## INVESTIGATION OF SI/SIGE FINFETS AND VERTICALLY STACKED NANOWIRES VIA ATOM PROBE TOMOGRAPHY

By

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## A DISSERTATION PRESENTED TO THE GRADUATE SCHOOL OF THE UNIVERSITY OF FLORIDA IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE DEGREE OF DOCTOR OF PHILOSOPHY

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To my wife, our families, and the shoulders of giants I stood upon

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# LIST OF ABBREVIATIONS

α-Si	Amorphous silicon
ALD	Atomic layer deposition
APT	Atom probe tomography
BF	Bright field
BSE	Backscatter electron
CVD	Chemical vapor deposition
DF	Dark field
DIBL	Drain induced barrier lowering
DP	Diffraction pattern
FET	Field effect transistor
FIB	Focused ion beam
FinFET	Fin field effect transistor
GeOl	Germanium on insulator
GRL	Germanium rich layer
HAADF	High angular annular dark field
HRTEM	High resolution transmission electron microscopy
IC	Integrated circuit
LE	Local electrode
MOSFET	Metal-oxide-semiconductor field effect transistor
PECVD	Plasma enhanced chemical vapor deposition
RTA	Rapid thermal anneal
ROI	Region of interest

S/D	Source and drain
SEM	Scanning electron microscope
SiGe	Silicon and germanium alloy
STEM	Scanning transmission electron microscopy
TED	Transient enhanced diffusion
ТЕМ	Transmission electron microscopy
TRD	Transient retarded diffusion

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# INVESTIGATION OF SI/SIGE FINFETS AND VERTICALLY STACKED NANOWIRES VIA ATOM PROBE TOMOGRAPHY

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The advancement in semiconductor technology has been shown to be dependent on shrinking transistors in accordance with Moore's Law. However, reductions in device size have resulted in increases of the contact resistance between metals and the source and drain regions. Reductions in these contact resistances have been achieved by strategically doping the S/D regions, especially in 3D devices. While many techniques exist to characterize these doping profiles, they fall short when attempting to describe 3D systems. Atom probe tomography has been demonstrated to be a promising method to quantify doping profiles in such systems, but still contains artifacts in reconstructions of heterostructures with varying evaporation fields. This work examines methods to remove such distortions and explore their viability in producing accurate data sets describing not only the overall device structure, but profiles of ion implanted dopants. A better understanding of doping profile extraction and profile accuracy post distortion correction is also investigated. Finally, a brand-new Ge diffusion process was discovered during the course of this work and will be presented along with applications in the fabrication of strained vertically stacked Si nanowires. Such a method offers brand new avenues of fabricating complex heterostructures

previously unobtainable using conventional photolithographic, deposition, and etch processes.

#### CHAPTER 1 INTRODUCTION

#### **1.1 Motivation**

Over the past several decades, advancements in the semiconductor industry have pushed the world into a new age of computing and information. Computers that used to be the size of rooms could now comfortably sit on a desk and provide even better performance. This miniaturization not only allowed for faster computing but made devices more accessible for the masses reducing the barrier for innovation. Now, every home and business can have a device capable of performing tasks orders of magnitude faster than could be previously done using more traditional methods. While devices continued to shrink, the sharing of information was limited to large floppy discs with low storage or large cumbersome spools of magnetic tape. With the advent of solid state memory, information that was typically stored in volumes of thick books could now be transferred onto a chip the size of a stamp. Such technology facilitated the creation of large server banks and high-speed telecommunications allowing for near instant communication between electronic devices and the sharing of data. Continued shrinking of devices vaulted the world into an age where computers became commonplace, even to the point where they fit in our pockets and were integrated into vehicles, airplanes, and even now into our clothing. Consumer demand is now higher than it ever has been, necessitating continued shrinking of devices and improved performance.

This shrinking of transistors has relied on scaling of planar devices since the 1960s for performance and device density improvements. However, further scaling of these devices has been hindered by short channel effects, necessitating new device architectures. FinFETs (fin field effect transistors) have been integrated into newer

devices, but performance issues remain as they are scaled down to the 7 nm node as defined by the International Technology Roadmap for Semiconductors (ITRS).<sup>1</sup> Nanowire transistors have proven to be of great interest for this continued scaling, but traditional techniques to characterize them have become less reliable and don't tell a wholistic picture of the structure. Atom probe tomography (APT) has been demonstrated as promising technique for characterizing these 3D devices with sub-nm resolution and excellent chemical identification.<sup>2</sup> Unfortunately, while this technique promises excellent results for homogeneous materials, several shortcomings still remain when analyzing 3D heterostructures including spatial distortions and poor counting statistics. Thus, improved APT methods are critical to continued scaling of these 3D devices.

#### **1.2 The Solid-State Transistor**

The shift away from vacuum tubes and physical mechanisms as a means for creating computational devices was facilitated by the invention of the solid-state transistor. This transistor was developed by Bardeen, Shockley, and Brattain at Bell Laboratories in 1947 and was dubbed the point contact transistor fabricated on a doped germanium substrate.<sup>3</sup> It was this invention that spurred research into the development of the bipolar junction transistor (BJT), junction gate field-effect transistor (JFET), and eventually, the metal-oxide-semiconductor field-effect transistor (MOSFET).<sup>4</sup> Thanks to their high switching speeds and low cost of production, MOSFET devices have become the device of choice for the fabrication of logic devices such as integrated circuits (ICs) and microprocessors. Combinations of n-type and p-type MOSFETs are now utilized in complementary metal-oxide-semiconductor (CMOS) logic to reduce power consumption when the device is in a static state. As MOSFET devices are the cornerstone of modern

IC logic and this work, they will be the main focus of this section describing transistor operation.

**Transistor operation**: In most logic applications, a transistor can be thought of simply as a switch, allowing current to flow through it when needed like a faucet in your kitchen. The switch, or in the case of the sink, a valve, contains a source and a barrier holding back the flow of current to the drain. Adjusting the degree to which the source is blocked by the barrier allows current to then flow from the source to the drain through some kind of channel. The MOSFET device consists of combining n-type and p-type doped semiconductors into p-n-p or n-p-n junctions where the two outside ends of the doped regions act as the source and drain (S/D), and the center acts as the channel. A gate electrode is placed over the channel region to control the flow of carriers through the channel. Figure 1-1 shows a schematic of an n-type MOSFET (NMOS) device illustrating the direction of current flow and relevant components. As this work focuses on n-type doing, n-type devices will be discussed from the remainder of this section.



Figure 1-1: Illustration and schematic of an n-type MOSFET device showing source, gate, and drain regions.

When the highly n doped (n+) source and drain regions are contacted with the p doped channel region, depletion regions form due to carrier recombination. This recombination in the depletion region causes a shift in the Fermi level back to the intrinsic level. However, the ionized dopants remain in their respective lattices inducing a voltage around the p-n junctions. This internal voltage (or built in potential) leads to band bending and results in the band structure depicted in figure 1-2 for the n-p-n junctions. It becomes apparent that at equilibrium conditions, a large barrier is present preventing the flow of electrons from the source to the drain. Such a prevention to current flow demonstrates the "off" state of the device and highlights a reason for it's use over other transistors. Some leakage current may be present as minority carriers are still located in the channel, but few make it across without recombining.

To induce a flow of electrons, the barrier height from source to drain must be reduced. Electrostatic interactions between the S/D can cause downward shifts in the conduction band allowing for easier transfer of electrons (figure 1-2). While not the most creative, this process was accurately named drain induced barrier lowering (DIBL) and can occur in devices with short channels. This lowering does help in reducing the barrier height but can lead to leakage currents if lowered too far. Control of this lowering process can be achieved by biasing the drain. Applying a positive voltage to the gate (V<sub>G</sub>) causes the metal-oxide-channel stack to act as a capacitor. As such, increasing electric fields across the dielectric lead to a buildup of charges in both the gate and channel regions. In the n-type device discussed so far, negative carriers are drawn from the substrate into the channel region. These electrons recombine with holes in the p-type channel creating a depletion region. Continued accumulation of electrons

eventually inverts the channel from p-type to n-type and lower the barrier height sufficiently for current to flow (figure1-2).



Figure 1-2: Band diagram for the source, channel, and drain in an n-type MOSFET device (left). DIBL and an applied gate voltage reduce the barrier height needed for electrons to travel from source to drain (right).

The voltage required to invert the channel bridging the source and drain is called the threshold voltage (V<sub>T</sub>). Any voltage lower than V<sub>T</sub>, including negative voltages, cause the device to be in an off state and lead to the desired rectifying behavior of the NMOS. In a p-type MOSFET (PMOS), the opposite it the case where the device is turned on with a negative voltage. As such, NMOS and PMOS devices can be combined to form a complementary MOS (CMOS) logic device like an inverter. Here, a high voltage input connects the device to ground by turning on the NMOS transistor on and turning off PMOS transistor. Conversely, low voltage inputs turn the PMOS transistors on and the NMOS transistors off connecting the output to power.

## 1.3 Transistor Scaling and Moore's Law

With the increasing demand for more computational power and improved microprocessor performance, scaling of large (100 µm) CMOS transistors was required. Miniaturization of devices such as MOSFETs leads to a linear increase in switching

speeds increasing the functional throughput of completed ICs. Power consumption is also reduced with a reduction in the overall device size requiring less energy per switching operation. Early shrinking of planar transistors was achieved using a simple scaling factor  $\alpha$  to produce a smaller MOSFET with very similar behavior.<sup>5–7</sup> To maintain a similar electric field in the scaled device, the voltage and dimensions are scaled down by  $\alpha$  while doping and charge densities are increased by the same scaling factor. This "constant field scaling" decreased the threshold voltage and drive current by  $\alpha$ , increased the circuit speed by  $\alpha$ , and increased the overall circuit density by  $\alpha^2$ . This type of scaling, in which the power density remains constant, became known as Dennard scaling. Scaling devices using a constant voltage was also explored which resulted in an increase in circuit speed by  $\alpha^2$  and a power density increase of  $\alpha^3$ . Unfortunately, this type of scaling results in increases of fields in the gate oxide by the factor  $\alpha$  calling into question the reliability of the gate oxide and velocity saturation. Table 1-1 illustrates the effects of ideal scaling on device parameters.

Parameter	Constant Field Scaling	Constant Voltage Scaling
Dimensions	1/α	1/α
Vdd	1/α	1
Fields	1	α
VT	1/ α	1
Current	1/ α	α
Power/Area	1	1/ α <sup>3</sup>

Table 1-1: Relative scaling effect for various device parameters assuming a scaling factor of  $\alpha$ .<sup>5</sup>

Gordon Moore took note of this transistor scaling in 1965 and observed that the number of transistors on a chip would double every two years.<sup>8</sup> This trend became known as "Moore's Law" and grew to be the scaling and performance target for the entire industry. Over the years, several groups began to predict the end of Moore's law as challenges in short channel effects,<sup>9</sup> limitations in lithography,<sup>10</sup> and breakdown of the gate dielectric<sup>11</sup> became apparent. However, changes in device design and careful material selection have allowed this level of scaling to continue.

Continued shrinking of transistors using the scaling factor method saw breakdowns in performance approaching the 90 nm node stemming from mobility degradation due to large vertical electric fields. To combat this, new materials were introduced into the device architecture. While at Intel, Thompson et al. developed a new logic technology in which the channel region of a MOSFET was placed in a strained state, thus increasing the carrier mobility.<sup>12</sup> For pMOS devices, SiGe was epitaxially deposited into the S/D regions introducing uniaxial compressive stress increasing the mobility of holes. Electron mobility in nMOS devices was increased by introducing biaxial and uniaxial tensile stress via a high stress SiN capping layer over the gate and S/D regions.

The introduction of strain helped scale CMOS devices down to the 65 nm node. However, continued scaling resulted in a reduction of the gate oxide thickness (SiO<sub>2</sub>) leading to a drastic increase in current leakage from the gate due to tunneling and a breakdown in Dennard scaling. This reduction of the gate oxide thickness is described in Equation 1-x which relates the capacitance between the gate and channel (C) with

the relative dielectric constant ( $\kappa$ ), permittivity of free space ( $\epsilon_0$ ), capacitor area (A), and dielectric thickness (t).

$$C = \frac{\kappa \epsilon_0 A}{t} \tag{1-1}$$

To maintain a constant capacitance when scaling, the thickness of the gate oxide must decrease. Mitigating the issue of a thin dielectric was achieved by implementing a gate oxide with a high dielectric constant (high- $\kappa$ ). It was found that HfO<sub>2</sub> provided such a  $\kappa$  value allowing for a physically thick layer which was electrically thin.<sup>13,14</sup> It was also found that the introduction of a metal gate (like TiN) can screen phonon scattering within the high- $\kappa$  dielectric preventing coupling to the channel which improves electron mobility.

#### **1.4 3D Transistor Architectures**

As mentioned previously, short channel effects stemming from a reduction in the gate length limit the ability to scale planar MOSFET devices. A way to control these short channel effects is to operate the device in a fully depleted mode using two or more gate electrodes. In this configuration, electric field lines from the source and drain terminate on the gates instead of affecting the channel region.<sup>15</sup> The most common multi-gate architecture consists of pulling the source, drain, and channel region out of the substrate surface creating a "fin" structure. While several multi-gate structures have been investigated to replace planar (or single gate) transistors including double gate,<sup>16,17</sup>  $\Pi$ -gate,<sup>18,19</sup> and  $\Omega$ -gate,<sup>20,21</sup> the trigate architecture has shown to have the most relaxed requirements and allow for improved manufacturability.<sup>22</sup> As such, these will be the main focus of this section concerning fins and all future references to finFETs will refer to the trigate architecture. Figure 1-3 shows schematics of these structures.



Figure 1-3: Schematics showing cross sections of various 3D MOS transistors explored to replace planar devices. Tan colors denote the Si substrate and channel region, blue represents the gate dielectric, and green represents the gate material.

The finFET transistor consist of a fin structure with the gate contacting the top and two sides of the fin. In this configuration fin widths and pitches (distance between fin centers) can be scaled more effectively than planar devices and maintain fully depleted operation. Work done by Doyle et al. demonstrated finFETs with ideal SS and DIBL behavior as well as drive currents greater than any previously reported non-planar devices.<sup>23</sup> In addition, the finFETs were found to be competitive with similarly sized planar CMOS transistors. These types of devices proved to be a great candidate for scaling past the 45 nm node<sup>24</sup> and were introduced by Intel at the 22 nm node. Scaling of these devices has continued down to the 7 nm node, however, limitations in fabrication and performance have necessitated the transition to a new architecture.

Gate all around (GAA) transistors have been shown to provide the best electrostatic control over the channel region by the gate compared to other multi-gate structures<sup>25,26</sup>. In particular, nanowire (NW) transistors have been studied heavily due to their ease in fabrication via selective etch<sup>27</sup> and vapor-liquid-solid growth.<sup>28</sup> For scaling to the 5 nm node, vertically stacked horizontal NWs have been largely considered promising easier integration with current technologies and increased drive current over single GAA transistors.<sup>29</sup> This technology has been implemented by IBM, but has not been released as a final product or benchmarked against 7 nm node devices.<sup>30</sup> VLS grown NWs have proven to be easier to fabricate than horizontal variety requiring fewer lithographic steps. While more easily fabricated, integration into a horizontal device requires placing the NWs into a solution and depositing them onto a substrate limiting the viability of integration at an industrial level. However, fabrication of vertical NW transistors using this growth technique remains as an interest for future device scaling.

#### **1.5 Contact Resistance**

While finFETs and GAA transistors run in a fully depleted mode, requiring no doping of the channel region, proper doping of the S/D regions is critical to overall performance. Doping in these regions is not only important for operation, but also to reduce the contact resistance between the S/D and their respective metal contacts. Contact resistance can be defined as the resistance of majority carrier transport between two materials with a potential barrier at their interface. As this contact resistance is the maximum contributor to the overall series resistance, reducing this resistance has become a crucial point of study, especially due to the increase in resistance with decreasing contact width.<sup>31</sup>

When a contact is made between a metal with a work function  $q\Phi_m$  and a semiconductor with work function  $q\Phi_s$ , the Fermi levels align at equilibrium due to charge transfer. When  $\Phi_m > \Phi_s$ , as is the case for n-type semiconductors, a depletion region is formed near the contact region raising the electrostatic potential of the semiconductor and creates an equilibrium contact potential V<sub>0</sub>. This contact is referred to as a Schottky barrier diode as current can only freely flow in one direction under bias.

For the n-type semiconductor, current flows when the device is operated under forward bias. Under these conditions, the contact potential is reduced to  $V_0 - V$  allowing electrons from the conduction band to diffuse across the depletion region. In contrast, under reverse bias, the potential is increased to  $V_0 + V$  preventing any diffusion of electrons from the semiconductor to the metal giving rise to diode characteristics. Figure 1-4 illustrates the metal-semiconductor contact at equilibrium and under forward bias.



Figure 1-4: Band diagram showing a Schottky barrier diode between a metal and semiconductor (left). Under forward bias (right), the barrier for electrons to travel from the semiconductor to the metal decreases. The red arrow 1 shows thermionic emission while 2 shows quantum-mechanical tunneling.

Under the forward bias condition, electrons with energy kT greater than the barrier height can diffuse into the metal via a thermionic emission process (red arrow 1 in figure 1-4). However, increasing doping levels in the n-type semiconductor to degenerate levels allows for quantum-mechanical tunneling of electrons through the depletion region (red arrow 2 in figure 1-4). When this transport process is achieved, the behavior of the contact can shift from Schottky to Ohmic (linear I-V characteristics).<sup>32</sup> The contact resistance between a metal and degenerately doped n-type semiconductor can be calculated using the active donor concentration (N<sub>D</sub>) via equation 1-2.

$$R_{c} = e^{\left[\left(\frac{2\sqrt{\varepsilon_{s}m^{*}}}{\hbar}\right)\left(\frac{\Phi_{Bn}}{\sqrt{N_{D}}}\right)\right]}$$
(1-2)

In this regard, high doping levels with a S/D can lead low contact resistances and improved device performance. Characterization of theses doping profiles is then critical to device development ensuring proper levels are achieved. However, the transition to 3D devices poses some problems as conventional characterization methods begin to fall short. While contact resistances are a key component of device performance, the characterization of doping levels within the device are the focus of this work.

#### **1.6 The Power of Atom Probe Tomography**

With the transition from planar transistor technologies to 3D architectures, characterization of doping profiles using traditional methods has become more difficult. Whereas analytical techniques such as energy dispersive x-ray spectroscopy are not sensitive enough to detect dopants (< 1 at%), several methods exist to measure doping concentrations with high sensitivity and spatial resolution.

Secondary ion mass spectrometry (SIMS) has traditionally been used to profile the depth and concentration of multilayer structures. This is achieved by bombarding a sample with a high energy ion beam which sputters away surface atoms. These secondary atoms can then be collected and identified by their mass to charge ration using a mass spectrometer. Traditionally in the semiconductor industry, this method is used to probe doping concentration, however, with the transition to 3D devices, SIMS is unable to provide the data necessary for a full understanding of the dopant distribution. Several groups have demonstrated the effectiveness of a technique coined 1.5D SIMS in which doping concentrations in a fin structure could be quantified<sup>33,34</sup>. While

concentrations for the top surface, fin sidewall, and fin bottom were obtained, no information concerning the lateral distribution of dopants through the fin were obtained.

To obtain electrical information in two dimensions, methods such as scanning spreading resistance microscopy (SSRM) can be employed. This method uses an atomic force microscopy tip in contact mode to scan across the surface of a sample<sup>35</sup>. The total resistance in the sample is then measured after applying a DC bias from which a spreading resistance term can be extracted. A carrier concentration can then be calculated for each scanned point producing a 2D concentration map with a spatial resolution of <1 nm<sup>2</sup>. SSRM proves to be better suited for dopant distribution analysis in 3D transistor devices than SIMS, however, only information on the relative concentration of active carriers is provided. Again, this method demonstrates some shortcomings in trying to provide an accurate picture in the distribution of dopants in 3D structures.

Atom probe tomography (APT) is a powerful characterization technique capable of providing elemental characterization with sub-nm resolution in 3D. This technique utilizes the phenomenon of field evaporation of surface atoms in conjunction with a time of flight (TOF) mass spectrometer and position sensitive detector to produce data sets with very high spatial resolution on the order of 0.3nm in the x, y, and z direcrections<sup>36</sup>. With the ability to detect single ions with high spatial resolution and chemical composition, APT offers itself as a premier technique for characterizing semiconductor devices.

An overview of the operation and specific parameters used in APT runs will be outlined in Chapter 2.2 including details concerning data reconstructions.

#### **1.6.1 History of Atom Probe Tomography**

APT has a rich history in the attempts to better characterize materials from both a structural and chemical viewpoint. The field emission microscope (FEM) was the direct ancestor to the current local electrode atom probe (LEAP) tool used in this work. It was born from the work by several groups in the early 20<sup>th</sup> century to study the theory of electron emission from solids. At the time, field emission of electrons required field strengths which exceeded those attainable from readily available equipment<sup>37</sup>.

Professor Erwin Müller employed the enhancement of electric fields at the apex of sharp points to reduce the necessary voltage required for field emission of electrons from a surface leading to the creation of the first FEM<sup>38</sup>. This was achieved by placing a needle shaped specimen with a tip diameter of ~10<sup>-6</sup> m under a negative potential sufficient to produce local fields on the order of 1 V nm<sup>-1</sup>. These fields were strong enough to field emit electrons via quantum mechanical tunneling onto a phosphor screen a few centimeters away. A projection of the tip apex was produced on the screen with a magnification on the order of 10<sup>4</sup> and a spatial resolution of 2 nm.

Müller continued to expand on his work with the FEM and in 1951 found that reversing the bias on the emitting tip resulted in the desorption of positive ions from the sample surface. This led to the development of the field ion microscope (FIM) which became the first imaging technique to image single atoms in a solid atomic lattice<sup>39</sup>. To mitigate the issue of a limited number of particles on the surface of the tip to ionize for imaging, hydrogen was introduced into the chamber to adsorb onto the sample. Surface atoms thus became emitters of positive ions which were then incident upon a detector mapping a projection of the surface atoms. It was also found that reducing the sample

to cryogenic temperatures resulted in an improvement in spatial resolution, or the ability to distinguish between different atomic positions<sup>40</sup>.

