Characterization of SiGe layers grown by Trisilane and Germane at low temperatures for BiCMOS application

CARMINE CAPPETTA
Characterization of SiGe layers
grown by Trisilane and Germane
at low temperatures for BiCMOS
application

Carmine Cappetta

Master's Degree Project

Stockholm, Sweden, 2014
Se io mi obliassi non arriverei
dove verità tremanti indugian,
rimarrei sospeso, inanelante;

tra quei fiotti della vòta vita
e la cagionevole essenza
scelgo di apparir del destin mio

creatore e unico padrone
Abstract

Low temperature epitaxy (LTE) of SiGe by chemical vapor deposition (CVD) has attracted dramatic attention during the last decade for CMOS and BiCMOS application. LTE relates to a temperature range of 350–650 °C. The low temperature budget provides the possibility of integrating epitaxy in the process line when the sensitive active parts are already present on the chips. In this case, the benefits of LTE are to avoid the thermal mismatch between different layers in the transistors, preventing damages to the poly gate and to ensure the integrity of thin gate oxide.

The challenge to deal with the lowering temperature process is the quality of epitaxial layer. In particular, the low temperature processing results in low growth rates and more oxygen (or water moisture) contamination in the layer growth. For these reasons, particular attention has been paid to islands formation at the interface and within the layer. Choosing appropriate Si precursor, optimizing the growth parameters and reducing oxygen (or moisture) contamination are critical issues for growing high quality epitaxial layers at low temperatures.

This thesis work presents the characterization of layers grown using Trisilane (Si$_3$H$_8$), Germane (GeH$_4$) and Hydrochloric acid (HCl) as precursor gases at LTE. Characterization tools were high-resolution scanning electron microscope (HRSEM), Secondary ion mass spectroscopy (SIMS), high-resolution x-ray diffraction (HRXRD) and noise measurements.
The results of this work suggest that only electrical measurements are most sensitive way to study the effects of low amounts of contamination on crystal quality of LTE grown structures. It also shows that the presence of HCl in the chamber increases the noise level in these types of structures. This higher level of noise is believed to be a result of higher defect density due to the etching caused by HCl and metal contamination that could arise from the aging and the corrosion of the pipelines.
I would like to give my sincere gratitude to all people who have helped me in this work.

First, I would like to thank Prof. Salvatore Bellone that gave me the chance to prove myself in a different environment and come here at KTH, always stimulating and encouraging me. With him I cannot forget to thank also Prof. Östling for accepting me and welcoming at EKT department and Docent Radamson for supervising me in this work and my thesis.

A special thank goes to Ing. Ahmad Abedin for his help and teaching me the device processing and electrical measurement parts of my work and to Mohdi Moeen for the explanations about x-ray measurements.

I would also like to thank all the professors I have met during these years, specially, Alfredo Rubino for his suggestions and instructions that revealed very useful in this thesis work.

I would not be the person I am now without my family, thus a particular thank goes to my mother, Helga, who is often too caring towards me even when I do not deserve that, my father, Donato, who always finds the right words somehow and my older sister, Gina, who is a fighter and gave me strength, besides of encouraging me in having always new interests since I was a child. With them there is also a great supporting by my grandparents, my uncles and cousins.
A lot of people say that friends are also close as family, so I have to thank also: Alessio, Alfonso, Carlo, Chiara, Emanuele, Fiorella, Francesco, Franco, Gaetano, Luca, Mattia, Raffaele, Roberto and Vincenzo that always supported me during these months. I wish to particularly thank three of my closest friends among the others, who gave me more than their friendship. Carmine, who is my friend since we were 3 years old helped me in so many ways during these years, together with his family, and with so much patience that he is like a brother to me (Cosimo, Sara, Mattia and Alessio I will not forget you and I hope nothing but the best for you); Nicola, who despite his hard work and his occupations never forgets to give me a call or to ask me how I feel (special thanks goes also to Carmen and Antonio, who care for me so much); Simona, who always succeed in making me happy, and is the only persons who seems to really understand me sometimes (about that I have also to thank her for some revisions of my work) together with her family which shared with me a lot of moments (Maria Rosaria, Anna, Rosanna, Claudia, Gaetano, Umberto and Davide have always been kind in my regards, words are not enough to describe what you have done for me).
## Acronyms and Symbols List

<table>
<thead>
<tr>
<th>Acronym</th>
<th>Description</th>
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<tbody>
<tr>
<td>ASIC</td>
<td>Application Specific Integrated Circuits</td>
</tr>
<tr>
<td>BiCMOS</td>
<td>Bipolar Complementary Metal-Oxide Semiconductor</td>
</tr>
<tr>
<td>CMOS</td>
<td>Complementary Metal-Oxide Semiconductor</td>
</tr>
<tr>
<td>cps</td>
<td>counts per second</td>
</tr>
<tr>
<td>CRT</td>
<td>Cathode Ray Tube</td>
</tr>
<tr>
<td>CVD</td>
<td>Chemical Vapor Deposition</td>
</tr>
<tr>
<td>DSP</td>
<td>Digital Signal Processor</td>
</tr>
<tr>
<td>DUT</td>
<td>Device Under Test</td>
</tr>
<tr>
<td>FBZ</td>
<td>First Brillouin Zone</td>
</tr>
<tr>
<td>FEG</td>
<td>Field Electron Gun</td>
</tr>
<tr>
<td>FFT</td>
<td>Fast Fourier Transform</td>
</tr>
<tr>
<td>FT</td>
<td>Fourier Transform</td>
</tr>
<tr>
<td>FWHM</td>
<td>Full Width Half Maximum</td>
</tr>
<tr>
<td>GR</td>
<td>Generation/Recombination</td>
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<tr>
<td>HBT</td>
<td>Heterojunction Bipolar Transistor</td>
</tr>
<tr>
<td>Abbreviation</td>
<td>Description</td>
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<tr>
<td>--------------</td>
<td>-------------</td>
</tr>
<tr>
<td>HH</td>
<td>Heavy Holes</td>
</tr>
<tr>
<td>HRRLM</td>
<td>High Resolution Reciprocal Lattice Mapping</td>
</tr>
<tr>
<td>HMDS</td>
<td>Hexamethyldisilazane</td>
</tr>
<tr>
<td>IBM</td>
<td>International Business Machine</td>
</tr>
<tr>
<td>IC</td>
<td>Integrated Circuit</td>
</tr>
<tr>
<td>ICT</td>
<td>Information and Communication Technology</td>
</tr>
<tr>
<td>IR</td>
<td>Infra Red</td>
</tr>
<tr>
<td>I/O</td>
<td>Input/Output</td>
</tr>
<tr>
<td>KTH</td>
<td>Kungliga Tekniska Högskolan</td>
</tr>
<tr>
<td>LH</td>
<td>Light Holes</td>
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<tr>
<td>LPF</td>
<td>Low Pass Filter</td>
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<tr>
<td>LTE</td>
<td>Low Temperature Epitaxy</td>
</tr>
<tr>
<td>LTI</td>
<td>Linear Time-Invariant</td>
</tr>
<tr>
<td>MQW</td>
<td>Multi Quantum Well</td>
</tr>
<tr>
<td>PDF</td>
<td>Probability Density Function</td>
</tr>
<tr>
<td>PECVD</td>
<td>Plasma Enhanced Chemical Vapor Deposition</td>
</tr>
<tr>
<td>ppb</td>
<td>parts per billion</td>
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<tr>
<td>PSD</td>
<td>Power Spectral Density</td>
</tr>
<tr>
<td>Acronym</td>
<td>Description</td>
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<tr>
<td>PR</td>
<td>Photoresist</td>
</tr>
<tr>
<td>RF</td>
<td>Radio Frequency</td>
</tr>
<tr>
<td>RGA</td>
<td>Residual Gas Analysis</td>
</tr>
<tr>
<td>RIE</td>
<td>Reactive Ions Etching</td>
</tr>
<tr>
<td>RMS</td>
<td>Root Mean Square</td>
</tr>
<tr>
<td>RPCVD</td>
<td>Reduced Pressure Chemical Vapor Deposition</td>
</tr>
<tr>
<td>rpm</td>
<td>Revolutions per Minute</td>
</tr>
<tr>
<td>RTA</td>
<td>Rapid Thermal Anneling</td>
</tr>
<tr>
<td>sccm</td>
<td>standard cube centimeters per minute</td>
</tr>
<tr>
<td>SE</td>
<td>Shielding Effectiveness</td>
</tr>
<tr>
<td>SEM</td>
<td>Scanning Electron Microscope</td>
</tr>
<tr>
<td>SNR</td>
<td>Signal to Noise Ratio</td>
</tr>
<tr>
<td>SRH</td>
<td>Shockley-Read-Hall</td>
</tr>
<tr>
<td>SW</td>
<td>Software</td>
</tr>
<tr>
<td>TCR</td>
<td>Temperature Coefficient of Resistivity</td>
</tr>
<tr>
<td>UV</td>
<td>Ultraviolet</td>
</tr>
<tr>
<td>VLSI</td>
<td>Very Large Scale Integrated</td>
</tr>
<tr>
<td>VPE</td>
<td>Vapor Phase Epitaxy</td>
</tr>
<tr>
<td>Abbreviation</td>
<td>Description</td>
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<tr>
<td>--------------</td>
<td>---------------------------</td>
</tr>
<tr>
<td>WGN</td>
<td>White Gaussian Noise</td>
</tr>
<tr>
<td>XRD</td>
<td>X-Ray Diffraction</td>
</tr>
<tr>
<td>μP</td>
<td>microprocessor</td>
</tr>
</tbody>
</table>
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1. Introduction

The recent developments in information and communication technology (ICT) systems have led to the need of high data rates and thus to higher working frequencies for all the system components (up to 100 Gb/s). A common problem is that these signals must be received and processed at high data rates too, eventually reducing the frequencies to match typical silicon CMOS processing speeds (up to 1.5 Gb/s) which requires a certain design complexity.

To overcome this issues without recurring to the significantly more expensive III-V groups based materials, in 1992 IBM developed a competitive technology for this applications based on silicon devices with some added silicon/germanium layers. Such a structure is based on heterojunctions obtained doping the silicon lattice with germanium atoms; the SiGe layers could be grown by epitaxial deposition upon a Si substrate. The intent was to make high frequency SiGe devices compatible with all the existing silicon technologies, with particular attention to CMOS in the intent to make BiCMOS devices integrating ultra high speed bipolar SiGe transistor with CMOS Si technology on the same chip; since this technology provides high performances and low power consumption it would be suitable for applications such as wireless ones. Such a solution is also very useful for telecommunications applications due to its low noise characteristics.
2. Framework of this Study

The aim of this thesis work is based on low temperature epitaxy (LTE) of SiGe layers using trisilane and germane as the main precursors, the effect of the oxygen contamination at the interfaces or within the SiGe/Si epi-layers.

The following thesis will be organized as follows:

✓ the importance of the introduction of SiGe technology and its developments is introduced with particular attention to BiCMOS applications;

✓ the process steps needed for device fabrication along with those to obtain the desired structures;

✓ a description of the equipment used during measurements will be provided; two types of measurements will be illustrated: first a x-ray diffraction system is used to obtain the rocking curves and thus important parameters regarding the strain in the structure with and without dopants and for different contaminants' concentrations; secondarly electrical measurements were performed on the device to determine the amount of oxide islands in the device and their influence, in particular they concern noise; in conclusion a SEM analysis to determine the goodness of the produced device is done;

✓ experimental results and conclusions on the conducted experiments are provided.
During the present work all the process steps in the making of the device, developed in KTH/Acreo’s clean room located in the Electrum Lab, the measurements and the equipment setups were personally performed and followed.

This work is a part of a project started in January 2013 and developed in cooperation with Texas Instruments and.
3. SiGe Importance in Electronic Applications

3.1. SiGe material properties

The boundaries of the material bandgap varies in a range contained between the bandgaps of germanium and silicon (0.66÷1.12 eV) and it is dependent upon the percentage amount of the latter. The SiGe material shows however an indirect bandgap since both silicon and germanium has the peculiarity of having the maximum of the valence band and the minimum of the conduction one are not at the same wavenumber, $k$, as shown in Figure 2.1.

![Figure 3.1 - Germanium and silicon bandstructures.](image)

Figure 3.1 - Germanium and silicon bandstructures. On the graph there are the significant point:
- $\Gamma$: center of the FBZ ($k = (0,0,0)$);
- $X, L$: boundaries along certain directions in three dimensional k-space (the directions chosen in this case are only an example) [1]
In this project Si/SiGe MQW structures have been studied in which SiGe layers showing a percentage amount of germanium approximately 24%. It is possible to calculate the value of the energy bandgap for this type of material, considering the fact that SiGe layer is subjected to strain and that the percentage amount of Ge is less than 30%, it is possible to calculate the value of the energy bandgap using the empirical formula [2]

$$E_{g}^{strain} = 1.107 - 0.896x - 0.396x^2$$  \hspace{1cm} (3.1)

where $x$ is the percentage amount of Ge in the alloy.

The strain due to the unrelaxed structure must be taken into account; this is related to a shift in Light Holes (LH) and Heavy Holes (HH) energetic levels that leads to higher carrier mobilities in the MQW structure.

### 3.2. Application Field

Some applications for transistors are explored in this part.

#### 3.2.1 Heterojunction Bipolar Transistors

A reason to develop a heterojunction bipolar transistor (HBT) were inherent to the scaling down of the devices' dimensions. In fact during this process several considerations have to be done in order to have a correct device behavior.

