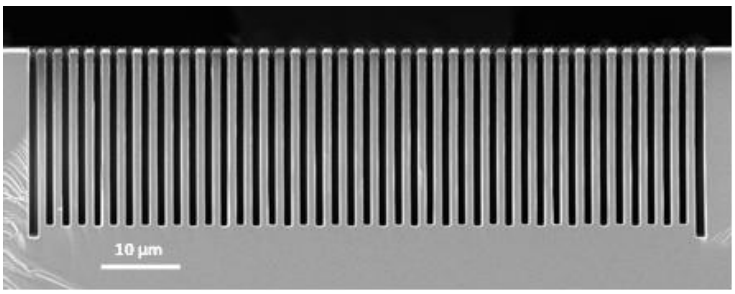

Fig. 1: High aspect ratio trenches obtained using the STiGer process

Decreasing the wafer temperature can also be useful to reduce the diffusion of reactive species at the surface. For example, when etching porous low K materials, the diffusion of reactive species can damage the material and modify the K value. Lowering the wafer temperature can help in maintaining the quality of the material as it was shown in [9, 10].
More recently, a cryogenic process was used for atomic layer etching (ALE) of SiO_2. Instead of initiating a plasma of C_4F_8 at the beginning of each cycle, the gas was simply injected and C_4F_8 molecules could physisorb at the SiO_2 surface. This step was followed by a plasma of argon to induce chemical reactions at the surface. An example is shown in Fig 2 where the evolution of the thickness of a SiO_2 layer measured by in-situ ellipsometry is shown versus time during 8 cycles for two different temperatures of -110°C and -120°C. At -110°C, no etching is observed whereas an etch per cycle of 0.3 nm is obtained at -120°C. Mass spectrometry analysis was also carried out to study the adsorption and desorption of different species during the process. The control of physisorbed species is crucial in this particular process: If the surface residence time of physisorbed molecules is too small, no etching occurs

during the argon plasma step. One of the advantages of using this cryogenic process for ALE relies on the low contamination of the reactor walls, avoiding process drift.

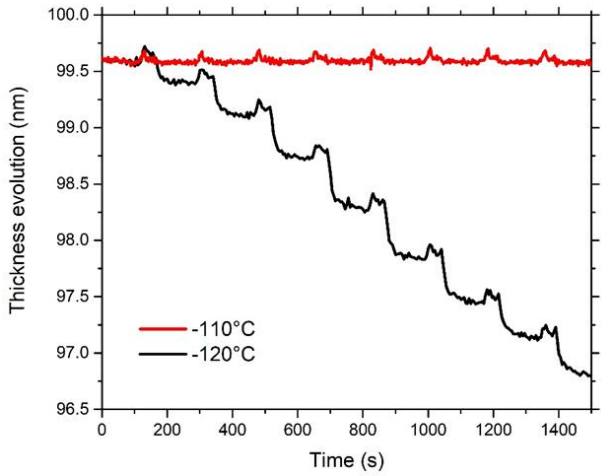

Fig. 2 : Thickness evolution of the SiO_2 layer versus time during 8 cycles for two different temperatures.

Finally, cryo ALE experiments have been investigated using SiF_4/O_2 plasma as a modification step, before the Ar plasma step. By reducing the temperature, the regime can be tuned from deposition to etching. In-situ XPS analyses were performed at IMN at Nantes. It was shown that the stoichiometry oft he deposited layer is significantly modified when the substrate is cooled. This property could be used to selectively etch Si_3N_4 layers while a deposition regime was obtained on Silicon.

All these different aspects of cryogenic processes will be presented more in detail to better understand the involved physical and chemical mechanisms.

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