Both the FEM and FIM allowed for very detailed microstructural studies of the tip material. One limitation of both characterization techniques is their inability to provide chemical information. In 1956, Müller discovered that individual atoms could be sublimated from the sample surface by increasing the applied voltage, and in turn, the field strength. This sublimation was referred to as field evaporation and could be controlled to allow for single atom evaporation from the sample<sup>41</sup>. With the addition of a time-of-flight (TOF) mass spectrometer to the FIM, Müller and Panitz developed the first atom probe microscope (AMP) in 1968<sup>42</sup>. After imaging the sample using FIM, ions could be field evaporated from the surface and identified providing both chemical and structural information.

In order to field evaporate sample atoms in the APM, the voltage required to produce a strong enough field was pulsed. By pulsing the voltage, the instrument was able to evaporate atoms one at a time. Whereas voltage pulsing could be applied to metal samples due to their high conductivity, it was not effective on semiconducting materials. This was due in part to the high resistivity of the semiconductors which attenuated the amplitude of the voltage pulse preventing field evaporation<sup>37</sup>. To combat this, Kellogg and Tsong developed the pulsed laser atom probe (PLAP) in 1980 to widen the list of characterizable materials<sup>43</sup>. In PLAP, a laser is pulsed onto the apex of the sample adding enough thermal energy to field evaporate atoms at a standing voltage. Laser pulsing also reduces the energy spread of ions thus improving the mass resolution of the instrument.

In the late 1980s and early 1990s, several groups utilized time of flight measurements and improved two-dimensional position sensitive detectors to develop three-dimensional imaging atom probes<sup>44–46</sup>. Surface atoms from samples with hemispherical tip geometries could be evaporated layer by layer. Position sensitive detectors developed using multiple microchannel plates would then allow for x-y-z coordinates to be calculate with a resolution approaching 0.3 nm. In conjunction with mass-to-charge ratio data collected by the TOF mass spec, 3D reconstructions can then be created using specialized software. This method was deemed 3D tomographic atom probe and became unmatched in characterizing materials properties in systems such as grain boundaries and thin films<sup>47</sup>.

Work done by Nishikawa and Kimoto in 1994 proposed the idea of using a local electrode to reduce voltage applied to the sample by increasing the potential field<sup>48</sup>. This would be achieved by reducing the distance between the tip and electrode which would have a conical shape. Kelly et al. used a similar design with an aperture at the apex of the conical electrode and developed the first local electrode atom probe (LEAP)<sup>49</sup>. In this design, samples would be mounted on microtip coupons and brought into proximity of the electrode. An applied bias and laser pulses would then evaporate atoms which would then be incident onto a detector. The principle and instrumentation for the LEAP system used in this work is outlined in Section 2.2.1.

#### **1.6.2 Dopant Mapping and Planar Transistors**

As described previously, characterizing doping profiles in semiconductor devices is crucial to understanding how a specific dopant incorporation process affects electrical characteristics. While SIMS does provide 1D concentration profiles with depth, it does

not allow for probing of doping concentrations in clusters or in multiple directions as would be required when fully characterizing a fabricated device.

Early demonstrations of dopant mapping using APT mainly focused on ion implanted samples as implant is a major dopant incorporation method for transistors.<sup>50</sup> Studies on defects arising from high dose implants were of special interest as chemical analysis proved difficult while maintaining the defect structure. Thompson et al. investigated the formation and evolution of arsenic Cottrell atmospheres in silicon post ion implant.<sup>51</sup> They were able to not only identify the location of the defects in the substrate, but also their elemental composition. Figure 1-5 shows correlative APT and TEM images for samples annealed at high temperatures in which dislocation loops were clearly visible. This result marked one of the first instances in which such defects were visualized in 3D with an accurate map of dopant atom positions. Studies were also carried out to improve of cluster analysis methods in APT data sets for doped semiconductors. Phillippe et al. developed a first nearest neighbor (1NN) distribution model for two phase systems in order to quantify the concentrations of clustered and dispersed atoms (B) in their solvent atoms (A).<sup>52</sup> This method was successfully applied to APT data sets of in situ doped Si layers with 1.5% As to quantify the fraction of dopants in a clustered phase. Comparable results were obtained with electrical measurements providing validation of the 1NN model.



Figure 1-5: TEM image of an As-implanted Si sample after a 600C anneal for 30 minutes followed by a 1000C anneal for 30 sec (a). a-b) APT reconstruction of annealed sample showing As atoms in dislocation loops where purple dots represent As and blue dots represent oxygen.<sup>51</sup>

APT proved to be invaluable when characterizing doping distributions in planar MOSFET devices. Work done by Moore et al. demonstrated the first 3D analysis of lateral dopant diffusion in the source and drain of a patterned structure. Such results were not previously attainable using conventional methods thus highlighting the viability of APT for this type of characterization. Additionally, this work was the first to demonstrate correlative techniques on a single sample comparing both TEM cross sections and simulation data showing good agreement between all methods. Future studies continued to leverage the high spatial resolution and 3D nature of APT data sets to investigate doping profiles including segregation of dopants at grain boundaries in the poly-Si gate<sup>53</sup> and variations in the electrical performance of n-type and p-type MOSFET devices.<sup>54</sup>

## **1.6.3 Characterization of 3D Transistors**

The real power of APT was shown off when groups began to characterize doping concentrations and interfaces in 3D transistor architectures ranging from FinFETs to nanowire FETs. In these systems, dopant distributions in multiple directions becomes critical to the overall electrical characteristics of the fully fabricated devices. Several sample preparation methods were developed via dual beam focused ion beam/scanning electron microscope (FIB/SEM)<sup>55</sup> to ensure the region of interest (ROI) was located close to the tip apex ensuring complete data sets. Miller et al. took advantage of SEM contrast differences in capped fin structures to carefully place the ROI at the apex of the completed tip.<sup>56</sup> In cases where a Si fin was capped with amorphous Si and all contrast in SEM was lost, platinum deposited in situ on top of the ROI during sample prep acted as a guide during annular milling.<sup>57</sup>

One of the first instances of investigating dopant distributions in 3D structures was work done by Kambham et al. studying ion implanted Si fins.<sup>58</sup> In this study, fins were implanted with boron at 10° and 45° using a variety of doses (atoms/cm<sup>2</sup>). APT reconstructions clearly showed a reduction in retained dose on the sidewalls of the fins with decreasing implant angle. This conclusion was validated by 1.5D SIMS measurements with good agreement when compared to a theoretical model. It was noted, however, that lateral concentration profiles (orthogonal to the fin sidewalls) were more accurately described in the atom probe data sets which are unobtainable using SIMS. Because finFET performance can be improved with uniform doping profiles in both the top and sides of the fin, accurate quantification of these sidewall profiles is necessary for device development. In cases where fin dimensions are larger than those required for completed APT tips, multiple data sets can be stitched together to fully

characterize the structure. Takamizawa et al. investigated self-regulatory plasma doped fins with a 100 nm width and 100 nm height.<sup>59</sup> These dimensions required several sample runs in order to probe doping profiles in the top, sidewalls, and trench of the fins. Results indicated doping concentrations at the fin top and trench were over an order of magnitude larger than those at in the sidewalls. However, conformal activation was achieved after thermal processing. Similar results were observed by Kim et al for even larger fin structures plasma doped with arsenic and phosphorus.<sup>60</sup>

Compositional and spatial analysis via APT has also been applied to fully fabricated 3D transistor devices. This characterization was not only to probe doping concentrations, but also the quality of interfaces and composition of deposited layers. Previously, this type of analysis was performed using an energy dispersive x-ray spectroscopy (EDS) system in a scanning TEM (STEM) on very thin cross sections.<sup>61–63</sup> An in depth study comparing results from both EDS and APT data sets of gated fins was done by Parikh et al. to demonstrate the viability of atom probe to provide higher resolution concentration profiles.<sup>64</sup> Both techniques provided accurate measurements of both layer thickness and composition, but APT was able to resolve lower z elements, such as boron, which are not detected in EDS. It was determined that APT provided a better outlook on characterization of future scaled devices thanks to its ppm sensitivity and sub-nm resolution. Dopant diffusion in a similar gated finFET plasma doped with arsenic was investigated by Kambham et al. further demonstrating the advantages of APT.<sup>65</sup> Upon thermal treatment, diffusion of As was observed from the S/D region into the channel region under the gate stack of HfO2 and TiN which would lead to poor
device performance (Figure 1-6). These results show the effectiveness of running fully fabricated transistors to gain a wholistic picture of the device.



Figure 1-6: a) Schematic of a gated finFET plasma doped with As and b) associated APT reconstruction showing As (orange), HfO<sub>2</sub> (black), and TiN (pink). Diffusion of dopants is observed from the S/D region of the fin into the channel under the gate.<sup>65</sup>

Atom probe characterization of 3D devices is not strictly limited to finFETs or traditionally fabricated devices. Vertical transistors fabricated from nanowires grown via a vapor-liquid-solid (VLS) method<sup>28,66</sup> have been heavily studied for applications in the 5 nm node and beyond.<sup>24</sup> APT is well suited for characterizing these nanowires as they are fabricated in an orientation requiring little to no sample prep as the wire itself acts as the LEAP tip. As the nanowire has no shank angle, it can run continuously at a constant operating voltage producing very large data sets (shank angle discussed further in Chapter 2-2). A main drawback of using the VLS growth method is the requirement of a metal nanoparticle to form a supersaturated eutectic liquid with a vapor precursor. As the precursor precipitates and forms the wire, atoms from the nanoparticle can incorporate themselves acting as deep level traps. Perea at al. demonstrated one of the

first instances APT was applied to VLS grown doped Ge nanowires and concluded their growth method was able to form nanowires without metallic contamination.<sup>67</sup> In conjunctions with this result, concentration maps illustrated a highly doped shell surrounding a underdoped core stemming from surface decomposition of the dopant precursor vapor onto the nanowire walls. Similar results of nonuniform doping concentrations during VLS growth of Ge nanowires was published by Connell et al. and were attributed to enhanced dopant incorporation at the VLS trijunction (Figure 1-7).<sup>68</sup> Investigations of alternative dopant incorporation methods into VLS nanowires have also been reported. Instead of doping VLS nanowires during growth, nanowires were transferred horizontally and doped via solid source from above and below. APT reconstructions revealed enhanced diffusion of C and P along the outside of the nanowire and a uniform distribution of B within. This diffusion behavior was believed to be due to the higher surface area to volume ratio at these small length scales.



Figure 1-7: a) 2D simulation of P concentration in the liquid droplet during VLS growth.b) Top down view of a portion of the APT reconstruction showing a nonuniform distribution of P atoms.

## **1.6.4 Limitations of Atom Probe Tomography**

While APT is presented as powerful technique rivaling other characterization methods such as TEM, EDS, and SIMS, several challenges are still present and overcoming them constitutes much of the research in the field. Reconstructing data sets relies on the assumption that the tip profile remains hemispherical throughout the entire run. As such, any slight deviation in tip shape can introduce volumetric distortions to the final data set including atomic density variations and compression/expansion of layers. Tip shape changes during evaporation are mainly caused by variations in field strength required to evaporate different atoms in the sample. This value is known as the local evaporation field threshold (F<sub>evap</sub>) and can be estimated using the Müller escape field equation shown in Equation 1-3 for a n+ charged ion.<sup>69</sup>

$$F_n^m = \left(\frac{4\pi\varepsilon_0}{n^3 e^3}\right) (K_n^0)^2 \tag{1-3}$$

$$K_n^0 = \Lambda^0 + H_n - \mathbf{n}\phi \tag{1-4}$$

The thermodynamic term  $K_n^0$  is defined by Equation 1-4 and is related to the zero-field binding energy ( $\Lambda^0$ ), sum of the first n (free space) ionization energies ( $H_n$ ), and the local work function of the surface after removal of the atom ( $\Phi$ ). The equation is better known now as Müller's formula providing only a prediction of the evaporation field as it does not consider the escape mechanism.

Different materials such as metals, semiconductors, and dielectrics have varying evaporation fields ranges. These variations arise from differences in the zero-field binding energy between atoms where large binding energies, like those in oxides, lead to higher evaporation fields and low binding energies, like those in most metals, result in lower evaporation fields. It is when these types of materials are evaporated simultaneously that artefacts are introduced to the tip shape, and in turn, the reconstructions. To maintain a constant evaporation rate (ions detected per laser pulse), a set voltage is applied to the APT specimen. Under this voltage, any atomic species with an evaporation field equal or lesser to the applied field will evaporate from the surface. Unfortunately, those species with a lesser evaporation field threshold will evaporate at a larger rate, eroding the tip more quickly in that particular region. It is this variation in evaporation that leads to tip shape deviations.

Modern 3D transistors contain a variety of materials include the metals, semiconductors, and dielectrics described previously making data analysis of APT difficult. A common distortion is the enhanced magnification of oxide layers between the channel and gate of a device. With a large evaporation field, oxides persist during runs until a smaller tip radius (local) has formed giving rise to focusing effects (Figure 1-8). This result was observed by Grenier et al. in finFETs where the gate oxide was enlarged by a factor of 2-3 while the Si fin was compressed by a similar factor.<sup>70</sup> Geometric distortions can also be present when running multiple interfaces with varying evaporation fields. Fully fabricated PMOS fins ran in a horizonal orientation have shown layer curvature arising from the evaporation of complex stacks and a SiGe S/D region.<sup>64</sup> The observation of these and other distortions signal the need for suitable correction methods to make APT analysis of 3D structures more viable.



Figure 1-8: a) Schematic of an APT tip containing a finFET with the gate oxide shown in blue. The evaporation field of the oxide is larger than that of the Si fin an cap.b) During evaporation, distortions in the tip shape are formed due to the preferential evaporation of the fin/cap over the oxide layers.

## **1.6.5 Distortion Correction Methods**

Several approaches to correct for artifacts in reconstructions have been developed. Corrections to density differences within multiphase reconstructions have been proposed wherein each phase in the data set is selected and reconstructed using each phases evaporation field.<sup>71</sup> While this adjusts the distribution of ions within the volume, atomic concentrations are for the most part preserved. Correlative methods using various microscopy techniques have also been reported.<sup>72</sup> Transmission electron microscopy (TEM) of sharpened tips has been used by several groups to provide an accurate representation of the material structure with which to compare APT reconstructions.<sup>73–75</sup> Electron tomography has also been used to produce 3D images of interfaces for identification of distortions in fins GAA device data sets.<sup>70,76</sup> Work has also been done to correct for these interfacial distortions in which interfaces know to be straight can be flattened improving steepness measurements.<sup>77</sup> These results served as

the basis for the Landmark Reconstruction method used in this work which will be described in Chapter 3.

## 1.7 Oxidation of SiGe Alloys

SiGe alloys have been incorporated into a variety of Si based devices in order to increase channel carrier mobility in MOSFET devices<sup>12,78</sup> and improve the performance of HBTs.<sup>79</sup> One large area of study for the integration of this alloy into large scale device fabrication is SiGe oxidation. When a SiGe alloy is oxidized, Si is preferentially oxidized and Ge is rejected resulting in a pileup of Ge at the Si/SiO<sub>2</sub> interface.<sup>80</sup> Fathy et al. performed oxidation experiments on Si implanted with Ge and found that the formation of a Ge rich layer (GRL) is due to a lower formation energy for  $SiO_2$  (E<sub>SiO2</sub> = -8.2 eV) than  $GeO_2$  (E<sub>GeO2</sub> = -4.7 eV).<sup>81</sup> As oxidation continues, the GRL formed increases in concentration up to a specific value governed by the oxidation temperature.<sup>82</sup> Once this concentration is reached, the Ge rich layer will maintain its thickness and continue to be rejected by the advancing oxide front provided there is Si below it to be oxidized.<sup>83</sup> High concentration Ge layers can be formed if the GRL meets a buried oxide layer. Continuing to oxidize a SiGe layer sandwiched between two oxides eventually results in the formation of a pure Ge layer followed by the consumption of the layer via oxidation (mixed oxide formation).<sup>83</sup> Figure 1-9 illustrates the formation of the GRL during oxidation and the diffusion of the bulk layer through the Si substrate. Several groups have shown the viability of using the oxidation of SiGe layers to form germanium on insulator (GeOI) substrates for fully depleted MOSFET devices.84-87



Figure 1-9: Schematic showing the oxidation of a SiGe allow (Ge fraction of 30%) on a Si substrate. The darker red layer denotes the GRL with a Ge fraction of 50%.

## **1.7.1 Temperature Dependence**

The maximum achievable Ge concentration in the pileup layer during oxidation has been found to be dependent on the oxidation temperature. XRD results from Long et al. demonstrated a peak Ge concentration value for oxidation temperatures ranging from 800 °C to 1000 °C and were found to be independent of oxide thickness and initial Ge content.<sup>82</sup> Their work also concluded that the increase in Ge content within the SiGe layer does not increase the oxidation rate of the layer, even compared to pure Si indicating that, for dry oxidation, Ge does not act as a catalyst for oxidation. Additional experiments by Long et al. helped in developing a model to describe the concentration of Ge in the pileup layer as a function of temperature and oxide thickness (oxidation time).<sup>88</sup> These models showed good agreement with experimental data for oxide thicknesses up to 45nm, but also indicated that Ge concentration would continue to increase with long oxidation time. Such a trend contradicts previous conclusions in which a peak concentration value is reached indicating the need for further refinement of the model.

Temperature settings in which the Ge fraction at the oxidation interface is kept static, increased, or decreased have been reported.<sup>89</sup> In particular, two regimes have been widely used based on the application of the condensed layer. The high temperature regime has been shown to form homogeneous SiGe layers as opposed to forming a GRL. This is due largely in part to the high interdiffusion coefficient of Ge in SiGe allowing for rapid redistribution of condensed Ge at the oxidizing interface. The low temperature regime produces GRLs with abrupt interfaces within the SiGe film as the interdiffusion coefficient is lessened considerably. While this regime has applications in GeOI devices, defects caused by the relaxation of strain energy between the epitaxial layers are commonplace.<sup>90</sup>

## 1.7.2 Proposed Mechanisms for SiGe Oxidation

Currently, there is no agreed upon mechanism to describe the diffusion of the Ge pileup layer through a Si substrate. Oxidation studies by LeGoues et al. showed that the SiGe layer suppressed the injection of interstitials and instead created an excess of vacancies.<sup>91</sup> This was observed by the suppressed diffusion of Boron normally enhanced by oxidation and led to the postulation that Si diffused up through the SiGe to the oxidizing interface. Excess vacancies were believed to account for the enhanced diffusion of Si thorough the GRL to the oxidizing interface. A diffusion model was proposed by Hellberg et al. to describe the oxidation of SiGe layers where silicon oxidation was enhanced by the presence of Ge<sup>92</sup>. It was proposed that GeO<sub>2</sub> was formed at the oxidation interface and then subsequently reduced by free Si atoms in the SiGe layer. According the ternary phase diagram of Si, Ge, and O at 1000K, GeO<sub>2</sub> and SiGe cannot coexist in equilibrium unless the Si fraction in the alloy is less than 3.2x10<sup>-19</sup>, or effectively 100% Ge. This conclusion is further supported by the free energy

change associated with the reduction ( $\Delta G = -356 \text{ kJ/mol } O_2 \text{ at } 1000 \text{K}$ ) and has been shown experimentally<sup>93</sup>.

Work done by Long et al. disproved the idea that oxidation of Si was enhanced by Ge.<sup>88</sup> Instead, they proposed a mechanism consisting of a balancing of three fundamental fluxes of Si: the flux of Si from SiGe into the oxide (J<sub>ox</sub>), the flux of Si through the SiGe layer (J<sub>pileup</sub>), and the flux of Si from the substrate into the SiGe layer (J<sub>substrate</sub>). It was determined that when J<sub>ox</sub> > J<sub>pileup</sub>, a pileup region is formed increasing the layers Ge fraction. However, once J<sub>ox</sub> = J<sub>pileup</sub>, a constant Ge fraction is reached, and the pileup layer continues to translate through the Si substrate. The rapidity at which the layer moved through a Si substrate once reaching it's maximum Ge fraction was attributed to a larger diffusivity of Si in Ge over Si in Si.<sup>94</sup> In cases where J<sub>substrate</sub> = 0, no Si remains to be oxidized and the layer condensed until reaching a Ge fraction 100% followed by mixed oxide growth. This description remains one of most accurate when referring to the dry oxidation of SiGe films.

#### **1.8 Summary and Statement of Research Goals**

The advancement in semiconductor technology has been shown to be dependent on reducing contact resistances by strategically doping S/D regions in 3D devices. While many techniques exist to characterize doping profiles, they fall short when attempting to describe 3D systems. Atom probe tomography has been demonstrated to be a promising method to quantify doping profiles in such systems, but still contains artifacts in reconstructions of heterostructures with varying evaporation fields. This work will examine methods to remove such distortions and explore their viability in producing accurate data sets describing not only the overall device structure,

but profiles of implanted dopants. A better understanding of the statistical nature of extracted doping profiles post distortion correction will also be investigated. Finally, a brand-new Ge diffusion process discovered during the course of this work will be presented along with applications in the fabrication of strained vertically stacked Si nanowires.

# CHAPTER 2 ANALYTICAL TECHNIQUES AND EXPERIMENTAL METHODS

#### 2.1 Materials Processing

## 2.1.1 Fin Fabrication

In semiconductor fabrication, the growth of single crystal channel, source, and drain regions is critical to device performance. Any defects, ranging from simple dislocations to large grain boundaries, can lead to scattering of electrons and severely reduce switching speeds and drive current. Growth of these single crystal layers is typically achieved using an epitaxial growth process where the grown layer lattice matches to the substrate. Historically, the most typical method for growing epitaxial Si onto a Si substrate was via a chemical vapor deposition (CVD) method. A silicon precursor like dichlorosilane was flowed through a quartz horizontal reactor with hydrogen as a carrier gas. Surface temperatures of ~1100 °C were achieved through lamp heating to drive the reaction

$$SiCl_2 \Leftrightarrow Si + 2HCl$$
 (2-1)

depositing Si onto the sample surface. High deposition temperatures are required for enhanced surface mobility of deposited atoms to ensure the grown layer remains crystalline and lattice matched.