First, during the scaling, the base width is reduced leading to an increase of the base doping concentration to avoid punchthrough problems and control the base resistance value; meanwhile the emitter doping level has to be high to ensure a sufficient injection of carriers from the emitter into the base and, knowing that the junction capacitance of emitter-base junction is given by
where $S$ is the junction area, $q$ is the elementary electrical charge, $\varepsilon_{\text{Si}}$ is silicon dielectric constant, $N_A$ and $N_D$ are the doping concentrations of acceptors and donors respectively, $\phi_f$ is the built in voltage and $V_D$ is the forward applied voltage, it is possible to predict an increase of the capacitance value due to the increase in the doping concentration. This higher capacitance value leads to an increase in the current value not to suffer extra delay problems; thus, in order to have a higher current, without suffering the Kirk effect, also the collector doping must be increased too. Summarizing, to obtain a higher cutoff frequency without using heterojunction bipolar transistors, a smaller current operating range and moreover high doping concentrations, leading to often unacceptable lower breakdown voltages, must be accepted. The simple scaling of silicon technologies is thus not satisfactory in most of applications.

Using HBTs instead it is possible to use different bandgap materials for the base, the collector and the emitter. Using different materials it is possible to vary the speed of the carriers, obtaining, for the same dimensions of the silicon technology, higher cutoff frequencies. In fact the speed of a bipolar transistor is mainly due to the passing time of a carrier in the base and to the time needed to charge and discharge the junction capacitances. Thus, in order to achieve better performances, usually thinner base region layers, higher charge carriers velocity and higher current densities are desired; all this characteristics could be achieved using HBT technology.

The technique is used because the emitter injection efficiency is related to the materials bandgap difference via the equation [3]
with $n_e$ electron concentration in the emitter region, $p_b$ hole concentration in the base region, $\nu_e$ is the effective velocity of electrons injected in the base from the emitter, $\nu_h$ is the effective velocity of the holes injected in the emitter from the base, $\Delta E_g$ is the difference between the bandgaps of the two materials, $k_B$ is the Boltzmann constant and $T$ is the absolute temperature.

In SiGe case a smaller bandgap material is used for the base. In particular different solutions for different problems can be used, e.g. it is possible to create narrow bandgap bases with low sheet resistance and lower junction capacitances to achieve very high frequencies or to have the same silicon technology performances in frequency. The advantage are a higher base doping to obtain a lower base sheet resistance or having a thicker base less doped to obtain higher breakdown voltages; among the other advantages also an higher Early voltage is achieved.

Thanks to the fact that the processes are very similar to those of silicon technology, SiGe technology has been very successful; in fact it is not necessary to change the equipment or the procedures, but only to adapt them, since the deposition and etching processes follow the same steps as in silicon based devices.

### 3.4. BiCMOS Technology

As reported before BiCMOS technology successes in the integration of HBT and silicon CMOS on the same chip.
This solution offers several advantages at a lower cost (compared with other technologies capable to increase the cutoff frequency and the device speed).

This technology finds application in both analog and digital circuit technologies.

### 3.4.1. Digital Applications

BiCMOS are very attractive for integrated circuits since it is possible to use the CMOS part of the circuit to develop the logic functions, having low power consumption, while developing the I/O circuits with HBT circuits, having high speeds, due to low time constants, to charge and discharge the I/O loads. Moreover this kind of logic shows a larger fanout than CMOS one.

Obviously the presence of HBTs produces an increase in terms of used area, but the percentage increase of area tends to decline for high numbers of CMOS in the structure; for this reason BiCMOS logics finds applications in gate arrays, that is to say in large cells, such as adders and shift registers. Other applications are SRAMs in which BiCMOS technology offers a power dissipation and a density similar to that of CMOS with higher speeds of the interface driver circuits.

### 3.4.2. Analog Applications

Analog applications for BiCMOS are particularly related to the integration on the same chip of analog and digital functions. In particular, often ASICs are developed using the CMOS technology while HBTs perform the analog interfacing with external systems.

The focus of this project has been centered on another analog application; the structures that were made during the processing could be in fact, with further developments, used as IR
detectors. Si/SiGe single crystalline MQWs structures has in fact been proven to be a good thermistor materials [4]. The reason to study these type of sensor is that it succeeds in obtaining a low 1/f noise (also known as pink noise) that is the main noise source in such type of detectors. In this type of bolometers the noise is related to the layers quality, their roughness and contact resistance.

### 3.4.2.1. Si/SiGe Bolometer

This type of material can be used as a thermistor because of the resistance variation of the Si/SiGe multilayers. This variation can be quantified via the TCR (Temperature Coefficient of Resistance) [4]:

\[ \beta = \frac{1}{R} \frac{\partial R}{\partial T} \]  

(3.4)

that, considering the typical semiconductor equation, can be rearranged to give:

\[ \beta = -\frac{1}{k_B T^2} (\frac{3}{2} k_B T + E_f - V) \]  

(3.5)

where \( k_B \) is the Boltzmann constant, \( T \) is the absolute temperature, \( E_f \) is the Fermi level of the considered material and \( V \) is the barrier energy.

To develop a bolometer structure usually a multi quantum well (MQW) stack of at least three layers is grown, since increasing the number of layers leads to an increase in the intersubband transitions in the valence band and thus to an increase of the TCR value [5].

### 3.5. Strained CMOS Applications

#### 3.5.1. Strain
Strain represents the relative change in shape or size of an object due to the application of an external force. The concept is related to that of stress, that is the internal force associated with the strain.

Strain could be either tensile or compressive; the former tends to measure the resistance of a material to a longitudinal stress without breaking, while the latter measures the ability of the material to resist to oppositely directed collinear forces that tends to crush the object under test.

![Figure 3.2 - Schematics of tensile and compressive strain applied to a structure](image)

**3.5.2. Use of the Strain in CMOS Technology**

The strain is generated when SiGe has been used as stressor material to induce strain in the channel region. Strain engineering is mostly directed to source/drain in MOSFETs. Figure 2.3 shows the process to generate strain in the transistor structures. Two types of strain design are realized: biaxial and uniaxial.
Experimental data [6] demonstrate that the uniaxial strain enhances the carriers' mobility. In particular, uniaxial compressive strain causes a large enhancement in hole mobility for low stresses.
For that reasons this kind of technology is also interesting for CMOS applications, since the mobility of the carriers in the inversion layer is strongly dependent on the applied electric field [6].

Figure 3.3 - Strained Si hole mobility vs. vertical electric field [6]
4. Processing

In this chapter processes needed for the construction of the device under test will be depicted. Most of the reported information about the processing have been taken from [7]. All the processing part has been performed on Si/SiGe structures with SiGe layers' thicknesses under the critical thickness of 60 nm (i.e. the thickness at which a relaxation in the layer could be seen) and in particular of 10 nm. Moreover intrinsic SiGe layers were considered and a low oxygen contamination (60 ppb) since the aim of this work is to study contaminations effect and not oxidation. Particular attention has also been paid to contacts' resistivity to obtain low values of this parameter using three layers contacts (NiSi/TiW/Al). These considerations allow to state that the noise in the structure produced from parameters not related to the contaminations is reduced and thus to have good noise measurements.

The processing has been performed on four wafers with different oxygen contamination (prior or during the epitaxial growth) and different HCl contamination of 80 mtorr.

Table 4.1 - Summary of processed wafers

<table>
<thead>
<tr>
<th>Wafer Recipe</th>
<th>Oxygen contamination (60 ppb)</th>
<th>HCl contamination</th>
</tr>
</thead>
<tbody>
<tr>
<td>0TIQWHCLO60DF</td>
<td>√ during</td>
<td>√</td>
</tr>
<tr>
<td>0TIQWSGO60DF</td>
<td>√ during</td>
<td>x</td>
</tr>
<tr>
<td>0TIQWHCLO60PF</td>
<td>√ prior</td>
<td>√</td>
</tr>
<tr>
<td>0TIQWSGO60PF</td>
<td>√ prior</td>
<td>x</td>
</tr>
</tbody>
</table>
4.1. Samples' Preliminary Cleaning

The process always starts with the wafers' cleaning; in fact virgin wafers need to undergo through chemical cleaning in order to remove the native oxide, an oxide that is formed on the top of the wafers when they are exposed to air and that typically presents a thickness of 1 nm, and make the silicon substrate available for further processes.

The cleaning involves the use of two acid solutions and it is so organized:

- Piranha solution (also known as 7-up solution) is prepared: it consists in 3 parts of sulfuric acid ($H_2SO_4$) and one part of hydrogen peroxide ($H_2O_2$); wafers are then immersed in the solution for 5 minutes. Piranha solution is useful to remove organic residues and to hydroxylate the surfaces. Two different reactions take place: the first reaction is dehydration

  \[ H_2SO_4 + H_2O_2 \rightarrow H_2SO_5 + H_2O \]

  that is a strongly favorable thermodynamically reaction and makes it dangerous to handle; the second reaction

  \[ 2H_2SO_4 + 2H_2O_2 \rightarrow 2H_3O^+ + 2HSO_4^- + O_2 \]

  presents strongly reactive species that success in breaking highly stable carbon or silicon hybridized bonds;

- wafers are immersed for 5 minutes in distilled water to dissolve the previously created hydrophilic groups;

- wafers are immersed for 10 seconds in a 5% hydrofluoric acid (HF) solution which provides good removal of the oxide through the reaction

  \[ SiO_2 + 4 HF \rightarrow SiF_4(g) + 2H_2O; \]

- wafers are immersed for 5 minutes in distilled water to remove reaction products;
wafers are put in the drier.

Cleaning of others compounds could be done via different techniques such as ultrasonic scrubbing or in situ etching sputtering to remove the particulates.

4.2. Epitaxial Growth

Wafers are then weighted and put in the reactor for the epitaxial growth. Every wafer is identified through an ID number written on the back for further operations.

Epitaxial growth is used to grow a single crystal layer on a single crystal substrate replicating the substrate periodicity, obtaining an energetically favorable process. Two types of epitaxy could be used for different purposes:

- if the film and the layer are made of the same material the process is called homoepitaxy;
- if the film and the layer are made of different materials the process is called heteroepitaxy;

in this project both techniques have been used, in particular heteroepitaxial growth since the aim is to build a multilayer SiGe/Si structure. Moreover different deposition types could be used from various matter phases; in what follows vapor phase epitaxy (VPE) technique is treated. Several VPE techniques have been developed, but, in particular for industrial applications, reduced pressure chemical vapor deposition (RPCVD) has become the most utilized thanks to a good tradeoff between the deposition rate and the film quality.

Several steps take place in an epitaxial growth process: first reactant gases, dopants and inert diluents (such as $H_2$) are put in the chamber; reactants are then carried to the substrate surface and adsorbed on it; the chemical reaction takes place forming the film and other reaction products which then have to be deadsorbed and carried away from the surface.
The major advantages of this technique, compared to others, are the low presence of contaminants that can be achieved, an accurately controlled doping concentration, the possibility to obtain an abrupt doping profile and to perform the process at low temperatures. On the other hand the main disadvantages are the auto-doping (limited by low temperature processing), the pattern shift and an increased processing complexity that results in increased costs.

4.2.1. Atomistic Model

A simple model to explain epitaxial growth is the atomistic one in which the film grows thanks to the migration of adatoms to steps on the substrate surface; the step position is indeed the most energetically favorable since two of the four chemical bonds of the species are already formed with the crystal. The value of the maximum growth rate depends upon the
temperature; that parameter has great importance because, if at a given temperature maximum growth rate is exceeded, polycrystalline growth instead of a crystalline one will occur, nullifying epitaxial deposition advantages. This could be explained in such a model as follows: when the maximum growth rate is exceeded adatoms have not got the time to migrate to the most energetically favorable spot resulting in polycrystalline growth; a way to overcome this problem (i.e. to increase the growth rate) is to increase temperature, thus increasing the surface migration rate and letting the adatoms reach the favorable sites; the temperature cannot be increased over a certain limit since one of the advantages of epitaxial growing process is to operate at low temperatures.

Figure 4.2 - Maximum growth rate for of single crystalline silicon formation as a function of the temperature [7]

Varying the temperature instead of the growth rate it is possible to obtain the dependence of the latter from the former and to recognize two different behaviors: as it could be seen from Arrhenius plot in Figure 2, at high temperatures growth rate is approximately constant and so gas phase mass control is the dominant process, while at low temperatures growth rate is
directly proportional to temperature because it is linked to the kinetic of surface reactions, also proportional to temperature.

![Figure 4.3 - Growth rate of silicon film from various silicon sources as a function of the temperature [7]](image)

Moreover, reactions can take place either distant from the surface or near it with the latter process preferable to the former because it shows better adhesion, higher density of the film and thus lower defects concentrations.

**4.2.2. Epitaxial Growth Model**

Usually epitaxial growth process is described as formed by five sub processes:

1. the reactants are transported to the substrate surface;
2. the reactants are adsorbed on the substrate surface;
3. the chemical reaction needed to the film formation takes place on the surface producing the film and other reaction products;
4. the reaction products are desorbed from the surface;
5. the reaction products are carried away from the surface.
Grove’s epitaxial growth model does not take into account all the five steps but only on the first three. The model assumes a flux of the reactants from the bulk of the gas to the surface, \(F_1\), and a second flux, \(F_2\), representing the consumption of the reactants at the surface. The two fluxes are approximated as

\[
F_1 = h_g (C_g - C_s) \tag{4.1}
\]

\[
F_2 = k_s C_s \tag{4.2}
\]

where \(h_g\) is the gas phase mass transfer coefficient, \(C_g\) is the reactants concentration in the bulk of the gas, \(C_s\) is the concentration at the surface and \(k_s\) is the surface reaction rate constant. In steady state conditions it is possible to equal the two fluxes and thus it is possible to write that

\[
C_s = \frac{c_g}{1 + \frac{k_s}{h_g}} \tag{4.3}
\]

and the process could be reaction controlled (in the case that \(k_s \ll h_g\) and \(C_s \cong C_g\)) or mass transfer controlled (in the case that \(k_s \gg h_g\) and \(C_s \rightarrow 0\)).

