

# From Lab to Fab: In-line SIMS for Process Control in Semiconductor Manufacturing

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## ABSTRACT

This paper demonstrates the successful lab-to-fab transition of dynamic secondary-ion mass spectrometry (SIMS). In comparison to traditional lab SIMS, the in-line version is optimized for automated wafer and measurement sequence handling and high throughput measurements in small areas. Key advantages are fast turn-around time, reduced scrap, increased yield, and the measured wafer can continue processing in the manufacturing line. The benefits of in-line SIMS in the production environment are demonstrated for several use cases: matching and monitoring the long-term stability of epitaxy tools on monitor wafers, process optimization and monitoring of epitaxial Si and SiGe layers on blanket and patterned wafers with blanket metrology targets, measurement of implant and dopant profiles on blanket and patterned wafers, and characterization of the Ge and B diffusion in multi-layer stacks stimulated by high-temperature annealing. Additionally, the characterization of the source/drain epitaxy in a fully integrated nanosheet gate-all-around transistor architecture is demonstrated and discussed. The results are compared to off-line lab SIMS and alternative methods where available.

**Keywords:** In-line metrology, SIMS, gate-all-around, nanosheet, epitaxy, diffusion, dopant profiling, implant profiling

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## 1. INTRODUCTION

Ever increasing process complexity drives the metrology needs and the transition of traditional lab techniques into semiconductor fabrication facilities. For example, X-ray diffraction (XRD), X-Ray fluorescence (XRF), X-ray reflectometry (XRR), X-ray photoelectron spectroscopy (XPS), spectroscopic ellipsometry, or optical scatterometry are invaluable in-line metrology techniques to optimize and monitor composition, thickness, strain, residues, and many more quantities on blanket films and fully integrated structures [1]. Most recently, Raman spectroscopy was introduced as an in-line metrology technique to measure composition and strain in nanosheet devices [2].

Secondary-Ion Mass Spectrometry (SIMS) is a widely used analytical method for characterizing the chemical composition of thin films, concentration and profile of dopants and implants, as well as trace element analyses since it provides the widest detection range for the concentration of a species of interest within a host matrix of all available analytical techniques [3]. So far, SIMS was only available as an off-line lab tool with limited throughput, minimum measurement area and maximum sample size limitations, and long turn-around times when transferring wafers from semiconductor fabrication facilities to offline measurement laboratories. Many applications can benefit from a fully automated SIMS option within the semiconductor fabrication ecosystem. For example, Si and SiGe epitaxial layers are grown with in-situ doping using gas phase adjustment [4]. The growth dynamics highly dependent on gas flow rates, chamber pressures, and temperature. This can lead to gradients even on blankets and requires careful process development and monitoring. For this application, in-line SIMS provides immediate feedback and speeds up the process development and epitaxial deposition tool recovery after preventative maintenance actions, for example. Different growth dynamics are expected for blanket film deposition compared to epitaxial growth within confined spaces such as source/drain epitaxy within the deep trenches of a nanosheet transistor structure [5]. Established in-line metrology solutions such as XPS or XRF typically do not have the necessary sensitivity and/or the high aspect ratios may limit their use to characterize the dopant profile in the source/drain material [1]. In-line SIMS can help to characterize the epitaxial growth in the source/drain region, including

dimensions and dopant profiles, and may be applied on production wafers that continue in the route to ensure process stability without sacrificing valuable wafers.

In this publication, dynamic in-line SIMS is introduced as the latest analytical technique that transitions from the laboratory environment into semiconductor fabrication facilities. The benefits of having a fully automated in-line SIMS tool for statistical process control, process development, epitaxial tool monitoring, and production wafer metrology are outlined. The potential application of in-line SIMS for process monitoring of source/drain epitaxial growth in fully integrated nanosheet transistors is highlighted.

## 2. EXPERIMENTAL DETAILS

SIMS utilizes a high-energy ion beam to sputter secondary particles out of the sample surface (Fig. 1 (a)). Positive or negative ions are extracted, separated by mass to charge ratio, and counted on a suitable detector [6]. In a dynamic SIMS measurement, the ion beam is rastered over a small area of the sample to remove material layer by layer. The species of interest are detected as a function of sputter time creating profiles as shown in Fig. 1 (b). The sputter yield for specific species depends on the matrix they are incorporated in. Therefore, quantitative analysis requires reference samples to determine erosion rates and relative sensitivity factors that allow to translate the profile from counts in dependence of time to concentration in dependence of sputter depth [6]. A small recess remains in the measurement area after completion of the measurement.