Formation of alloy layers such as SiGe can be fabricated in a similar manner via a heteroepitaxial process. Deposition of these layers utilizes the same equipment but flows a germanium precursor in addition to dichlorosilane and hydrogen. For most epitaxial deposition applications, germane (GeH<sub>4</sub>) is used as a Ge precursor. Adjusting the relative flow rates of both precursors allows for control of the Si and Ge ratio based on the desired application. As both elements are miscible with each other, a

homogeneous alloy is formed.<sup>95</sup> However, differences between the Si and Ge lattice constants ( $a_{Ge} > a_{Si}$ ) necessitates a compression of the deposited layer lattice in the xand y-directions (lateral) and a stretching in the z-direction (vertical) to lattice match to the Si substrate. Because of this distortion, a biaxial compressive strain is developed on the deposited film. To prevent the strained layer from relaxing and forming defects, Ge concentrations must be kept below critical concentrations based on the layer thickness.<sup>96</sup>



Figure 2-1: a) Schematic showing the fabrication of multilayer Si/SiGe fin structures. b) High magnification HAADF-STEM image showing Si/SiGe interfaces and c) low mag image showing cross section of completed fin.

Achieving the required high aspect ratios for 3D structures requires anisotropic removal of the semiconducting material. Once layers have been deposited, features patterned by photolithography can be formed via a dry etch processes. The most commonly used process to make high aspect ratio Si features is a deep reactive ion etching method known as the Bosch process. In this method, alternating steps etch and passivate the sample to produce nearly vertical structures. Etching of Si is achieved using a plasma formed from a  $SF_6$  precursor which dissociates into individual fluorine atoms. Fluorine then bonds with Si surface atoms forming a volatile SiF<sub>4</sub> compound which leaves the surface. To prevent etching material underneath the photo mask, Ar is introduced to the plasma along with an applied electric field drawing the Ar ions to the surface. This introduces a directional physical etch in conjunction with the chemical etch. Even with the addition of Ar, some isotropic etching still occurs. Between etch phases, carbon compounds (typically C<sub>4</sub>F<sub>8</sub>) are introduced into the chamber which deposit a passivation layer. Resuming the etch removes passivation layers at the bottom of trenches but not the sidewalls thanks to the direction sputtering of the Ar ions. Continued etching down through the material can then resume revealing structures with flat sidewalls. The process is repeated multiple times until the desired etch depth is reached.

Fin structures in this work were fabricated on commercially available 300 mm (100) Si wafers using similar CVD deposition and dry etch techniques described above. To produce superlattice Si/SiGe fins, alternating layers of Si and SiGe were epitaxially deposited onto Si wafers cleaned with HF. Switching the germane precursor on and off during the run allowed for the formation of sharp interfaces between the layers. The

abruptness of the change was dependent on whether the gas was being turned on or off. Sharper interfaces were formed when turning the gas on with more diffuse (relatively) interfaces forming when the gas was turned off due to residual germane precursor left in the chamber. Fin patterns were formed in the <110> direction using photolithography and a deep reactive ion etch (DRIE). Targeted spacing between fins was greater than 200 nm to prevent shadowing during ion implants or the meeting of oxidation fronts from neighboring fins which would halt further oxidation. Figure 2-1a shows illustrations of the deposition, patterning, and etch steps to form the fins. HAADF-STEM images demonstrating the formation of high quality interfaces, defect free layers, and the final fin structure are presented in Figure 2-1b and Figure 2-1c.

#### 2.1.2 Ion Implant

Tailoring the electrical properties of specific regions in a MOSFET device is carried out through the introduction of dopant atoms (intentional impurities). When sitting on a lattice site, an n-type dopant like arsenic will donate its extra electron increasing the number free carriers in the solid. The same is true with p-type dopants like boron that provide excess holes. Increasing the number of free carriers, or doping, in semiconductors is the backbone of transistor fabrication and requires accurate dopant incorporation.

Ion implantation has been the mainstay in industry for dopant incorporation over solid source diffusion or plasma doping thanks to its control of doping depth and concentration. In this system, dopant atoms are ionized from a gas source and accelerated via an applied electric field creating an ion beam. Mass selection is carried out using an analyzer magnet which filters out ions without the proper mass-to-charge ratio. These filtered ions are then incident onto a beam stop, allowing an isotopically

pure ion beam to continue towards the sample. Once the proper ions have been selected, they are accelerated to a user-defined energy and rastered across the sample through deflector coils. It is the acceleration energy that defines the implantation depth of dopants in the sample. This depth is referred to as the projected range ( $R_p$ ). Because the incident dopant atoms are ionized, a charge integrator can calculate an implanted dose (atoms/cm<sup>2</sup>) based on the current required to neutralize the implanted charges. Therefore, adjusting the amount of time the ion beam is incident upon a sample can control the overall dose ( $\Phi$ ). Adjusting this dose in turn adjusts the peak doping concentration observed at  $R_p$ . Figure 2-2 shows a schematic of an ion implant system as well as a Monte Carlo simulated implant concentration profile from TRIM.<sup>97</sup>



# Figure 2-2: Schematic of a beamline ion implant system (left) and a simulated concentration profile for a 10 keV As implant at 7° into Si with a 1 x 10<sup>15</sup> cm<sup>-2</sup> dose.

Implanting single crystal materials with high energy ions isn't without its

drawbacks. Once implanted ion energies drop below 100 keV within the lattice,

electronic stopping (interactions with the electron cloud) of dopant atoms within the

target gives way to nuclear stopping.<sup>98</sup> At this stage, collision cascades occur through

the lattice creating interstitial and vacancy pairs known as Frenkel pairs. Some of these

point defects diffuse to and recombine with one another in a process known as dynamic annealing. Repairing damage from the ion implant process in semiconductors is critical for proper performance of fabricated devices. Defects in the lattice can act as scattering points for charge carriers and can propagate into epitaxial layers grown above them. High-temperature annealing is used to repair ion implant damage, providing enough thermal energy for diffusion of atoms back to lattice sites. Annealing also allows for implanted dopants to move onto lattice sites, enabling the donation of excess charge carriers. This process is known as activation.

At high doses, the target layer can even incur enough damage to become amorphized. In the case of amorphized layers, solid phase epitaxial regrowth can occur using the undamaged lattice beneath as a template.<sup>99</sup> Regrowth in samples amorphized by the implant process occurs without the formation of extended defects in the regrown layer and can even lead to higher levels of activation.<sup>100,101</sup> For non-amorphizing implants, gettering of excess interstitials can occur, forming extended defects known as dislocation loops.<sup>102,103</sup>

## 2.1.3 Thermal Processing

As mentioned in Chapter 2.1.1 and 2.1.2, high temperature processing is required for select fabrication steps involving diffusion or growth. These techniques are sensitive to temperature fluctuations and processing times necessitating careful control to ensure repeatable and accurate experiments. A variety of thermal processing techniques are available based on the required temperature and times. This work utilized two methods: rapid thermal annealing and tube furnace annealing.

Furnace anneals have historically been used in the literature and industry for annealing allowing for high volume and throughput. Tube furnace anneals were carried

out in a fused silica tube heated by heating coils and controlled by an internal thermocouple. Temperature variations within the tube were still present necessitating additional and more precise measurements. A second thermocouple was inserted into the open end of the tube furnace to measure which zone along the tube provided the required temperature. Anneals in this system were reserved for timescales over 5 minutes in length due to the slower ramp rates when compared to system heated by lamps. Contributions from the thermal ramp (both up and down) could then be minimized when compared to those received while the sample is at the set temperature. Atmosphere control was achieved by a gas inlet located at one end of the tube and flowing the gas outlet through a bubbler. Using this set up, both dry O<sub>2</sub> and inert gases like Ar could be introduced into the tube furnace at atmospheric pressures. Gas flowrates of 5 L/min were targeted using a standard flowmeter (rotameter).

Shorter anneal times were achieved using a rapid thermal annealing (RTA) system. Instead of heating coils, sample heating is achieved using halogen lamps allowing for maximum ramp rates in ranges of hundreds of °C per second. Heating in these systems is also surface sensitive unlike equilibrium annealing performed in furnaces, thus reducing required thermal budgets. The system used in this work was an AG Associates HeatPulse 4100 allowing for annealing of wafers 200 mm or smaller. To prevent loss of energy from undesired heating, the chamber, stage, and pins used to hold samples were made of quartz absorbing little to no radiation produced by the lamps. Proportional integral derivative (PID) controllers were utilized for optimal temperature control by calculating the required lamp input power. Temperature readings

from a thermocouple below the sample provide feedback to the PID controller modulating the lamp power. Recipes for each anneal temperature were created by a trial and error method adjusting control parameters until the proper temperature and ramp rates were achieved.

## 2.1.4 Focused Ion Beam

In order to prepare samples for both transmission electron microscopy (TEM) and atom probe tomography (APT) analysis, a dual focused ion beam and scanning electron microscope (FIB/SEM) was implemented. Both TEM and APT require very small sample volumes with critical dimensions approaching <50 nm from very specific regions. Sample preparation methods like plan view ion milling (TEM) or electropolishing (APT) can provide the necessary critical dimensions for their respective analysis, but they lack the ability for fine control of site specific placement. As this work focuses on nanoscale 3D transistors, site specific sample preparation is critical for accurate characterization. With achievable beam sizes down to ~2-5 nm, the dual beam FIB/SEM has been demonstrated to be perfect for sample preparation.

Sample preparation via the FIB/SEM is achieved through a phenomenon known as sputtering where high energy ions incident on a surface lead to the ejection of target atoms from a solid. The FIB utilizes a liquid gallium ion source in which heated gallium flows onto a tungsten needle wetting the tip.<sup>104</sup> A bias is applied to the needle creating a large electric field leading to field evaporation of Ga ions located at the tip apex. Magnetic lenses then focus the Ga beam down onto the sample in a similar fashion to a SEM. Upon Ga ions striking the surface, secondary electrons are emitted which can be collected to form an image. Imaging samples via the ion beam is generally discouraged as the high energy ions damage and sputter away surface material. Instead, imaging

and sample placement is carried out using the SEM, protecting the surface. To ensure both beams are observing the same areas, samples are brought to a stage height where the two beams are coincident and at the horizontal center of the objective lenses (known as the eucentric height).

Site specific milling is carried out by rastering the ion beam over a user defined region. Mill depth is controlled by the number of passes the beam makes over a specific region and is controlled by the mill time. This mill time is largely influenced by the accelerating voltage and beam current where larger voltages and currents reduce overall mill times. When milling large amounts of material, these high voltages and currents are desirable. During finer milling steps, the beam current must be reduced to reduce the overall beam size allowing for more precise removal of material.

Deposition of protective capping layers is also achievable in FIBs using metal organic gases. These gases are flowed across the sample while the Ga beam rasters in a defined region depositing the metal compound. In a vast majority of FIBs, platinum is the most typical material used for in situ deposition of protective layers. The need for protective layers stems from the cascading damage introduced to a material when struck with a high energy ion. When characterizing single crystal materials, damage from the ion beam can amorphize regions of interest erasing important structural information. The large atomic mass of Pt makes it a prime candidate for capping samples as the implantation depth of Ga is greatly reduced when compared to Si.

In this work, a FEI Helios NanoLab 600 is utilized for a majority of TEM and APT sample prep. Specifics on the preparation procedures can be found in Chapter 2.2.2.2 and Chapter 2.3.

#### 2.2 Atom Probe Tomography

#### 2.2.1 Principle and Instrumentation

As described in Chapter 1.6.1, the modern atom probe tool can be described as a combination of FIM, pulsed laser systems, time of flight measurements, a position sensitive detector, field enhancement devices, and complex reconstruction and analysis software. It is these devices that make up the LEAP system used in this work allowing for high resolution sub-nm chemical and spatial analysis needed for investigating 3D electronic devices.

The system used in this work is a LEAP 5000 XS system developed and built by CAMECA. A schematic of this system is illustrated in Figure 2-3 including dimensions of typical APT tip samples. These specimens are placed in proximity to a local electrode (LE) while a bias is applied creating a strong electric field at the apex of the tip. Enhancement of the field is facilitated by the LE reducing the voltage necessary of field evaporation to occur. Further reduction of the required field is achieved by pulsing a UV laser ( $\lambda$  = 355 nm) onto the tip. Once an atom has been evaporated from the tip surface and passes through the LE, it enters the field free region where it is incident on the position sensitive detector. Here, the ions time of flight is recorded as well as its X and Y coordinates on the detector. With these data points, atom by atom reconstructions are possible with atomic identification.



Figure 2-3: Schematic of the LEAP system (left) showing the position of the sample tip, local electrode, laser, and detector. Tip shape dimensions (right) are also shown.

Field evaporation is a process by which the ionization of an atomic species becomes more preferable than remaining in a neutral state under an applied electric field. A typical energy well diagram is shown in Figure 2-4 with the most probable bonding distance located at the energy minimum. The application of an external potential field reduces the potential energy for larger separation distances and in turn reduces the barrier for bonding. Once ionized, a positively charged ion would then be repelled by the positively charged surface and follow trajectories orthogonal to equipotential lines.



Internuclear Separation

While field evaporation of ions in semiconductors can occur at high fields produced by large voltages (4000-10000 V), stress from these voltages can lead to sample fracture. Laser pulses onto the sample can reduce the required voltage needed for evaporation to occur, but details of the mechanism by which this occurs is still under debate. One theory suggests that the field component of the laser induces field evaporation of atoms at the surface.<sup>37</sup> Another suggests that surface atoms can overcome the ionization barrier from the applied field through electronic excitation or thermal heating from laser interactions with the solid.<sup>105</sup> Both theories have been supported through experiments, but in most instances, the addition of thermal energy to evaporate atoms seems most likely. Laser-assisted evaporation is utilized in this work as a means of reducing the required voltage necessary for evaporation to occur in semiconducting materials thus reducing the odds of sample failure through fracture. To prevent overheating of the sample, the laser is pulsed at rates ranging from 50 kHz to 1 MHz to allow for sufficient cooling.

Figure 2-4: Potential well diagram illustrating the lowering of the well with an applied external potential.

Voltage control in the LEAP system is critical to ensure uniform evaporation of atoms from the surface. Feedback systems adjust the voltage such that the number of detected ions per laser pulse is kept constant. This value is known as the detection rate (DR) and is normally presented as a percentage (# ions detected per single pulse x 100). The evaporation field (F) at the apex of a tip with tip radius r (see Figure 2-3), dimensionless geometric factor k (typically 3.5 for conical tips), and applied voltage V can be calculated by Equation 2-2.

$$F = \frac{V}{kr}$$
(2-2)

It becomes apparent that as atoms are evaporated from the surface under a constant applied voltage, the radius of the tip increases, thus reducing the evaporation field and DR. The feedback system then increases the voltage to main the preset DR and allow for continued collection of ions. Under these conditions, a field strength just under the threshold for evaporation is produced. Ions can then be evaporated one at a time by laser pulses allowing for single ion identification.

Chemical identification of the evaporated ions is carried out using time of flight mass spectrometry measurements. Time measurements are taken between the laser pulse and subsequent detection of an ion on the microchannel plate positioned just before the position sensitive detector. A mass to charge ration can then be calculated using Equation 2-3:

$$\frac{m}{q} = -2eV\left(\frac{t}{L}\right)^2 \tag{2-3}$$

where m is the ion mass, q is the ion charge, V is the accelerating voltage (or applied voltage), t this the time of flight, and L is the flight distance. This mass to charge ratio can then be used to define the atomic species present in the reconstruction.

#### 2.2.2 Sample Preparation

For field evaporation to occur at reasonable voltages (2000-10000 V), samples must be milled into needle shaped specimens with a tip radius on the order of 100 nm (Figure 2-3). Sharp tips enhance the fields generated by an applied voltage and help reduce the applied stress on the samples. Traditionally, metallic APT samples could be fabricated via a method know as electropolishing. In this method, metals were formed into wires with a diameter on the range of 0.2 mm and immersed into an acidic solution. A platinum loop was then placed around a portion of the wire hooked up to a DC voltage supply to act as an electrode. This set up created an electrolytic cell etching the metal wire in the region encompassed by the loop. Moving the loop up and down allowed for the formation of a necked region until the wire fractured under its own weight. Two completed APT tips were then produced with smooth surfaces and appropriate tip radii. While this process proved to be a quick preparation method, it was only applicable to bulk materials and did not provide the ability to place specific interfaces or devices at the tip apex.

Site-specific sample preparation of APT using a dual beam FIB/SEM was achieved by applying a similar lift out and mounting technique for preparing TEM samples (see Chapter 2.1.4). Miller et al. formed samples by milling trenches into the substrate at 30° relative to the surface on either side of a protective deposited Pt strip forming a long wedge.<sup>55</sup> The wedge would then be released from the substrate and lifted out via a manipulating probe. Multiple samples could then be created by placing

the free end of the wedge onto a flat top Si microtip, welding it in place via Pt deposition from the FIB, and freeing the sample from the bulk wedge. Annular milling masks could then be used to sharpen the mounted sample down to a needle with a tip radius of ~200-100 nm. Final sharpening was done with lower beam voltages on the order of 2-5 kV to sharpen the sample further (~50-30 nm) and remove regions damaged by the high voltage Ga beam.

#### 2.2.2.1 Sample capping layer

Capping layers deposited on APT samples serve a dual purpose. The first is to protect the sample from damage incurred via the high voltage ion beam during milling steps. The second is to create a more uniform evaporation surface and fill gaps within the material. This becomes critically important when running 3D devices like finFETs as open space between the fins would prevents the formation of a tip with a smooth conical shape. Capping layer material selection is largely dependent on the material system being investigated. To prevent introducing spatial distortions to the reconstructed data set, the evaporation field of the capping layer should closely match that of the sample. With similar evaporation fields, the capping layer and sample material will evaporate at a more uniform rate preventing deviations in the overall tip shape.

For this work, amorphous silicon ( $\alpha$ -Si) was chosen as the capping layer which provided a similar evaporation field to the primarily crystalline Si sample. A plasmaenhanced chemical vapor deposition (PEVCD) process was used to deposit  $\alpha$ -Si onto the fin structures via a STS 310 instrument operating at 350 °C. While an epitaxial deposition process described in Chapter 2.1.1 could provide a quality uniform layer, high processing temperatures could lead to undesirable diffusion introduced dopants. Striking a plasma via an RF source supplies much of the energy required for a reaction

reducing the processing temperature. A silane precursor was utilized flowing with an inert nitrogen carrier gas to control the chamber pressure. PECVD was also chosen over sputtering, a physical vapor deposition process, as it provided better conformity along the uneven surface. While small voids were still present between fins after PECVD deposition, they were removed during APT sample sharpening steps.

#### 2.2.2.2 Sample lift out

Alterations to Miller's preparation method were made for samples investigated in this work to make completed samples more robust and improve yield. This entailed increasing the distance between the tip apex and the Pt weld as well as increasing the width of the tip below the sample. Such changes increased the strength of the completed tip and prevented the APT laser from aligning to the weld. Figure 2-5 illustrates the new lift-out method completed using the FIB/SEM outlined in Chapter 2.1.4. Instead of milling out large angled trenches beneath the protected surface, a staircase style mill normal to the surface was performed similar to standard TEM sample preparation. Milling using this pattern led to shorter mill times over using a rectangular pattern to remove all of the material in the trench. Larger mill depths on the order of ~5 µm could then be achieved, over 2x deeper when compared to the standard method. The undercut step was performed by tilting the stage 15° and milling away a portion of the bottom of the cross section from either side (see step 3 in Figure 2-5). Additionally, material connecting one side of the sample to the substrate was removed turning the sample into a cantilever. Finally, a manipulating probe was contacted and welded to the freed side of the sample. Milling away the final connected side of the cross section freed it from the substrate allowing it to be lifted out via the manipulating probe. Mounting was achieved using the standard methods outlined by Miller et al.<sup>55,56</sup>



Figure 2-5: Schematic depicting the lift out method for APT samples in this work. 1) A protective Pt strip is deposited. 2) a staircase style mill is performed defining the cross section to be lifted out. 3) Partial release of the wedge. 4) Lift out using a manipulating probe.

# 2.2.2.3 Sample sharpening

Sharpening the mounted samples was completed using a TESACN LYRA

FIB/SEM. Due to the site-specific nature of this preparation, great care was taken to

ensure fins were centered with the tip apex during all annular milling steps. Centering of

the fins was achieved by adjusting the position of the annular milling pattern laterally

after every subsequent mill. As the capping layer used to protect the samples during milling was the same material as the substrate and portions of the fins (Si), visualization of the fins using the secondary electron detector of the SEM became difficult. However, mass contrast between Ge and Si could be observed using the backscatter electron (BSE) detector, even at high magnifications. In this mode, heavier elements like Ge and Pt appear brighter than Si as they have stronger elastic scattering interactions with incident electrons. Figure 2-6a and Figure 2-6b show BSE images of a tip sharpened from showing four fins, to one centered fin respectively. This demonstrates not only a good centering method, but also excellent visualization of the fins.

Final sharpening was carried out using a low ion beam voltage of 2V to remove material damaged by the high voltage beam and further sharpen the sample. In addition, this process also allowed for positioning of the fin near the apex of the APT tip. Apex positioning was critical for improving yield as having to evaporate large amounts of capping material increased the tip radius once reaching the fin, thus increasing the applied voltage and stress. Live imaging of the sample during the mill process allowed for fine control of the apex position. A completed APT tip is shown in Figure 2-6c with the fin ~50 nm from the apex demonstrating the precision achievable on the instrument using the BSE detector.



Figure 2-6: SEM images (using a BSD) of APT tips at various stages during the milling process with a) four fins showing and b) one fin centered. c) Completed tip with measurement lines for reconstruction purposes.

# 2.2.3 Data Reconstruction

In order to visualize data collected during APT runs, specialized software is utilized to reconstruct the evaporated volume. Integrated Visual and Analysis Software (IVAS) developed by CAMECA uses outputs from the LEAP 5000 instrument to calculate not only atomic positions, but atomic identity as well. This section serves to provide a basic understanding of the reconstruction process, variables used to adjust the dimensions of the reconstructed volume, and brief discussion of analysis tools used in the software package.