The growth rate is then given by

\[
V = \frac{F}{N_1} = \frac{k_s h_g C_g}{k_s + h_g N_1} \tag{4.4}
\]

where \(N_1\) is the number of atoms of SiGe in a unit volume of the film. Now, writing \(C_g = C_T Y\) with \(C_T\) the total number of molecules in the gas per cm\(^3\) and \(Y\) the mole fraction of the reaction species, it is possible to say that the growth rate increases increasing the concentration of reactants in the gas phase. However this is true for low \(Y\) values, since for
higher values an etching process will occur, due to the fact that \( HCl \) is one of the reaction products.

### 4.2.3. Chemical Reactions and Compounds Used

For silicon deposition silane (\( SiH_4 \)) precursor is usually used to form the film, following the reactions [9]

\[
\begin{align*}
SiH_4 + 2_- & \leftrightarrow SiH_3 + H \\
SiH_3 + - & \leftrightarrow SiH_2 + H \\
2SiH_2 & \leftrightarrow H_2 + 2SiH \\
2SiH & \leftrightarrow H_2 + 2Si + 2-
\end{align*}
\]

where ‘_’ symbolize an incorporation site on the surface. In this project however trisilane, another compound of silane group, has been used. Trisilane has a structure similar to propane’s one and it is entered in the chamber in liquid state. Trisilane decomposition reaction could be presented as

![Trisilane molecular structure](image)

\[
\begin{align*}
Si_3H_9 + - & \leftrightarrow Si_2H_6 + SiH_2 \\
Si_2H_6 + 2Si + 2- & \leftrightarrow 2SiH_3 + 2Si \\
SiH_3 + Si & \leftrightarrow SiH_2 + SiH \\
SiH_2 + Si & \leftrightarrow 2SiH \\
2SiH & \leftrightarrow 2Si + H_2 + 2-
\end{align*}
\]

Trisilane is preferred to silane because it allows to have higher growth rates at lower temperatures and also because, since it is introduced in the chamber as a liquid, it is possible to avoid contaminations at the same time and have a purer compound using the fractional
distillation technique. For these reasons it has become the standard choice together with germane for the epitaxial growth of SiGe films. However the typical silicon epitaxial growth processes cannot happen at temperatures below 420 °C. Below that temperature the absorption of H₂ in the film takes place degrading the film quality. Particular attention must be paid using trisilane because it is a pyrophoric material, that is to say that it spontaneously ignites in air.

For germanium deposition it is possible to choose among two different compounds:

 ✓ digermane (Ge₂H₆) could be a good solution but it is expansive and often not immediately available;

 ✓ germane (GeH₄), on the contrary, represents a cheap solution and it is very easy to find it on the market.

For the previous reasons the latter compound is preferred to the former and it is the one considered in the following. The presence of germanium in the chamber allows to carry on the SiGe epitaxial growth process at lower temperatures, of approximately 300 °C, since it helps the H₂ desorption filling the dangling bonds. Germane is always entered in the chamber together with HCl to form the film and meanwhile regulate the percentage amount of germanium in it, since the acid preferably etches silicon atoms over germanium ones. Germane is also a flammable and toxic gas so, as for trisilane, particular attention must be paid during processing to clean room safety. The process, similar to silane's one, follows the reactions
Different lattice constants, atomic densities and bandgaps are found varying the percentage amount of germanium and silicon in the structure (that crystallizes in a sphalerite lattice structure). The percentage amount of germanium in the structure could be adjusted introducing more or less hydrochloric acid ($HCl$), varying its partial pressure in the chamber. It is thus possible to our purposes to obtain $SiGe$ films with a percentage amount of germanium between 13% and 26%.

For the purpose of the project, wafers have been also exposed to oxygen contamination at different temperatures and different oxygen partial pressures before and during the $SiGe$ epitaxial growth.

Doping of the films is performed using boron and phosphorus for a $p$- and $n$- doped film, respectively.
4.2.4. Some Issues in Epitaxial Growth

Sometimes after the film growth it could be found that:

- the pattern has been shifted from the original pattern on the substrate;
- the pattern has been distorted from the original pattern on the substrate;
- the pattern is not reproduced at all on the film (washout).

Such effects depend on substrate crystallographic orientation, growth rate, temperature, gas precursors and pressure involved in processing. It was empirically demonstrated that the use of silanes without chlorine reduces pattern shift and distortion, while the presence in the deposition chamber of $Cl_2$ or $HCl$ tends induce the shift. For this purpose also reactor's architecture is an influential parameter.
LTE has got some advantages compared to traditional epitaxial techniques. In fact it avoids strain relaxation of the layers, it limits dopant outdiffusion and the thermal mismatch in the considered structure. Those features are of particular importance in BiCMOS applications since in the CMOS the gate oxide, the poly gate and thin gate oxide layer suffer the thermal mismatch, while in the HBT a boron outdiffusion could arise and moreover a SiGe strain relaxation is not desirable.

At the same time in LTE issues relative to the precursor gas quality could arise, leading to oxygen ($O_2$) and water ($H_2O$) contaminations. These contaminants cannot come from the carrier gas ($H_2$) or from the HCl, since these two gases have dedicated purifier systems; as been explained before the trisilane source is preferred to others also for its purity, thus the contaminations do not come from it either. It is possible to state that the possible contamination sources are represented by the chamber handling, load-locks purging and impurities in the germanium gas or in the carrier gas. These impurities could be incorporated at the interface between the layer and the substrate, inside the layer itself, or both at the interfaces and in the layer.

4.4. Photolithography

Photolithographic process consists in a large number of sub processes to create the patterns defining the structure that is intended to make. The sub processes involve a photoresist (PR) coating, a selective exposition of the same through an appropriate masking, its development and finally an etching process to remove the undesired layer's zones and the resist.
4.4.1. Photoresist

Photoresist is a material which may dissociate after exposing to light (e.g. UV, visible light or X-rays). Photoresist material could be one of two types:

- **positive photoresist**: when the exposed regions are made soluble and then a positive image of the mask is produced on the resist;
- **negative photoresist**: when the non-exposed regions are dissolved by the developer and a negative image of the mask is produced.

The positive PR higher resolution compared with the negative's one has made it the standard choice for VLSI applications.

Thus the PR achieves two goals: replicating the mask image on the underlying layer and protecting the other areas of the same layer. To perform this and other tasks PR material should possess certain properties.
4.4.1.1. Photoresist Parameters

✓ Resolution: it specifies the process' ability to print minimum size images under conditions of reasonable manufacturing variations. A lot of limitations on resolution can occur: hardware limitations (e.g. light diffraction, lens aberrations, system's mechanical stability); material limitations (since the resolution is influenced by the resist composition); process characteristics (e.g. softbake and postbake processes, development and etching);

![Figure 4.2 - Photolithographic process example with positive PR](image)

✓ Sensitivity: input energy required to cause a specified degree of chemical response in the resist. For photochemical reactions a parameter known as a photoefficiency is defined:

\[
\Phi = \frac{\text{# of photo-induced events}}{\text{# of photons absorbed}}
\]  

(5.1)
a high sensitivity is desired but if this parameter is too high resist may undergo thermal reactions at room temperature, thus having a too short shelf life and showing exposure uniformity control problems;
- **Etch resistance and thermal stability:** PR has to show some resistance to wet and dry etching processes to protect the substrate from etching itself and a certain stability to process’ temperature variations;
- **Adhesion:** to increase adhesion, that is fundamental also in wet etching processes, pre and post deposition baking cycles are used together with adhesion promoters like HMDS;
- **Viscosity:** it is a key parameter to determine the thickness of the resist coating together with spin speed.

### 4.4.1.2. Photoresist Processing

The wafers have to undergo to a dehydration baking and to a priming (in the latter process HMDS is often the standard choice) in order to improve PR adhesion to the wafer.

Wafers are then ready to be coated with resist producing a uniform, adherent and defect free polymeric film of a certain thickness over the entire wafer. To do so a spin coating technique is used to produce resist films with uniformities across the wafer of $\pm 100 \text{ Å}$.

After the coating the wafers are subjected to a temperature step to drive off solvents from the resist, improve its adhesion and anneal the stresses caused by shear forces present in the spinning process.

Exposure is one of the most important steps in photolithography. In this process photochemical transformations occur within the resist. Parameters such as the resist film thickness are crucial in this process since it determines the time of exposure and the process reproducibility.
The last process related to PR is its development to create the image on the wafer which will serve as a pattern for subsequent processes. In our processes a single wafer spray development system has been used in which the developer is sprayed on the spinning wafer. Usually to increase PR's resistance to etchants also a post baking step is performed.

After, the development regions of the layer not covered by resist anymore could be removed together with the left photoresist by etching processes, replicating the mask pattern on the layer.

In the process a positive PR with a thickness of 1.05 μm has been considered (5000 rpm), deposed with a Maximus 804 model tool.

4.4.2. Optical Aligners and Masking

Machines used to impress the resist are composed of different systems to illuminate the PR and provide it the necessary energy for the resist transformation, to focus the circuit patterns and allow a controlled exposure time; moreover they use a moving system to move the wafer and to align the tool to the previously printed patterns on the wafer.

Three methods which realize the transfer of patterns on PR exist: contact printing, proximity printing and projection printing. In this work, the projection printing has been used. In projection printing a lenses system is used to transfer the mask image on the photoresist.
Masks are usually made of transparent fused quartz with the pattern defined by a chrome metal absorbing film. Two pattern tools are utilized in IC processing: reticles and masks. The former contains a pattern image that has to be stepped and repeated to expose the entire wafer and it found large application in step and repeat processing; the latter is useful to transfer pattern to the whole wafer in one exposure.

Particular attention must be also paid to alignment marks; in fact they are critical since usually a lot of different processes have to be done on a single wafer to develop a single structure and thus not having an alignment system could result in unacceptable devices at the end of the processing. Different auto align systems have been developed to this purpose.

The exposition and masking processes are conducted in a XLS-Stepper machine which characteristics are summarized in the following table.
<table>
<thead>
<tr>
<th>Feature</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Exposure wavelength</td>
<td>365 nm</td>
</tr>
<tr>
<td>Magnification</td>
<td>1/5 X</td>
</tr>
<tr>
<td>Resolution</td>
<td>0.65 µm</td>
</tr>
<tr>
<td>Depth of Focus</td>
<td>1.25 µm</td>
</tr>
</tbody>
</table>

### 4.5. Etching

Etching is in general a process in which part of the material is removed from the substrate; this section is particularly focused on post photolithography etching that usually takes place after PR developing to reproduce the needed pattern, precisely removing the undesired material. The most important parameter in etching processes is the etch rate that specifies the rate at which the material is removed from the substrate. It is good to have high etch rates since it will result in higher throughputs; on the other hand it is impossible to efficiently control too high etch rates and so a trade-off is usually found. Etching could also be characterized by being isotropic, when it exhibits the same etch rates in all directions, or more or less anisotropic, in the case it etches along preferable directions; it is completely anisotropic in the case it proceeds only in one direction.

#### 4.5.1. Wet Etching

Wet etching is an isotropic process so it is not adequate to build structures that involves features less than 3 µm wide; nevertheless this kind of processes are largely used because not every process involves the dimension specified above and they are reliable, high throughput
and low cost processes. A wet etching process could be subdivided in three sub processes: diffusion of reactant to the reacting surface; reaction; diffusion of reaction products from the surface. Obviously the slowest process will influence the reaction dynamics and will control the etch rate. In the present project dry etching is used particularly for native oxide etching that does not require particular attention in micrometric and sub micrometric dimensions; thus the processes considered above for wafers' cleaning are also wet etching processes.

**4.5.2. Dry Etching**

The main reason to apply dry etching, as said before, was the possibility to develop anisotropic etching and then to create nano-scaled features; this process also uses smaller amount of chemicals if compared to wet etching technique.

![Dry etching techniques](image)

Figure 4.12 - Dry etching techniques [7]
A plasma is defined as a partially ionized gas composed of electrons, ions and other neutral species; when a plasma is maintained in a particular pressure range [1÷750 Pa] a glow discharge is formed, in which it is approximately possible to say to have the same concentration of negative and positive ions. Basically reactive species needed to the process are generated from the inert molecular gas and from the etchant gases. The reactive species are so generated in the plasma and they are then diffused to the surface of the material to produce the etching chemical reaction; products from this reaction have to be volatile to be then diffused into the bulk of the gas. Thus thanks to plasma etching it is possible to develop systems capable of etching the surface bombarding it with energetic ions and at the same time producing, via the gas glow discharge, reactive species for chemical etching. Reactions occurring in the plasma are called homogeneous reactions, while the ones occurring at the surface are called heterogeneous reactions.

A plasma is generated using an oscillating RF sources with a frequency of 13.56 MHz or 100 kHz; that source partially ionizes the gas, until it reaches the plasma state.