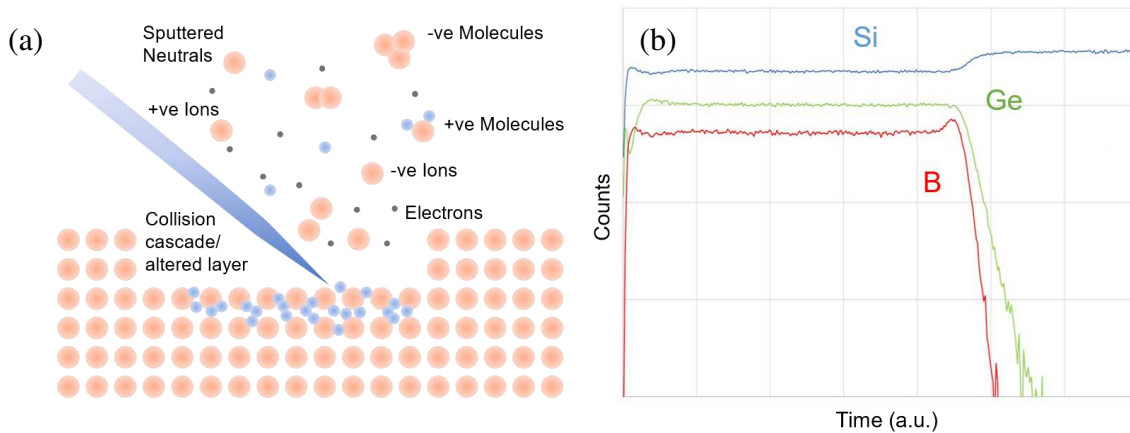


Figure 1. (a) Illustration of the principle of a SIMS measurement at the microscopic scale. (b) Raw data profile of a dynamic SIMS measurement while sputtering through a SiGe:B layer on Si. Raw detector counts for all three species of interest are traced in parallel over time. The drop in B and Ge counts indicates the interface between the SiGe:B layer and the Si substrate.

Dynamic in-line SIMS requires similar considerations as lab SIMS measurements with some constraints specific to the semiconductor fabrication environment. The species of interest need to be identified and, depending on the tool mass resolution, potential mass interferences from isotopes of neighboring elements or cluster ions composed of lighter elements need to be considered, especially when studying higher masses [6]. Depending on the species used for the primary ion beam and the element of interest, higher secondary ion yields can be achieved either for positive ions or negative ions [6]. The sputter and ionization yields for dopants and implants depend on the host matrix. Therefore, reference wafers of known concentration are needed for absolute calibration of the y-axis of the sputter profile graph in Fig. 1 (b). Relative results can be obtained if no suitable reference wafers are available. Similarly, the sputter rate for any given primary beam condition and sample composition needs to be calibrated on blanket reference films of known thickness in order to translate the time axis in Fig. 1 (b) to a depth information. The ability to focus the ion beam, the penetration depth of the ions defined by the beam energy, and acceptable signal-to-noise considerations restrict the minimum suitable raster size. For in-line measurements, small raster sizes down to  $50 \times 50 \text{ } \mu\text{m}^2$  are desired for high throughput measurements in the scribe lines with low impact on topography. In-line SIMS is optimized for measurements on production wafers intended to continue the manufacturing process. Therefore, in-line SIMS may be considered as a “non-destructive” solution to monitor parameters of interest at various process steps and correlate material properties to in-line electrical test results for faster process optimization, tighter process control, and improved yield. Depending on the application and desired parameters of interest, SIMS requires tailored measurement solutions: while small raster sizes reduce the time for high throughput, deeper

profiling may require larger raster sizes to avoid side wall effects, for example. High beam energies increase the sputter yield and therefore sensitivity, low energy beams provide improved depth resolution. Insulating samples might require charge compensation, for example, by directing free electrons to the sputtered surface.

The Nova Metrion in-line SIMS tool merges some of the benefits of traditional lab SIMS with the requirements of a semiconductor production environment. It is a fully automated, recipe-driven tool for 300mm wafers and high wafer throughput. A contamination-free oxygen ion source is combined with multiple detectors for parallel detection of several species of interest. A small sputter area is used suitable for patterned wafer metrology on production wafers. The beam energy and current are adjustable to meet the application needs. The basic system components of the Metrion tool are shown in Fig. 2.

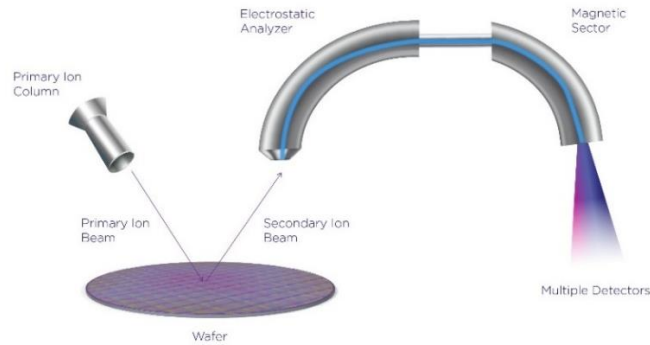


Figure 2. Main components of the Nova Metrion in-line SIMS tool. A contamination-free oxygen ion source is combined with multiple detectors for parallel detection of multiple species of interest.

### 3. RESULTS AND DISCUSSION

To provide meaningful information about a sample of interest, an in-line SIMS tool needs to deliver accurate and repeatable data that correlate well with lab SIMS measurements while focusing on high throughput with minimum sample preparation. A comparison of in-line SIMS data with lab SIMS results obtained for a Si:P film grown epitaxially with in-situ doping is shown in Fig. 3. The in-line SIMS measurement performed on a 300 mm Si wafer is optimized for a compromise between high throughput and data accuracy. Despite a 16 times smaller sputter area, shorter integration time, lower beam current, and accordingly higher noise in the measurement data, the overall shape of the data correlates well with the lab SIMS measurements performed on a diced wafer piece. Utilizing a known reference, both data sets can be calibrated to report the same values for the P concentration.

