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# Interface and Abruptness Characterization of Si/SiGe Superlattice Epitaxial Films

**The process of applying both PCOR-SIMS$^{SM}$ and STEM imaging to the same Si-SiGe multilayer sample demonstrates the individual strengths of both techniques and how they can be combined to form a complete understanding of the sample.**

In recent years the interest in Si/SiGe superlattice structures for a variety of semiconductor applications has grown. These layers are typically on the order of several nanometers with the desire to have extremely sharp or smooth interfaces. Characterization of such intricate layers for layer thickness, %composition and interface abruptness is extremely challenging. At EAG Laboratories, we have developed several metrologies to address this complex characterization problem. In this article we will discuss two commonly used techniques; Secondary Ion Mass Spectrometry (SIMS) and Scanning Transmission Electron Microscopy (STEM). Each technique has its own unique capabilities, but when used together they provide comprehensive characterization solution, especially for thin layers.

## Secondary Ion Mass Spectrometry (SIMS)

An energetic focused ion beam rastered on the sample of interest causes atoms and molecules to be ejected from the top few monolayers. Figure 1 below illustrates this process at an atomistic level.

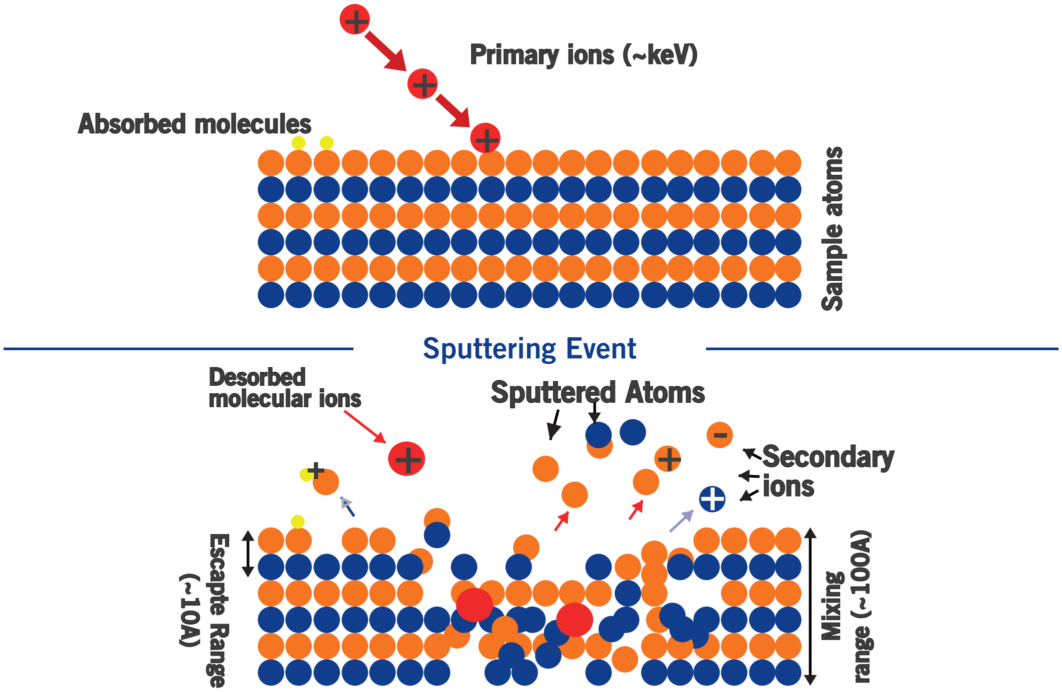


Figure 1: Basic sputter process in Dynamic SIMS. The mixing range is proportional to the depth resolution, i.e., layer definition resolution.