Due to the fact that plasma etching has an isotropic component that would be a problem in the case of high mesa walls etching, another technique has been developed. This technique known as RIE (Reactive Ions Etching) use both ionic bombardment and chemical etching processes to etch the material. Obviously, since two different types of etching occur, a lot of different parameters are involved in the process and thus particular attention must be paid to those in order to have a reproducible and useful process. The two processes are essential in this case since the ion bombardment ensures the anisotropic behavior while the chemical etching guarantees the selectivity of the process.
In this technique the ion direction is directed towards the wafer and a higher energy is used in the process. As shown in Figure [4.13], the potential distribution changes varying the electrodes' areas; moreover in the case of asymmetric electrodes, i.e. in the case of one electrode is smaller than the other, it is possible to establish that, due to electrons' and ions' mobility disparity, the anode will tend to be more negatively biased. Following the developed considerations it is convenient to put the wafers on the anode because the voltage drop will make the ions acquire more energy leading to a better etching process. To understand why the potential distribution is like that it is possible to develop the following reasoning: in an asymmetric electrode system, the smaller electrode will have a higher RF electron density due to the fact that the current has to be constant between the electrodes and the carriers at the cathode are slower than the ones at the anode, leading to a more negative potential (the smaller it is the electrode, the bigger it will be the potential in absolute value).

![Figure 4.13 - Potential distribution in a parallel plate plasma etcher with a grounded electrode](image)

The voltage used is in a range of [100÷700] V with a pressure of [10÷100] mtorr to obtain an even more directional etching. The fact that ions are sensitive to the atomic bonds forces rather than on the composition of the substrate guarantees a good anisotropic behavior.
Moreover the plasma etching does not degrade the PR adhesion as some wet etching processes do.

To perform dry etching it is possible to use barrel etchers; in the project a P5000 tool implementing this technology has been used. In this type of machine the wafers are held by a support in a vertical position and are positioned in a metal cage.

Because of the difficulties in process' to control peculiar monitoring equipment must be used with particular attention to endpoint detection tools that allows process' reproducibility, limiting overetching issues and increasing process' throughput. Four methods are commonly used to determine the endpoint: laser interferometry, optical emission spectroscopy, direct observation by an operator, mass spectroscopy; among them the most widely used is optical emission spectroscopy because its implementation is quite easy, shows high sensitivity and provides information about both etching species and products. This information must be taken into account to decide if the etching action has to be terminated before its assigned time.

In this work dry etching and in particular plasma etching is used for the definition of the mesa structures and thus for Si and SiGe etching and moreover for metal etching after making the contacts and also to remove passivation SiO₂. A list of dry etching processes for various purposes is reported in the table below.

<table>
<thead>
<tr>
<th>Compound to etch</th>
<th>Compounds used in the process</th>
<th>Time [s]</th>
<th>Rate [nm/min]</th>
<th>Endpoint Overetch</th>
</tr>
</thead>
<tbody>
<tr>
<td>Si / SiGe</td>
<td>Cl₂</td>
<td>10 sccm</td>
<td>300</td>
<td>400</td>
</tr>
<tr>
<td></td>
<td>HBr</td>
<td>30 sccm</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>He</td>
<td>10 sccm</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Passivation process is crucial in VLSI technologies because the oxide manages to mask the underlying layers from chemical and mechanical damages. Thickness of the passivation layer is usually the main interest parameter since good passivation is achieved increasing it; however the thickness value cannot become too high due to the next metallization step and to the tendency of thicker films to easily crack. For this reason a trade-off is needed to be found for any specific application.

Other passivation layer's purposes are to prevent corrosion, to exhibit low and compressive stress, to be impermeable to high diffusing impurities and to have good adhesion and thickness uniformity in addition to provide good step coverage.

\( SiO_2 \) is used as insulator to passivate the structure; in this project no oxidation process was used since this type of processes tend to grow the oxide reducing \( Si \) layer thickness. For this

<table>
<thead>
<tr>
<th>Layer</th>
<th>( CHF_3 )</th>
<th>( CF_4 )</th>
<th>( Ar )</th>
<th>( BCl_3 )</th>
<th>( Cl_2 )</th>
<th>( N_2 )</th>
<th>100</th>
<th>150</th>
<th>-</th>
</tr>
</thead>
<tbody>
<tr>
<td>( SiO_2 )</td>
<td>15 sccm</td>
<td>5 sccm</td>
<td>50 sccm</td>
<td>40 sccm</td>
<td>30 sccm</td>
<td>40 sccm</td>
<td></td>
<td></td>
<td>-</td>
</tr>
<tr>
<td>( Al )</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>20%</td>
</tr>
<tr>
<td>( TiW )</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>15%</td>
</tr>
</tbody>
</table>
reason PECVD at low temperature (300°C) and low pressure was used instead, depositing the material according to the reaction:

$$SiH_4 + O_2 \rightarrow SiO_2 + 2H_2.$$  

This kind of reaction takes also place before the PR deposition to protect the SiGe layer during etching; the $SiO_2$ is then entirely removed and then deposited again to have a good control of the insulator thickness.

The passivation step is carried on in a Plasmalab 80Plus (Oxford PECVD System) Chamber A (Pekka) with a processing time per wafer of 210 s at a temperature of 300 °C, obtaining a growth rate of 65.54 nm/min.

4.7. Metal Contacts

Where a contact is needed in the structure an opening in the isolation film must be provided in order to ensure the contact itself. Processes needed to perform the opening in the passivation layer are photolithographic and etching steps similar to those described above.

Aluminum is the most used metal in IC contact making but establishing ohmic contact between silicon and aluminum, showing controlled specific contact resistivity is not a simple task. This is due to the fact that Al is also a silicon n dopant and thus the contact's parameters strongly depend on the underlying Si layer's doping and on surface's conditions. Obviously such a situation is not acceptable because of an intrinsic lack of reproducibility in the process. To overcome this problem in the project a multilayer ohmic contact structure to silicon has been used forming the contact through three different layers.
The first contact layer used is formed by nickel silicide (NiSi) to obtain low contact resistance. The silicide film is formed from a thin Ni film deposited over the silicon layer and treated by RTA. The NiSi film is formed by consuming the underlying silicon layer; the process to realize it is known as salicidation (self aligned silicidation). Compared to other compounds NiSi shows low consumption of silicon; in fact all the deposited nickel approximately reacts with silicon so that the amount of consumed Si is determined by the deposited Ni amount. The Ni silicide material shows low resistivity (~14 μΩ·cm), that helps reducing Johnson noise, low formation temperature (450°C) and a good thermal stability range (450–700 °C) [10].

The other two layers are made of an aluminum layer on top of a titanium/tungsten (TiW) layer; both of them are deposited using a sputtering technique and ensure low resistivity (TiW: 60–90 μΩ·cm; Al: 2.65 μΩ·cm).

The thicknesses obtained in the process for the various materials composing the contact are

Table 4.4 - Metal contacts thicknesses

<table>
<thead>
<tr>
<th>Material</th>
<th>Thickness [nm]</th>
</tr>
</thead>
<tbody>
<tr>
<td>NiSi</td>
<td>12</td>
</tr>
<tr>
<td>Al</td>
<td>700</td>
</tr>
<tr>
<td>TiW</td>
<td>50</td>
</tr>
</tbody>
</table>
4.8. Processing steps

Having described all the basically needed processes to perform a complete structure it is now possible to describe the required structure in detail and understand how it is possible to build it on a wafer using the above processing.

A Si/SiGe mesa multi-layer structure has been chosen since, how the electrical transport through it may reveal the presence of defects or contamination in SiGe layers [11]. Figure 4.6 shows a schematic view of how mesas were processed and contacts were formed.

Figure 4.14 - Processing steps: (a) epitaxial growth of the layers by RPCVD; (b) lithography; (c) formation of the mesa structure by dry etching; (d) PR removal; (e) SiO₂ deposition; (f) contact lithography and etching; (g) metal deposition; (h) contact pads formation; schematic of where to connect the probes [12]
On every die of the wafer contains four arrays of mesas: 25x25\(\mu\)m\(^2\), 50x50\(\mu\)m\(^2\), 100x100\(\mu\)m\(^2\) and 200x200\(\mu\)m\(^2\). The different structures were developed to have several testing structures and also to further evaluate the influence of the structure's dimensions on the electrical characteristics of the device.
5. Noise Measurements

5.1. Measurement Setup

Noise measurement is a sensitive electrical characterization method which can provide information about the presence of defects and impurity in the epi-layers. For that reason during the years different ways to measure noises have been developed.

The first problem to solve dealing with noise is the one of being able to recognize the noise from the signal; in fact usually noise voltage amplitudes have values from pV to nV and are then very difficult to separate from the signal to directly measure them. For these reasons ad hoc systems are used to obtain the signals' spectra and hence the noise characterization of the device. Thus measurements are performed in the frequency domain using a spectrum analyzer and a noise amplifier to increase the noise amplitude and make it detectable. All the system is also shielded from external noises such that it is possible to study only the parameters arising from the device itself.

The measurements are all performed using a shielded Cascade 11000 probe station with temperature controlled chuck in which it is also possible to do laser interferometric measurements to determine the sample’s temperature.

5.2. Low Frequency Noise Measurement Bias Network

The DUT is inserted in a bias circuit useful to bias the device at a certain voltage. This is done using the network shown in Figure 5.10.
 Basically the network is composed by a 2V battery and variable resistors that, varying their resistance values, can vary the voltage value on the DUT. A voltmeter is also present in the circuit to monitor the interest voltages in the same; through a switch it is possible to measure the voltages $V_d$ and $V_{dut}$, a voltage reference, $V_{ref}$, is also present in the network. The capacitance parallel to the DUT is useful to stabilize the voltage on the same in case of fluctuations.

The resistance values that the various resistors can assume are reported in Table 5.1.
Table 5.1 - Bias circuit resistances' possible values

<table>
<thead>
<tr>
<th>Pos.</th>
<th>R1 [Ω]</th>
<th>R2 [Ω]</th>
<th>R3 [Ω]</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>49,9</td>
<td>49,9</td>
<td>0</td>
</tr>
<tr>
<td>2</td>
<td>75</td>
<td>75</td>
<td>49,9</td>
</tr>
<tr>
<td>3</td>
<td>100</td>
<td>100</td>
<td>100</td>
</tr>
<tr>
<td>4</td>
<td>124</td>
<td>124</td>
<td>200</td>
</tr>
<tr>
<td>5</td>
<td>150</td>
<td>150</td>
<td>499</td>
</tr>
<tr>
<td>6</td>
<td>200</td>
<td>200</td>
<td>1k</td>
</tr>
<tr>
<td>7</td>
<td>499</td>
<td>499</td>
<td>4,99k</td>
</tr>
<tr>
<td>8</td>
<td>10k</td>
<td>10k</td>
<td>10k</td>
</tr>
<tr>
<td>9</td>
<td>2k</td>
<td>2k</td>
<td>20k</td>
</tr>
<tr>
<td>10</td>
<td>4,99k</td>
<td>4,99k</td>
<td>50k</td>
</tr>
<tr>
<td>11</td>
<td>10k</td>
<td>100k</td>
<td></td>
</tr>
</tbody>
</table>

Obviously the resistors in the network suffer of the thermal noise problem, but it is possible through a calibration process to maximize the DUT's collected noise and minimize the noise from the rest of the circuitry; in particular measurements on the resistive circuit alone are performed to obtain the background noise coming from it, shorting the DUT's branch.

The noise on the DUT is then available to be amplified by a noise amplifier that amplifies the low voltage signal picked at the DUT's terminals. The amplifier's gain is set to be $10^2$ to have an acceptable output range for the output of the conditioning circuit. Finally the signal is ready to be processed by the spectrum analyzer.
All networks are shielded in a metal cage in order to avoid external interferences to the measurement circuit.

### 5.3. Shielding

Usually to shield the system from external noise sources a metal coverage is used and thus this coverage allows reducing the system susceptibility. In the case of study the most important thing is to shield the conditioning circuit (composed by the noise amplifier and the bias circuit) and the device under test (DUT).

The shielding causes the incident electromagnetic external wave to produce a reflected wave and a transmitted one.

![Electromagnetic waves representation in presence of a metal shield](image)

*Figure 5.2 - Electromagnetic waves representation in presence of a metal shield*
The shielding effectiveness (SE) is then defined in terms of the electric or the magnetic field as:

\[
SE_{dB,E} = 20 \log_{10} \frac{|E|}{E_i}
\]

(5.1)

\[
SE_{dB,H} = 20 \log_{10} \frac{|H|}{H_i}
\]

(5.2)

The phenomenon is however not so simple and other contributing sources must be taken into account. In particular there are different terms that contribute to SE's final value and it is possible to distinguish three contributions: first reflection metal absorption (arising from the metal finite conductivity) and the term which is related to multiple reflections in the metal.

It is then possible to write that

\[
SE_{dB} = R_{dB} + A_{dB} + M_{dB}
\]

(5.3)

where \(M_{dB}\) is a negative term that tends to decrease the SE value. Moreover the frequency at which these events occur is of interest since skin effect is taken into account in the absorption term.

For a uniform plane wave it could be verified that

\[
R_{dB} = 20 \log_{10} \frac{1}{4 \zeta_0 \zeta_2} \left(\zeta_0 + \zeta_2\right)^2
\]

(5.4)

and it is the same for electric and magnetic fields, however while the electric field undergoes to a great reflection at the first interface and to a little one at the second, the magnetic field shows a dual behavior and, having a little first reflection term, it will also have a greater contribution of the multiple reflection term, that, as seen before, tends to worsen the shielding.
For this reason the shield's thickness is often the parameter to increase to have a good shielding to the magnetic field; in fact since

$$A_{dB} = 20 \log_{10} e^{t/\delta}$$

(5.5)

where \( t \) is the thickness of the shield and \( \delta \) is the penetration depth defined as \( \delta = \sqrt{\frac{2}{\omega \mu \sigma}} \) with \( \omega = 2\pi f \), \( \mu \) the material permittivity and \( \sigma \) the material conductivity.