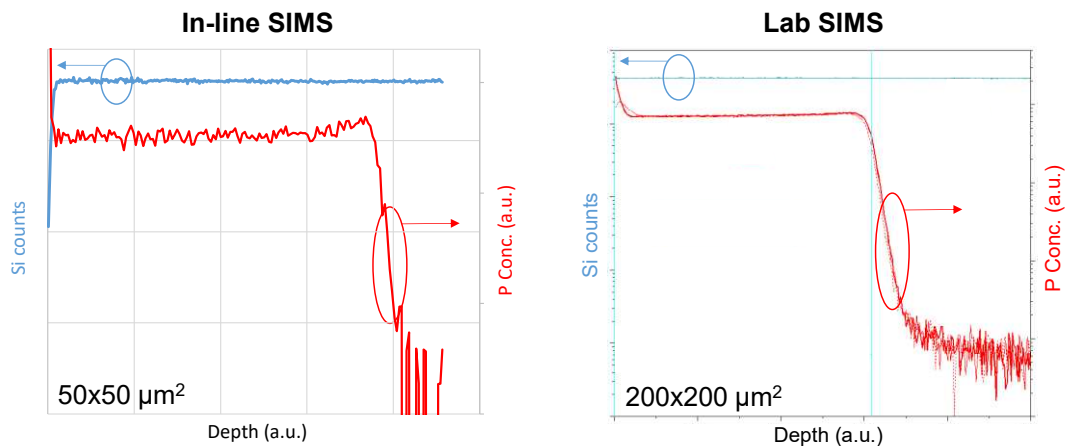


Figure 3. Comparison between an in-line SIMS measurement and a lab SIMS measurement for a Si:P film epitaxially grown on a standard 300 mm Si wafer. The in-line SIMS tool measured on the full wafer and measurement settings were optimized for fast data acquisition on a  $50 \times 50 \mu\text{m}^2$  raster area, while the lab SIMS tool was optimized for best data quality and measured on a small piece of the diced wafer with a 16 times larger raster area ( $200 \times 200 \mu\text{m}^2$ ) and much higher beam current.

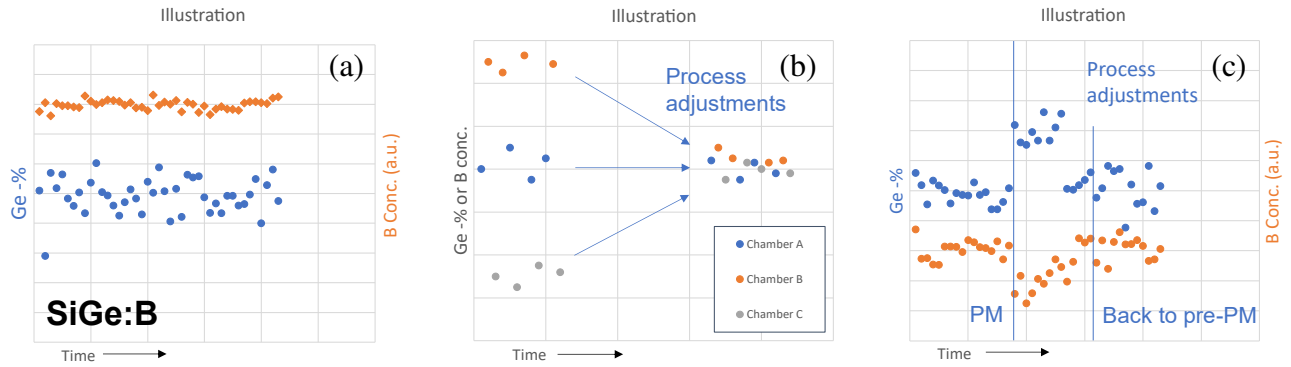


Figure 4. Illustrations highlighting the benefits of in-line SIMS for: (a) monitoring the epitaxy tool stability for an in-situ doped SiGe:B film growth process on Si, (b) matching of three epitaxy tool chambers utilized for the same SiGe:B growth process, and (c) epitaxy tool recovery for a SiGe:B growth process after a preventative maintenance action on the epitaxy tool.

The following subsections highlight applications that benefit from having an in-line SIMS tool available in the semiconductor fabrication facilities.

### 3.1 Epitaxy process characterization and monitoring

Epitaxially grown layers of Si and SiGe are foundational building blocks of nanosheet transistor devices. The composition profile of each layer, the thickness, as well as the profile of any intentional or unintentional dopants are crucial for the device performance. In-line SIMS can provide a direct and fast measure for these critical material properties either on dedicated tool monitor wafers with blanket films or on patterned production wafers with blanket film metrology pads. Illustrations for three different scenarios are shown in Fig. 4. Monitoring both Ge fraction and B concentration in epitaxially grown and in-situ doped SiGe:B layers is critical for a consistent source/drain process (Fig. 4 (a)). In-line SIMS provides a direct measurement of both parameters without any correlation as, for example, present in XRD measurements. When several different chambers on a deposition tool are set up to run the same epitaxy process, tool matching is an important step to provide consistent layer properties. For the SiGe:B material system, in-line SIMS enables immediate feedback for simplified and fast chamber matching (Fig. 4 (b)). Every epitaxy tool must undergo preventative maintenance actions at some point during which critical components such as heaters are exchanged and the chambers are cleaned. This changes the growth dynamics inside the chamber, and seasoning and process parameter adjustments will be necessary to retain previous layer properties. The immediate feedback of the in-line SIMS tool enables fast tool recovery after the preventative maintenance action to significantly increase the up-time of the epitaxy tool (Fig. 4 (c)).