Reconstruction begins by selecting the ions which will make up the final tip volume. The selection process entails providing boundaries to the voltage profile and detector event histogram which results in removing ions at the edge of the detector or those detected during the initial evaporation turn on stage. After ion selection, elemental identification is carried out using the accumulated mass spectrum calculated from TOF measurements (see Chapter 2.2.1). Relative peak locations in this spectrum correspond to specific elemental isotopes within the sample. For example, Si is present in atomic masses of 28, 29, and 30 for singly charged ions. Under APT evaporation conditions, Si is more likely to be evaporated in a 2+ charge state causing a shift in the detected mass to charge ratio (m/q). In this case, the mass to charge ration is reduced by a factor of two displaying Si ions in the mass spectrum under 14, 14.5, and 15. The relative size of these peaks will be proportional to the elements natural abundance (if not isotopically pure). Once identified, mass spectrum peaks can be assigned an element or molecule providing chemical identification to the associated detected ions.

Once chemical identification of all relevant ions is completed, 3D rendering of the volume can begin. As ions were evaporated from the surface onto the position sensitive detector, their X and Y positions were recorded. Because ions are evaporated one by one during laser pulses, a sequence of hitting history can be obtained. From this sequence, ions evaporated from the same layer can be identified providing a means of calculating Z coordinates. However, the positions of these ions on the 80 mm detector are magnified from the sub 100 nm tip apex. The magnitude of the magnification can be calculated using Equation 2-4:

$$\eta = \frac{L}{\xi r} \tag{2-4}$$

where  $\eta$  is the magnification, L is the distance from the tip apex to the detector,  $\xi$  is dimensionless projection parameter between 1 and 2 known as the image compression factor (ICF), and r is the tip radius. The tip radius can be determined by rearranging Equation 2-2 and inputting the applied voltage and evaporation field producing Equation 2-5.

$$r = \frac{V}{kF}$$
(2-5)

With this, the original X and Y coordinates can be expressed as

$$X = \frac{X_a d_x - \frac{d_x}{2}}{\eta}$$
(2-6)

$$Y = \frac{Y_a d_y - \frac{d_y}{2}}{\eta}$$
(2-7)

where  $X_a$  and  $Y_a$  are the relative coordinates on the detector and  $d_x$  and  $d_y$  are the physical extents of the active area of the position sensitive detector in their respective directions. The numerator of both Equation 2-6 and 2-7 convert the relative coordinates into true distances.<sup>106</sup> Finally, the Z coordinate is calculated from the ion hitting order history in which each ion hitting a detector in the same area A<sub>d</sub>, the Z value is changed by a distance  $\delta Z_i$  given by

$$\delta Z_i = \frac{\Omega_i \eta^2}{\zeta A_d} \tag{2-8}$$

where  $\Omega_i$  is the atomic volume of the ith ion in the analysis phase and  $\zeta$  is the detector efficiency (number of ions detected/number of ions evaporated).

With the corrected X, Y, and Z coordinates, a 3D reconstruction of the tip volume is created. As tip shapes vary based on sample preparation, more inputs are required to tailor the shape of the reconstruction to match the shape observed in SEM or TEM. Two common methods of determining tip shape are the use of voltage profiles or the use of a single half shank angle and initial tip radius. Reconstructing using the voltage profile mode calculates the tip radius for every evaporated layer using Equation 2-5 based on the evaporation field of the primary sample element. However, materials with several interfaces of varying evaporation fields can see large shifts in applied voltages introducing distortions into the reconstructed volume. Using a measured half shank angle and initial tip radius (Figure 2-3), a conical reconstruction with smooth sides can be produced. In most cases, this type of reconstruction more accurately reflects the shape of the APT specimen. More recently, an additional reconstruction algorithm was created which utilizes imported TEM or SEM images of the completed tip. A dimensional profile can then be made from these images and applied to the reconstruction more accurately representing the tip shape than a shank angle reconstruction. Further improvements to the tip shape can be made by adjusting the ICF, effectively adjusting the magnification. Changes to this factor can be made until identifying features within the volume are correctly proportioned or scaled appropriately.

**Reconstruction analysis tools:** Once a volume has been reconstructed, data can be extracted via a number of methods beside simple visualization. The most common is the use of data pipes to probe variations in atomic concentration with distance. To produce these profiles, 3D regions of interest (ROI) such as rectangular prisms are created with specific dimensions and placed such that interfaces, clusters, etc. are within the ROI volume. Concentration vs. distance plots can then be created in the desirable direction (most often the longest direction). Sensitivity is determined via user defined bin sizes which break up the volume into sections along the analysis direction. Atomic concentrations can then be calculated in each section by dividing the number of a selected ion by the total number of ions. Due to the relatively small size of reconstructed volumes, small bin sizes are discouraged as large errors are expected due to poor counting statistics with very few numbers of ions. An example of a 1D

concentration profile is shown in Figure 2-7 for an As ion implanted Si fin. A 10x10x30 nm ROI was created and positioned near the fin/cap interface. A peak concentration of As at the interface (defined by the O peak concentration) is clearly observed.





Figure 2-7: Reconstruction of an As ion-implanted Si fin (above) with As isosurfaces in pink and SiO ions in white. 1D concentration profile (below) showing atomic concentrations of As (pink), O (blue), and Si (gray).

To facilitate in the observation of interfaces, surfaces of constant concentration,

or isosurfaces, can be produced for specified ions. Such surfaces can define interfaces

between layers or the bounds of precipitates within the sample volume. In the case of precipitates, isosurfaces can aid in counting as well as producing size distributions for a more advanced analysis. Figure 2-7 shows As isosurfaces (pink) defining regions of As with a concentration of 0.5 at%. It becomes more evident that a majority of the dopants are located near the surface of the fin. Proximity histograms (or proxigrams) can also be produced by moving isosurfaces +/- a few nm and computing the change in concertation. This method is most useful when characterizing precipitates or clusters and defining decay rates (distance required for a 10x reduction in concentration) at their interfaces.

## 2.3 Transmission Electron Microscopy

Transmission electron microscopy (TEM) is another powerful technique for characterizing the structural and chemical compositions of materials. While TEM does not provide the 3D positional information generated in APT data sets, direct observation of a sample's structure free from volumetric distortions is achievable (under proper conditions). As transistor devices have continued to scale from the micron regime down to the sub 10 nm regime, characterization methods at these length scales and below have become increasingly more important.

High-magnification imaging using TEM is achieved by taking advantage of the particle-wave duality of electrons. Accelerating electrons using voltages on the order of 80-200 kV can produce an electron beam with wavelengths down to ~2.73 pm, vastly outperforming microscopes using visible light ( $\lambda$  from 380 to 750 nm). A series of magnetic lenses and apertures focus the beam down onto a prepared specimen near the middle of the TEM column. As the beam passes through the specimen, electrons interacting with the material are scattered and incident onto a phosphor screen or CCD

detector. Information gained from this process is highly dependent on the structure, orientation, and quality of the specimen as well as the selected imaging mode.

Imaging single crystal materials leads to the formation of distinct diffraction patterns (DP) which can be observed by projecting the focused back focal plane of the objective lens onto the phosphor screen. In this pattern, scattered electrons from specific families of planes form pairs of points determined by Bragg's Law:

$$\lambda = 2d\sin\theta \tag{2-9}$$

where  $\lambda$  is the wavelength of the incident electrons, d is the interplanar spacing within the crystal for the specific set of planes, and  $\theta$  is the angle of incidence. Varying the zone axis, or plane normal to the incident electron beam, can produce different DPs. In this work, all TEM specimens are orientated along the (110) crystallographic plane as structures are fabricated in the [110] directions. A DP for this orientation is shown in Figure 2-8.

By selecting specific points in the DP with the objective aperture and blocking others, different imaging modes can be achieved. The simplest is known as bright field (BF) and requires imaging using only the transmitted spot blocking all scattered electrons. Contrast from mass and diffracted planes can be obtained using this mode where dark regions are areas in which high scattering occurs. Including at least one pair of Bragg spots with the transmitted spot enables high-resolution TEM (HRTEM) imaging. In Si and Ge, diffracted spots from the {111}, {200} (allowed in the DP by double diffraction), and {220} family of planes are typically used for HRTEM imaging. Interference from these scattered electrons allows for lattice imaging in which the projected image resembles the crystal structure. It is important to note lattice fringes do

not represent actual atoms, but only the interference between planes. Dark field (DF) imaging only selects individual Bragg spots in the DP while the sample is tilted to enhance the scattering at that point. In this mode, bright regions correspond to heavily diffracted areas and is typically used for defect imaging. Figure 2-8 shows illustration of objective aperture placements for the three imaging modes.



Figure 2-8: Illustration of a Si (110) diffraction pattern observed in TEM and objective aperture placements for bright field, high resolution, and dark field imaging.Specimen preparation for cross-sectional TEM analysis is carried out using a

dual beam FIB/SEM<sup>107</sup> in a very similar manner as APT samples (see Chapter 2.2.2.2). Trenches are milled out on either side of the region of interest protected by a strip of FIB deposited platinum. Lift out is achieved by a manipulating probe (Omniprobe) after which the lamella is welded onto a copper TEM grid. The lamella is then thinned down
until transparent to electrons and cleaned of layers damaged by the high energy ion beam via a low voltage milling step. Once completed, the grid can be placed into a TEM for analysis.

**High-angle annular dark-field scanning TEM:** Converging the electron beam into a probe and rastering it over the specimen is known as scanning transmission electron microscopy (STEM). In this mode, intensities from individual columns of atoms can be detected at resolutions lower than traditional HRTEM approaching point to point resolutions of 0.78 Å in aberration corrected tools. Images in this work were captured using the imaging mode known as high-angle annular dark-field (HAADF) STEM. As the name suggests, an annular detector is used collecting electrons scattered from atomic columns at high angles. Scattering angles of these electrons is strongly dependent on Z, or the atomic number, with heavy elements scattering electrons more strongly than lighter elements. Therefore, larger signals are observed at the annular detectors for higher Z elements giving rise to mass contrast (or Z contrast) in addition to highresolution imaging. Figure 2-9 illustrates the stronger scattering from heavier elements as well as a sample HAADF-STEM image showing contrast between Si layers and a SiGe alloy. With such high resolutions individual columns of atoms can be observed, even the space between Si atoms in the {110} plane with a separation of 1.35 Å.



Figure 2-9: Schematic showing enhanced scattering of electrons in Ge over Si during HAADF-STEM imaging (left). HAADF-STEM image of a Si/SiGe superlattice demonstrating high resolution and Z-contrast (right).

## CHAPTER 3 IMPROVEMENTS TO YIELD AND DATA ANALYSIS FOR ATOM PROBE TOMOGRAPHY OF SI/SIGE FIN STRUCTURES

#### 3.1 Introduction

Over last decade, atom probe tomography (APT) has been of large interest for the characterization of semiconductor structures to aid in the continued shrinking of devices. This scaling requires the precise understanding of atomic positions in devices for proper characterization. APT has shown to be unparalleled in the characterization of dopants<sup>60,65,108</sup>, interfaces<sup>109</sup>, and film composition<sup>64,110</sup> in 3D transistors with a depth resolution of 0.1 nm and a lateral resolution of 0.3 nm.

In order to produce reconstructions which accurately reflect the geometry of the 3D structures, preparation of samples becomes crucial. Typically, samples are prepared using a standard FIB lift out technique where a cross section is mounted onto a post and sharpened to a final tip radius less than 100 nm.<sup>56</sup> In the case of device analysis, several groups have developed site specific sample preparation methods to ensure the region of interest (ROI) is located at the apex of the tip.<sup>57,111,112</sup> Positioning of the region of interest within the apex is crucial to minimize artifacts arising from nonuniformity. Skewness of the ROI laterally within the apex during milling can lead to distortions such as compression of a fin and expansion of a capping oxide.<sup>113</sup> Even worse, poor positioning can lead to milling away of parts of the ROI, thus reducing the amount of information obtained from the APT run. As sample preparation for this characterization method is laborious, careful consideration of the positioning of structures is vital to the reduction of artefacts.

One of the largest challenges with running 3D architectures such as finFETs or gate all around (GAA) transistors is the simultaneous evaporation of various layers

comprised of semiconducting, dielectric, and metallic materials. Each material has a different evaporation field threshold (F<sub>evap</sub>) which describes at what field strength ionization of surface atoms occurs.<sup>41</sup> Previous work with layered structures demonstrated that when multiple layers evaporate simultaneously but at different rates, the specimen departs from an ideal hemispherical shape.<sup>114,115</sup> Variations in tip curvature in turn vary the magnification of each layer leading to density artifacts in the final reconstruction and can distort morphologies such as interfaces or precipitate shapes. Similar artifacts have been observed in both fins and GAA devices by Grenier et al. who observed severe compression of the Si channel region surrounded by both a gate oxide and metal.<sup>76</sup> This compression was attributed to the lower evaporation field of the Si compared to its surrounding materials.

Several approaches to correct for artifacts in reconstructions have been developed. Corrections to density differences within multiphase reconstructions have been proposed wherein each phase in the data set is selected and reconstructed using each phases evaporation field.<sup>71,116</sup> While this adjusts the distribution of ions within the volume, atomic concentrations are for the most part preserved. Correlative methods using various microscopy techniques have also been reported.<sup>72</sup> Transmission electron microscopy (TEM) of sharpened tips has been used by several groups to provide an accurate representation of the material structure with which to compare APT reconstructions.<sup>73–75</sup> Electron tomography has also been used to produce 3D images of interfaces for identification of distortions in fins GAA device data sets.<sup>70,76</sup> Work has also been done to correct for these interfacial distortions in which interfaces known to be straight can be flattened improving steepness measurements.<sup>77</sup> These results served as

the basis for the Landmark Reconstruction method used in this work which will be described in more detail later in this paper.

This work seeks to understand and correct distortions observed in APT data sets of multilayer Si/SiGe fins. Such structures have applications in the fabrication of stacked nanowire transistors<sup>117,118</sup> which are being investigated as potential device structures for the 5 and 3 nm nodes<sup>24</sup> as they provide superior electrostatic control over the channel region.<sup>26</sup> More importantly, as device scaling continues, the contact resistance at the source and drain becomes incredibly important necessitating a precise understanding of dopant distributions in these locations.<sup>119</sup> Previous work has shown that when running a pure SiGe fin, compression of the fin is observed in the reconstruction due to departures from the hemispherical tip shape.<sup>120</sup> This was rectified utilizing a specialized density correction method in conjunction with field evaporation simulations. However, no studies have explored the distortions present in 3D multilayer heterostructures or their effect on doping distributions. We present a method which combines density corrections with a newly developed interface flattening tool which accurately removes distortions allowing for improved analysis of these multilayer materials systems.

#### **3.2 Experimental Details**

The multilayer fin structure used in this study are presented in Figure 3-1. The first structure consists of four alternating layers of 15 nm thick Si and Si<sub>0.7</sub>Ge<sub>0.3</sub> which were deposited on 300 mm (100) Si wafers. These wafers were then patterned and etched to form 50 nm wide and 120 nm tall fins with a 360 nm pitch running in the <110> direction. A wide pitch was chosen to minimize the presence of voids formed during the capping process. Carbon was deposited over the fins to act as a protective

layer during cross sectional lift out for transmission electron microscopy (TEM) analysis. TEM specimens were prepared via an in situ lift out technique using a FEI Helios Nanolab 600 focused ion beam (FIB). A modified method was used with wedge premilling to ensure samples were thin enough for accurate analysis.<sup>107</sup> High resolution TEM images were captured using a JEOL2010 operating at 200 kV. Figure 3-1a shows a TEM cross section of the final structure where darker layers correspond to SiGe and brighter layers to Si. A schematic of the fin showing layer dimensions is shown in Figure 3-1b.



Figure 3-1: TEM cross section and schematic representation of a Si/SiGe multilayer fin.
Atom probe specimens were fabricated using a standard lift out technique in a
dual SEM/FIB (FEI Helios NanoLab 600). Prior to this preparation, a 350 nm thick layer
of amorphous Si (α-Si) was deposited using STS 310PC plasma enhanced chemical
vapor deposition system at 300 °C. This layer not only acts as a protective layer during
sample prep to prevent ion induced damage, but also has an evaporation field close to

that of crystalline Si allowing for more uniform evaporation of the Si portions of the fin. After lift-out is complete, samples are prepared by mounting sections to vertical Si posts on a microtip coupon. Annular milling was then performed to form needle shaped structures using a TESCAN LYRA FIB/SEM instrument. The initial mill steps were done using a beam energy of 30kV to remove a majority of the material at a reasonable rate. A beam energy of 5 kV was then used for the final milling steps to sharpen the APT tips within a target tip radius of 20 nm. This lower beam energy limits the amount of ion beam induced damage in the specimen while also allowing for controlled placement of the fins at the apex of the tip. As ROI placement within the sample crucial, an electron back-scatter detector was utilized to visualize the fin in the tip (Figure 3-2a and Figure 3-2b).





Prepared APT samples were analyzed using a LEAP 5000 instrument from

CAMECA utilizing a 355 nm wavelength laser. Specimens were cooled to 50 K and a

base pressure of 1e-10 torr was targeted. The detection rate (number of ions evaporated per laser pulse) was set to 0.05% to aid in preventing fracture events and improving yield (more discussed in Chapter 3-5). Laser parameters were set to an energy of 30 pJ and pulse rate of 200 kHz.

Data sets from the LEAP system were reconstructed and analyzed using the IVAS 3.8.0 software package from CAMECA. Reconstructions were completed using initial tip radii and shank angles obtained from FIB images after annular milling. The image compression factor, x projection, and y projection coordinates were adjusted until the layered interfaces (horizontal) were flat and layer spacing closely matched that observed in TEM cross sections. The new Landmark Reconstruction tool was utilized to correct interfaces known to be flat from TEM. It operates by calculating best fit planes to a collection of user-defined concentration or density isosurfaces. Each isosurface is then sampled with an array of points to map out the local displacement with respect to its best fit plane. Using these displacement maps, the distance between each ion and its bounding surfaces (at least one, at most two) is calculated. The distances are defined by a line normal to the best fit plane of each of its bounding surfaces, by interpolating between the array of sampling points on the surface. For each bounding surface of an ion, new target positions are calculated that would maintain the ion's relative distance if those surfaces were moved to have zero displacement distance from the target fit plane. For an ion with two bounding surfaces the two target positions may be different, possibly requiring it to move in opposite directions to satisfy each. For these ions, the final new position is a linearly weighted combination. The weights for each target

position are determined by the ions original distance from its corresponding bounding surface (nearer surface given more weight).

#### 3.3 Distortion Observation in Undoped Si/SiGe Fins

Vertically stacked nanowires are of great interest for continued transistor scaling due to there superior electrical properties over finFETs and planar devices. Si/SiGe superlattice fins are a building block for producing such devices where SiGe is etched away to form horizontal nanowires.<sup>121</sup> A detailed understanding of the distributions of atoms within these structures is crucial for device development ensuring specific metrics are achieved. Distortions within SiGe heterostructures has been previously observed where a deviation in tip shape occurs when running a SiGe fin surrounded by an oxide<sup>120</sup>. This departure from an ideal tip shape leads to a reduction in magnification of the region and subsequently, a reduction of the fin width. A similar result was observed when evaporating a Si/SiGe superlattice fin. Figure 3-3a shows a reconstruction for this structure where a reduction in the widths of all SiGe layers is clearly observed. However, as the run progresses and the Si layers begin to evaporate, the reconstructed layer width closely returns to that observed in TEM cross section. This reduction and subsequent correction to the layer widths produces an undulation in the reconstructed sidewalls.

A plot of the detector event histogram (measuring the number of ion hits per area on the detector) during evaporation from a SiGe layer to a Si layer is shown in Figure 3-3b with the corresponding elemental identification shown via the detector ion map in Figure 3-3c. The mass spectrum used to determine the elements on the detector ion map is shown in Figure 3-3d. Larger counts of Ge from the SiGe layers are shown at the center of the detector compared to the surrounding oxide, Si layer, and  $\alpha$ -Si cap.

This points to enhanced evaporation of the SiGe layer which would cause a deviation from the desired hemispherical tip shape. However, the similarity in evaporation of the Si layer and the  $\alpha$ -Si capping layer shown in the detector histogram indicates both have a very similar evaporation field. Thus, as the Si layer begins to evaporate, the tip regains a more hemispherical shape and in turn more accurately reflects the actual fin dimensions. As the Si layers do not fully return to the width observed in TEM, it is expected that the tip does not fully recover a hemispherical shape before evaporating into the next SiGe layer.



Figure 3-3: Reconstruction of the multilayer Si/SiGe fin demonstrating compression of the SiGe layers when compared to TEM cross sections(a). (b) Detector event histogram and (c) detector ion map showing enhanced evaporation of Ge (Ge atoms in red and O atoms in blue). (d) Labeled mass spectrum from the APT data set.

# Distortion correction using the Landmark Reconstruction method: To

correct for this undulation in fin sidewall, the Landmark Reconstruction tool in IVAS

3.8.0, utilizing planar registration (flattening), was used. This registration algorithm works with layers at any orientation, but assumes the interfaces span the entire analysis volume (i.e. do not terminate within the volume). Additionally, multiple layers can be flattened at once so long as the interfaces are all nominally the same direction. For this data set, the sidewalls of the S/SiGe fin will be corrected to be flat which necessitates the selection of a subvolume such that the sidewalls run the length of the data set. To satisfy the above criterion, the ROI was reduced to omit the oxide at the top of the fin and in the trench at the base (Figure 3-4a). The interfaces for correction were created via isoconcentration surfaces (isosurfaces) defined at 0.7 ionic% of C + O + O<sub>2</sub> + SiC + CN. To reduce noise artifacts in the surfaces, grid parameters were set at 1 nm<sup>3</sup> voxels and 6 x 6 x 6 nm delocalization. Isosurfaces selected for planar registration are shown in Figure 3-4b (overlaid in Figure 3-4a) and represent those produced at the oxide/fin interface. These layers were then flattened and forced to be parallel as described in Section 3.2.



Figure 3-4: Schematic showing the application the Landmark Reconstruction process to a distorted Si/SiGe fin. (a) Distorted APT data set with a 0.7 ionic% C + O + O<sub>2</sub> + SiC + CN isosurface defining the fin sidewalls. (b) The isolated isosurfaces used for the planar registration process. (c) Isosurfaces with the bestfit planes overlayed (shown in red). The color scale on the isosurface denoted distance from the best fit plane with red being the farthest and blue

being the closest. (d) Sideview for one of the isosurfaces and associated best fit planes showing high and low points.