In this study, the term \( A_{dB} \) is made sufficiently large that the shield could be considered a good conductor and the term due to multiple reflections, \( M_{dB} \), could then be neglected (\( A_{dB} > 15 \text{ dB} \)) [13].

### 5.4. Spectrum Analyzer

In order to obtain the DUT's noise, PSD, perform measurements and signal analysis, a spectrum analyzer is needed. Such a tool is capable to obtain the signal's spectrum from its waveform using a Fast Fourier Transform (FFT) algorithm.

In the project a HP89410 vector spectrum analyzer has been used; it is possible to build a block diagram of the same to clarify its behavior and then describe the mode of operation of the tool.
First the input signal is modified by an attenuation network and filtered by a LPF in order to fit it to the instrument's amplitude range and reduce its harmonic content to be able to study the frequency range of interest. Then the resulting signal undergoes to an analog to digital conversion before being led to the digital mixer. As in all spectrum analyzers, the mixer, nevertheless it is a digital one, has the function to shift the signal to approximately the central frequency of the following filter (in that case the digital filter shown in Figure 5.12); the frequency shift is related to local oscillator frequency. The filter is particularly important in this process, since the resolution depends mainly upon the filter bandwidth; however a trade-off between resolution bandwidth (i.e. the filter's bandwidth) and the frequency range is needed because the time required for the measurement is dependent on the inverse of the resolution bandwidth and on the frequency range. Moreover in noise measurements it is not recommended to use an overly high resolution bandwidth since higher resolution bandwidths, minimizing the noise floor level, imply a better noise rejection making thus noise more difficult to be detected. The signal is then processed by a microprocessor and undergoes also

![Figure 5.3 - Spectrum analyzer block diagram](image)
to an elaboration using DSP modules before being sent to the processor, for storage or further elaborations, and to the display.

Particular attention must be paid also to the choice of the window function to use in the process; a window function is needed because of the signal non periodicity that makes the signal's components to be present over a large frequency range. Choosing the window function properly is important to determine the resolution of the measurement and other parameters; e.g. the rectangular window has got good amplitude accuracy, while the Blackman one shows good frequency resolution. A short and non-exhaustive list of the possible windows that can be used and of their characteristics in term of width of the main lobe ($\Delta\omega_m$), amplitude of the secondary lobe ($\alpha$) and maximum approximation error ($\delta$) is reported in Table 5.2.

Table 5.2 - Window functions and their parameters (M is the number of samples composing)

<table>
<thead>
<tr>
<th>Window</th>
<th>$\Delta\omega_m$</th>
<th>$\alpha$[dB]</th>
<th>$\delta$[dB]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rectangular</td>
<td>$\frac{4\pi}{M+1}$</td>
<td>-13</td>
<td>-21</td>
</tr>
<tr>
<td>Bartlett</td>
<td>$\frac{8\pi}{M}$</td>
<td>-25</td>
<td>-25</td>
</tr>
<tr>
<td>Hanning</td>
<td>$\frac{8\pi}{M}$</td>
<td>-31</td>
<td>-44</td>
</tr>
<tr>
<td>Hamming</td>
<td>$\frac{8\pi}{M}$</td>
<td>-41</td>
<td>-53</td>
</tr>
<tr>
<td>Blackman</td>
<td>$\frac{12\pi}{M}$</td>
<td>-57</td>
<td>-74</td>
</tr>
</tbody>
</table>
5.5. Remarks

Some considerations are necessary about the measurement system before moving on to the measurements discussion is required. Every tool and circuit added in the system is a noise source and that has to be taken into account developing considerations about the measurements.

Empirically has been proved that the low noise amplifier introduces a $1/f$ noise and so spectrum analyzer does. From the amplifier datasheet it is possible to get that it introduces a noise PSD of $1.6 \times 10^{-14} \frac{V^2}{Hz}$. From the spectrum analyzer's datasheet it is not possible to get such information; however it can be seen from experimental results that the noise introduced by this instrument is much lower than the one introduced by the amplifier. It has to be underlined that the noise introduced by the amplifier is not always negligible even if it is lower than that the one introduced by the bias circuit.

The bias circuit is affected by Johnson-Nyquist's noise since the resistors composing the circuit are all obviously subject to carriers' fluctuations in conductors. Knowing the circuit scheme it is in this case possible to develop an analysis to determine the contribute to the PSD of this system; in this type of reasoning a noise voltage source is introduced for any resistor in the circuit and the analysis is conducted via the superposition principle, since the circuit to consider is a linear one [5]. However it would be only a preliminary analysis since after that an experimental characterization of the circuit is needed to take into account and to correct this effect in the measurements.
In the following discussion and measurements all the effects described above has been taken into account and corrected; it is thus possible to refer in what follows only to noise produced by the DUT, that in the end is the only one on which the study is focused.

The measure of interest is, in particular, the evaluation of the $k_{1/f}$ factor through $PSD - f$ curves analysis. Among the settings of the instrument a tool to change the display setting from linear to logarithmic on both axes is available; that type of visualization is way more comfortable for the noise constant evaluation. It is known in fact that, from equations [C.12] and [C.13] and their discussion, it is possible to say that in such a graph the curve of interest will be represented by a line with a negative slope given by $-k_{1/f}$ since in the region of interest is when $\lambda \approx 1$. The expected results from the analysis are to find that the samples show higher noise levels when treated with oxygen and hydrochloric acid. With this in mind it is possible to finally present, examine and discuss the experimental results obtained from noise measurements.
6. Results and discussions

In this chapter, material and electrical characterization of SiGe layers are presented. RGA, SEM and SIMS results are applied to understand the influence of the various growth parameters and oxygen treatment on the epi-layer quality.

6.1 Quality and profile of SiGe layers

In the following measurements, the quality of epi-layers is discussed through the full-width-half-maximum (FWHM) of SiGe peak where the composition and the layer thickness are determined by the position of SiGe peak relative to Si substrate peak and the distance between the two fringes in the rocking curve. The strain type is compressive in our epi.layers (i.e. the layer peak is always to the left of the substrate one). It is of interest to study how the strain and FWHM of layer peak vary when the growth parameters and oxygen exposure are changed of the epi-layers. All the measurements are referred to the plane indicated with the Miller indices [004].

It has to be underlined that the sample series in this part covers a large range of oxygen exposure and are not the ones to be later processed.

The description of the wafer's recipes in the figures and in the Table 4.2 is provided as following:

- the first 5 digits are the same for any sample, with the second and third (TI) indicating the project, and the fourth and fifth (SG) indicating the composition of the layer (silicon - germanium);
if a B is reported it means that the sample has been boron-doped;

if a H is reported it means that the sample has been processed with HCl and if no other contaminant is introduced in the chamber the following digits indicate HCl partial pressure in mtorr;

if a O is reported it means that the SiGe layer is grown with oxygen, either at the interface of Si/SiGe or within the epi-layer. The oxygen amount is in ppb range which is usually obtained at LTE.

a T is reported followed by a number, indicating the growth temperature used in the process in Celsius degrees.

6.1.1. Interfacial oxygen and temperature effect

It is known that the growth temperature is an important parameter for the growth kinetics and defect generation, propagation and interaction during epitaxial growth. Therefore, the effect of oxygen at different growth temperature on the crystallographic quality has been investigated in the beginning of this work. A list of grown samples is summarized in Table 6.1.

The formed oxygen islands (after surface treatment) may affect the layer quality. By increasing the amount of exposing oxygen (increasing the exposure time or partial pressure of oxygen in purging gas), fringes disappear and strain relaxation in epi-layers gradually increases. In this case the FWHM of SiGe peak is increased and the position of layer peak shifts to the higher incident angles for higher strain relaxation. In case of trisilane-based epitaxy at 500 °C is not sensitive to interfacial oxygen according to HRXRD (see Fig.6.1) The FWHM does not change in presence of oxide islands. Surprisingly the strain amount has slightly increased with increasing oxygen at the interface. This behavior is not the same for the SiGe growth at 600 °C (see Fig.6.2). The strain reduction occurs already at 800×10⁻⁹ torr.
Table 6.1 - Results Table

<table>
<thead>
<tr>
<th>Sample</th>
<th>Wafer Number</th>
<th>Si %</th>
<th>Ge %</th>
<th>Thickness (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>OTISGBH20T500</td>
<td>2912005225</td>
<td>81,500</td>
<td>18,500</td>
<td>42,675</td>
</tr>
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<td>OTISGBH40T500</td>
<td>2912005249</td>
<td>79,245</td>
<td>20,755</td>
<td>50,075</td>
</tr>
<tr>
<td>OTISGH20T500</td>
<td>2912002269</td>
<td>75,384</td>
<td>24,616</td>
<td>35,850</td>
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<tr>
<td>OTISGH40T500</td>
<td>2907822070</td>
<td>75,684</td>
<td>24,316</td>
<td>34,300</td>
</tr>
<tr>
<td>OTISGBH20T600</td>
<td>2907822096</td>
<td>80,215</td>
<td>19,785</td>
<td>192,475</td>
</tr>
<tr>
<td>OTISGBH40T600</td>
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<td>21,212</td>
<td>200,975</td>
</tr>
<tr>
<td>OTISGH20T600</td>
<td>2907822263</td>
<td>79,545</td>
<td>20,455</td>
<td>201,725</td>
</tr>
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<td>OTISGH40T600</td>
<td>2907822265</td>
<td>78,691</td>
<td>21,309</td>
<td>149,850</td>
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<td>OTISGBO200T500</td>
<td>2907822234</td>
<td>85,096</td>
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<tr>
<td>OTISGBO200T600</td>
<td>2907822127</td>
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<td>14,691</td>
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<td>OTISGO200T500</td>
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<td>69,400</td>
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<td>15,340</td>
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<td>16,606</td>
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<td>OTISGBH5T600</td>
<td>2907822051</td>
<td>83,704</td>
<td>16,296</td>
<td>100,475</td>
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<td>OTISGBOH20020T5</td>
<td></td>
<td>81,105</td>
<td>18,895</td>
<td>51,025</td>
</tr>
<tr>
<td>OTISGBOH20020T6</td>
<td></td>
<td>80,330</td>
<td>19,670</td>
<td>201,750</td>
</tr>
</tbody>
</table>
Figure 6.1 - Rocking curves of samples grown at a temperature of 500 °C with different oxygen partial pressure contaminations

Figure 6.2 - Rocking curves of samples grown at a temperature of 600 °C with different oxygen partial pressures
6.1.2. Interfacial oxygen and boron incorporation in SiGe

The effect of interfacial oxygen is different when the SiGe layers are doped. The oxygen may affect the strain in SiGe which is a dominant factor for incorporation of dopants in SiGe matrix. Fig. 6.3 show the rocking curves of epi-layers B-doped SiGe grown on a series of oxygen samples.

All the boron-doped SiGe peaks are located closer to the Si substrate indicating strain compensation in presence of substitutional boron in SiGe matrix (concentration of $10^{20}$ cm$^{-3}$).

The oxygen treated samples (below 800 ppb), a slight shift in the position of SiGe is observed to the lower incident angles (to the left) which indicate higher strain amount (or less strain relaxation). For higher oxygen partial pressure (1600 ppb) stain relaxation takes place due to the poor interfacial quality.

![Figure 6.3 - Rocking curves of samples boron doped, grown at a same temperature, with different oxygen partial pressures](image)

(counts/s)

(counts/s)
6.1.3. \textit{HCl} effect

The concentration of \textit{HCl} is a fundamental parameter for the Ge content in SiGe layers since the hydrochloric acid preferably etches silicon over germanium. It is then expected that higher \textit{HCl} concentrations will lead to higher \textit{Ge} percentage amount in the layer, thus leading to a left shift of the layer peak in the rocking curves meaning a more compressive strain in the structure.

This is confirmed by the rocking curves and by the simulation parameters extracted via X'Pert Epitaxy SW (see Table 6.1)

However another trend arises from the reported Figure 6.4. It is in fact possible to notice that the curves tend to shift back to the right showing a less compressive strain. It is believed that this behavior is due to the fact that at partial pressures higher than 100 mtorr \textit{HCl} etches significantly not only silicon but also germanium atoms.

![Image of rocking curves]

\textbf{Figure 6.4 - Rocking curves of samples grown at a same temperature with different \textit{HCl} partial pressures}
To understand how HCl influences the percentage amount of germanium in the layer and the growth rate it is possible to consider the results reported in Figure 6.5.

**Figure 6.5 - HCl effects on the layer growth and on the germanium content in the layer itself for different HCl partial pressures and at two different growth temperatures (500 °C and 600 °C)**

It is then clear that higher HCl concentrations produce a decrease in the growth rate of the layer due to an etching of Si or Ge atoms. Moreover, as stated before, there is a critical value for HCl partial pressure where the germanium etching will become significant and it will result in a lower percentage amount of Ge in the SiGe layer. For these reasons it is preferable to do not work with HCl concentrations over 100 mtorr and to work with HCl partial pressures of approximately 40 mtorr that allows to control the percentage amount of germanium in the layer and makes the process controllable.
6.2. Estimation of critical size oxide islands

Although the XRD results show the oxygen has no significant change on the strain and FWHM of SiGe layers but our observations from HRSEM analysis reveals a critical value for oxygen partial pressure where the oxide islands are formed. As it could be noticed from Figure 6.6, the pressure of such contamination in the chamber 400 ppb, since after this value island formation could be detected.

From those measurements it is also possible to obtain the size of the formed islands and, taking into account the measurements on all the samples, it is possible to obtain the following graph (Figure 6.7). The graph shows that to obtain islands' sizes below the critical size (0.02μm²) partial pressures of 200 ntorr maximum are required.

