



# Accurate depth profiling of dry oxidized SiGeC thin films by extended Full Spectrum ToF-SIMS

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## ABSTRACT

The abundance of work on SiGe based devices demonstrates the importance of compositional characterization of such materials. However accurate SIMS depth profiling of SiGe, and especially of SiGe/silicon dioxide interfaces can be difficult due to matrix effects.

Therefore, we highlight here the improvements brought by the extended Full Spectrum protocol, presented in previous works and allowing minimization of matrix effects. Previous studies on this protocol showed that it was extremely precise and reproducible for Ge and impurity quantification in non oxidized matrices. In this study we thus investigated its accuracy for *simultaneous quantitative depth profiling* of both matrix elements (Si, Ge, O) and impurities (C) in  $\text{Si}_{0.82}\text{Ge}_{0.16}\text{C}_{0.02}$  layers annealed in oxidizing atmosphere, by comparing results with more classic protocols. The profiles provided by the extended Full Spectrum protocol were found to be more accurate than the others, especially around interfaces. This results in a better comprehension of the behaviour of SiGeC layers under oxidizing anneal and thus allows the fabrication of very well controlled three dimensional Ge nanowire structures for next generation devices.

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## 1. Introduction

Nowadays, Complementary Metal Oxide Semiconductor (CMOS) planar technology presents limits in terms of scaling, performance and power consumption. Nanowire (NW) technology appears to be an attractive alternative [1]. It allows the improvement of electrostatic gate control and, consequently, the reduction of short channel effects. Nevertheless, NWs' low diameters limit the current density in devices. To overcome this issue, nanowires can be stacked one upon the other, with a current density gain per area [1]. Rather high electron mobilities have been achieved in Si NW-based nMOS transistors [1]. Hole mobility in p-type MOS devices can be slightly improved by switching from Si to compressively strained  $\text{Si}_{0.8}\text{Ge}_{0.2}$  NWs, as shown in Ref. [2]. Higher Ge content SiGe (up to 60%) should yield higher hole mobilities and thus higher performance pMOS devices [3]. The condensation technique allows the Ge enrichment of low Ge content SiGe layers by a selective oxidation of Si compared to Ge [4]. This technique has been used to fabricate high Ge content SiGe-On-Insulator (SGOI) substrates, with excellent hole mobilities achieved in planar pMOS devices built on top [2]. However, Ge enrichment can also be used to obtain Ge nanowires [5]. In Ref. [6], Saracco et al. proposed a new top-down method based on the Silicon-On-Insulator (SOI)

technology to fabricate 3D suspended Ge-rich nanowires by Ge condensation, which is completely CMOS compatible. In-situ alloying with C atoms of the SiGe layers during epitaxy can help reduce the compressive strain in these layers and thus avoid strain relaxation during the fabrication process [6,7]. An in-depth knowledge of the behaviour of such layers during high temperature dry oxidation (such as Ge and C diffusion and/or condensation pattern, oxidation rate, etc.) would in that respect be most useful for process optimization.

In this paper, we thus report on the accurate characterization, using Time of Flight Secondary Ion Mass Spectrometry (ToF-SIMS), of the structural properties of enriched SiGeC test layers obtained with the process presented in Ref. [6]. SIMS (and ToF-SIMS) usually suffers from non linearity of its Relative Sensitivity Factors (RSF) when profiling through films with various compositions. This is particularly the case in enriched hetero-structures, which successively feature a silicon dioxide ( $\text{SiO}_2$ ) layer, a Ge-rich SiGe layer, and a Ge-poor SiGe layer followed by the substrate (Si or SOI). Such dramatic changes in matrix material undoubtedly lead to hampered SIMS quantification of the different elements of interest (Ge and impurities if present), especially at the interfaces between those materials. To overcome those matrix effects, we resorted to the extended Full Spectrum (ext. FS) protocol proposed in Ref. [8]. This protocol, which allows simultaneous quantification of matrix elements, was shown to be particularly useful for the study of annealed intrinsic or doped (with B, P or C) SiGe/Si superlattices [9]. It was also found by Ferrari et al. [10] that the protocol helped reducing

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matrix effects through oxides. Recent results also show the accuracy of this approach for quantification of Ge and dopants in SiGe/Si stacks [8] and of Si, O, Hf and N in silicon and hafnium oxide layers [11]. We will discuss in this paper its accuracy for the quantitative depth profiling of matrix elements in dry oxidized SiGeC layers by confronting its results with those obtained using more established protocols. We will then use it to highlight typical behaviour of SiGeC layers during dry oxidation.