APT reconstruction of the Si/SiGe fin after planar registration removes undulation from the sidewalls and accurately corrects fin dimensions (Figure 3-5). Both the Si and SiGe layer thicknesses closely match those measured in TEM cross section. A 1D concentration profile trough one of the SiGe layers is shown in Figure 3-5b demonstrating that a Ge fraction of 29% is measured throughout a majority of the layer. This displays good agreement with the deposition parameters for epitaxial growth. Small variations in the Ge fraction are evident near the sidewalls of the fin with a max increase of ~2% on the right side of the fin. These variations were observed in all four SiGe layers are believed to be caused by the planar registration process as a 0.5% variation within the layer was measured in the distorted data set. Additionally, high angle annular dark field scanning transmission electron microscopy images (sensitive to z) show no variation in brightness across the SiGe layers indicating a homogeneous concentration (Figure 3-5c). The effect of correcting fin width on layer thickness and interfacial abruptness was also explored. A concentration profile through the entire reconstructed fin before and after the distortion correction is shown in Figure 3-5d. Surprisingly, a byproduct of the correction process was a change in decay length (distance required for an order of magnitude change in concentration) of the Si/SiGe interfaces. An average measured decay length of 4.8 nm/dec for the distorted data set was improved to 2.1 nm/dec through the planar registration process. This result is apparent in the concentration profile with an increased magnitude in slope moving from layer to layer. The reduction in decay length could most likely be attributed to the reduction in lateral curvature of the Si/SiGe interface from stretching the SiGe layer.

Further investigation of the Landmark corrected data set shows some surface roughness is still present on the sidewalls of the fin, observable at the outer layer of the native oxide (Figure 3-5a). These small undulations could be due to the initial roughness in the isosurfaces used to produce the corrected data set. Figure 3-4d illustrates this roughness with small variations in distance from the best fit plane at the high and low points (denoted at red and orange areas). However, carful inspection of the fin sidewalls in Figure 3-5d does indicates some variation in fin width near the Si/SiGe interfaces most likely caused by differences in the etch rates between S and SiGe during fin formation. Based on this observation, it is believed the correction process is removing large-scale local magnification while maintaining roughness information. Additionally, flaring of the fin toward the base is not taken into account using the registration process as the selected isosurfaces are forced to be parallel. In this instance, some error could be introduced into the reconstruction. Selecting ROIs with parallel sidewalls could alleviate this issue. After the correction process, it also becomes important to consider effects on regions outside of the fin structure, i.e. the capping layer. In order to produce the desired structure, ions in the capping layer were moved outward from the reconstruction center transferring the severe sidewall undulation to the reconstruction boundary. It is important to note the reconstruction boundary is not the edge of the specimen tip as atom probe does not capture data from the entire tip radius. While the tip geometry may not be completely free from undulations, the distortion introduced to the reconstruction edge is beyond what would be expected as the SiGe compression has been shown to be caused by departures from a hemispherical tip shape. In this study, the  $\alpha$ -Si acts purely as a protective layer

during sample preparation and can be considered sacrificial. Thus, it becomes important to understand how flattening interfaces can affect adjacent layers when working with more complex heterostructures. Properly identifying and selecting interfaces which are known to be flat (and potentially parallel) is crucial to accurately correct data sets and prevent introducing new distortions.



Figure 3-5: Reconstruction of Si/SiGe fin after application of the Landmark Reconstruction method (a). (b) 1D Ge concentration profile horizontally through a SiGe layer (black dotted line). (c) HAADF STEM imaged of the Si/SiGe fin structure where bright regions correspond to SiGe and darker regions to Si (black on sides of fin denote oxide and protective carbon). (d) Vertical 1D Ge concentration profiles through both the distorted and corrected data sets showing the abruptness of the Si/SiGe interfaces.

# 3.5 Improvements to Sample Yield for Si/SiGe fin APT Tips

Although laser pulsing reduces the required voltage for evaporation in

semiconducting samples, tip failure due to high stresses is still common, especially

when running 3D devices with dielectric layers. This presents a large problem when attempting to make APT a more routine method for characterizing devices and an industry level where turnaround time for analytics can lead to holds on production costing company's large sums of money. This section seeks to provide methods for improving the yield probability for running 3D heterostructure fins which represent an actual device structure used for next generation transistors.

## 3.5.1 Yield Improvement with Upgraded APT Equipment

Over the past several years, CAMECA has developed improvements to their LEAP systems allowing for better control of run parameters and a more self-sufficient feedback program. The LEAP 3000 toolset was upgraded to the LEAPP 4000 with the addition of a 355 nm UV laser replacing the old 515 nm green laser. Improved detector efficiencies (number of ions detected/number of ions evaporated) up to 80% were then implemented in the LEAP 5000 system (up from 40-50%) as well as improvements to the LEAP control system. These control system improvements allowed for automatic laser scans and focusing steps ensuring optimal parameters during the entirety of runs.

Moving from the LEAP 300 system to the LEAP 4000 allowed for an increase in sample yield from 0.08 to 0.19. Improvements to the yield were attributed to the change from a green laser to a UV laser. When a laser is pulsed onto a semiconductor, carriers are injected into the conduction band. Subsequent recombination of these carriers back into the valence band generates heat<sup>122</sup> and in combination with the applied electric field cause field evaporation. However, in the case of Si/SiGe heterostructures, the green laser light is more strongly absorbed by the SiGe layers than the Si leading to preferential heating. Temperature differences between these layers have previously been shown to cause distortions with reconstructions due to preferential evaporation.<sup>123</sup>



Figure 3-6: Improvements to APT sample yield via the use of updated equipment utilizing UV lasers and improved software.

Further improvements to sample yield were made by transitioning to CAMECA's latest instrument, the LEAP 5000. Using the system's improved laser control algorithms, sample failure due voltage spikes at interfaces were lessened. This included transitions from the  $\alpha$ -Si capping layer to the single crystal fins as well as between Si/SiGe layers within the fin. A small increase in yield was observed on this instrument up to 0.24. Improvements to data sets were also made with an increase in the detector efficiency allowing for increased sensitivity. Figure 3-6 shows the improvements in yield based on the instrument used. The parameters and yield calculations can be found in Appendix A.

#### 3.5.2 Improvements to Yield Through Detection Rate Reductions

While transitions to newer instruments made slight improvements to sample yield, it became clear that further improvements would require adjustments to other run parameters. A majority of sample failure was attributed to high stress build up within the tip when high voltages were applied. Fracture points varied from the cap/fin interface, the fin/substrate interfaces, to even catastrophic failures in which a majority of the tip was melted from plasma formation. As such, it became necessary to find a way to reduce the applied stress on the tip to reduce these fracture events.

Reductions to the applied stress on APT tips was achieved by reducing the set detection rate (DR). This variable dictates at what rate ions are detected based on the number of laser pulses and is typically expressed as a percentage.

$$DR = \frac{number \ of \ detected \ ions}{single \ laser \ pulse} \times 100 \tag{3-3}$$

Reducing this rate reduced the applied voltage and in turn reduced the strength of the field applied to the sample apex. Figure 3-7 demonstrates how lowering the DR reduced the voltage profile throughout the run. It should be noted that while reducing the DR lowers the applied voltage, it also lengthens the amount of time necessary to complete a run. Longer runs will still experience high voltages (>6000 V) near their completion, but at that time, the tip radius will have increased improving the specimen's resistance to fracture.



Figure 3-7: Schematic showing the reduction in applied voltage over time to the APT specimen with a reduction in the set detection rate.

Reducing the detection rate from 0.5%, which is standard for a vast majority of runs, to 0.1% saw a large increase in yield from 0.24 to 0.69. Continued improvements were made to yield by decreasing the DR further to 0.05%. Providing even more gentile acquisition conditions increased the yield to 0.86, a record yield level for this work. While this low DR showed vast improvements to yield, it was in conjunction with several drawbacks. Low DRs translate to long run times which may be undesirable in an industry setting. A data set like the one shown in Figure 3-2a containing over 30 million ions required a run time of over 27 hours. An additional drawback is the increase in background levels associated with low DRs. These larger background levels are believed to be caused by an increase in available time between event detection where uncontrolled field evaporation events can occur. Increase in the sample yield with a reduction in the DR is shown in Figure 3-8 with the associated run parameters and yield calculations shown in Appendix A.

During several runs on the LEAP 5000 system, small non-catastrophic fracture events know as ruptures occurred which could be observed as small spikes in the applied voltage. In most cases, small portions of the capping layer would fracture off the tip apex exposing the fin and creating a rough surface. As this leads to a discontinuity in the reconstructed data set, only ions collected after the rupture event could be used to create the 3D reconstruction. Data from these reconstructions was still usable once the tip regained its hemispherical shape. These types of runs were still considered successful and counted towards the overall yield.



Figure 3-8: Change in the probability of APT sample yield by adjusting the detector efficiency on LEAP 5000 system.

## 3.6 Conclusions

Previous observations of compression in SiGe fins capped in high evaporation field materials like SiO<sub>2</sub> have been demonstrated to occur in multilayer Si/SiGe fins. We have shown that such distortions can be corrected using a new method called Landmark Reconstruction developed by CAMECA in the IVAS 3.8.0 reconstruction software. This method identifies interfaces which are known to be flat and separated by a particular distance and adjusts the distribution of ions within the APT reconstruction to achieve those dimensions. When applied to the distorted Si/SiGe fin structures, undulations present in the sidewalls were corrected and dimensions in the reconstruction closely matched those in the TEM cross sections. Concentration profiles through the SiGe layers showed comparable Ge fractions to those used in the epitaxial growth process indicating good accuracy in the correction method. This work highlights not only methods to correct spatial distortions in multilayer heterostructures, but some of the limitations in dopant analysis. It should also be noted that the Landmark Reconstruction method can be applied a variety of material systems in which interfaces known to be flat can be corrected.

## CHAPTER 4 ARTIFACTS IN APT SI/SIGE FIN DOPING PROFILES

#### 4.1 Introduction

With continued transistor scaling to 3D architectures, contact resistance between metals and semiconductors in the source and drain has become a limiting factor to overall device performance as is the maximum contributor to the overall device series resistance. Reducing contact resistance in these areas has become a crucial point of study due large in part to an increase in resistance with decreasing contact width.<sup>31</sup> A common method to overcome these large resistances is to increase doping concentrations within the source and drain such that they are degenerately doped. In ntype contacts, this allows for electrons to tunnel through the Schottky barrier allowing for ohmic contact behavior and a reduction contact resistance.<sup>32</sup> As such, a clear understanding of dopant distributions is critical to develop low resistance contacts and devices with improved performance. Secondary ion mass spectrometry (SIMS) has previously been shown to provide doping profiles within 3D devices,<sup>34</sup> however, these profiles cannot probe diffusion the x or y directions. Scanning spreading resistance microscopy (SSRM) does provide 3D doping information, but only on the active fraction,<sup>2</sup> neglecting inactive dopants which are typically in higher concentration for highly doped layers.

APT has shown to be adept and analyzing 3D doping concentrations, but as described in Chapter 3 of this work, evaporation of heterostructures can cause large distortions in the reconstructed volume. The same Landmark Reconstruction method, as well as an atomic density correction, is applied to doped Si/SiGe multilayer fins to produce more accurate profiles of ion implanted arsenic. This chapter also aims to

explore some limitations in dopant profiling using corrected APT data sets and what future the method has in its current state.

#### **4.2 Experimental Details**

Doped fin structures were fabricated using similar techniques those described in Chapter 3.2, but with only two alternating layers of 7 nm thick Si<sub>0.75</sub>Ge<sub>0.25</sub>, a SiN capping layer, and a pitch of 200 nm. Longer etch times were used to produce a 100 nm tall fin (excluding the nitride) with the alternating layers at the apex shown in Figure 4-1a. The fin structures were then implanted with As using an ion energy of 1 keV and an implant angle of 45 degrees to ensure reasonable dose retention in the sidewall.<sup>58</sup> A dose of 1.0 x 10<sup>15</sup> cm<sup>-2</sup> was targeted on either side of the fin for a total implant dose of 2.0 x 10<sup>15</sup> cm<sup>-2</sup> within the fin. Stopping and Range of Ions in Matter (SRIM) and Florida object-oriented process simulator (FLOOPS) were used to simulate as implanted profiles for comparison to data collected from APT. Figure 4-1b shows a schematic of the implant parameters. Capping the fin in SiN allows for a better study of the implant profiles within the apex layers as it prevents a double dose at the fin top. Finally, anneals at 900 °C were performed using a rapid thermal anneal system.

APT specimens where fabricated using the same FIB lift out and sharpening methods described in Chapter 3-2. Prepared APT samples were analyzed using a LEAP 5000 instrument from CAMECA utilizing a 355 nm wavelength laser. Specimens were cooled to 50 K and a base pressure of 1e-10 torr was targeted. The detection rate (number of ions evaporated per laser pulse) was set to 0.05% to aid in preventing fracture events and improving yield (more discussed in Chapter 3-5). Laser parameters were set to an energy of 30 pJ and pulse rate of 200 kHz.



Figure 4-1: TEM cross section of doped Si/SiGe fin and schematic showing implant conditions. APT tips were fabricated similar to those in Chapter 3.

### 4.3 Distortion Correction of Doped Si/SiGe Fins

Similar to results observed with the undoped multilayer fins, severe compression in the SiGe layers is present in the doped structures (Figure 4-2). Additionally, an enhancement in magnification of the native oxide layer is also present displaying an oxide thicker than the SiGe layer. It is interesting to note that magnitude of the compression in this reconstruction is larger than that of the previous data set. The difference in compression of fins with varying thicknesses was previously explored using field evaporation simulations.<sup>120</sup> It was concluded that the ratio of high evaporation field material (capping layer) to low evaporation material (fin) in the tip determined the amount of compression with thicker capping layers and thinner fins leading to more compression. Our data shows good agreement with this result in which the compression factors (ratio of actual fin width to compressed fin width) for the undoped and doped fins were 3.1 and 5.4 respectively.



Figure 4-2: Reconstruction of an As implanted Si/SiGe fin annealed for 5 seconds at 900 °C. Compression of the SiGe layers is clearly observed. Silicon atoms are shown in gray, germanium atoms are shown in red, and oxygen atoms are shown in blue.

# **4.3.1 Reconstruction Correction using Atomic Density Calculations**

While the SiGe layers have been compressed by a factor of 5.4, all the evaporated ions from the layer are still detected (with a detector efficiency of 80%) and present in the reconstruction. As a result, the atomic density of all species present in the SiGe layers must have increased past their theoretical values. Therefore, volume corrections can be applied to the reconstruction or obtained concertation profiles to more accurately reflect dimensions observed in TEM cross sections similar to work done by Melkonyan et al.<sup>120</sup> This section seeks to provide a comparison of density corrected profiles with profiles obtained using the Landmark Reconstruction method in Chapter 4.4.2.



Figure 4-3: Germanium atomic density calculations across the center of the top SiGe layer. Bin size was set to 1 nm in the x-direction and 5 nm in the y and z-directions. A theoretical Ge atomic density was calculated for a Si<sub>0.75</sub>Ge<sub>0.25</sub> film and is displayed on the atomic density plot as a dotted line. Ge atoms are shown in red and oxygen atoms are shown in blue.

Atomic density calculations were carried out only for Ge atoms in the top SiGe layer of the fin. A rectangular reduced ROI was created within the original reconstruction with an x-dimension of 36 nm and y and z-dimensions of 5 nm. This ROI was then placed orthogonal to the fin sidewalls and centered on the SiGe layer (Figure 4-3). Data from the reduced ROI file was uploaded into MATLAB using code shown in Appendix B. This code retrieved the X, Y, and Z-coordinates of every ion in the ROI as well as their associated mass to charge ratios and placed them in a 4 by x vector (ROI vector) with x being the total number of ions in the volume. Using mass to charge ranges obtained from the data set's mass spectrum, all ions besides Ge can be filtered out of the ROI vector. A histogram with 1 nm wide bins in the x-direction was then produced counting the total number Ge atoms in each bin. Dividing the total number of atoms in each bin by the bin volume returns the Ge atomic density.

Figure 4-3 shows the calculated Ge atomic density with respect to distance in the x-direction of the ROI. For comparison, the theoretical Ge atomic density for a  $Si_{0.75}Ge_{0.25}$  film was calculated via Equation 4-1: where  $n_A^{Ge}$  is the atomic density of Ge,  $N_{Ge}$  is the number of Ge atoms in a single unit cell of  $Si_{0.75}Ge_{0.25}$ , and  $a_{SiGe}$  is the lattice constant for  $Si_{0.75}Ge_{0.25}$ .

$$n_A^{Ge} = \frac{N_{Ge}}{(a_{SiGe})^3}$$
(4-1)

The lattice constant for a  $Si_{1-x}Ge_x$  alloy was calculated using a model based on the Ge fraction (x) and returns a value in Å.<sup>124</sup>

$$a_{SiGe} = 5.431 + 0.20x + 0.027x^2 \tag{4-2}$$

Assuming two Ge atoms per Si<sub>0.75</sub>Ge<sub>0.25</sub> until cell, a theoretical atomic density of 9.7 atoms/nm<sup>3</sup> was found. As Si and Ge are miscible in one another, it is assumed this density is constant throughout the alloy. The limit was plotted in Figure 4-3 as a dotted line. Atomic densities exceeding 70 atoms/nm<sup>3</sup> were found in the center of the SiGe layer falling to ~12 atoms/nm<sup>3</sup> at the edge of the fin clearly well above the theoretical Ge atomic density. As the middle of the SiGe layer was most likely the center of the concave tip shape, it would stand to reason this section would be the most compressed due to alterations in the ion flight paths.



Figure 4-4: APT Ge concentration profiles before and after an atomic density correction for a Si/SiGe fin annealed for 5 seconds at 900 °C.

To correct for these large atomic density values, each 1 nm wide bin within the layer ROI was scaled in the x-direction such that the atomic density matched the theoretical value. For example, the bin width for the fin center was adjusted from 1 nm to 7.3 nm in order to scale the calculated atomic density to the theoretical atomic density. Width values for Ge detected in the SiO<sub>2</sub> layer were also corrected such that the oxide layer was 2 nm wide (corrected from > 4 nm). This change in width was then applied to the width values in a concentration profile produced in the IVAS software (also using 1 nm wide bins). Figure 4-4 shows Ge concentration profiles before and after applying the atomic density corrections. Corrections to the profile increase the calculate layer width and sharpened the SiGe/SiO<sub>2</sub> interfaces more accurately reflecting the actual fin dimensions. However, this correction method fell short as the fin width calculated by APT data was still narrower than the actual fin width observed in TEM images. Additionally, the method proved inconsistent between reconstructed data sets with only a 20 second anneal sample approaching the correct layer width (Figure 4-5).

Finally, this correction method could only be applied to concentration profiles calculated from small APT volumes and not applied to the actual 3D reconstruction. This limits the methods applications and increases the turnaround time for producing corrected profiles.



Figure 4-5: APT Ge concentration profiles after an atomic density correction through a single SiGe from samples annealed for 2, 5, 10, 20, and 30 sec at 900 °C. Variations in the corrected fin width are present between some of the data sets.

It becomes clear that simply correcting the atomic densities within small volumes will not provide sufficient corrections to the heavily distorted APT data sets. Instead, a method correcting the actual reconstruction must be used to provide ease of producing meaningful data in a reasonable time frame.

# Effect of atomic density corrections on implanted profiles: The effect of

correcting APT data sets through atomic density adjustments on doping profiles was

also explored. Figure 4-6 shows implanted arsenic profiles after annealing for 2, 5, 10,

20, and 30 seconds at 900 °C in an argon ambient. Atomic concentration values (in

#/cm<sup>3</sup>) were calculated from the extracted atomic percentages by multiplying them by

the known number density of atoms in a  $Si_{1-x}Ge_x$  (where x = 0.25) lattice.<sup>124</sup> Much like the Ge profiles shown in Figure 4-x, these profiles do not present an accurate reflection of the correct fin width observed in TEM. While the width of these profiles is incorrect, several other issues remain when attempting to use these profiles to investigate As diffusion.





Variations in the peak concentrations for varying anneal times is present on both the left and right side of the fins. While some variation is expected as diffusion occurs, lower peak concentrations for longer anneal times is not observed. Instead, the peak concentration for the 2 second anneal is below that of the 30 second anneal questioning the validity of the profiles. Additionally, all of the doping profiles exhibit a concentration plateau in the center of the fin between  $1 \times 10^{19}$  to  $2 \times 10^{19}$  cm<sup>-3</sup> indicating a diffusivity well above any presented in the literature.<sup>125–127</sup> The formation of these profile shapes is believed to be caused by the stretching of regions with high atomic densities. When compressed, the distorted SiGe sidewalls are much closer together intermixing their implanted As profiles. Merely stretching the profiles can leave data points meant for the fin edge in the middle of the profile giving rise to the incorrect doping profiles. A schematic illustrating this stretching issue is shown in Figure 4-7. This error wasn't visible in the Ge profiles as the layers are homogeneous, so variations in the lateral position of a data point wouldn't affect the overall profile. Much like the conclusions made from Section 4.3.1, improved analysis of doping profiles will require corrections to the entire data set.



Figure 4-7: Schematic illustrating the formation of distorted dopant profiles using the atomic density correction.

## 4.3.2 Application of Landmark Reconstruction to Distorted Doped Fins

Similar to the undoped fins structures, the Landmark Reconstruction process was applied to the doped fins in hopes of producing similar results. Defining adequate interfaces for the Landmark Reconstruction method proved difficult with large levels of compression in the SiGe layers. A density correction was applied to the data set to reduce the level of compression and allow for better surface identification. This density correction method differs from the one described in Chapter 3.4.2 as it is built into the IVAS software package. Groups of atoms are created iteratively with density variations being corrected by small volume adjustments. Unfortunately, this method does not provide corrections to a similar magnitude as our density correction method. Figure 4-8b shows the reconstruction after density correction indicating a widening in the SiGe layers as well as a reduction in the native oxide thickness. However, this correction alone is not enough to completely remove all distortions from the reconstruction.