Figure 6.6 - SiO₂ islands formation for various concentration of O₂ in the chamber (Trisilane flow: 2gr/hr; Germane flow: 49sccm; Tempeareure 600°C)
6.3. Residual Gas Analysis (RGA)

RGAs are used to monitor the quality of the vacuum and to detect traces of impurities in low-pressure gas environments. The impurity levels down to $10^{-14}$ torr can be measured by this instrument.

Thus the partial pressure of exhaust gases during the exposure step reveals the interaction conditions between the surface and the gas itself. Mass spectrometer of RGA tool has been connected at the exhaust gas line close to the reactor process to measure the residual gases after the process. In all experiments, the background of oxygen and water amount in the carrier gas ($H_2$) was as low as $10^{-10}$ and $10^{-9}$ torr respectively as shown in Figure 6.8.
Thus, this amount of oxygen and water before the intentional exposure of $O_2$ will not affect the measurement and it is negligible. Exposure at different $O_2$ partial pressures and different temperatures explores various reacting modes of $O_2$ with silicon surface. The partial pressures of oxygen for an exposure time of seven minutes as a function of the temperature are shown in Figure 6.9.
The exposure condition is constant and the partial pressure of oxygen is varied for different growth temperature. There is a large variation of measured oxygen partial pressure, indicating that the only effect that can change the consumption of oxygen is the temperature defining the deposition regime. Increasing the temperature from 550 °C to 650 °C the partial pressures of oxygen decreases showing that at higher temperatures the consumption of the inlet gas becomes higher too, if compared to the previous case. Moreover, increasing the temperature the deposition mode will change from being reaction controlled to be mass transport controlled (this is completely related to the activation energy of oxygen).
6.4. Oxygen profile in SiGe layers

SIMS is a technique used to analyze the composition of solid surfaces and thin films by sputtering the surface of the specimen with a focused primary ion beam, collecting and then analyzing the ejected secondary ions. The mass/charge ratios of the secondary ions are measured with a mass spectrometer thus determining the elemental, isotopic, or molecular composition of the surface. SIMS shows elemental detection limits ranging from parts per million to parts per billion.

It is important also to determine the oxygen level for each exposure. SIMS analysis from multilayer samples where the exposure time and temperature have been varied are shown in Figure 6.10a and 6.10b. The obtained results show that the oxygen amount increases increasing the partial pressure or the process temperature and also that increasing the value of these parameters will lead to shallower islands in the structure.

Figure 6.10 - SIMS spectra of oxygen treated Si multilayer: a) versus different oxygen partial pressures; b) with 400 ppb oxygen level but at different process temperatures
6.5. Electrical characterization

6.5.1. Growth of SiGe for electrical characterization

The prepared SiGe/Si multi-layer structures for electrical measurements are illustrated in 6.11 and 6.12.

![Figure 6.11 - Wafers epitaxial growth parameters](image)

Figure 6.12 shows all samples in this project where oxygen contaminations of 20 and 60 ppb were planned to be investigated. In this work, only oxygen contamination of 60 ppb has been considered.
Figure 6.12 - Complete scheme of the samples to process and analyze in the project

Figure 6.13 shows the PSD of four SiGe layers. $k_{1/f}$ factor were obtained from the slopes of these curves, considering the frequency range of interest ([10÷10$^4$] Hz, where the main contributions to noise level are related to flicker and GR noise as it is possible to notice in Figure 6.13). The collected the data of interest are summarized in Table 6.2.
Table 6.2 - Summary of calculated $k_{f}$ parameters for different mesas’ sizes

<table>
<thead>
<tr>
<th></th>
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<th></th>
<th></th>
<th></th>
<th></th>
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</thead>
<tbody>
<tr>
<td>25x25</td>
<td>$3 \cdot 10^{-14}$</td>
<td>$4 \cdot 10^{-13}$</td>
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<td>$6 \cdot 10^{-12}$</td>
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<td>$5 \cdot 10^{-11}$</td>
</tr>
<tr>
<td>50x50</td>
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<td>$9 \cdot 10^{-13}$</td>
<td>$4 \cdot 10^{-12}$</td>
<td>$7 \cdot 10^{-12}$</td>
<td>$3 \cdot 10^{-12}$</td>
<td>$3 \cdot 10^{-10}$</td>
</tr>
<tr>
<td>100x100</td>
<td>$3 \cdot 10^{-14}$</td>
<td>$4 \cdot 10^{-13}$</td>
<td>$6 \cdot 10^{-13}$</td>
<td>$1 \cdot 10^{-12}$</td>
<td>$2 \cdot 10^{-12}$</td>
<td>$5 \cdot 10^{-11}$</td>
</tr>
<tr>
<td>200x200</td>
<td>$3 \cdot 10^{-14}$</td>
<td>$1 \cdot 10^{-15}$</td>
<td>$7 \cdot 10^{-14}$</td>
<td>$8 \cdot 10^{-14}$</td>
<td>$2 \cdot 10^{-13}$</td>
<td>$2 \cdot 10^{-12}$</td>
</tr>
</tbody>
</table>

Figure 6.11 - Noise measurement results on the processed samples
The value of the considered parameter tends to increase also in the case of oxygen contamination without entering HCl in the chamber. In this case, an increase of factor of 10 is observed regardless if the contamination happens before or during the SiGe epitaxial growth. That is due to incorporated SiO$_2$ at the interface and within the layer.

Moreover, as it could be noticed, another case in which the $k_{1/f}$ is considerably higher is the one in which both HCl and oxygen contaminants are considered; in fact the measurements show an increase of about two orders of magnitude compared with the reference sample. That could be due to metal contaminants in the chamber, coming from the pipe, in presence of HCl during the growth and also to higher defects concentrations due to the etching process; more analysis on that are required as a future development of this study.

Another expected trend was the one regarding the decrease of the factor, and thus of the noise, considering larger structures; this trend is confirmed by experimental data, where the noise factor increases with the decrease of the mesa's size. That could be due to lateral defects in the structures due to processing which is more salient for mesas with smaller areas.
7. Conclusions

The whole work is composed by two parts: one related to the material characterization and one to the electrical characterization of the obtained structures.

From the material characterization it has been possible to obtain how the SiGe layer growth is influenced by parameters such as temperature, boron doping and hydrochloric acid pressure during the LTE process. It has been possible to recognize that:

- No oxide islands were observed for oxygen level below 200 ntorr. A critical size of 0.02μm² is estimated for oxide islands to affect SiGe growth.

- Increasing HCl partial pressures tend to increase the compressive strain in the structure and to produce layer with higher Ge percentage amount.

- The interfacial oxygen (Si/SiGe) may affect the incorporation of B in SiGe layers. This is due to reduction of strain in SiGe matrix caused by low quality interface (Si/SiGe).

The noise measurements were performed to obtain further information on the layer quality and on the electrical performances.

The performed noise measurements show that in the range of interest, [1÷10⁴] Hz, the predominant noise factor is the flicker one and it is thus possible to evaluate the noise level extracting the $k_{1/f}$ factors as the absolute value of the measurement waveforms' slope.

It has been found that the noise level varies with the oxygen and hydrochloric acid partial pressures and in particular it tends to increase increasing the value of these parameters. Also
in the case both of them are used significantly higher noise levels have been found if compared to the reference sample's ones. It has been also possible to notice that an increase in the structure's size will lead to lower noise levels due to lateral defects in the multilayer structures.
8. Future work

Future developments of this work are related to the understanding of how HCl presence in the chamber during LTE influences is necessary. In fact, from previous studies it was known that HCl could produce metal contaminations in the structure due to the etching of the pipe walls. For that reason, in this study the pipe has been changed and lower noise level have been found, compared with the previous case. It is however possible that the aging of the pipe and thus the etching of its walls could lead in time to unacceptable metal contaminations in the LTE and thus an analysis of this problem is required.
Appendixes
Appendix A. X-Rays

A.1. X-Rays Production

X-rays are an electromagnetic radiation with wavelength between 10 pm and 10 nm (frequency $f \in [3 \cdot 10^{16} \div 3 \cdot 10^{19}]$ Hz). Such wavelengths are comparable to interatomic distances in crystals, hence x-ray diffraction represents the main technique to study and determine solid's crystal structure.

![Electromagnetic Spectrum](image)

Figure A.1 - Electromagnetic Spectrum

The simplest way to produce x-rays is to use a cathode ray tube (CRT) in which the cathode is led to high temperature to obtain electrons emission due to thermionic effect; the electrons are then accelerated to hit on a metallic anode creating the x-ray beam leaving from a beryllium window. X-rays are produced because the accelerated electrons excite anode's electrons so that they could reach a higher energy level and then return to the previous equilibrium state emitting photons with energy given by Planck equation

$$\Delta E = h \cdot \nu$$  \hspace{1cm} (A. 1)

from which it is possible to obtain, using the relation $c = \nu \cdot \lambda$, that
with $\Delta E$ expressed in keV. From the latter equation is evident that in order to obtain x-rays, whose wavelength were specified before, a energy in a $[0.124\div124]$ keV range must be used; usually an energy of 10 keV is used to obtain a x-ray beam with a wavelength around 0.1 nm.

The intensity of the radiation emitted by the target is a function of wavelength and target's composition; as metal target copper (Cu), aluminum (Al), molybdenum (Mo) and magnesium (Mg) are usually used. A typical pattern of a wavelength - intensity graph is shown in Figure A.3; two peaks due to electrons' disexcitation could be noticed. In particular the first peak from the left, $K_\beta$, represents transitions from 3p to 1s orbital, while the second, $K_\alpha$, represents 2p to 1s transitions. $K_\alpha$ shows an intensity almost two times higher than $K_\beta$, because the former type of
transitions is energetically more likely than the latter. For this reason usually $K_\alpha$ beam is used to conduct the experiments while $K_\beta$ beam is filtered.

**A.2. X-Ray Diffraction**

**A.2.1. Miller indices**

Miller indices are three indices useful to identify directions, axes or planes in a crystallographic structure. They represent a coordinate system for the considered Bravais lattice.

To determine the Miller indices it is possible to follow a procedure which starts from picking three lattice vectors $(a_1, a_2, a_3)$, describing the elementary cell, and choosing a plane not intercepting the axes' origin; then it is possible to determine the interceptions between the axes and the plane. The inverses of the so calculated interceptions are calculated and in the case they are in form of a fraction the set of number has to be multiplied for an entire $N$ such that the new set will be made up by integer numbers, thus $N$ is the least common multiple of the denominators of the fractions. Finally negative indices are signed with a $\bar{\cdot}$ and the Miller indices are obtained. Examples of Miller indices for various crystallographic planes are reported in Figure A.4.
In other words it is possible to say that the set of indices \((h,k,l)\) identifies a plan intercepting the points \((\frac{a_1}{h}, \frac{a_2}{k}, \frac{a_3}{l})\) and so the indices are proportional to the inverses of the intercepts of the plane in the lattice vectors' system of coordinates. Obviously, if one of the indices is zero it means that the plane is parallel to that component of the system's coordinates (i.e. the plane and the considered component does not have any interception point) [16].

**A.2.2. Bragg's Law**

The so produced x-ray beam is used to examine crystals to identify the material of which they are made, their grains' size and orientation. In fact when the beam is directed on the crystal with a certain incident angle, \(\theta\), supposing that the considered planes are identified by Miller indices \((100)\), it is possible to measure a reflected beam due to the presence of mobile
charges in the planes; to do that a detector is placed at an angle $\theta$, knowing from reflection's theory that the incident and the reflected angles are congruent. Rays reflected by different planes can cause constructive or destructive interference according to the difference between rays' optical paths. In particular the constructive interference condition is given by Bragg's law, establishing that the optical paths' difference must be an integer multiple of the wavelength, $\lambda$; that is to say that

$$2 \cdot d \cdot \sin \theta = n \cdot \lambda \quad (A.3)$$

where $n$ is the diffraction order and $d$ is the crystallographic distance between one plane and the next. In this way it is possible to get the angle $\theta$ for which the intensity of the reflected ray is maximum, having fixed $n$, $d$ and $\lambda$.

**A.2.2. Laue's Conditions**

Using Bragg's condition it is then possible to predict diffraction's directions, but not to calculate diffraction's intensity, which is directly proportional to the scattered electric field. Assuming that the incident radiation is a travelling plane wave which electric field could be modeled by equation

$$E(\vec{r}, t) = E_0 e^{j(\vec{k} \cdot \vec{r} - \omega t)} \quad (A.4)$$
with \( \vec{k} \) wave vector having module \( \frac{2\pi}{\lambda} \) and directed along wave propagating direction. The incidence of this plane wave on a lattice atom will result in a scattering process with a spherical wave generation in the form

\[
E_{scf} = fE(\vec{r}, t) \frac{e^{i\vec{k}' \rho}}{\rho}
\]  

(A.5)

in which is possible to recognize a phase factor, an attenuation factor, due to the wave propagation, and a constant, \( f \), which takes into account atom's features and that is proportional to the atomic number of the species, \( Z \); \( \vec{k}' \) is the reflected wave vector, having module \( |\vec{k}'| = |\vec{k}| \) and same direction as \( \vec{R} \), the vector identifying detector's position. Simplifying the previous formula for \( |\vec{R}| \gg |\vec{r}| \) and taking into account all possible \( \vec{r} \) values it is possible to obtain

\[
E_{sc} = \frac{E_0 e^{i(|\vec{k}|R-\omega t)}}{R} fQ
\]  

(A.6)

with \( Q = \sum_{n_1=1}^{N_1} e^{-j \alpha n_1 \Delta \vec{k} \cdot \vec{a}} \sum_{n_2=1}^{N_2} e^{-j \alpha n_2 \Delta \vec{k} \cdot \vec{b}} \sum_{n_3=1}^{N_3} e^{-j \alpha n_3 \Delta \vec{k} \cdot \vec{c}} \), known as structure factor, that is the Fourier discrete transform of the electronic density, and \( \Delta k = \vec{k}' - \vec{k} \). To maximize the scattered field the following conditions should then be satisfied

\[
\Delta \vec{k} \cdot \vec{a} = 2\pi h \quad \Delta \vec{k} \cdot \vec{b} = 2\pi k \quad \Delta \vec{k} \cdot \vec{c} = 2\pi l
\]  

(A.7)

where \( (h,k,l) \in \mathbb{N} \) are the Miller indices and the conditions, known as Laue's conditions, are equivalent to Bragg's law.