## 2. Experimental details

For this study, three identical samples were grown on different wafers with an Epi Centura Reduced Pressure - Chemical Vapor Deposition (RP-CVD) industrial cluster tool from Applied Materials. Samples consist of epitaxially grown,  $\sim 70$  nm thick  $\text{Si}_{0.82}\text{Ge}_{0.16}\text{C}_{0.02}$  layers following the procedure described in Ref. [12]. Samples were grown at  $650^\circ\text{C}$ , 20 Torr on SOI wafers with a  $\sim 70$  nm thick Si layer on top of the buried oxide (BOX).  $\text{SiH}_2\text{Cl}_2$ ,  $\text{GeH}_4$  and  $\text{SiCH}_6$  were used as gaseous precursors for the epitaxy of the SiGeC layers. The SiGeC layer thickness was chosen lower than critical thickness of plastic relaxation to guarantee that the structure is strained and without any crystalline defects [13]. One sample was kept as deposited, and the two other were then annealed in a Tempress horizontal furnace for 30 min or 60 min at  $900^\circ\text{C}$  in dry oxygen, in order to initiate the Ge enrichment process. The oxidation temperature used was lower than the Ge melting temperature ( $T_{\text{Ge}} = 937^\circ\text{C}$ ) in order to preserve crystalline quality during the whole process. Previous studies have shown that the process used to enrich the SiGe layer led to a conservation of the total amount of Ge [14].

ToF-SIMS data was obtained with a TOF SIMS V from ION-TOF GmbH with a Caesium or Oxygen ion source for abrasion and a Bismuth liquid-metal ion source for analysis, all beams being incident at  $45^\circ$ . When using oxygen sputtering, profiles were acquired by analysis of positive secondary ions, with the same data treatment protocol as the one used by Jiang et al. [15] (even though the  $45^\circ$  incidence imposed by our instrumental setup can yield to significant surface roughening while profiling). In the case of caesium abrasion, profiles were acquired by analysis of positive secondary ions following the MCs protocol [16–18] and by analysis of negative secondary ions, following the ext. FS protocol [8]. The latter enables precise simultaneous profiling for dopant and matrix species and is thus preferred for this study, MCs and  $\text{O}_2^+$  abrasion protocols being only used to compare with the ext. FS profiles. Caesium abrasion was carried out with a  $500\text{ eV Cs}^+$  beam ( $250\ \mu\text{m} \times 250\ \mu\text{m}$  raster), while oxygen abrasion was performed with a  $500\text{ eV O}_2^+$  beam ( $400\ \mu\text{m} \times 400\ \mu\text{m}$  raster). For analysis, a  $25\text{ keV Bi}_3^+$  beam was used for the MCs protocol to enhance  $\text{MCs}_n^+$  ion yields, while a  $25\text{ keV Bi}^+$  beam was employed for  $\text{O}_2^+$  abrasion and ext. FS protocols  $80\ \mu\text{m} \times 80\ \mu\text{m}$  (for ext. FS and MCs) or  $125\ \mu\text{m} \times 125\ \mu\text{m}$  (for  $\text{O}_2^+$  abrasion protocol) rasters in the centre part of the sputter crater were used for data collection. The analysis chamber pressure was kept between  $7 \times 10^{-8}$  Pa and  $1 \times 10^{-7}$  Pa while profiling with caesium. It was set at  $2 \times 10^{-6}$  Pa while profiling with  $\text{O}_2$  (with a slight oxygen flooding). Cycle time for ext. FS protocol was set to  $80\ \mu\text{s}$  to allow observation of secondary ions up to  $\text{Ge}_6^-$  ( $m = 435.5\ \mu$ ). No electron flooding was applied as no significant charge compensation was observed during profiling of the samples, as attested by the mass resolution  $M/\Delta M > 3000$  obtained on the whole mass range. However, due to its intrinsic normalization method, the output of quantified profiles obtained with the ext. FS protocol is necessarily in atomic % (at%), i.e. it relates to the matrix composition and not directly to concentrations, as it is usually the case in SIMS. To transform these values into concentrations in  $\text{at}/\text{cm}^3$ , one needs to model the density of the layers, which we did using the same procedure than in Ref. [8] for SiGe and Si layers. For silicon dioxide, a standard