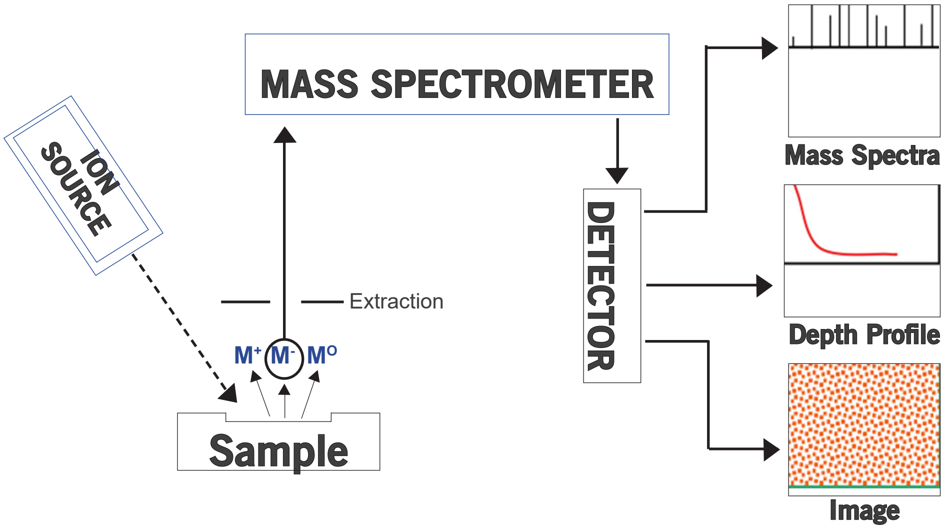


Figure 2: Schematic of a SIMS Instrument

The ejected or sputtered atoms from the sample that are ionized are called secondary ions. The ions can be single atoms or clusters of atoms. The ions are then extracted and passed through a spectrometer that separates the ions by mass and are collected into a detector to generate a mass spectrum, a depth profile or a chemical map. The schematic shown in Figure 2 illustrates this process. For layered structures like Si/SiGe we use depth profile mode, otherwise known as Dynamic-SIMS. Using our propriety SIMS intensity calibration/ quantification method, i.e., PCOR-SIMS$^{SM}$ [1], we can measure %Ge, layer thickness and evaluate interface sharpness with <5% uncertainty. To achieve such accuracy in nm layers, the broadening artifact inherent to dynamic-SIMS, known as atomic mixing, must be minimized. See Figure 1 for illustration of this phenomena, which is strongly dependent on the incident primary ion beam kinetic energy and the angle of incidence. Therefore, to achieve the necessary depth resolution these two parameters must be optimized. Figure 3 shows several SIMS depth profiles of Ge measured from the same SiGe/Si multilayer sample. In this example, the same layer was measured using four different primary ion beam conditions resulting in different depth resolutions. The SIMS condition 1 had the highest primary beam energy and the condition 4 had the lowest. As the atomic mixing effect is reduced by lowering the primary beam

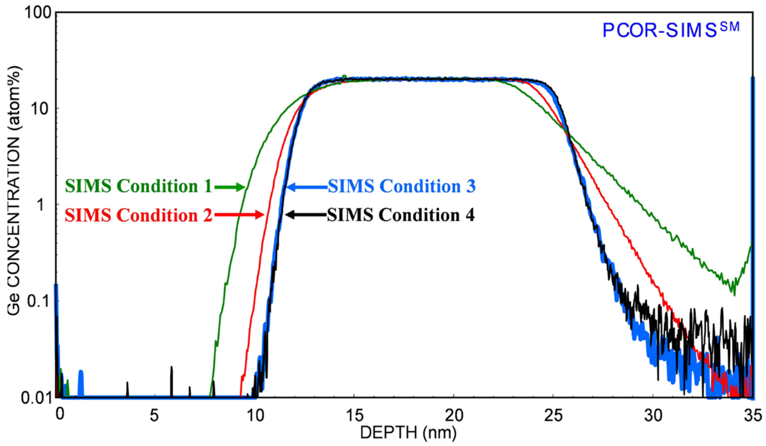


Figure 3: The results of four different primary ion beam conditions while profiling a multi-layered Si/SiGe sample. Reducing the impact energy minimizes the atomic mixing artifact which leads to progressively more accurate characterization of the SiGe layer.