**B.2.3. Reciprocal Lattice**

Introducing three vectors such that $\vec{A} \perp (\vec{b} \wedge \vec{c})$, $\vec{B} \perp (\vec{a} \wedge \vec{c})$, $\vec{C} \perp (\vec{a} \wedge \vec{b})$ and that $\vec{A} \cdot \vec{a} = 2\pi$, $\vec{B} \cdot \vec{b} = 2\pi$, $\vec{C} \cdot \vec{c} = 2\pi$ it is possible to generate a reciprocal lattice in the transformed space. The reciprocal lattice is generated by all the points obtained by the following relation

$$G = h\vec{A} + k\vec{B} + l\vec{C}$$  \hspace{1cm} (A. 8)

with

$$\vec{A} = 2\pi\frac{\vec{b} \times \vec{c}}{\vec{a} \cdot \vec{b} \times \vec{c}} \quad \vec{B} = 2\pi\frac{\vec{c} \times \vec{a}}{\vec{a} \cdot \vec{b} \times \vec{c}} \quad \vec{C} = 2\pi\frac{\vec{a} \times \vec{b}}{\vec{a} \cdot \vec{b} \times \vec{c}}$$  \hspace{1cm} (A. 9)

If $\vec{a} \perp \vec{b} \perp \vec{c}$ the previous conditions reduce to

$$\vec{A} = \frac{2\pi}{\vec{a}} \quad \vec{B} = \frac{2\pi}{\vec{b}} \quad \vec{C} = \frac{2\pi}{\vec{c}}$$  \hspace{1cm} (A. 10)

and $\vec{G} = \Delta \vec{k}$. From the reciprocal lattice approach it will be easier to find diffraction conditions thanks to Ewald's sphere method. In this method $\vec{k}$ vector is translated to point to axis' origin and a sphere centered in the origin of the vector with its module as radius is then built. Any point of the reciprocal lattice intercepted by the sphere clearly satisfies Laue's conditions providing a diffraction peak [16].
A.2.4. Wigner - Seitz Cell and First Brillouin Zone

The Wigner - Seitz cell is a mean to describe crystal's symmetry and draw conclusions on material properties related to symmetry found. It is defined as the locus of points in the lattice space that are closer to the considered lattice point than to any other lattice point. Construction of the cell starts with the choice of a lattice point, tracing the segments joining this point to all nearby lattice points and their segment axis too. Considering then the planes identified by any previous segment axis it is possible to build the smaller volume enclosed in this planes that is by definition the Wigner - Seitz cell.

Figure A.7 - Wigner - Seitz primitive cell construction example
The first Brillouin zone is nothing but the Wigner - Seitz primitive cell built considering the reciprocal lattice and it gives information about the nature of the examined material; in particular, knowing the material FBZ it is possible to establish if the material is a conductor, a semiconductor or an insulator, since the FBZ defines material's energy bands and their properties.

**A.3. Powder Diffraction Method**

The importance of this method is linked to the fact that this is a high accuracy and non-destructive method of analysis. The setup is similar to that shown in Figure A.8 but, since it is difficult to vary the radiation's incident angle because of the dimensions of voltage generators, it is preferable to vary both the sample's position, by an angle $\theta$, and the detector's position, by an angle $2\theta$; that's why the method is often referred to as $\theta$ - $2\theta$ method. In addition, commonly a mirror to increase the intensity of the beam, useful for quickest measurements, and several monochromators, to contain the beam divergence and increase the resolution, are used before the beam is sent to the sample.

In a $\frac{\omega}{2\theta}$ - intensity graph, also known as rocking curve, peaks will be detected when Bragg's condition is satisfied for planes parallel to external surface; in general in Bragg's condition $d$ shows a dependency on all three spatial coordinates and varies according to what type of crystalline...
structure is being studied. If the structure is not known, hypothesis on it can be formulated and then compared with experimental results to find what the crystal's structure really is.

It can be noticed that intensity peaks tend to decrease while increasing 2θ value; that's because atomic density along the incident direction decreases and with it planes' reflections too.

An equation to express the intensity according to which

\[ I = Q^2 p \left( \frac{1+\cos^2 2\theta}{2 \sin^2 \theta \cos \theta} \right) A(\theta) e^{-2M} \]  

(A.10)
can be empirically found; the equation's factors are listed below:

- **Q** is the previously defined structure factor;
- **p** is called multiplicity factor and takes into account the fact that a set of vectors may have the same length in the reciprocal lattice showing then diffraction peaks for the same Bragg's angle. The multiplicity factor is simply the number of different planes characterized by Miller indices that contribute to the same reflection;
- **L₀P₀** is a factor defined by the experiment's geometry, composed by two terms: Lorentz factor \( L₀ = \left( \frac{1}{\sin^2 \theta \cos \theta} \right) \) and polarization factor \( P₀ = \left( \frac{1+\cos^2 2\theta}{2} \right) \);
- **A(θ)** is an absorption factor dependent on the incident angle;
- **e⁻²M** is a temperature factor due to atomic planes vibrations causing a background noise in the spectrum.

**A.3.1. Rocking Curves**

A rocking curve for a structure formed by a substrate and a layer, as in the case of study, is presented in Figure A.9.
It is possible to distinguish two different peaks with other fringes due to the thickness of the sample's strata; the broader and lower peak is relative to layer's diffraction (since the layer is less thicker then the substrate) while the narrower and higher peak is relative to substrate's diffraction. The position of the layer peak depends on the lattice mismatch between the substrate and the layer itself (for bigger lattice mismatches the two peaks tend to show a greater separation) and on the kind of stress that the layer induces on the substrate (whether it is tensile or compressive strain); shift of the layer peak could also indicate strain variation from which it is possible to determine dopants concentration according to contraction/expansion coefficient determination.

In this type of measurements the Full Width Half Maximum (FWHM) values are of interest. The FWHM is defined as the distance, on the independent variable axis, between the
points of the curve for which the dependent variable assumes half maximum values; it is possible from that to obtain curves' standard deviation from the maximum.

Different factors influence the broadening of the rocking curve and must be taken into account such as instrumental broadening, due to equipment used, angular rotations at dislocations and strain broadening, due to dislocations presence in the lattice, and crystal size broadening, due to crystals' dimensions and defined empirically by Sherrer's formula [18]

\[
B = \frac{K\lambda}{L \cos \theta}
\]  \hspace{1cm} (A. 11)

where \(B\) is the peak width, \(K\) is an empirical constant, \(\lambda\) is the wavelength of the incident x-rays, \(L\) is the linear dimension of the particle and \(\theta\) is the Bragg's angle.

Thanks to this type of measurements it is possible to estimate layers' dislocation density and lattice parameters.

**A.3.2. High Resolution Reciprocal Lattice Mapping (HRRLM)**

Reciprocal lattice mapping consists in a series of rocking curves obtained varying the \(\omega\) parameter and forming contour maps in a \(\omega - \frac{\omega}{2\theta}\) graph which is inherent to reciprocal lattice space, but gives information on mismatch factors, defects, dislocations.
A.4. Tools

For high resolution X-ray diffraction (XRD) a Philips' X'Pert PRO X-ray diffraction system has been used [19]. The system consist of:

- a console, providing the working environment for the system (a microprocessor system to measure and control electronics and a high tension generator). The console presents also a panel to power on the instrument, put the system in standby operation mode, switch on or off the inspection light and the high tension generator and close the shutter;
- a goniometer, providing the two main axis for x-rays measurement (0, 20) and on which the x-ray tube, the incident and diffracted beam optics and the sample stage are mounted;
- a ceramic diffraction x-ray tube mounted onto the goniometer in a tube shield;
- optical modules for the incident and diffracted beams;
✓ a sample stage on which to mount the sample to perform the measurements on the same;

✓ a detector, to measure the intensity of the diffracted x-ray beam.

In the measurements made no filter has been used to increase to an acceptable level the counts per second at the detector.

![X’Pert PRO System](image)

**Figure A.11 - X’Pert PRO System**

The sample has to be mounted on the sample stage and fixed on that, usually using specific adhesive tape. After that the machine is managed by remote, using a dedicated software named X’Pert Data Collector. Using that SW it is possible to vary the position of the sample stage along the various directions and it is also possible to tilt the sample varying φ and ψ angles. Moreover it allows to vary the current and voltage values; in the project of interest the voltage has been imposed to 45 kV and the current to 40 mA.
The SW initially provides standard setups for rocking curves measurements allowing also to vary this settings to better examine crystallographic planes of interest. Before starting the measurement the operator has to optimize three parameters in order to increase the counts per second (cps) on the detector and thus have a good measurement. The three parameters are ω, φ and ψ angles; the expected procedure requires several iterations to find the cps maximum at the parameters' variations. A good level of cps for the measurement of interest is assumed to be approximately \(10^6 \frac{\text{counts}}{s}\).

So the parameters of interest for the sample stage position are:

- ω, angle between the incident x-rays and the sample surface;
- \(2θ\), angle between the incident x-rays and the detector;
- ψ, sample tilt;
- φ, in plane sample rotation;
- x,y, in plane displacement of the sample;
- z, vertical displacement of the sample.

After that optimization it is possible to run the measurement of interest; knowing that the structure is made by a SiGe layer on a Si substrate, from x-rays diffraction theory it is expected to have two peaks, with the substrate peak higher than the layer one. It is also useful at this point to perform high resolution measurements decreasing the time per step and adjusting the measurement range for each measurement.

Rocking curves for sixteen different wafers grown with different values of oxygen, hydrochloric acid, boron and temperature has been measured to recognize the impact of this parameters on the structure's stress. After the measurements another SW has been used, X'Pert Epitaxy, to develop comparisons between the various wafers' rocking curves, to fit the
rocking curves and to estimate some parameters of interest, such as the percentage amount of Ge in SiGe.

X'Pert Epitaxy compares the experimental rocking curve to curves contained in its database and it estimates the layer thickness and the percentage amount of compounds in the layer; the correct thickness evaluation is possible if and only if the rocking curve shows fringes that the SW can use as a reference to fit the database models.

In this type of SW particular attention must be paid by the user to build and simulate a good reference sample to compare with the empirical curve; in the case of study it has been possible to use a virtual reference sample composed by a the Si layer with on top a SiGe layer with a 100 μm thickness and a 15% percentage amount of Ge in it. This sample has then been modified in the simulation parameters (e.g. the thickness, the percentage amount of the compounds, monochromators used and magnification) to obtain curves more similar to the one resulting from the measurements. Finally an automatic fitting is done on the obtained reference curve to have a better matching and from that curve the parameters of interest are extracted [20].
Figure A.12 - Example of a simulation and fitting result; is it possible to recognize the experimental result curve (blue), the simulated curve (red) and the fitted one (green)
Appendix B. SEM

Scanning Electron Microscope (SEM) is an important analysis tool because of its capability to provide high magnification, resolution and depth of field compared to optical microscopy. SEM's resolution could be up to 10Å with a magnification in a range of 10x to 500,000x. Using SEM it is possible to gather information about film thickness, step coverage, morphology and edge profile after etching.

A SEM is composed by seven principal systems: a vacuum system, an electron beam generation system (Electron Gun), an electron beam manipulation system, a beam/specimen interaction system, a detection system, a signal processing system and a display and recording one [21].

Figure B.1 - SEM's basic structure [22]
B.1. Vacuum System

A good vacuum system is required to obtain a correct generation and control of the electron beam. The required vacuum level is achieved using different types of pumps to reach pressures up to $10^{-11} \div 10^{-10}$ Torr.

B.2. Electron Beam Generation

In a SEM a source is used to create a beam of electrons then accelerate to energies of 500 eV ÷ 40 keV; the electron beam is focused to a small diameter and directed to the surface of the sample following a raster-scan pattern on the same.

![Image of raster-scan pattern](image)

Figure B.2 - Example of a raster-scan pattern [23]

B.3. Electron Guns

The electron gun is used to produce electron beam with desired energy; three main types of electron guns are used:
The most used type of electron guns are FEGs nowadays and thus the following discussion will be focused on them.

B.3.1. FEG

The FEG's cathode consists in a sharp metal tip with a diameter under 200 nm; a voltage difference, called extraction potential, is imposed between the cathode and the anode, generating an electric field, concentrated at the cathode's tip, which induces the electrons emission; a second grounded anode is used to accelerate the carriers and thus to confer them with more penetrating power as the voltage is raised.

The FEGs require the tip to stay contaminant free and so ultra-high vacuum conditions are required ($10^{-11} \div 10^{-10}$ Torr).

FEGs present, compared with the other technologies, greater brightness values (by two or three orders of magnitude) where brightness is identified as beam current per unit area per solid angle [22]

$$B = \frac{4i_p}{(\pi d_p \alpha_p)^2} \quad (B.1)$$

Where $i_p$ is the probe current, $d_p$ is the probe diameter and $\alpha_p$ is the probe convergence angle; moreover brightness increases linearly with accelerating voltage.