Using the same procedure outlined in Chapter 3, isosurfaces of 1 ionic% O, SiO, and SiO<sub>2</sub> were produced to define the sidewalls of the fin. The surfaces were then flattened using best fit planes and forced to be parallel with a separation of 30nm. The resultant reconstruction is shown in Figure 4-8c demonstrating good agreement with dimensions calculated in TEM cross section. Some small undulations still remained at the interfaces between the Si and SiGe layers, however, the Landmark Reconstruction method cannot be applied in this instance as it would require truncating the sidewalls and capping layer of the data set to ensure Ge isosurfaces defining the interfaces do not terminate within the volume.

To validate the accuracy of the reconstruction, atomic density profiles for Ge within the SiGe layers were produced at each step in the correction process (Figure 4-8d). In the initial distorted reconstruction, an abnormally large atomic density is observed at the center of the layer exceeding the calculated atomic density of by over an order of magnitude. Performing a density correction drastically improved the density profile, but deviations at the fin center were still present. Finally, application of the Landmark Reconstruction produced an atomic density profile that closely matches the expected value while also improving the layer thickness. This result demonstrates that not only are fin dimensions being corrected, but the underlying distribution if atoms as well.



Figure 4-8: Reconstruction of the doped Si/SiGe fin before any corrections showing large compression in the SiGe layers (a). (b) Data set after a density correction. (c) Reconstruction after performing the Landmark Reconstruction method demonstrating flat sidewalls and more accurate dimensions. (d) Atomic concentration profiles for the original reconstruction (black), density corrected reconstruction (red), and Landmark Reconstruction data set (blue). Inset shows a magnified profile for the landmark data set. (e) Arsenic concentration profiles for the three reconstruction (a-c) through the lower SiGe layer (shown in black outlined box).

The effect of removing spatial artifacts on doping profiles was carried out using

1D concentration profiles taken orthogonal to the fin direction. Figure 4-8e shows

arsenic concentration profiles across the lower SiGe layer (denoted by dashed box) for

each step in the correction process. Atomic concentration values were calculated from

atomic percentages using the same method described in Section 4.3.1.1. Peak

concentrations of ~1.6 x 10<sup>20</sup> cm<sup>-3</sup> are observed for each side of the fin in the original

reconstruction with a minimum concentration of  $\sim 2 \times 10^{18}$  cm<sup>-3</sup> found at the center.

Subsequent corrections to the data set increase the separation of peak concentration

positions as well as lower the arsenic concentration at the center of the fin. It should be noted that as the profiles widen, the peak concentration values near the sidewalls do not decrease indicating that the relative atomic densities for each atomic species are scaling similarly in these regions.



Figure 4-9: Comparison of the 1D As concentration profile after a 5 second anneal at 900 °C along one sidewall and FLOOPS simulation.

As previously described, several studies concerning doping distributions in 3D transistor structures relied on SIMS measurements to validate profiles obtained from APT data sets. Unfortunately, only profiles in the z-direction (orthogonal to the Si/SiGe interfaces) are obtainable using this method requiring validation of dopant distributions to be carried out via simulations. Florida object-oriented process simulator (FLOOPS) software was used to model the diffusion of an as implanted profile using the same annealing conditions as the APT specimen. The diffusion model implemented in FLOOPS was the dopant-defect pair model<sup>128</sup> where atomic As migrates through the lattice with the presence and interaction of vacancies. Starting point defects were input from trim outputs, and bulk I-V recombination and diffusion were accounted for using

literature values.<sup>129</sup> Characterizing active arsenic and vacancy interactions (including nearest neighbor distances, binding energies) were implemented following literature values.<sup>128,129</sup> Figure 4-9 shows the comparison between the APT concentration profile on one side of the fin with the simulated data set from FLOOPS. While there is good agreement between the experimental and simulated data, some discrepancies are visible. Near the fin edge (SiGe/SiO<sub>2</sub> interface), a larger concentration is observed in the simulated data set, but moving into the fin, the reverse is seen. Such error could be in part to introductions of volumetric adjustments to correct for distortions arising from tip shape deviations.

The overlapping of ions during evaporation between adjacent high and low evaporation field materials have previously been reported showing intermixing of the two regions in reconstructions.<sup>130</sup> In a recent study of SiGe fins, intermixing of oxygen from a deposited oxide layer into the fin was detected leading to a loss in spatial resolution which was unable to be restored.<sup>120</sup> Figure 4-10a clearly shows this trajectory overlap is occurring in the doped APT data set as oxygen is present within SiGe layers. Oxygen concentrations of ~6% are observed at the SiGe layer edges which drops to ~0.8% at the fin center (Figure 4-10b). Because a large portion of the arsenic dose is contained in the oxide and in the fin close to the SiGe/SiO<sub>2</sub> interface, ion trajectory overlap of arsenic ions is also expected. Thus, the center of the fin could see an enrichment of arsenic with the edge seeing a depletion. In addition, the severe compression of SiGe layer leads to an overlap of arsenic atoms at the fin center in the distorted data set overestimating the arsenic concentration. After applying the Landmark Reconstruction method, oxygen is still present within the center of the SiGe layer but

reduced to ~0.6% (Figure 4-10c). It would stand to reason then that arsenic introduced to the center of fin through ion trajectory overlap would still remain after the correction. As described in section 2, moving ions within the reconstruction using two bounding surfaces uses a linear weighted combination of the ions target positions. Ions at the center would most likely see less movement than those near the edge as the weight from each bounding surface would be similar in magnitude. These results explain the discrepancy between the extracted APT doping profile and the simulated profile with larger concentrations of arsenic present in the reconstructed data set center.

It is important to note such error could also be due to an unclear understanding of the diffusion kinetics associated with the lateral indiffusion of arsenic into a strained SiGe layer in close proximity to Si interfaces. This not only highlights the need for further investigation of doping in such heterostructures, but also the limitations in characterizing doping profiles using APT.



Figure 4-10: Oxygen (O<sup>+)</sup> atoms (blue spheres) are present within the distorted SiGe layers whose bounds are denoted by red 10% Ge isosurfaces (a). 1D concentration profiles through the center of the lower SiGe layer before (b) and after (c) the Landmark Reconstruction quantify the oxygen concentrations.

# 4.4 Viability of APT for Dopant Diffusion Analysis in Heterostructure Fins

To investigate the viability of the APT to investigate dopant diffusion in Si/SiGe

fin systems, the Landmark Reconstruction method was applied to an as implanted

sample. Figure 4-11a shows a distorted APT reconstruction containing both Si and SiGe layers. Applying a density correction and the Landmark Reconstruction yields a data set more closely resembling the structure observed in TEM (Figure 4-11b and 4-11c). Some discontinuities are present in the upper SiGe layer and lower Si layer which were not adjusted by the Landmark method. It is believed that the isosurfaces utilized in the correction process did not extend fully on both sides of the truncated layers due to the geometry of the reconstruction. As a result, they were not stretched t the same extent as the middle layers.



Figure 4-11: Distorted, density corrected, and Landmark corrected APT reconstruction of a Si/SiGe implanted with 1 keV As at 45° with a dose of 1e15 cm<sup>-2</sup>.

Concentration profiles across the Si layer were extracted using a cylindrical data pipe with a diameter of 3 nm located at the center of the layer. The extracted arsenic profile was then adjusted to correct for a thicker oxide by reducing the distance values in the x-direction such that the measured oxide thickness matched that observed in
TEM. A corrected arsenic profile is shown in Figure 4-12. It should be noted that the arsenic concentrations were calculated in the same fashion as the profiles in Section 4.3.2 using calculated number densities of  $Si_{1-x}Ge_x$  (where x = 0.25). The retained dose within the layer can be determined by calculating the area underneath the concentration curve. Using Riemann sums (as entire curve is concave down and contains increasing and decreasing sections), the dose can be approximated as  $1.0 \times 10^{14}$  cm<sup>-2</sup>. The validity of this profile was tested using a TRIM simulation with implant conditions matching those described in Section 4.3 and the approximated dose. Figure 4-12 shows the simulated data overlaid onto the experimental data demonstrating excellent agreement and accuracy of the corrected APT data set.



Figure 4-12: APT As concentration profile from the center Si layer of a Si/SiGe fin and the associated TRIM simulated profile (no anneal).

# 4.4.1 Dose Retention in Si/SiGe Fin

A total dose of  $1.0 \times 10^{15}$  cm<sup>-2</sup> was targeted for each side of the fin with the top of the fin receiving both implants for a total dose of  $2.0 \times 10^{15}$  cm<sup>-2</sup>. However, dose calculations for the extracted as implanted arsenic profiles from the Landmark corrected

APT data set indicated a retained dose of only  $1.0 \times 10^{14} \text{ cm}^{-2}$  (Section 4.4). This result points to a dose loss of 90% which was very unexpected. Similar dose retention values were observed in SiGe layers annealed for 5 seconds at 900 °C To verify this dose loss is real and not an artefact of the APT reconstruction process, the total dose received by the top of the fin of the 5 second anneal sample was calculated. It should be noted the 5 second anneal reconstruction was used as this region was lost in the as implanted sample due to a small rupture event. Extracting the dopant profile and correcting for an enhanced oxide thickness returned a total retained dose of  $2.2 \times 10^{14} \text{ cm}^{-2}$ , well in line with the 90% dose loss observed in the fin sidewalls. This dose retention value was used in all reconstructed doping profiles to ensure accurate reconstruction of the profile from the distorted data sets.

Dose loss in fins implanted at high angles and low energies has been previously reported. Kambham et al. observed a dose retention of 75% when implanting boron into a Si fin at 45°, a beam energy of 5 keV, and a dose of 8 x 10<sup>14</sup> cm<sup>-2</sup>.<sup>58</sup> Similar experiments were performed Duffy et al. implanting arsenic into bulk Si wafer at 45° with a dose of 1 x 10<sup>15</sup> cm<sup>-2</sup> to mimic implants into a fin.<sup>131</sup> In their work, they varied the implant energy from 3-7 keV investigating its effect on dose retention. No large differences were observed, but their range of energies did not extend low enough to compare to our data sets. At energies as low as 1 keV, the projected range is within 3 nm of the surface meaning surface sputtering can remove large amounts of dopant atoms during the implant. Additionally, backscattering at low energies would become more likely reducing the retained dose. Dose loss of this magnitude was not predicted by TRIM simulations, but it should be noted that a 1D simulation might not accurately

depict the collision and cascading occurring in the 3D structure. To better understand the origin of this large dose loss, 2D Monte Carlo simulations of the fin structure will most likely need to be performed.

The extracted arsenic profile from the corrected lower SiGe layer was found to have an integrated dose of ~ $4.0 \times 10^{13}$  cm<sup>-2</sup> when using a data pipe of similar size to the Si layer (Figure 4-12). This dose was ~60% lower than the previously calculated doses determined to be the actual retained dose. Using 0.40% As isosurfaces, it becomes apparent in Figure 4-13 that the Si layer has retained more of the As dose that either of the SiGe layers. Currently, it remains unclear as to whether this is an artefact of the reconstruction process or is caused by tip shape deviations during evaporation. It should also be noted these isosurfaces suggest the As groups together in small high concentration clusters along the fin wall. Peak concentrations within these regions can reach ~1.1 at%, but in regions of 8 nm<sup>3</sup> or less. Like the variations in dose between the layers, this grouping of dopant atoms could be a result of the distortions introduced to the reconstruction due to preferential evaporation of the SiGe layers. Co-evaporation, as described in Section 4.3.2, could be the main cause of this dopant redistribution.

It should be noted that adjusting the size of the data pipe around the high concentration regions can adjust the peak concentration of As within 1D concentration profile. This is due to an increase in the relative ratio of As ions to substrate ions within the calculating voxel. An adjustment could allow for better matching of the extracted dose to the known dose within the fin. One drawback of narrowing the data pipe and reducing the number of ions used for calculations is an increase in the standard

deviation of the measurement. Using Equation 4-3, the reduction in standard deviation  $\sigma$  with a reduction in counted atoms n<sub>i</sub> and change in concentration c<sub>i</sub> becomes apparent.

$$\sigma = \sqrt{\frac{c_i(1-c_i)}{n_i}} \tag{4-3}$$

Therefore, care needs to be taken as to not reduce the data pipe diameter too far where the error becomes larger than the measured value. Reducing the data pipe to 2 nm in diameter retuned an improved dose of  $\sim$ 7.0 x 10<sup>13</sup> cm<sup>-2</sup>, just 30% below the actual dose.



Figure 4-13: Arsenic distributions identified with 0.40% isosurfaces (right) from the right side of the Si/SiGe fin (TEM on left). Ge atoms are shown in red, oxygen in blue, and arsenic in pink. Large concentrations of arsenic are present in the Si layer than the SiGe layer.

For ease of analysis and to accurately compare this profile to the other, a dose correction was performed on the extracted profile. This involved multiplying all concentration data points by 1.42 to increase the integrated dose to 1.0 x 10<sup>14</sup> cm<sup>-2</sup>. After the dose correction, the extracted APT profile shows decent agreement with the profile produced by TRIM of As into a Si<sub>0.75</sub>Ge<sub>0.25</sub> layer (Figure 4-14). The SiGe layer density was calculated from an interpolation equation.<sup>132</sup> It is currently unclear as to the validity of performing a dose correction on an APT profile. Dose corrections are

performed on SIMS profiles due to sputter rate differences between layers, but only by varying the depth of specific data points, not the concentration values. While there are differences in the evaporation rates between layers in APT, concentration values are still below what they would need to be for an accurate dose calculation. Some merit is given to this adjusted profile as accurate peak concentrations can be found when reducing the data pipe diameter significantly. More work will need to be done to validate the adjustment of doping concentrations for dose corrections.



Figure 4-14: APT As concentration profile after dose correction from the lower SiGe layer of a Si/SiGe fin and the associated TRIM simulated profile (no anneal).

# 4.4.2 Limitations in Accurate Dopant Profiling

One of the primary goals of using APT to investigate dopants in 3D semiconductor devices is not only to investigate dopant distributions, but also investigate how dopants diffuse in complex 3D structures. As industry departs from planar transistors and pure Si finFETs, a clear understanding of dopant diffusion in multilayer heterostructures which act as potential precursors to nanowire transistors becomes increasingly important. In these structures, changes to intrinsic and extrinsic diffusivities can be induced by the introduction of strain through lattice mismatch or even through nm scale high aspect ratio structures. This section seeks to explore the viability of APT to produce accurate doping profiles for various anneal times in the hope of extracting diffusivity values.

Figure 4-15 shows an overlay of the corrected arsenic profile within the Si layer before any heat treatments with two profiles extracted from the Si layer after a 5 second anneal at 900 °C. The profile shown in blue was extracted from the right side of the Si layer while the red profile was extracted from the left side of the Si layer (Figure 4-xc). Both profiles have a dose  $\sim$ 1.2 x 10<sup>14</sup> closely matching that of the as implanted profile discussed in Sections 4.4 and 4.4.1. The profiles were adjusted such that the oxide thickness matched that observed in cross sectional TEM images. It becomes apparent that different magnitudes of diffusion are predicted by either side of the Si layer. The left side of the layer predicts faster diffusion of arsenic while the right side predicts slower diffusion. To understand which side more accurately describes the diffusivity of arsenic through the layer, a diffusivity value was extracted from each profile using FLOOPS.

To extract a diffusivity value, FLOOPS solves a partial differential equation describing the concentration dependent diffusion process using finite element analysis. A set diffusivity is used to produce a diffused profile from an initial as implanted profile using Equation 4-4: where D is the diffusivity, C<sub>As</sub> is the concentration of arsenic in the matrix, and t is the diffusion time.

$$\frac{\partial C_{AS}}{\partial t} = \nabla \left( D \nabla (C_{AS}) \right) \tag{4-4}$$

This process maintains the dose as the profile is diffused. To accurately predict the diffusivity, D is adjusted iteratively such that the error between the simulated profile and the experimental profile is minimized.



Figure 4-15: APT concentration profile for As implanted into the left (red) and right (blue) sides of the Si layer before (black) and after a 5 second anneal at 900°C. More diffusion is clearly observed on the left side of the layer.

Figure 4-16 shows the FOOPS simulated profile overlaid onto the extracted APT profile from the right Si layer sidewall. Very good agreement is observed between the two profiles which produced a diffusivity value of 2 x 10<sup>-17</sup> cm<sup>2</sup>/s. Some deviation from the APT is observed farther into the fin, but these concentration values are near the noise floor for the method. An overlay of the simulated and experimental profiles from the left side of the Si layer is shown in Figure 4-17. A poor fit is observed stemming from the non-uniformity of the APT arsenic profile which increases the error associated with the diffusivity calculation. This poor fit can also be due to the simplicity of the model

which doesn't consider several point defect interactions. However, as expected, the diffusivity was larger with a value of  $2 \times 10^{-16}$  cm<sup>2</sup>/s, an order of magnitude over the right side of the layer. Unfortunately, it becomes difficult to say which side more accurately describes diffusion within the layer.



Figure 4-16: Overlay of the right APT diffused profile (5 seconds at 900 °C) and FLOOPS simulation. A diffusivity of  $2 \times 10^{-17}$  cm<sup>2</sup>/s was calculated.



Figure 4-17: Overlay of the left APT diffused profile (5 seconds at 900 °C) and FLOOPS simulation. A diffusivity of 2 x 10<sup>-16</sup> cm<sup>2</sup>/s was calculated.

Pichler produced a regression curve for calculating the diffusivity of arsenic in bulk Si based on the diffusion temperature.<sup>133</sup> At 900 °C, a diffusivity value of ~7 x  $10^{-17}$  cm<sup>2</sup>/s is calculated. While this value is close to the one predicted for the right side of the fin, this diffusivity does not take strain, interfacial, or transient effects into account. The effect of strain on diffusivity in Si has been found to be negligible, but enhancements have been seen close to SiGe interfaces.<sup>134,135</sup> At 5 seconds, this anneal is well within the transient enhanced diffusion (TED) rage where excess interstitials created during the implant increase the diffusivity of As in Si.<sup>136</sup> Several groups have shown that the enhancement can range from 2-50 times the steady state diffusivity based on the relative As concentration, implanted dose, and anneal temperature.<sup>137–139</sup> Under the assumption of a peak concentration of ~3 x  $10^{20}$  cm<sup>-3</sup> and a 900 °C anneal, an increase in the diffusivity on the order of 4-5x is expected. Thus, assuming TED is occurring

during the 5 second anneal, the left side of the fin showing more diffusion agrees better with expected literature values.



Figure 4-18: Comparison of As doping profiles in the Si and SiGe layers in a Landmark corrected fin annealed for 5 seconds at 900 °C.

Figure 4-18 compares the diffused profiles from the left sidewall of the Si layer and the SiGe layer (Figure 4-9) after a 5 second anneal at 900 °C. More diffusion is apparent in the SiGe layer over the Si layer with As reaching a concentration of 1 x 10<sup>19</sup> cm<sup>-3</sup> ~8.5 nm from the oxide interface in the Si layer and ~13 nm from the oxide interface in the SiGe layer. This result is consistent with differences in As diffusivities calculated in Si and SiGe systems at similar temperatures and times.<sup>125</sup> Enhancement of the As diffusivity in SiGe is attributed to a chemical effect, but is retarded to an extent do to compressive strain due to the lattice mismatch. However, it should be noted that both profiles are more similar than would be expected with this difference in diffusivity values. Work done by Eguchi et al. found that As diffusion in ion implanted SiGe alloys undergoes transient retarded diffusion (TRD), opposite of the TED observed in Si.<sup>127</sup> In SiGe, As diffuses via a combination of interstitial and vacancy mechanisms similar to Si, but with a larger vacancy fraction. Thus, due to the excess of interstitials introduced by the implant, As diffusion is retarded in SiGe while being enhanced in Si. Even with TRD present in SiGe and TED in Si, after 5 seconds, the diffusivity of As in SiGe would still be slightly greater than that in Si pointing to good accuracy of the APT reconstructions and their ability to capture these small differences.

#### 4.5 Discussion

The work outlined in this chapter demonstrates the first time dopant diffusion studies in multilayer Si/SiGe have been performed. Results from APT profiles and correction procedures bring up a tough discussion for the future of analyzing dopants using atom probe. Correcting for errors present in the initial reconstruction can potentially introduce new distortions into data sets making analysis difficult. Currently, we cannot adequately quantify the propagation of error associated with moving atoms via the Landmark Reconstruction. However, improvements in profile shape are still observed indicating that while the process does distort the original data set, the resulting corrected profile more accurately reflect the actual dopant profile shape.

Issues with consistencies in profile shape were also apparent based on data pipe position within the Si and SiGe layers. When a data set returns two plausible profiles as shown in Figure 4-15, which one should be chosen? Even if one produces a more accurate value, what would cause an inaccurate profile to be produced from a corrected data set? It becomes difficult to make these decisions without purposefully omitting data sets and cherry-picking data that agrees with our expectations. Additionally, counting statistics can lead to large errors in concentration calculations. When data points are calculated from only a handful of ions, the associated error could be larger than the

variations between points. As such, this section shows that more work is required before adequate analysis of dopant diffusion can be completed.

## 4.6 Conclusions

With Si/SiGe multilayer structures proving to be candidates for nanowire transistor fabrication, accurate measurements of doping profiles is critical to their development. Distortions from tip shape variations led to a sever compression in SiGe layer widths in these fins making dopant analysis difficult. It was found that these distortions created regions with high atomic densities well above theoretical values. Attempting to correct these densities for reduced ROIs did not produce accurate doping profiles necessitating the correction of the entire reconstructed volume. Applying the Landmark Reconstruction described in Chapter 3 removed undulations in the fin sidewalls and even corrected the large atomic densities observed in the SiGe layers. Arsenic profiles through the Si and SiGe layers were produced showing these samples only retained ~90% of the implanted dose. The dose loss was attributed to the large implant angle and low implant energy. Extracted profiles from the as implanted Si and SiGe layers showed good agreement with TRIM simulations validating the correction procedure. However, a small dose correction to the SiGe layer As profile was required. Diffusion of As in a sample annealed for 5 seconds at 900 °C was apparent in both the corrected Si and SiGe layers demonstrating the viability of APT to perform dopant diffusion analysis on thin films < 10 nm. However, variations in the implanted dose with data pipe width and diffusion lengths bring that result into question when analyzing corrected data sets. It becomes apparent that more work in characterizing diffusion profiles in these nanoscale heterostructures is needed. Producing distortion free data

sets or departing from the hemispherical assumption is most likely the best avenue to take in improving doping characterization studies using APT.