density was used ( $\rho(\text{SiO}_2) = 2200\text{ kg}/\text{m}^3$  [19]). Accurate variable sputter rate was determined thanks to the procedure described in Ref. [8]. In addition, the sputter rate in  $\text{SiO}_2$  was determined using a reference layer measured by spectroscopic ellipsometry. In average, the sputter rate in  $\text{SiO}_2$  was found to be  $\sim 1.4$  time this in Si using  $500\text{ eV Cs}^+$  sputter and therefore quite close to this of SiGe with high Ge content situated right below the oxide layer. The variation of sputter rate at the interface of  $\text{SiO}_2$  and SiGe was therefore accounted by an error function running for  $\pm 2$  nm around the interface as determined by O half signal. Eventually, a HF dip was performed on parts of the three wafers to remove the oxide layer produced during enrichment (or to remove the native oxide layer on the as deposited sample) prior to ToF-SIMS analysis.

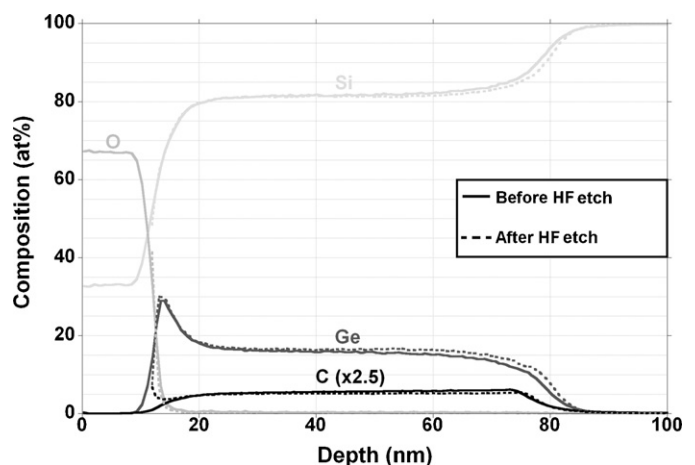
## 3. Results and discussion

### 3.1. Accuracy of the ext. FS protocol

Profiles of the wafer oxidized for 30 min in dry oxygen at  $900^\circ\text{C}$  (with and without HF removal) obtained with the ext. FS protocol are displayed in Fig. 1. These profiles display features which are typical of the ext. FS protocol, i.e., a simultaneous quantification of all matrix elements combined with a good signal/noise ratio. Briefly, from what we can observe in Fig. 1 (full lines), the ext. FS protocol succeeds in quantifying all matrix elements in the successive layers. Precisely we can list:

- (i) A  $\text{SiO}_2$  layer, where the average composition is found to be 66.8 at% O and 33.1 at% Si, remaining 0.1 at% being Ge and C related noise (this region is only observed as a native layer on the as deposited sample);
- (ii) Then, a Ge enriched SiGeC layer, with a varying Ge composition as a function of depth, typical of condensation processes;
- (iii) Then, a SiGeC layer with stable Ge and C concentrations, of average composition 16.2 at% Ge and 2.2 at% C, corresponding to the original SiGeC layer;
- (iv) And finally, the Si layer underneath, where the average composition is found to be 99.8 at% Si, the remaining 0.2 at% being Ge, O and C related noise.

An enrichment is clearly observed on the Ge profile. By contrast, no such behaviour is seen on the C profile. The profile of the sample oxidized for 60 min (not shown in Fig. 1) exhibits similar features.



**Fig. 1.** Ext. FS ToF-SIMS profiles of the Si, the Ge and the C atoms in the SiGeC layer oxidized for 30 min under dry oxygen at  $900^\circ\text{C}$  without (full lines) or with (dashed lines) a full HF etching of the oxide layer. The depth scale of the latter sample has been shifted so that the depth of the Ge peak is the same for both configurations. Notice the ordinates, in at% and in linear scale.

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