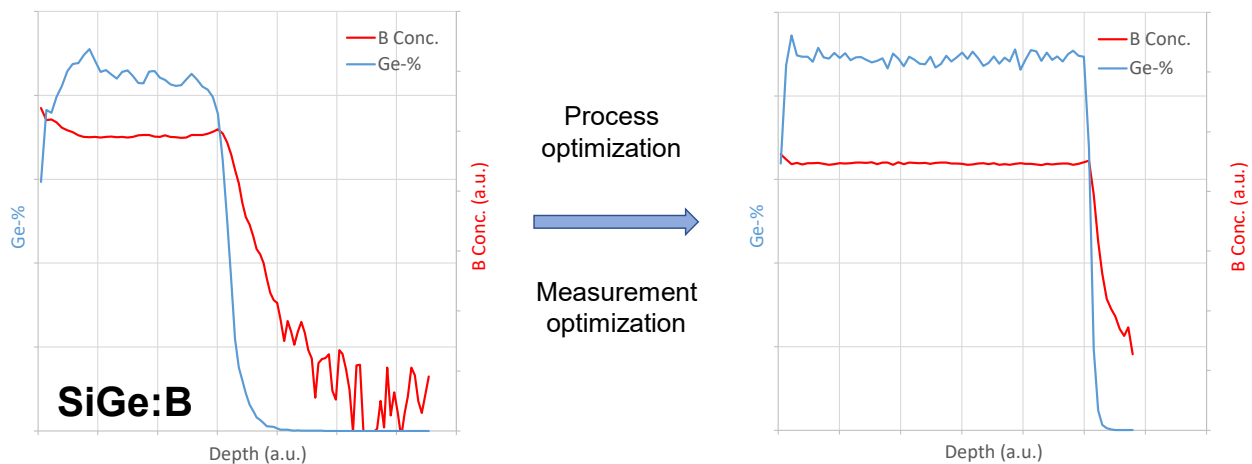


Figure 5. In-line SIMS profile for a SiGe:B epitaxy process before and after process and measurement optimization.

During process development, in-line SIMS may provide quick access to the exact composition and dopant profile of a film of interest. Figure 5 shows measured in-line SIMS profiles for a SiGe:B layer before and after process and measurement optimizations. Before the optimization, surface and interfacial accumulation of B dopants as well as an inhomogeneous Ge fraction profile are observed. After process optimization, homogeneous B and Ge fraction profiles are obtained with significantly reduced interface accumulations. At the same time, the measurement itself was optimized by adding an endpoint detection algorithm that recognizes once the signal for a given species drops, i.e., the point at which the sputter process captured the entire film of interest. This minimizes the measurement time and improves throughput, limits the impact of the measurement on production wafers, and extends the detector lifetime.

Figure 6 shows a comparison between in-line SIMS measurements and lab SIMS data for a Si/SiGe multilayer blanket stack. The in-line SIMS measurement indicates the presence of a growth anomaly at the interface between the bottom SiGe layer and the Si substrate which was confirmed by the lab SIMS measurements. Instead of a Ge signal with a rather symmetric profile, an accumulation of Ge at the interface between the bottom sheet (Bot NS) and the Si substrate is apparent from both data sets. Note, that an inverse trend for the Mid NS Ge fraction profile is apparent in the lab SIMS measurements that is also reproduced in the in-line SIMS data. Such anomalies are not detectable by any alternative metrology techniques.

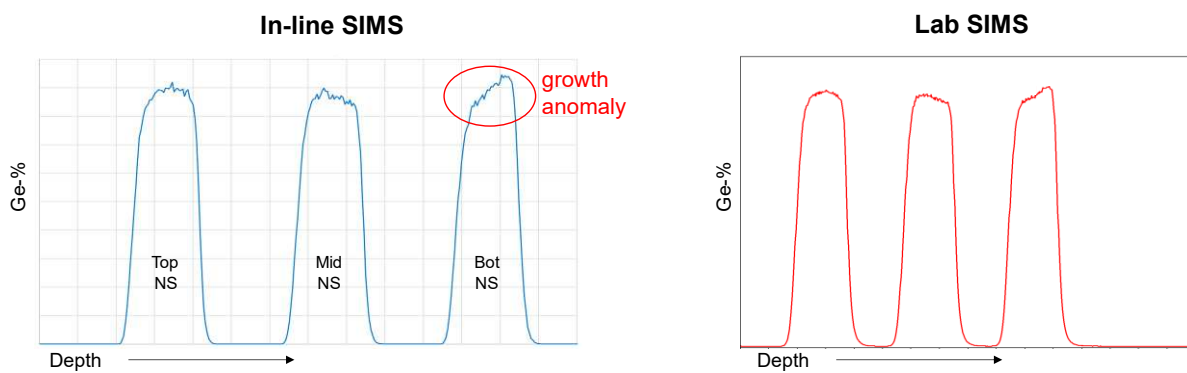


Figure 6. In-line SIMS measurements and lab SIMS data for a Si/SiGe multilayer blanket stack on a 300 mm Si wafer. A growth anomaly for the bottom SiGe sheet (Bot NS) is revealed.

