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## **Investigating the nucleation of Al<sub>2</sub>O<sub>3</sub> on clean Si in ALD : plasma post and pre-processing**

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This study is dedicated to the investigation of the nucleation behavior of Al<sub>2</sub>O<sub>3</sub> deposited by ALD via TetraMethylAlumina (TMA) and H<sub>2</sub>O on clean Silicon. We already showed in previous studies<sup>1,2</sup> that this nucleation may follow a island growth regime, with a seed density ranging approximatively at 0.5% of the Silicon surface density. This is supposed to lead to the growth of non-continuous layers, for thicknesses lower than 3nm. In this work, we first study the oxidation in plasma of layers (supposedly Al<sub>2</sub>O<sub>3</sub>) grown after a few number of cycles (13 to 50). The fact that there is a noticeable oxidation of layers thinner than 5nm confirms the non-continuous character of these last.

We then try to optimize the in-situ plasma pre-processing, for which we already demonstrated<sup>1,2</sup> that the creation of an intermediate layer by nitridation of the Si gave an instantaneous linear growth regime of Al<sub>2</sub>O<sub>3</sub> i.e. a seed density at 100% on the initial surface. We tried to optimize this intermediate layer, using different source of gases instead of N<sub>2</sub>-NH<sub>3</sub> used previously<sup>1,2</sup>, which presented the drawback to give a relatively thick layer (1.5nm) with a low material density. We compare three different compounds for the plasma pre-processing : N<sub>2</sub>, N<sub>2</sub>+O<sub>2</sub> and O<sub>2</sub>, by performing 30cycles of Al<sub>2</sub>O<sub>3</sub> deposition. Observations by TEM and EDX show that the pre-treatments involving N<sub>2</sub> are effective and optimum, creating a thinner interface (0.5 nm), and a continuous layer of Al<sub>2</sub>O<sub>3</sub> with a thickness of 3nm, as expected for a continuous growth. Using plasma O<sub>2</sub> only does not give so positive results, with a thicker interfacial layer and a thinner deposited Al<sub>2</sub>O<sub>3</sub>.

Finally, an attempt was made using in-situ plasma SF<sub>6</sub> pre-treatments. The purpose was to avoid the creation of an intermediate layer, as obtained by nitriding/oxidizing plasmas. We hoped to improve the nucleation on Si by removing the Si-H bonds leaved on the Si surface by wet cleaning with diluted HF, because these Si-H bonds were supposed to be at the origin of the low density of seeds for ALD deposition. The first experiments exhibit unattended results : the deposition kinetic is strongly reduced, with a complex dependence with the parameters of the plasma (time , pressure and power). After further "optimization", we obtain a case were no deposition is observed till 120cycles, and an apparent deposited thickness of 12nm after 250cycles. For this last experiment, we observed, for the first time, by AFM nuclei of Al<sub>2</sub>O<sub>3</sub>, confirming that, for this case the initial seed density is around 0.002% of the Si surface density. Assumptions for the explanation of these results are made, involving cleanness of the surface, and leading for promising results in micro-electronic applications.

[1] : Gakis et al., J. Appl. Phys., 2019[

2] : Gakis et al. RAFALD 2019 Toulouse