# CHAPTER 5 INVESTIGATION OF GERMANIUM DIFFUSION ALONG OXIDIZING SI/SIO2 INTERFACES

### 5.1 Introduction

In microelectronics, there has been an increasing interest in non-planar 3-D structures to maintain transistor scaling. The development of these devices has been motivated by the need to combat short channel effects stemming from the reduction in gate length<sup>7,140</sup>. Device architectures such as finFETs<sup>141</sup>, omega-gate<sup>142</sup>, and gate all around (GAA) transistors<sup>143</sup> have been shown to suppress these short channel effects. In particular, GAA transistors can provide superior electrostatic control over the channel region<sup>26</sup> and are currently of great interest to continue scaling down to the 5 nm node<sup>24</sup>. As the cross-sectional area of nanowires is small relative to that of a fin, multiple nanowires are generally fabricated in a stack to increase the overall drive current<sup>27,144,145</sup>. With improved scaling, several studies have investigated the potential of these GAA transistors for a variety of applications beyond logic and memory including solar cells<sup>146,147</sup> and sensors<sup>148,149</sup>.

Improvements to charge carrier mobility in devices can be obtained by straining the channel region<sup>12</sup>. This can be achieved in planar devices by depositing Si onto a SiGe buffer layer on a Si substrate. More recently, coherently strained Si-Si<sub>x</sub>Ge<sub>1-x</sub> coreshell nanowires have been fabricated vertically using vapor-liquid-solid growth and chemical vapor deposition techniques<sup>150</sup>. While such work shows the potential for strained nanowires in transistors, issues with integration into current device technologies are present. If a process was developed to strain stacked horizontal nanowires down to 2 nm, improvements to device performance and scaling to smaller technology nodes would be more feasible.

When a SiGe alloy is oxidized, the oxidation potential of Si is sufficiently greater than that of Ge such that Si is preferentially oxidized and Ge is rejected resulting in a pileup of epitaxial, single crystal SiGe at the SiO2/SiGe substrate interface<sup>80,81,151</sup>. During this process, a Ge rich layer is formed which continues to increase in concentration up to a value of 36-64% which is governed by the oxidation temperature<sup>82</sup>. Once this concentration is reached, the Ge rich layer will maintain its thickness and continue to be rejected by the advancing oxide front provided there is Si below it to be oxidized<sup>83</sup> and the temperature is sufficient for the Ge to diffuse into the Si. This process has been widely investigated for use in the fabrication of Ge-oninsulator (GeOI) substrates for CMOS applications<sup>86,87,152–155</sup>.

Whereas previous studies on Ge condensation focused on blanket SiGe layers, no investigations on the oxidation of an interface consisting of adjacent SiGe and Si layers have been performed. Oxidation experiments have been conducted on superlattice fin structures of alternating Si and SiGe layers, offering such an interface, but did not incorporate temperature regimes in which Ge pileup would occur and instead fully oxidized the SiGe layers<sup>117,118</sup>. In this chapter, a truly unexpected observation is reported. During the oxidation of a superlattice Si/SiGe fin, enhanced Ge diffusion along the adjacent Si/SiO<sub>2</sub> interfaces was observed for the first time. Subsequently, the Si layers were effectively encapsulated in SiGe forming strained nanowires with a rounded cross-section and controllable diameters down to 2 nm. We found that a temperature process window exists in which the Si layers are encapsulated by this new interfacial Ge diffusion process without substantial interdiffusion of Ge into the Si nanowires. This process enables the fabrication of nanowires well below current lithographic limits and

offers new design and optimization options for strained nanowire channels in future CMOS devices.

## 5.2 Initial Observation of Ge Lateral Diffusion

Many advancements in the fields of science have stemmed from results obtained by mere happenstance. In 1895, Wilhelm Röntgen observed the fluorescing of a spare barium platinocyanide screen from a cathode ray tube completely covered in cardboard while studying cathode ray fluorescence.<sup>156</sup> This marked the first observations of x-rays which now have numerous applications in a variety of fields. Similarly, in 1928, Alexander Fleming found that a mold produced in staphylococci cultures killed all the bacteria in its immediate surroundings.<sup>157</sup> Further experiments led to the discovery of penicillin which became the world's first antibiotic. While discoveries like this are sometimes made on accident, it is through rigorous scientific experimentation that meaningful results, conclusions, and application can be made. The work presented in this chapter is no different. This section seeks to describe the series of experiments and results which led to the initial observation of lateral Ge diffusion. "One sometimes finds, what one is not looking for." -Alexander Fleming

Experiments to improve the yield for APT runs of Si/SiGe superlattice fins resulted in some puzzling data. As described in Chapter 3, one of the main sources of tip fracture was believed to be due to poor adhesion of the amorphous capping layer to the crystalline fin and substrate. To remedy this, 5 nm of atomic layer deposition (ALD) SiO<sub>2</sub> was deposited onto the fins to promote better adhesion of the 300 nm PECVD deposited  $\alpha$ -Si layer (Figure 5-1d). Faceting of the Si layer sides along the {111} planes was attributed to slight etching from the plasma deposition process. A 650 °C anneal in an argon ambient was then performed for 1 hour in an attempt to improve the film



Figure 5-1: Results motivating the exploration of SiGe oxidation and its effect on Si/SiGe superlattice fin structures. a) APT reconstruction of a Si/SiGe fin with a 5 nm ALD oxide and 300 nm α-Si deposited cap followed by a 650 °C anneal for 1 hour (Ge = red, Si = gray, O = blue). d) Associated TEM cross section of structure. b) Reconstruction showing clusters via 40% Ge isosurfaces. c) Proxigram for surface identified in b) showing radial change in concentration. e) High mag TEM image showing potential evidence of high concentration Ge cluster in oxide. quality and produce regions with short-range order. APT samples were fabricated using the methods outlined in Chapter 2.2.2 and ran on a LEAP 4000 instrument at Oak Ridge National Laboratory. A reconstruction of the APT specimen is shown in Figure 5-1a where the oxide and Si/SiGe layers are clearly visible using a 15% Ge isosurface and a 25% O isosurface. Additionally, small Ge clusters within the ALD deposited oxide were observed. These clusters were only observed in the annealed sample and not in the unannealed sample.

Creating 40% Ge isosurfaces further identified high concentration Ge clusters ranging from 2-5 nm in diameter (Figure 5-1b). A cluster proxigram (proximity histogram) analysis was performed on the largest cluster in which concentration values for specified elements are calculated based on their radial distance from the defined isosurface. Figure 5-1c shows an increase in Ge concentration up to 92 at% at the center of the cluster indicating the cluster is almost pure Ge formed from a 30% Ge layer. Plateauing of the concentration outside of the fin to  $\sim 10$  at % is owed to contributions from the adjacent SiGe layer. To verify these clusters were not an artefact of the APT reconstruction process, HRTEM images were taken of the annealed sample. A small cluster located in the ALD oxide beside the SiGe layer shown in Figure 5-1e demonstrating validity of the APT results. Low contrast between the cluster and the oxide is most likely due to the large differences in their signal contribution to the CCD detector. The oxide runs the full thickness of the cross section while the cluster most likely on contributes a few nm. It becomes apparent that the formation of these clusters is not controlled by a Fickian diffusion process dependent on a concentration gradient.

These results suggest a different diffusion mechanism is responsible in which Ge is segregated from the SiGe and SiO<sub>2</sub> layers.

Research into this segregation phenomenon revealed a process in which the oxidation of a SiGe layer lead to the preferential oxidation of Si and the rejection of Ge back into the layer. This resulted in the formation of an enriched Ge layer with achievable Ge concentrations up to ~67% at 800 °C.<sup>82</sup> Specifics of the process are discussed in Chapter 1.7. The formation of SiGe quantum dots within thermally grown SiO<sub>2</sub> has also been demonstrated utilizing this segregation process at temperatures ranging from 900-950 °C.<sup>158,159</sup> Similar to this type of growth, we hypothesized that during the 1 hour anneal at 650 °C, excess oxygen within the ALD SiO<sub>2</sub> layer reacted with the SiGe layers segregating out some Ge forming the observed high concentration clusters. To support this idea, a 7 hour anneal at 650 °C in dry O<sub>2</sub> was performed on the superlattice Si/SiGe fins. Similar to the results observed in the APT data sets, clusters of SiGe were observed to have spun off from the fin and become imbedded in the thermally grown oxide (Figure 5-2a). A high magnification image of a cluster is shown in Figure 5-2b demonstrating maintained crystallinity even when segregated into the oxide.

With an improved understanding of the oxidation kinetics of SiGe alloys, we looked to apply this process to Si/SiGe fins at higher temperature regimes with the hopes of concentrating Ge within the SiGe layers. Work by LeGoues found the oxidation rate of SiGe was enhanced compared to Si when performed under wet conditions.<sup>160</sup> Using this result, we set out to oxidize our multilayer fin structures at 900 °C such that the SiGe layers would oxidize faster than the Si layers creating a structure similar to that shown in Figure 5-3. In this configuration, the wider Si layers would increase the surface

area available for gate contact in a completed device while also being strained by the concentrated SiGe layers. However, after oxidation, a completely different structure was observed. Ge was observed to have diffused around the Si layers encapsulating them



Figure 5-2: High resolution TEM images of a Si/SiGe fin oxidized for 7 hours at 650 °C. Segregation of SiGe clusters in the grown SiO<sub>2</sub> are evident





Figure 5-3: Schematic of the predicted structure of an oxidized Si/SiGe fin and the experimental result after oxidizing at 900 °C for 20 minutes. Lateral diffusion of Ge is observed on the sides of the Si layers.



fully in SiGe (Figure 5-3). Additionally, rounding of corners in the encapsulated Si layers was observed pointing to potential applications as rounded Si nanowires. A literature search indicated this result had never been seen before opening the door for the first ever investigation of this new Ge diffusion phenomenon.

#### **5.3 Experimental Details**

The initial fin structures were fabricated on commercially available 300 mm (100) Si wafers. Alternating layers of 15 nm thick Si and Si<sub>1-x</sub>Ge<sub>x</sub>, where x=0.3, were deposited for a total of 4 layers each resulting in a stack height of 120 nm. Fin patterns 45 nm wide were formed in the <110> direction using photolithography and a deep reactive ion etch. Samples were analyzed with cross-sectional HAADF-STEM and high resolution TEM using a probe aberration-corrected JEM-ARM200CF instrument with a STEM resolution of 0.78 Å. Images were collected with a probe convergence semiangle of 22 mrad and an inner collection angle of 76 mrad. Additional HRTEM images were captured using a JEOL 2010F. Cross-sections were prepared using a focused ion beam (FIB) system and taken orthogonal to the fin direction. A HAADF-STEM image of the Si/SiGe superlattice fin is shown in Figure 5-4a where the brighter areas correspond to SiGe and the darker areas to the Si layers due to higher atomic number of Ge. A larger magnification of the alternating layers is shown in Figure 5-4c demonstrating the epitaxial structure is defect free. To prevent the oxidation fronts of neighboring fins from meeting and halting further oxidation, a fin spacing of 360 nm was used. Fin oxidations were carried out in a conventional tube furnace and a rapid thermal annealing system at temperatures between 650 °C and 1000 °C in a flowing dry O<sub>2</sub> ambient. Prior to annealing, samples were dipped in 6:1 buffered oxide etch (BOE) for 30 seconds to remove the native oxide. The grown thermal oxide was left intact for TEM analysis.



Figure 5-4: Cross-sectional HAADF-STEM images of the SiGe/Si superlattice fin structure (a) before oxidation and (b) after 40 min at 900 °C in O<sub>2</sub>. Brighter areas correspond to SiGe layers and darker areas correspond to Si layers. The black region surrounding the fin is SiO<sub>2</sub> and protective carbon. For ease of discussion, the Si nanowires are labeled top, middle, and bottom excluding the top layer of Si as it is partially consumed by oxidation. Higher magnifications (c) before oxidation and (d) after 40 min at 900 °C in O<sub>2</sub> show no defects in the crystal structure and a rounding of the newly formed nanowires.

Atom probe tomography (APT) samples were prepared using an in situ FIB lift-

out procedure<sup>111,112</sup>. Prior to lift out, the thermally grown oxide was removed using a 6:1

BOE solution and a 300 nm amorphous Si layer was deposited over the fins using a

PECVD process. Removal of the oxide was necessary as it has a larger evaporation

field than the surrounding Si and Ge. The difference in evaporation fields could lead to

artifacts in the APT reconstruction such as local magnification, out of sequence

evaporation, and increased background level. Large differences can even cause

fracture of the tip. To protect the fins from damage during the milling process, a strip of

Pt was deposited orthogonal to the fin direction. Finally, lift-out and annular milling of the

APT tips was performed. Initial milling was performed using a 30 kV beam; the final milling was done at 2kV to limit the amount of ion beam induced damage to the specimen. These lift-out specimens were mounted to pre-sharpened copper posts that were created by slicing a TEM grid in half. The holder for the grid is compatible with the FIB, TEM, and APT instrumentation<sup>161</sup>. This allowed for TEM imaging of the specimens in a Philips CM200 microscope before and after APT analysis. Figure 5-5 show these TEM images as well as a combination of the two identifying the evaporated volume. Imaging aided in both the targeting of the fins at the apex of the tip and the generation of accurate APT reconstructions.

Before APT Run





Combined Images

Figure 5-5: TEM images of an APT tip before and after a run. Combining these images allows for identification of the evaporated volume aiding in reconstruction.

The APT analysis was performed using a local electrode atom probe (LEAP) 4000X Si from Cameca utilizing a 355 nm wavelength laser. During the run, the specimen temperature was set to 50 K and the base pressure was 5.12e-11 Torr. To reduce the number of background counts and improve the sample yield, a detection rate (number of ions detected per laser pulse) of 0.5% was targeted and a laser energy of 40 pJ was used. APT data sets were reconstructed using Cameca's IVAS 3.6.8 program.

To ensure fin dimensions in the APT reconstructions were accurate, high magnification TEM images of each tip were captured. These images were then used to create a tip profile during the 3D reconstruction process.

## 5.4 Lateral Ge Diffusion and Nanowire Formation During Oxidation

Cross-sectional HAADF-STEM images of an oxidized fin are shown in Figures 5-4b and 1d after oxidizing for 40 minutes at 900 °C. When compared to the starting fin structure in Figure 5-4a, the Si layers have reduced in width and Ge has diffused around them encapsulating the newly formed Si nanowires in SiGe while remaining single crystal. This type of Ge diffusion along an Si/SiO2 interface, which we will refer to as lateral diffusion, has never been reported and is critical to forming a defect free encapsulated Si nanowire.

To better study the lateral Ge diffusion around the Si layers and the formation of encapsulated Si nanowires, a time series study was performed, oxidizing the fins for 5, 10, 20, 30, 40, and 45 minutes at 900 °C with a dry oxygen ambient. Figures 5-6a through 5-6d show the formation evolution of the Si nanowire from the starting fin structure. After 5 minutes (Figure 5-6b), Ge is present on the sides of the Si layers diffusing a distance of 7.5 nm (from each layer) with a uniform thickness of approximately 1nm. For comparison, the time for Ge to diffuse 7.5 nm in bulk Si was calculated using Einstein's approximation (Equation 5-2) relating diffusion time t to the mean diffusion distance x and diffusivity D. A regression curve derived from several reported measurements was used to calculate the Ge diffusivity at 900°C using the Boltzmann's constant k and temperature T (in Kelvin).<sup>133</sup>

$$D = 1.72 \cdot 10^3 e^{-\frac{4.830 eV}{kT}} cm^2/s$$
(5-1)

$$t = \frac{x^2}{2D} \tag{5-2}$$

A diffusion time of 25.6 hours is required for Ge to diffuse a similar distance through bulk Si indicating the lateral diffusion process is over 2 orders of magnitude faster. Further oxidation of the fin structure results in continued shrinking of the Si layer width and an increase in thickness of the SiGe on either side of the Si fin as Ge is rejected from the oxidizing interface (Figure 5-6c). As the process continues, the Si layers begin to round (Figure 5-6d). Schematics shown in Figure 5-6 illustrate these competing Ge diffusion processes.



Figure 5-6: HAADF-STEM images and schematics of the middle Si layer (a) before oxidation and after oxidation at 900 °C for (b) 5 minutes, (c) 20 minutes, and (d) 45 minutes. The lateral diffusion of Ge along the Si/SiO<sub>2</sub> interface is indicated in the schematic by the vertical arrows labeled 1. This process initially encapsulates the Si layers illustrated in the 5 minute oxidation images. As Ge continues to laterally diffuse, it is rejected by the advancing oxide front indicated by the horizontal arrows labeled 2.

The change in width of the Si nanowires with oxidation time is shown in Figure 5-7a where both the top and middle wire widths are presented. Initially, the nanowire width decreases in a fashion comparable to a complementary error function indicating the loss of Si exhibits Fickian behavior. Between oxidation times of 25 and 30 minutes,



Figure 5-7: (a) Si nanowire widths plotted as a function of oxidation time for both the middle and top wire positions. Samples were oxidized at 900 °C. (b) HAADF-STEM image of a Si/SiGe superlattice fin oxidized for 45 minutes at 900 °C in a dry O2 environment. (c) A nanowire with a diameter of 2 nm was achieved at the top wire position.

the nanowire width remains mostly unchanged. This regime marks where the nanowires

begin to become rounded as width dimensions were calculated across the center of the

Si layer. Rounding is most likely due to a reduction in surface energy of the Si nanowire.

After 30 minutes, the wire thickness continues to decrease until sharply falling near the

45-minute mark. This trend is analogous to spherical dissolution<sup>162</sup> and could be considered a form of cylindrical dissolution<sup>163</sup>. A minimum nanowire width of 2 nm was achieved at the top wire position after a 45-minute oxidation (Figure 5-7b and 5-7c). Thus, size control of these nanowires is possible by tuning the oxidation time and fin geometry.

The change in width of the laterally diffused SiGe layers on either side of the middle Si nanowire is shown in Figure 5-8. Width values were measured even with the center of the Si nanowire in a similar fashion to the Si wire with measurements. As the oxidation time increases, the width of the laterally diffused SiGe layer increased approaching a steady state value on the order of 3.7 nm after 40 minutes. At 45 minutes, the Si nanowire had begun to shrink not only in the x-direction, but also in the y-direction. In this regime, the laterally diffused SiGe layer increased in width past the steady state thickness and is considered an outlier as the growth is in a different regime.



Figure 5-8: Change in the laterally diffused SiGe layer width with oxidation time. A steady state width of 3.7 nm is shown via the dotted line.

The defect free nature of the nanowires formed after oxidation proved to be an interesting result as oxidation of Si is known to inject interstitial point defects due to volume mismatches between Si and SiO<sub>2</sub>.<sup>164</sup> Aggregation of these interstitials can lead to extrinsic extended defect formation degrading the performance of a fabricated device. SiGe has previously been shown to suppress the injection of interstitials into a Si substrate during oxidation<sup>160,165</sup> by either reducing interstitial generation due to Ge being more closely lattice matched to SiO<sub>2</sub><sup>166</sup> or reducing interstitial formation energy from SiGe to Si.<sup>167</sup> As such, the SiGe layers would be free from injected interstitials maintaining their single crystal structure. In the case of the Si layers, recent work by Martin at al. found that a SiGe film as thick as a few monolayers can suppress the injection of interstitials into a Si substrate.<sup>168</sup> Thus, once Ge has laterally diffused across the Si layers, interstitial injection is suppressed allowing for the formation of a single crystal structure regardless of oxidation time.

#### 5.4.1 Fin Shape During Oxidation

It becomes apparent after 5 minutes that the width of the SiGe layer is wider than the Si layer indicating faster oxidation of Si over SiGe. However, as discussed in Chapter 5.2, the oxidation rate of Si and SiGe is the same under dry conditions. BF TEM images of this sample show the grown oxide perfectly conforms to the undulating sidewalls and has a constant thickness (Figure 5-9) demonstrating similar oxide growth for both Si and SiGe. This result implies that while different amounts of each layer were consumed, a constant oxide thickness is still produced. The uniform thickness of the oxide layer could be explained by a redistribution of SiO<sub>2</sub> along the sidewalls of the fin during growth. EerNisse found that mechanical stresses during oxidation could be relieved at high growth temperatures via a viscous flow mechanism.<sup>169</sup> Additionally,

modeling work by Senez et al. demonstrated that oxides grown on convex and concave surfaces, much like what we observe in our fin, are tensely and compressively strained respectively and flow to relieve to these stresses.<sup>170</sup> When implemented into simulations, these curved surfaces show similar oxide growth reduced from that of a flat surface.<sup>171</sup> As such, Figure 5-9 points to variations in the initial growth rates between Si and SiGe contrary to previous results. The variations in oxide thicknesses are most likely masked by viscous SiO<sub>2</sub> flow which relived stresses in the film built by the undulating fin shape.



Figure 5-9: BF TEM image of a fin oxidized for 5 minutes in dry O<sub>2</sub> at 900 °C. Oxide thickness is constant along fin sidewalls.

Continued oxidation of the Si/SiGe fin structure leads to a reduction in the undulations present in the sidewalls. After 40 minutes, the sidewalls had flattened and became more uniform remaining as such until the fin was completely oxidized (Figure 5-10). The nonuniformity of the sidewalls yields a variety of surface orientations including {111} planes with varying surface energies, and thus, varying surface free energies calculated using the Kelvin equation.<sup>172</sup> High temperature oxidation provides an avenue for a reduction in the surface free energy smoothing the "rough" sidewalls.<sup>173</sup> The high

mobility of Ge atoms at the oxidizing interface facilitates the redistribution of surface atoms.



Figure 5-10: HAADF-STEM image of a Si/SiGe fin oxidized for 40 minutes in dry O<sub>2</sub> at 900 °C.