### 3.2 Ion implantation and diffusion experiments

A common application of SIMS is the characterization of ion implant profiles. In order to dope substrates or locally vary undoped blanket films or patterned wafers, highly energetic ion beams of well-defined beam energy and controlled ion current are directed at the sample. Accurate simulation tools provide good predictions of the resulting implantation profile for any given implant condition and sample system [7]. A comparison of in-line SIMS measurements with simulated data for a multi-step implantation process of B in Si wafers is shown in Fig. 7. Excellent agreement between the measured and simulated implant profiles is observed. The in-line SIMS recipe contains an algorithm to provide the maximum concentration, position, and width of the implant peak to provide quantitative feedback for process monitoring and optimization.

The implant process is generally well controlled such that the measured total ion dosage for a given implant step can be utilized to accurately design and characterize SIMS calibration standards [6]. However, it is much harder to predict the effect of high temperature annealing on such profile. Here, the in-line SIMS excels compared to lab SIMS tools as the same wafer can be processed repeatedly and the impact of different anneal temperatures and durations can quickly be characterized, for example, to explore the temperature budget for an entire production process. Figure 8 shows in-line SIMS data for a Si/SiGe blanket stack deposited on a B implanted Si wafer. For the as-grown wafer, the Ge fraction is shown in addition to the B concentration to indicate the position of the SiGe sheets. The wafer was cycled through consecutive anneal steps of three different temperatures, one mid temperature and two high temperature steps (T1 and T2). For repeatable processing and reliable performance of a nanosheet transistor device patterned into this layer stack, it is crucial to avoid any diffusion of B into the Si and SiGe sheets. For low and mid temperature annealing, no B diffusion is apparent, i.e., the as-grown and annealed B profile are virtually identical. After the first high temperature anneal step, the

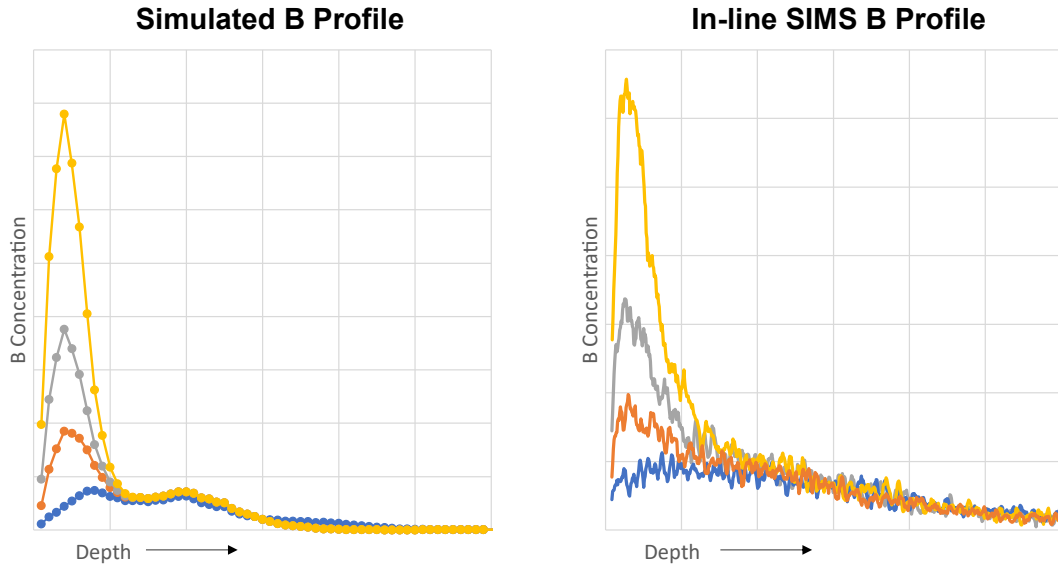


Figure 7. Simulated and measured in-line SIMS profile for an implant experiment of B implanted at different doses and ion beam energies into a 300 mm Si wafer.

B profile is significantly broadened, with reduced B peak maximum value. Specifically, B diffuses into the first SiGe and Si sheets, with some accumulation within the SiGe layer. The subsequent second high temperature anneal step further broadens the B concentration peak and significant diffusion of B into the second SiGe sheet and beyond is revealed. In-line SIMS provides fast and quantitative means to evaluate the temperature budget for this material system.

In nanosheet transistor devices, etch rate dependence on the Ge fraction in SiGe layers is utilized to selectively etch specific layers [8]. Diffusion of Ge from SiGe into Si layers can lead to undesired feature shapes and needs to be avoided. A layer stack containing a high-Ge SiGe layer followed by a bare Si layer, a low-Ge SiGe layer and a Si cap was epitaxially grown on a 300 mm Si wafer. XRD, in-line SIMS, and a high-temperature anneal step were cycled five times to study the Ge diffusion in this layer stack. The last anneal step was five times longer than each of the other anneal steps. Figure 9 shows the in-line SIMS data for this diffusion experiment. Minor Ge diffusion only is found for the low-Ge SiGe layer. The in-line SIMS data indicates a very small increase in layer thickness with apparent small extension of the layer downwards into the Si layer while the interface between the Si cap and the low-Ge layer is aligned for all measurements. Significant