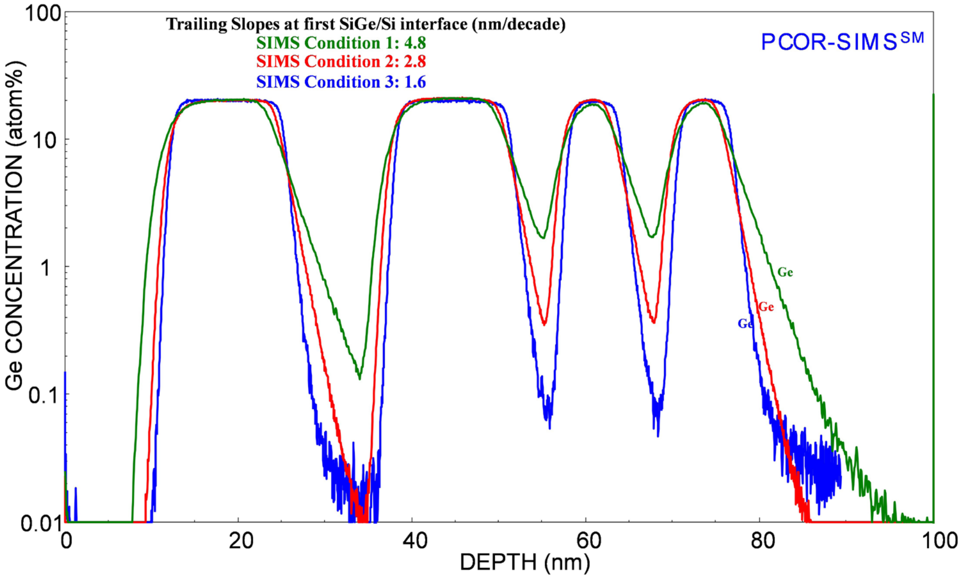


Figure 4: Three different SIMS atomic mixing modes leading to three different depth resolutions measured by interface decay length (nm/decade).

energy, the SIMS depth resolution is increased, and we measure the distribution that is closer to the real profile. The optimum measurement conditions are realized when further reduction of primary beam energy results in the same SIMS distribution as the black and blue curves show in Figure 3.

In SIMS data the interface abruptness is typically measured as decay length (nm/decade) of the SiGe/Si trailing or leading edges, as shown in Figure 4. In Figure 3 the decay length decreases with improvement in SIMS depth resolution with SIMS condition 1, 2 and 3, but when we use ultra-high depth resolution conditions such as condition 4, we no longer measure a lower decay length. This is because condition 3 has sufficiently good depth resolution and improving the depth resolution further does not change the sharpness.

The analytical conditions chosen for a SIMS analysis depend on many factors such as depth, elements required etc. Total analytical time is also a key parameter that is considered, as higher depth resolution analysis requires much longer analytical times compared to lower depth resolution analysis. Once the optimum SIMS conditions are chosen, the application of PCOR-SIMS$^{SM}$ protocol with NIST traceable standards allows %Ge measurement with <2% relative uncertainty and with layer thickness determination with <5% relative uncertainty. Having several orders of magnitude of composition detection sensitivity, SIMS is also useful for Ge or Si inter-diffusion studies during growth.

Although PCOR-SIMS$^{SM}$ provides quantitative chemical composition information and interface abruptness measurements when we minimize the atomic mixing artifact, we still may not have a complete understanding of the SiGe/Si layer's structural morphology.

## Scanning Transmission Electron Microscopy (STEM)

Scanning transmission electron microscopy (STEM) is the standard analytical technique to probe epitaxial interface behavior at the highest resolutions; aberration-corrected STEMs (AC-STEM) available at EAG can achieve 70 pm spacial resolutions [2]. A highly focused beam of high energy electrons (200 keV) is scanned over a thinned section from a sample, typically under 100 nm thick, and various detectors are placed around

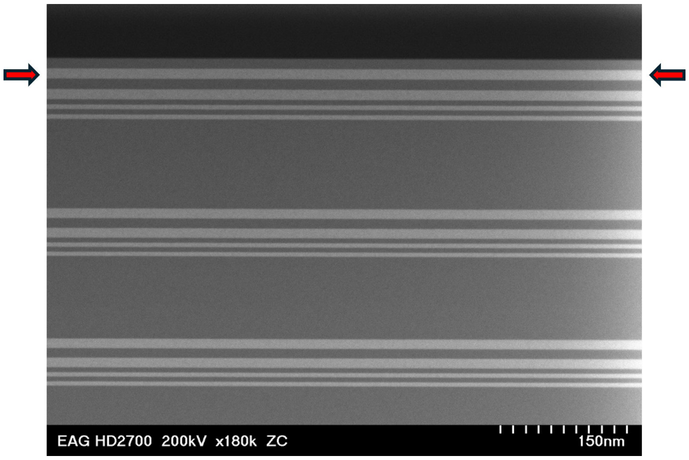
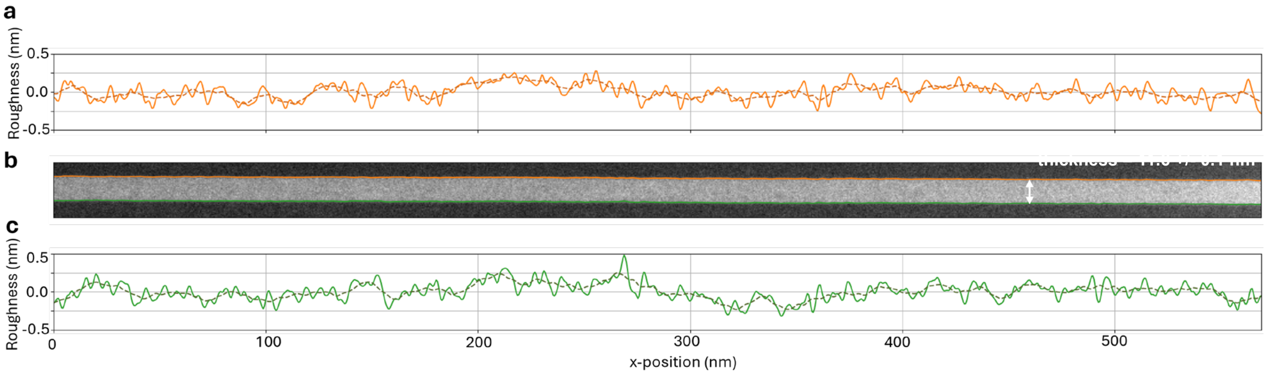