Finally, variations in fin width with along the y direction (vertical) are observed once sidewall undulations have been removed. This result points to a larger rate of oxidation for the top of the fin than the bottom. Diffusion limited oxidation at the bottom of the fin is most likely not the case as the atmosphere surrounding the fin is 100% dry oxygen. Variations in oxidation rates based on the curvature of Si structures has been reported concluding concave surfaces, like those locate at the base of the fin as it transfers to the substrate, demonstrated retardation in oxidation when compared to planar surfaces.<sup>174</sup> A byproduct of this variation in oxidation rates is differences in nanowire radii within the fin with larger nanowires located at the base and smaller nanowires located at the fin apex. Applications requiring uniform nanowires would require taller fins to ensure uniform oxidation in regions with Si layers.

## 5.4.2 Strain Mapping

Due to the lattice mismatch between SiGe and Si, the deposited SiGe layers are in a state of compressive strain. As Ge diffused around the Si layers during oxidation, the strain state of the newly formed Si nanowires changed. Geometric phase analysis (GPA) of atomic resolution HAADF-STEM images was utilized to quantify the strain in the oxidized samples<sup>175</sup>. The SiGe lattice below the Si wires was used as a reference for the strain calculations. The STEM images used for the strain analysis were taken from a cross section which contained multiple fins that underwent the same thermal oxidation process. It was found that the variations in diameters of the nanowires in the top and bottom positions between different fins in the same cross section were small and therefore would not lead to large variations in the strain field. As such, any error in the strain measurement would then be due to standard measurement error and not sample variation. Several FIN structure measurements confirmed the strain measurement has about less than 0.5% variation.

Figure 5-11 shows strain mapping of the top wire from the 40-minute oxidized sample. A maximum of -2% strain in the x direction ( $\varepsilon_{xx}$  Figure 5-11b) was found on the sides of the Si wire with the center showing closer to -1% strain. Strain in the y direction ( $\varepsilon_{yy}$  Figure 5-11c) shows some fluctuation, but the profile within the Si wire shows very little variation from the SiGe layer. A dilation of the  $\varepsilon_{xx}$  and  $\varepsilon_{yy}$  maps (Figure 5-11d) show the Si wire is in a net tensile strain state with approximately -1% strain and demonstrates a more wholistic view of the strain fields within the wire. Larger  $\varepsilon_{xx}$  tensile strains were observed for samples oxidized for 20 minutes up to -3% (Figure 5-12). Due to the shorter oxidation time, the Ge concentration on the side of the wire was larger than the concentration above or below leading to a larger lattice mismatch in the x

direction. This can be observed in Figure 5-6c where the Ge rich regions on either side of the Si wire have a brighter intensity than the regions above or below. However, no long-range order of the strain field is observed in the y direction similar to the 40-minute oxidized sample.



Figure 5-11: Strain measurements were performed on a HAADF-STEM image using GPA software in Gatan DigitalMicrograph (a). Strain maps for the 40 min oxidation at 900 °C were created in the (b) x direction ( $\epsilon_{xx}$ ) and (c) y direction ( $\epsilon_{yy}$ ). A maximum  $\epsilon_{xx}$  of -2% was calculated on the sides of the wire. (d) A dilation, or Dxy, of the  $\epsilon_{xx}$  and  $\epsilon_{yy}$  shows the overall strain profile.



Figure 5-12: HAADF-STEM image of the center wire of a fin an oxidized for 20 minutes at 900 °C in a dry O<sub>2</sub> environment with associated strain fields.

# 5.4.3 Atom Probe Tomography Analysis

A 3D APT reconstruction of the top nanowire after a 40-minute oxidation process

is shown in Figure 5-13a. The rounded Si nanowire can clearly be observed at the

center of the reconstruction with Ge present on either side. Figure 5-13b displays a 1D concentration profile through the center of the nanowire in the x direction. Peak Ge concentrations of 46% are observed in the center of the laterally diffused Ge layers, increased from effectively 0% before oxidation. These concentration values correlate well with the results of Long et al<sup>82</sup> for SiGe oxidations at 900 °C. The decay length (defined as the distance required for a change in concentration by one order of magnitude) of Ge into the nanowire was calculated the be 1.2 nm/dec indicating a fairly abrupt interface between the nanowire and surrounding SiGe. Variations between the measured fin dimensions in TEM and the dimensions obtained from APT data are attributed to distortions in the reconstruction stemming from preferential evaporation of the SiGe layers. These variations lead to changes in the local tip radii varying the magnification of different layers.<sup>120</sup> However, these distortions primarily affect the dimensions of the reconstructions, not the concentrations of the species as atomic density variation affect all detected atomic species within the volume.

A reconstruction cross-section was taken down the length of the oxidized fin is shown in Figure 5-13c. When displaying only Ge atoms, the reconstruction demonstrates the middle Si nanowire runs the length of the fin and is not broken up by variations in oxidation rates. This further illustrates the feasibility of forming nanowires utilizing the oxidation and lateral diffusion process. While this result shows the continuity of nanowires > 5 nm in diameter, no data has been obtained to show the continuity of the 2 nm diameter Si nanowire obtained in the top position of the 45 minute oxidized sample.



Figure 5-13: Atom probe tomographic reconstruction of the top wire region from a sample oxidized for 40 minutes at 900 °C in a dry O2 environment (a). Only Ge atoms are presented and are shown in red. (b) 1D Ge concentration profile taken along the dotted region in a). (c) A reconstructed cross-section taken down the length of the top wire shows the nanowire is continuous through the oxidized fin. Narrowing of the wire on the right side of the image is attributed to distortions in the reconstruction.

# **5.4.4 Temperature Effects**

Oxidation experiments were also carried out at 800 °C and 1000 °C to investigate

the effect of temperature on nanowire formation. To ensure the experiments were

comparable to those performed at 900 °C, equivalent oxidation times were targeted

such that the thickness of the SiO<sub>2</sub> layer grown for each temperature were comparable.

Figure 5-14 shows BF TEM images of Si nanowires at 800 °C and 1000 °C for oxidation

times comparable to 5 minutes, 20 minutes, and 40 minutes at 900 °C demonstrating

similar oxide thicknesses. Corresponding HAADF-STEM images for the 800 °C and

1000 °C samples are shown in Figure 5-15. At 800 °C, some Ge diffuses down the Si/SiO<sub>2</sub> interface encapsulating the Si layers much like the original experiments. However, continued oxidation does not increase the thickness of these Ge rich layer as the width of the fin decreases. Additionally, the Si layers no longer form into rounded wires, but into more boxlike wires similar to what was observed in previous publications<sup>117,118</sup>. Increasing the oxidation temperature to 1000 °C resulted in an increased rate of interfacial Ge diffusion relative to that of the Si layer is comparable to that at 900 °C for 20 minutes but the fin width is larger for the 1000 °C sample. More importantly, because of its higher activation energy, the rate of diffusion of Ge into the Si wire increases more than the oxidation rate and leads to interdiffusion at the Si/SiGe nanowire interface. This interdiffusion is apparent in Figure 6f, and would result in a roughening of the Si wire post-etch, potentially limiting electron mobility<sup>176</sup>.

The temperature study indicates there are competing activation energies of several diffusion processes during the Ge encapsulation process. At low temperatures, the oxidation of Si and SiGe is dominant over the lateral diffusion of Ge along the oxidizing Si interface as well as the interdiffusion of Ge into Si, leading to limited encapsulation of the Si layers. Segregation of Ge clusters into the growing oxide was even observed during oxidation experiments at 650 °C (Figure 5-2). However, at higher temperatures while the rate of lateral Ge diffusion increases, Ge interdiffusion into Si also increases resulting in poorly defined Si nanowires. This leads to a process window in which the combination of thermal oxidation, Ge segregation, and lateral Ge diffusion

along the interface can occur without Ge interdiffusion, leading to controllable fabrication of Si nanowires of variable dimensions.



Figure 5-13: TEM bright field images of the FIN oxidized at 800 °C for (a) 45 minutes, b) 160 minutes, and (c) 300 minutes as well as 1000 °C for (d) 2 minutes, (e) 3 minutes 35 seconds, and (f) 7 minutes and 30 seconds. Oxide thicknesses are visible to show that oxidation experiments at different temperatures with similar oxidation times were targeted.


Figure 5-14: HAADF-STEM images of cross-sections of fin samples oxidized at 800 °C for (a) 45 minutes, (b) 160 minutes, and (c) 300 minutes and 1,000 °C for (d) 2 minutes, (e) 3 minutes 35 seconds, and (f) 7 minutes 30 seconds. Minimal lateral Ge diffusion around the Si layers is observed at 800 °C with no change in thickness of the Ge rich layer after continued oxidation. After oxidizing at 1,000 °C for 7 minutes and 30 seconds, the interdiffusion of Ge into the Si wires becomes apparent.

As was previously shown in Figure 5-8, a steady state width of the laterally diffused SiGe was observed for 900 °C oxidation experiments. Width measurements were taken from the 800 and 1000 °C samples at their steady state thicknesses for comparison. Table 5-1 shows these thickness values demonstrating an increasing laterally diffused width with increasing temperature. While this width does increase with temperature, it is important to note that previous studies have found increasing the oxidation temperature decreases the achievable pileup concentration.<sup>82,88</sup> As such, to better compare these layers, the overall laterally diffused width was multiplied by the

temperature dependent pileup Ge fraction. This yielded a layer thickness if all Si atoms were removed forming a pure Ge layer. Unfortunately, no concentration data was obtained for the 800 and 1000 °C samples. Because APT data from Chapter 5.4.3 showed good agreement with the literature results for 900 °C oxidation, the assumption that these results would agree with the 800 and 1000 °C layers was made. Thickness values obtained by multiplying the pileup concentrations with the laterally diffused SiGe layer thicknesses are shown in the right column of Table 5-1. Very good agreement is observed between the pure Ge thickness calculations for each oxidation temperature. This result indicates the total amount of Ge in the pileup layer is independent of oxidation temperature once reaching the steady state width regime. This result would allow for the prediction of the pileup layer thickness based on the chosen oxidation temperature.

1000 °C and their associated pure Ge thickness. *Assumed from Long et al <sup>2</sup>							
	Layer Thickness	Pileup	Pure Ge Layer				
Temperature (°C)		•	2				
	(nm)	Concentration (%)	Thickness (nm)				
	(1111)						
800	2.64	*65	1 70				
800	2.04	CO	1.72				
900	3.64	46	1.67				
1000	5.00	*34	1.70				

Table 5-1: Calculation of laterally diffused SiGe layer thickness for temperatures 800-1000 °C and their associated pure Ge thickness. \*Assumed from Long et al<sup>82</sup>

### 5.5 Discussion

As previously stated, the estimated rate of this lateral Ge diffusion is at least 2 orders of magnitude faster than bulk diffusion of Ge through crystalline Si. This high diffusivity suggests an alternative diffusion mechanism to traditional interstitial and substitutional diffusion of Ge in Si. It is suggested that the diffusion process may depend on a redox enhanced diffusion (RED) reaction at the interface. It is possible Ge atoms are initially oxidized at the oxidizing interface forming GeO bonds and then reduced by Si in the lattice. As there is a larger net concentration of Si in the Si rich layers than in the SiGe layers, reduction of GeO by Si in the Si layers would result in apparent lateral diffusion. This process would repeat until the Si layer was effectively encapsulated in a thin SiGe layer. Such an oxidation and reduction process has been observed in Ge nanocrystals<sup>177</sup> and modeled for blanket oxidation<sup>178</sup>. Further oxidation and inert ambient experiments will need to be performed to determine if a RED mechanism can explain the observed diffusion.

**Planar dependence of Ge lateral diffusion:** Careful observation of high magnification HAADF-STEM images yielded a very interesting result. After a 40-minute oxidation at 900°C, Ge had diffused along the top Si layer sidewalls, but not across the top of the layer/fin (Figure 5-15). Similarly, no lateral Ge diffusion was observed along the trench adjacent to the Si/SiGe fin even after oxidizing at 900°C for 50 minutes (Figure 5-16). These observations point to a planar dependence for lateral Ge diffusion across an oxidizing Si/SiO<sub>2</sub> interface as the top of the fin and adjacent trenches are {100} surfaces while the fin sidewalls are {110} surfaces. It should be noted that lateral diffusion along {111} surfaces is present as Ge diffused around corners at both the tip apex and base. It is still unclear as to why this planar dependence occurs for the lateral diffusion process. One explanation is that the {100} surface has a lower packing efficiency than the {110} or {111} surfaces reducing the likelihood of adjacent Si atoms reducing GeO bonds. A more likely explanation is that the surface energy of a Si {100}

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surface is larger than those for {110} or {111} surfaces and is also larger than the {100} surface energy for Ge.<sup>179</sup> As such, and energy barrier is in place unable to be overcome by the thermal energy provided in the oxidizing anneals. Further experiments will need to be performed in order to fully explain and prove the planar dependence of lateral Ge diffusion.



Figure 5-15: HAADF-STEM images of the Si/SiGe fins before (left) and after (right) a 40-minute oxidation at 900 °C. Inset shows a higher magnification image of the oxidized fin apex showing lack of lateral Ge diffusion along fin top.



Figure 5-16: HAADF-STEM images of the Si/SiGe fins before (left) and after (right) a 50-minute oxidation at 900 °C. No lateral diffusion is observed in the trench adjacent to the fin.

### **5.6 Conclusions**

A novel method to fabricate horizontally stacked strained Si nanowires via the oxidation of Si/SiGe superlattice fin structures has been shown. During this process, it was found that Ge diffused along the Si/SiO<sub>2</sub> interface and was subsequently rejected by the advancing oxide front. Such a process has never before been observed. Further oxidation of the structure consumed the Si layers during Ge pileup forming rounded Si nanowires. Size control of these nanowires, even down to 2 nm, was achieved by tuning the oxidation time. Due to the lattice mismatch between the Si and surrounding SiGe, the resulting encapsulated Si nanowires also showed biaxial strain up to -2%. Furthermore, we have concluded that there is a temperature process window in which significant lateral Ge diffusion occurs without the interdiffusion of Ge into the Si nanowires. These nanowires have the potential to be implemented in strained CMOS devices or in more conventional nanowire transistors where the SiGe is selectively etched away. While this lateral Ge diffusion process was observed during the formation of strained Si nanowires, we believe its applications are more far reaching than what has been presented. Such a process which forms defect free epitaxial heterostructures can be applied to the fabrication of devices which would otherwise be difficult to obtain using conventional methods. This includes extremely small Hall sensor arrays formed by crossed nanowires, strained Si quantum dots, and air gap transistors.

## CHAPTER 6 CONCLUSIONS AND FUTURE WORK

#### 6.1 Conclusions

This work has demonstrated results aimed at contributing to the further scaling of 3D transistors and their characterization. Standard methods to characterize doping concentrations within 3D devices fall short in proving a wholistic picture of their distributions. Atom probe tomography has shown to provide the necessary resolution and 3D capabilities for accurate quantification of these profiles. However, distortions in reconstructions arise when running 3D heterostructures with multiple interfaces and evaporation fields. We have shown these distortions can be corrected via a sidewall flattening method deemed the Landmark Reconstruction. This method successfully removed undulations in the sidewalls of Si/SiGe fins and provided accurate dimensions matching those observed in TEM. The Landmark Reconstruction method was also extended to doped Si/SiGe fins to determine its viability in probing doping profiles. While there was good agreement between the extracted profiles and simulated data, some error was present most likely stemming from co-evaporation of dopants with oxygen at the oxide interface. Additionally, small inconsistencies in the shapes of extracted profiles in different regions of the same layer pose a question as the extent APT can provide accurate diffusion information. With that, it is believed the application of the Landmark Reconstruction method to distorted heterostructures is a critical step to making APT a routine characterization method for the semiconductor industry.

We have also demonstrated the first observation of lateral Ge diffusion along an oxidizing Si/SiO<sub>2</sub> interface. This phenomenon was observed during the oxidation of Si/SiG fins when SiGe diffused around the Si layers and resulted in the formation of

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rounded Si nanowires. HAADF-STEM images showed sharp interfaces between the SiGe cladding and Si nanowires as well as a net tensile strain within the nanowires. APT analysis also shows the nanowires run the length of the oxidized fins and the pileup concentration of Ge matches well with literature values. The temperature dependence on nanowire formation was also explored. It was found that a temperature window exists in which lateral diffusion of Ge occurs without significant indiffusion of Ge into the Si nanowire roughening the interface. Additionally, initial observations have concluded there is a critical Ge content achievable in the laterally diffused SiGe layer allowing for tailoring of this layer's thickness based on the oxidation temperature. This novel diffusion process has a wide variety of applications not only in the fabrication of 3D transistors, but also in the formation of strained Si quantum dots and other heterostructures unobtainable using conventional fabrication methods.

#### 6.2 Future Work

While this work has explored how to correct for distortions created during an APT run, correction processes can only go so far to produce accurate reconstructions and concentration profiles. Future work investigating Si/SiGe fins should focus on preventing the introduction of distortions in the first place. Similar to experiments performed by Melkonyan et al.<sup>120</sup>, the high evaporation field capping material should be removed after the final low kV clean step (final sharpening) via a wet etch. It is expected compression in the SiGe layers will be nonexistent allowing for better characterization of doping profiles and opening the door for improved As diffusion studies.

As this work demonstrates the first observation of the lateral Ge diffusion phenomenon, more experiments will need to be done to determine the mechanism with which Ge diffuses along an oxidizing interface. The first step will be to confirm that

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oxidation is driving force for the lateral diffusion process. Oxidation experiments of wide fins with a single SiGe layer near the apex should allow for a long (110) interface to observe this diffusion. Performing both oxidizing and inert anneals should provide the answer to this question. Additionally, creating fins with (111) sidewalls will help investigate the planar dependence of the diffusion process. Investigation into the mechanism by which lateral diffusion occurs will be paramount to its implementation into more advanced applications. Finally, the process should be applied to new Si/SiGe structures such as multilayer pillars and crosses to probe its application in creating novel structures.

# APPENDIX A APT SAMPLE YIELD AND RUN PARAMETERS

	LEAP 3000	LEAP 4000	LEAP 5000	LEAP 5000 – Low DR	LEAP 5000 – Min DR
Facility	University	Colorado School of	University	University of Alabama	University of Alabama
	of Alabama	Mines and Oak	of Alabama		
		Ridge National Lab			
Laser Wavelength	515	355	355	355	355
(nm)					
Detection Rate (%)	0.5	0.5	0.5	0.1	0.05
Sample Temperature	80	80	50	50	50
(K)					
Number of Runs	37	47	41	26	7
Successful Runs	3	9	10	18	6
Probability of Yield	0.08	0.19	0.24	0.69	0.86

# APPENDIX B ATOMIC DENSITY CALCULATION MATLAB CODE

This code calculates the atomic density for several 1 nm wide voxels within an APT data set and plots them versus distance.

clear all;

close all;

%%%Load APT Data%%%

filename = 'R86\_01790-v01-roi-cross-section.pos'; %pos file

XYZM=posRead(filename);

%%%Select only Ge atoms%%%

XYZGe = rangeGe01790(XYZM); %Range for 01790

Ge = XYZM(2,:); %Uses all ions for calculation

% Ge = XYZGe(2,:); %Uses only Ge ions for calculation

dist = ceil(max(Ge))-floor(min(Ge));

GeDensity = zeros(1,(dist));

x = 0:1:(dist-1);

m = floor(min(Ge));

for i = floor(min(Ge)):ceil(max(Ge));

for n = 1:length(Ge);

if Ge(n)>i && Ge(n)<(i+1)

```
GeDensity(i-m+1) = GeDensity(i-m+1)+1;
```

end

end

end

xdist = range(XYZGe(1,:));

zdist = range(XYZGe(3,:));

%%% Volume of voxel calculate in nm. Used to determine the atomic density

%%% of Ge in the distorted reconstruction

volume = xdist\*zdist\*1; %%%1 denotes 1nm in the y direction (bin size)

GeDensity = GeDensity/volume; %% in atoms/nm^3

bar(GeDensity)

title('Ge Atomic Density in Distorted Reconstruction')

xlabel('Distance (nm)')

```
ylabel('Atomic Density (atom/nm^3)')
```

The posRead function extracts the position and mass/charge ratio data from the

.pos file and converts it into a 4 by x matrix compatible with MATLAB functions.

%rnd file data read function

```
function [XYZM] = posRead(filename)
```

lengthL = dir(filename);

DataNumber = lengthL.bytes/16;

fileID = fopen(filename,'r','b');

XYZM = fread(fileID,[4,DataNumber],'single','b');

XYZM=single(XYZM);

fclose('all');

end

All atoms with the exception of Ge are removed from the data set using a range function with mass/charge inputs acquired from the mass spectrum for the particular data set.

% range of Ge atom selection function

function [pos] = rangeGe01790(POS)

Ge1 = (POS(4,:)>73.861)&(POS(4,:)<74.023);

Ge2 = (POS(4,:)>72.854)&(POS(4,:)<73.030);

Ge3 = (POS(4,:)>71.869)&(POS(4,:)<72.015);

Ge4 = (POS(4,:)>69.875)&(POS(4,:)<70.040);

Ge5 = (POS(4,:)>75.876)&(POS(4,:)<76.017);

Ge6 = (POS(4,:)>37.891)&(POS(4,:)<38.043);

Ge7 = (POS(4,:)>36.433)&(POS(4,:)<36.570);

Ge8 = (POS(4,:)>36.925)&(POS(4,:)<37.063);

Ge9 = (POS(4,:)>35.457)&(POS(4,:)<35.575);

Ge10 = (POS(4,:)>34.960)&(POS(4,:)<35.034);

Ge = Ge1|Ge2|Ge3|Ge4|Ge5|Ge6|Ge7|Ge8|Ge9|Ge10;

pos = POS(:,Ge);

pos(4,:) = [];

end

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## **BIOGRAPHICAL SKETCH**

Will Brewer was raised in Castle Rock, Colorado where he developed an interest in science by performing home experiments with his parents and grandfather. This led him to the University of Colorado at Boulder where he received his undergraduate degree in chemical engineering. There, he met his now wife Meg who shared his interests in the sciences, music, and the sport of ultimate frisbee. After graduating, he and his wife moved to Boise, Idaho where Will began working at Micron Technology as a manufacturing development engineer. With aspirations of doing industrial research in semiconductor devices, he attended the University of Florida to pursue a Ph.D. in Materials Science and Engineering under Dr. Kevin Jones. Will graduated with his Ph.D. in August of 2018. He and his wife are moving back to Boise where he will continue work at Micron as a process development engineer.