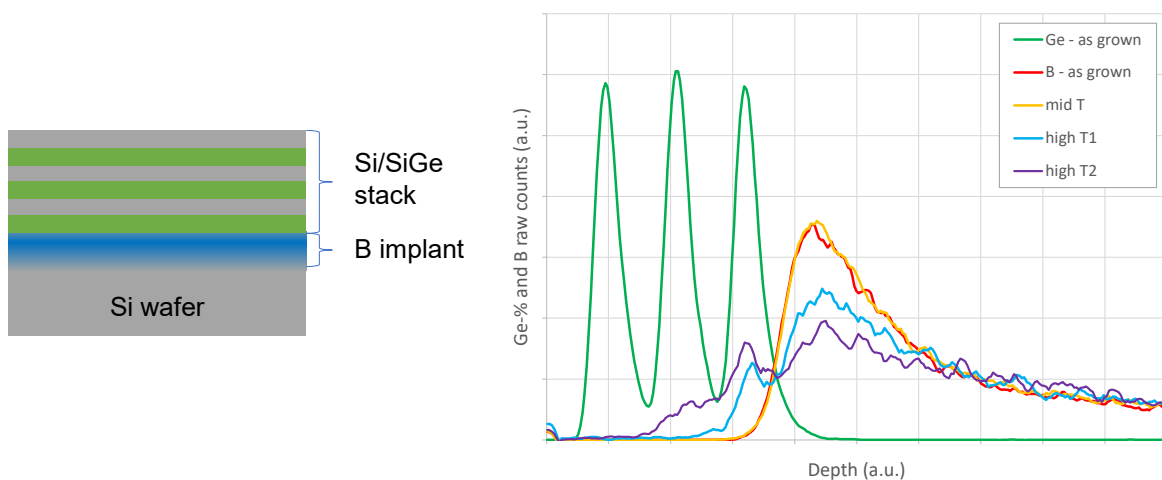


Figure 8. In-line SIMS profiles for a Si/SiGe blanket stack deposited on a B implanted Si wafer. For the as-grown wafer, the Ge fraction is shown in addition to the B concentration to illustrate the position of the SiGe sheets. The wafer was cycled through consecutive anneal steps of three different temperatures, one mid temperature and two high temperature steps.

Ge diffusion is observed for the high-Ge SiGe layer; the average Ge concentration reduces with each anneal step and appears to level off with the last anneal step. Additionally, a rounding of the originally sharp profile is observed at the layer edge with an increase of the layer thickness by diffusion of Ge into the surrounding Si layer and substrate. The diffusion behavior for this stack depends on the initial Ge concentration in the SiGe layer and is driven by the anneal temperature and potential temperature gradients. The anneal temperatures applied here would be too high for a manufacturing flow since they do lead to diffusion of Ge from a high-Ge SiGe layer into surrounding Si.

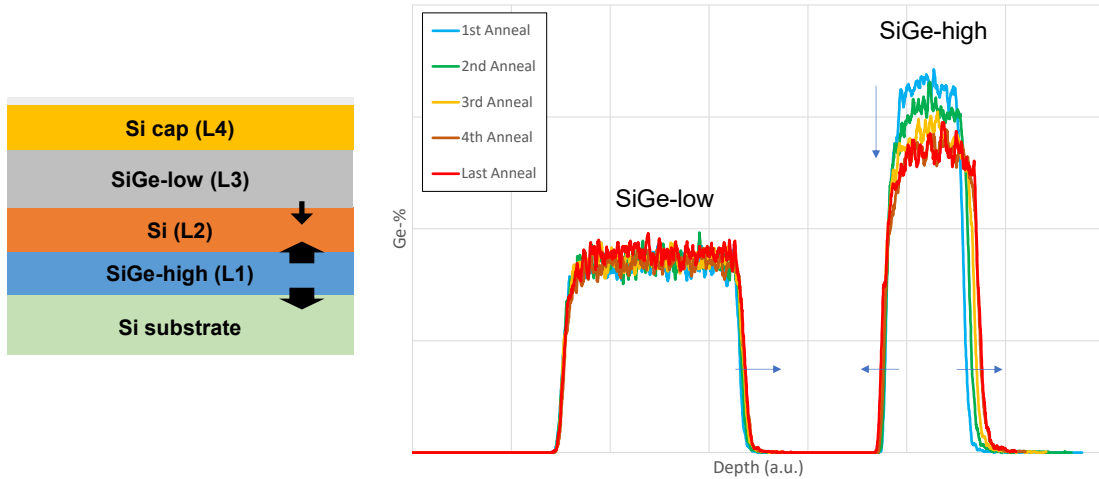


Figure 9. In-line SIMS data for an anneal experiment to study the Ge diffusion in a stack of a high-Ge SiGe layer, bare Si layer, a low-Ge SiGe layer, and a bare Si cap. Data after five anneal steps are compared with the last anneal step being five times longer than each of the previous anneal steps.

A comparison of the extracted layer thicknesses and Ge fractions for both SiGe layers for the in-line SIMS measurements with results from XRD measurements are shown in Fig. 10. The same trends are observed for in-line SIMS and XRD. However, no complementary metrology technique can provide the same detail as the in-line SIMS measurements since model assumptions have to be made for the data analysis such as sharp interfaces or specific Ge gradient profiles, for example. Only in-line SIMS can reveal the Ge profile for each layer and give a direct read of the diffusion behavior.