Figure 5: STEM HAADF of SiGe quantum wells in Si viewed along <110> zone axis. The red arrows indicate the SiGe layer analyzed in Figure 6.

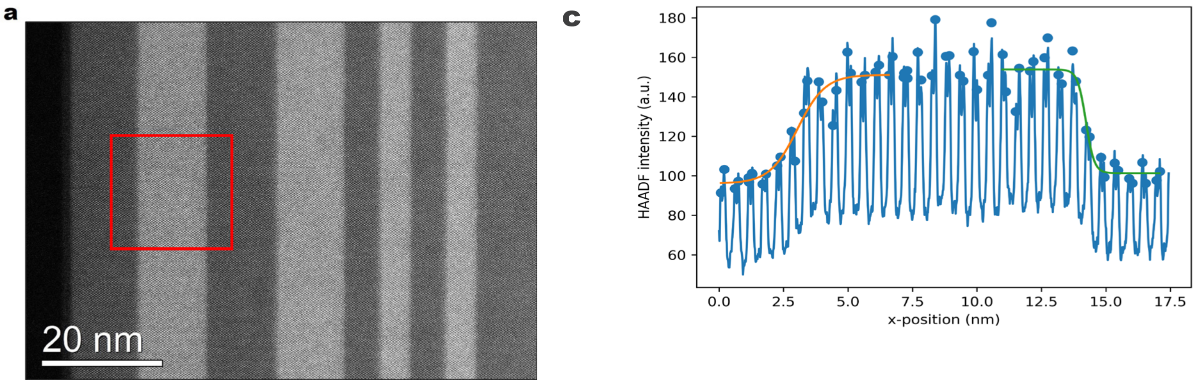
the sample to collect unique types of information. Figure 5 above shows a high angle annular dark field (HAADF) image of a Focused Ion Beam (FIB) prepared lamella (~60 nm thick) taken from the same bulk SiGe/Si sample as reported in the previous SIMS data. Here the sample is oriented with the surface at the top of the image. HAADF images are formed by collecting the transmitted electrons that have been elastically scattered to high angles, where the intensity of the signal at each pixel varies according to the average atomic number of each atomic column. This intensity is related approximately to $Z^{1.8}$. Since the thin lamella has a uniform thickness, the layers with the atomic columns that appear brighter have increased Ge content (~20 at% from SIMS data) relative to the others that are darker (pure Si). Although intensity patterns in HAADF images in Figures 5, 6, 7 show

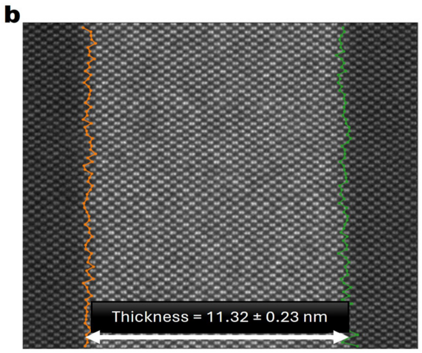


**d**

| | $R_a$ (nm) | $R_{rms}$ (nm) | Width (nm) |
|---|---|---|---|
| Top | 0.1 | 0.1 | 1.1 |
| Bottom | 0.1 | 0.1 | 1.1 |

Figure 6: Low magnification interface analysis | a) Measured roughness of the top interface. Dashed line indicates a rolling average over a window with 11 nm width. b) HAADF image of an SiGe layer with the position of the top and bottom interfaces overlaid in orange and green, respectively. The interface position is determined by gaussian fitting after applying a Sobel filter to the image. Thickness is measured as the average difference in position between the interfaces; the uncertainty is the standard deviation of all thickness measurements. c) Measured roughness of the bottom interface. Dashed line indicates a rolling average with the same window as a to reflect change in figure d) A table of the average roughness ($R_a$), root-mean-square roughness ($R_{rms}$) and width for each interface, defined based on the gaussian fitting.