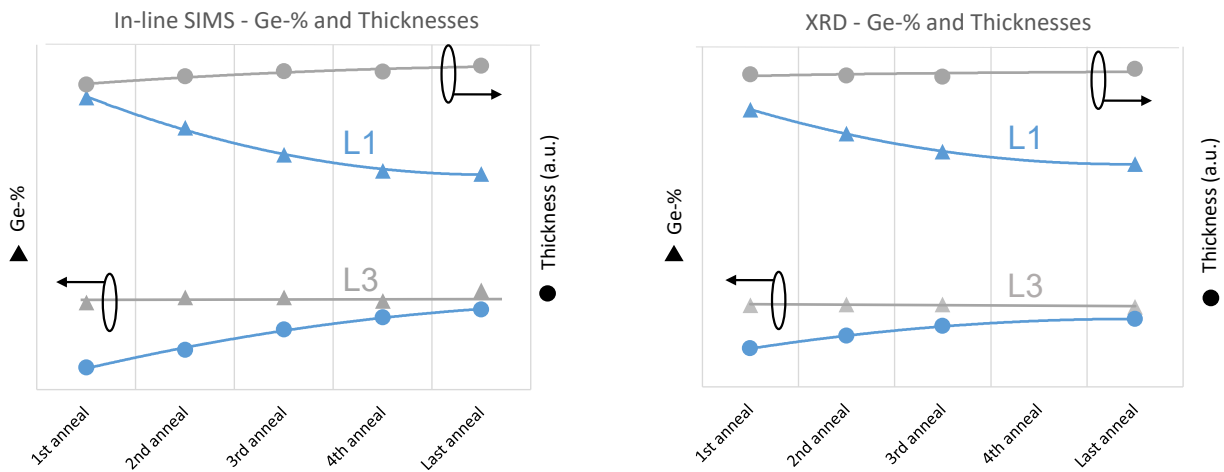


Figure 10. Comparison of the in-line SIMS results and XRD results for the Ge fraction and thicknesses of the high-Ge SiGe layer (L1) and the low-Ge SiGe layer (L3) for the layer stack and in-line SIMS profile shown in Fig. 9.

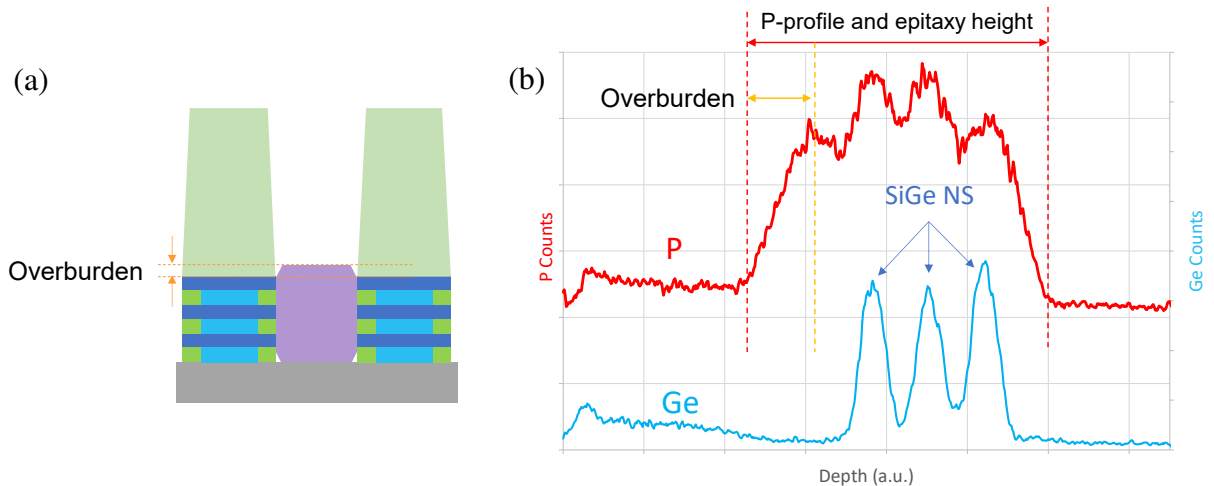


Figure 11. (a) A schematic drawing of a nanosheet transistor structure after source/drain epitaxy for a cut across the gate. The “overburden” parameter is indicated in the drawing. (b) Raw in-line SIMS data for P and Ge species for an nFET nanosheet transistor measured after in-situ doped Si:P epitaxy. Measurements were performed on fully integrated metrology targets in the scribe line.

### 3.3 In-line SIMS on structure measurements

The ultimate goal of in-line SIMS is to measure fully integrated product wafers and correlate material properties to device performance. A challenging application is the characterization of the source/drain epitaxy within the trenches of a fully integrated device architecture. A schematic drawing of a nanosheet transistor structure after source/drain epitaxy for a cut across the gate is shown in Fig. 11 (a). Optimum device performance requires the epitaxially grown “diamonds” to fully cover the Si nanosheets indicated in light blue with controlled “overburden”, i.e., epitaxial growth above the top Si sheet. Raw in-line SIMS data for P and Ge species for a nFET nanosheet transistor measured after in-situ doped Si:P epitaxy is shown in Fig. 11 (b). The measurements were performed on fully integrated metrology targets in the scribe line. The raw Ge counts indicate the position of the SiGe nanosheets. The position of the P profile relative to the SiGe sheets allows to draw conclusions on the quality of the source/drain epitaxial diamond growth. Here, the profile encompasses all SiGe nanosheets which indicates that the epitaxy diamond fully covers all nanosheets as intended and the overburden can be extracted as the distance between the onset of the P signal to the first peak in the P profile. Currently, there is no alternative in-line metrology solution that can monitor the P profile in source/drain epitaxial Si:P in the trenches of a fully integrated nanosheet transistor device. Quantitative analysis of the P concentration requires careful consideration of the individual material volumes in this complex 3-dimensional structure. Such volume information can be obtained through optical scatterometry, for example, and then utilized for SIMS data analysis.

Even without quantitative analysis of the P profile, the raw P data provide critical details on the epitaxy quality and can be used to detect failure modes during the growth process. The in-line SIMS results for a set of fully integrated metrology targets measured in the scribe line of the same wafer is shown in Fig. 12. On these targets, the NS width is varied while the lateral space between the NS stacks is kept constant. Schematic drawings for a cut through the epitaxy diamonds across the NS are shown in Fig. 12 (a). The size of the epitaxy diamond relative to the NS dimensions is in agreement with results of scatterometry measurements (not shown) on the same targets performed before the in-line SIMS measurements. The raw P profile for the smallest NS width shown in Fig. 12 (b) indicates an issue with the epitaxial growth. The upper edge of the P profile barely covers the top Si sheet indicated by the first peak in the P profile with the largest NS width. The bottom edge of the P profile barely extends past the Ge peak associated with the bottom SiGe sheet revealing that the source/drain epitaxy is not properly growing from the bottom NS channel. Again, quantitative results can be enabled by combining in-line SIMS with optical scatterometry in a hybrid metrology approach.



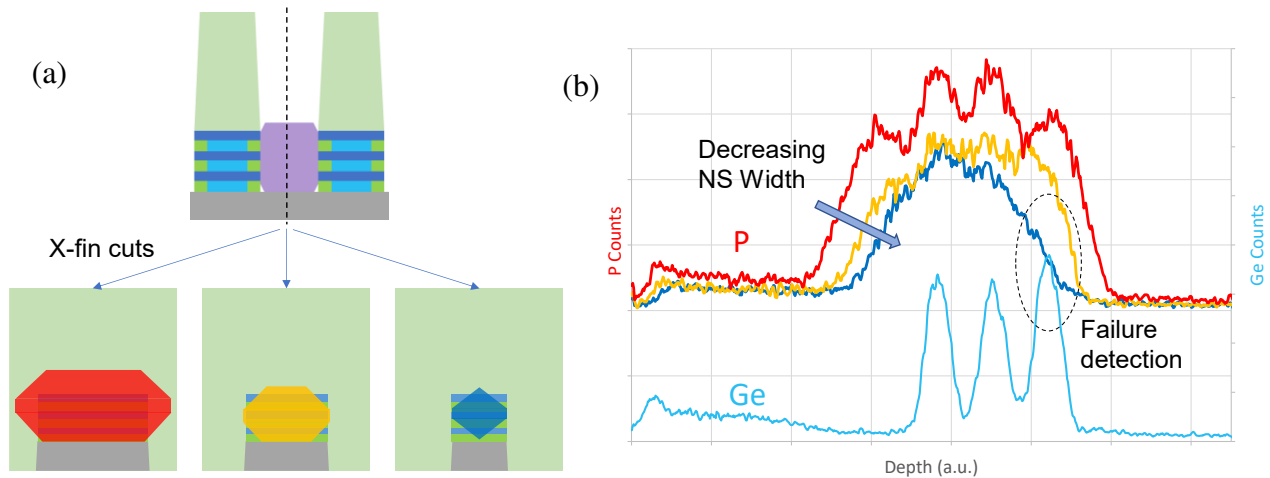


Figure 12. (a) Schematic drawings for a cut through the epitaxy diamonds across the nanosheets (NS) for a set of fully integrated metrology targets in the scribe line with different NS widths but same spacings. The size of the epitaxy diamond relative to the NS width and stack height approximates the results of optical scatterometry measurements (not shown) on the same targets performed before the in-line SIMS measurements. (b) Raw in-line SIMS data for P and Ge species measured on the targets outlined in (a). An area of potential device failure due to incomplete epitaxy diamond coverage of all three nanosheets for the smallest NS width is highlighted.

#### 4. SUMMARY AND CONCLUSIONS

In an environment of ever-increasing metrology demands, dynamic SIMS is the latest technique that transitions from the laboratory to semiconductor fabrication facilities. Fully automated in-line SIMS enables process characterization and monitoring of epitaxy tools and on product wafers at unprecedented turn-around times. Epitaxy tool stability for all parameters of interest can be monitored without correlation between parameters. Chamber matching and fast recovery after preventative maintenance allow for tighter process control and increased tool uptime. Immediate feedback results in faster process development cycles; for example, temperature budgets can easily be explored to avoid undesirable material diffusion. While locally invasive, the sputter crater for in-line SIMS is small enough without globally impacting the wafer to allow sending product wafers back to production, thus providing an overall “non-destructive” metrology solution that keeps the wafers moving. This enables process monitoring at critical steps on the same blanket or patterned wafer and direct correlation of material properties with in-line test results. Measurements on fully integrated proxy targets in the scribe line allow characterization and monitoring of source/drain epitaxy within 3-dimensional nanosheet transistor structures. Raw data analysis can provide critical process parameters such as the epitaxy diamond height and overburden, therefore allowing for early failure mode detection. Hybrid scatterometry and SIMS metrology can enable quantitative analysis of the dopant profile in nanosheet transistor devices.

In conclusion, in-line SIMS provides wafer-level materials characterization for process development, high-volume manufacturing, and statistical process control. Fully automated hardware and measurement sequences, with recipe-driven measurement on 300 mm wafers enable high throughput, accuracy, and repeatability for material analysis on monitor and production wafers.

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