**d**

| | $R_a$ (nm) | $R_{rms}$ (nm) | Width (nm) |
|---|---|---|---|
| Top | 0.11 | 0.13 | 0.96 |
| Bottom | 0.13 | 0.17 | 0.81 |

Figure 7: Atomic resolution interface analysis | a An atomic resolution HAADF image of an SiGe [110] multilayer sample rotated 90 deg CCW with the surface on the left and the substrate on the right. b Zoomed in region of the first SiGe layer outlined by the red box in a. The orange and green lines mark the position of the left and right interfaces respectively, based on sigmoid fitting of atomic column peak intensities. The thickness of the layer is measured by averaging the difference in position across the entire layer, uncertainty is given as standard deviation across all thickness measurements. c A line profile of the intensity across an individual atomic row. The blue line is a line profile of the HAADF intensity while the blue points indicate the peak column intensities used for fitting. Orange and green lines show fitting results for each interface. d A table of the average roughness ($R_a$), root-mean-square roughness ($R_{rms}$) and width for each interface, defined based on the sigmoid fitting.

the same composition pattern as seen in the SIMS profiles, the composition information is merely qualitative. STEM spectroscopy techniques such as energy dispersive x-ray spectroscopy (EDX) or electron energy-loss spectroscopy can be performed, however, the accuracy for standardless based spectroscopy is still limited as compared to PCOR-SIMS$^{SM}$. The key strength of STEM compared to SIMS lies in combining high spatial resolution imaging with quantitative image processing to measure critical dimensions and structural properties. To measure the layer thickness and assess the abruptness of the interfaces, EAG can provide a customized strategy encompassing sample preparation, image acquisition, and a robust set of image analysis tools suitable for your specific analysis requirements. One such image analysis protocol available is outlined in the app note: “Statistical Analysis of Multilayer Structures in Electron Microscopy Using Image Processing” [3]. Figure 6 shows an example of how this protocol can be applied to evaluate the upper most SiGe layer in Figure 5 over an ~500 nm wide region. This strategy can be very helpful for observing longer range variance and roughness with nm to sub-nm precision. Tabulated measurements can be obtained from one or all layers quite easily. If even finer interfacial details are required, the analysis can be approached by a different computational strategy called Atom-by-Atom Distance Mapping [4] [5] as shown in Figure 7. For this approach, high-quality atomic resolution HAADF images are required. From these images each atomic column is located very precisely using 2D gaussian fitting, and from this positional data, atoms in each lattice plane normal to the layer of interest are extracted and the interfaces fitted with a sigmoid function, as illustrated in Figure 7(c). This enhanced plane-by-plane procedure gives rise to additional statistics providing layer thickness, interface width, and roughness with 100-10 pm precisions. Based on our fitting process, the width of the trailing (right) interface of the first layer was measured to be 0.81 nm. For comparison, applying the same sigmoid fitting process to the SIMS profile in Figure 3 yields a width of 1.75 nm, comparable to the value measured based on the decay length, but larger than the value measured from STEM.

## Conclusion

This application of both PCOR-SIMS$^{SM}$ and STEM imaging to the same Si-SiGe multilayer sample demonstrates the individual strengths of both techniques and how they can be combined to form a more complete understanding of the sample. PCOR-SIMS$^{SM}$ provides quantitative chemical information that is generally unavailable in STEM imaging because the sputter yields and rates measured in SIMS are directly tied back into quantitative compositions using calibrated NIST traceable reference data. In addition, SIMS can detect concentrations < 1 atom%, making it essential when investigating diffusion or matrix intermixing between layers. However, atomic mixing and directionality artifacts in SIMS make it very difficult to evaluate whether broadening appearing at an interface is due to roughness, inter-diffusion, or analysis settings. Although these artifacts can be minimized by optimizing the SIMS acquisition, complementary high-resolution STEM imaging combined with image processing can help to provide accurate quantitative structural interface measurements. With an atomic resolution image, it is possible to measure atomic positions with 100-10 pm accuracy which further enables measurement of the plane-by-plane interface roughness and calculation of statistical quantities that describe the interface over the full field-of-view. Therefore, both PCOR-SIMS$^{SM}$ and AC-STEM analyses are required for complete characterization of these interfaces.

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