The peculiarity to have enough probe current in a probe of small diameter allows FEG SEM to reach the high resolution it does; it happens because the ability to achieve small probe diameters is directly related to the diameter of the electron beam exiting the gun; a small diameter corresponds to a higher spatial coherency. Another important parameter is the
temporal coherency, that is to say that the electrons emitted from the gun have approximately the same wavelength; there is always a certain spread in electrons’ energy, but to attain low accelerating voltage imaging it is important to have as lower energy spread as possible. Obviously vacuum has to be maintained to obtain a sufficient coherent beam.

**B.4. Electron Beam Manipulation**

**B.4.1. Electromagnetic Lenses**

Electromagnetic lenses are used to reduce magnification related to the beam and to focus the beam on the specimen; the former task is accomplished thanks to a system of condenser and objective lenses, while the latter is obtained thanks to the use of objective lenses only. In this case the object to demagnify is the diameter of the electron beam which has to be reduced to reach greater resolutions. Electromagnetic lenses can vary their focal point by simply varying the amount of current running through them.

Having high magnification comes with a cost; in fact reducing the probe diameter causes the probe current to decrease while a sufficiently high current is essential to achieve acceptable image contrast and a good SNR.

An electromagnetic lens is composed by a coil of copper wires inserted in an iron torus; the current flowing through the copper wires creates a magnetic field in the torus bore which forces the electron beam to converge, since the magnetic field is weaker in bore's center while it gets stronger along bore's radius.
B.4.2. Lens Aberrations

The most important lens defects are spherical aberrations, chromatic aberrations and astigmatism. All these defects contribute to form a disk of minimum confusion rather than a focal point.

- **Spherical aberration**: it is due to the magnetic field non uniformity which results in a series of focal points, instead of a single point, forming a disk of finite size. To reduce this effect circular apertures are introduced into the beam path; the apertures reduce the disk size, reducing also the probe current. Also the apertures' dimensions have to be taken into account; in fact small apertures, that are needed to reduce the spherical aberration, give rise to diffraction effects and so, usually, a trade-off between the two effects has to be found;
✓ **Chromatic aberration**: it is due to different electrons' energies and it is produced because lenses deflect lower energy electrons more strongly. In this case a disk of finite size instead of a focus point is produced too;

✓ **Astigmatism**: the effect is related to the imperfect lens symmetry that results in an oblong beam and thus in an ellipse of minimum confusion; applying current differentially to a ring of stigmator coils around the objective lens and so producing a compensating field causes the beam to assume a more symmetric shape.

### B.4.3. Effects of Other Parameters on Aberration

#### B.4.3.1. Accelerating Voltage

The accelerating voltage could be varied in a [1 - 30] kV range; an increase of the operating voltage lead to a decrease in lens aberration and to an increase of probe current, but on the other side it can cause charge up and damage nonconductive and beam sensitive specimens besides obscuring surface details, due to an increased depth of penetration.

#### B.4.3.2. Emission Current

Increasing the emission current will cause an increase in probe current, but at the same time it can cause charge up and damage nonconductive and beam sensitive specimens.

#### B.4.3.3. Probe Diameter

Decreasing the probe diameter, varying the condenser lens current, will enable greater resolution and decrease lenses' aberration, but it will also decrease probe current leading to a lower SNR and to a worse image contrast.
**B.4.3.4. Objective Aperture Size**

Decreasing the aperture size will lead to a decrease in lens' aberration, probe current and beam's convergence angle.

**B.4.3.5. Working Distance**

Increasing the working distance will increase the depth of focus, but will also increase the size of the probe and the aberrations leading to lower resolution.

**B.5. Beam Interaction**

The detection is possible only after that the electron beam have hit the specimen so that the primary electrons penetrate it; these electrons will probably travel into the specimen before hitting other particles and being then scattered. During this process an interaction volume is defined in which scattering events take place, known as reaction vessel. The larger is the reaction vessel the higher will be the signal (and so the SNR), on the other hand, smaller reaction vessels tend to give better resolution.

![Figure B.4 - Reaction vessel][23]
B.5.1. Backscattered electrons

Higher energy electrons, also called backscattered electrons, are those which suffered elastic collisions with target's atoms and so, after that, they still possess the majority of their incident energy.

B.5.2. Secondary electrons

Lower energy electrons, also known as secondary electrons, are produced by inelastic collision of the primary beam and the shell electrons of the sample's atoms; in these collisions, primary electrons dislodge a specimen electron from its surface. Due to their low energies only electrons produced near the sample's surface escape the surface and are then detected (they usually carry the most of the signal used for morphology studies).

The most important feature about imaging in the secondary mode is that images' contrast and soft shadows are similar to that of a specimen illuminated with light and so image interpretation is easy because the images appear more familiar to the operator.

B.5.3. Auger Electrons

When an electron is emitted from the inner shell of one of the specimen atoms it leaves behind a vacancy that could be filled by other electrons. The process releases a certain amount of energy which in some cases could lead to the emission of an electron from the outer shell; the process is called Auger effect, thus electrons produced during it are called Auger electrons.

B.5.4. X-Rays
X-rays could provide important information on materials nature; that is due to the fact that the generated x-rays have a wavelength (and an energy) peculiar of the type of atom which originated it, together with the layer's thickness and structure's strains, according to the previously developed theory.

**B.5.5. Cathode Luminescence**

Some specimen molecules could react and become fluorescent after being exposed to the electron beam; when the reaction happens in SEM it is called cathode luminescence. The phenomenon produces photons that can obviously be detected. It is also possible to label structures with fluorescent molecules and then detect them using cathode luminescence techniques. However this is usually not the purpose for which a standard SEM is used.

**B.5.6. Specimen Current**

When the primary electrons undergo to enough scattering their energy decreases and they are absorbed by the sample, for that reason in this case a current arises in the specimen. Changes in this parameter could be viewed and detected. This currents are usually led to ground, otherwise they could induce a negative charge on the region impacted by the electron beam interfering with the measurements. A solution to this problem is to ground the samples and coating them with conductive material.

**B.5.7. Transmitted Electrons**

In the case the specimen is thin enough some electrons from the primary beam may pass through it; these electrons, known as transmitted electrons, provide information about the atomic density of the sample (the higher the atomic number the darker the shadow will be, until no electron pass through the specimen).
However observing the energy spectrum of emitted electrons it is possible to recognize three main contributions to that at different energy levels coming from backscattered, secondary and Auger electrons (as empirically found).

Figure B.5 - Emitted electrons energy spectrum [7]

**B.6. Signal Detection and Manipulation**

The most important task of a SEM is to give topographical information that are mainly due to secondary electrons. The secondary electrons detector magnetically attracts the electrons by a potential applied to a ring around the detector (called Faraday Cup) and then it accelerates them by mean of a second potential of approximately $10^1\text{kV}$ on a target scintillator; image contrast is related to the number of electrons arriving at the detector from the sample, and thus it is related to the number of emitted electrons for incident electron.

For secondary electrons emission the yield depends on the work function of the material; it is higher for oxides and wide bandgap materials than for silicon. According to that principle metals, oxide and silicon patterns are clearly distinguishable from one another.

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Hitting the scintillator the electrons cause photons to be emitted; then they are led to a photomultiplier. The photomultiplier generates several electrons for any photon and makes possible to amplify and control the signal by mean of an external voltage control.

The detector should be as closer as possible to the primary beam without interfering with it to collect the most of the secondary electrons coming from the sample.

The signal is then converted to an image on a CRT screen; the screen is composed by pixels that can assume values in the shades of gray, from black to white. Adjustments on contrast and brightness parameters could be made varying controls.

![Diagram of secondary electron detector](image)

**Figure B.6 - Secondary electron detector [21]**

### B.7. Display and Record System

The important features in a display system are:

- **Brightness**: it is the value of each individual pixel composing the image; the higher the overall pixel value, the brighter the image;
Contrast: it is the difference between two pixels values; the overall contrast is the difference between the higher and the lower pixels values in the picture;

Resolution: it represents the ability to distinguish between two points; factors influencing the resolution are the beam spot size, the working distance, the aperture size and the voltage. Poor resolution will result in poorly defined edges and out of focus images;

Magnification: to change the device's magnification the only thing to do is to change the size of the pixels on the specimen. Magnification increases reducing the scanned area according to the law

\[ M = \frac{\text{Monitor Scanned Area}}{\text{Specimen Scanned Area}} \]

The main controllable parameters that define SEM's imaging modes are beam accelerating voltage (kV), probe convergence angle (\(a_{pc}\)), half of the angle of the cone of electrons converging onto specimen, probe current (\(i_p\)), incident current on the specimen necessary to imaging signals generation and probe diameter or spot size (\(d_p\)), diameter of the final beam at the specimen surface [23]. The various parameters are interrelated in many ways, so usually trade-offs are found to obtain the required performances;

Depth of field: it is the region of acceptable sharpness in a range around the focal point; it is a function of the distance between the sample and the final lens. Adjusting the working distance bringing the sample closer to the final lens will increase the resolution and decrease the depth of field, while increasing this distance will do the vice versa;
Noise: to have high quality images a high signal to noise ratio (SNR) is needed. Imposing a high scan speed will lead to lower SNR and thus to do not detect small size objects in the observation. A combination of high beam current and slow speed is required in order to detect small size objects with low contrast in a SEM.
Appendix C. Noise

C.1. Introduction

The term noise is related to undesired signals arising during measurements and caused by different processes; noise is also a stochastic process and thus during measurements it represents a disturb source, causing fluctuations from the real value of the quantity under measure. The study of noise is of particular importance to determine the detection limits; in fact noise sets an upper limit to the system gain (due to the fact that noise worsens the signal linearity) and a lower limit to the signal detection (due to the fact that signals component with amplitudes lower than that of the noise are indistinguishable from the noise itself and then cannot be detected).

Figure C.1 - Signal without and with noise example
In what follows additive noises will be considered such that a generic signal could be represented via the sum of three different terms:

\[ X(t) = \bar{X} + \hat{x}(t) + n(t) \]  \hspace{1cm} (C. 1)

where \( \bar{X} \) is the average signal, \( \hat{x}(t) \) is the time varying component of the signal and \( n(t) \) is the superimposed noise signal.

There are several quantities of interest in the study of random processes and signals. First, values are often referred to root mean square (RMS) values defined as

\[ x_{RMS} = \lim_{T \to \infty} \sqrt{\frac{1}{T} \int_{-T}^{+T} |x(t)|^2 dt} \]  \hspace{1cm} (C. 2)

that is a statistical measure of the magnitude of a varying quantity.

In the characterization of signals it is also possible to separate energy signals from power ones. A energy signal is a signal of non zero and finite energy, where the energy of a generic signal \( x(t) \) is calculated as

\[ E_x = \int_{-\infty}^{\infty} |x(t)|^2 dt \]  \hspace{1cm} (C. 3)

However a lot of signals treated in electronics do not belong to this group but are power signals; a power signal is a signal showing a non zero and finite power where the power of the signal is defined as

\[ P_x = \lim_{T \to \infty} \frac{1}{T} \int_{-T/2}^{T/2} |x(t)|^2 dt \]  \hspace{1cm} (C. 4)

The noise spectrum represents the distribution of power along the frequency range, allowing to know at what frequencies the most important signal components are located. The
spectrum is also known as Power Spectral Density (PSD) which can be calculated by considering a range of the entire signal (since the considered signals are power signals) and multiplying it for a window function such that

\[ x_T(t) = x(t) \text{rect} \left( \frac{t}{T} \right) = \begin{cases} x(t) & -T/2 \leq t \leq T/2 \\ 0 & \text{otherwise} \end{cases} \]  

where \( x_T(t) \) is the signal after being multiplied by the window function.

The PSD can then be defined as

\[ S_x(f) = \lim_{T \to +\infty} \frac{|F_{x_T}(f)|^2}{T} \]  

(C.6)

where \( F_{x_T}(f) \) is the Fourier Transform (FT) of the signal after the multiplication for the window function.

Properties of the PSD function are that it is never negative (\( S_x(f) \geq 0 \)), if the signal is real then the PSD is an even function (\( S_x(f) = S_x(-f) \)) and that it is possible to calculate the power of the signal knowing the PSD simply integrating it over the whole range of frequencies (\( P_x = \int_{-\infty}^{+\infty} S_x(f) \, df \)). Moreover, from Wiener-Khinchin theorem it is possible to state that

\[ S_x(f) = \hat{\Im}(R_x(\tau)) = \int_{-\infty}^{+\infty} R_x(\tau) e^{-j2\pi f \tau} \, d\tau \]  

(C.7)

that is to say that the PSD is the FT of the autocorrelation function of the process. Also there is another way to express the PSD in function of the RMS value of the signal as

\[ S_x(f) = \frac{x_{\text{RMS}}^2}{\Delta f} \]  

(C.8)

where \( \Delta f \) is the unit of the bandwidth, that is to say that it represents the power carried by the signal in a bandwidth of 1 Hz around the considered frequency.
PSD is a powerful mean to analyze circuits since, for linear time-invariant (LTI) systems, there is a theorem stating that

$$S_y(f) = S_x(f)|H(f)|^2 \quad \text{(C.8)}$$

where $S_x(f)$ is the PSD of the signal to measure and $H(f)$ is the system frequency response.

The most known noise process is White Gaussian Noise (WGN) characterized by having a null expected value and a certain standard deviation $\sigma$; it is equally distributed along all frequencies and it presents a probability density function (PDF)

$$p(x) = \frac{1}{\sigma \sqrt{2\pi}} e^{-\frac{x^2}{2\sigma^2}} \quad \text{(C.9)}$$
However this is not the only noise process to take into account; in particular noise processes with different parameters arise in different measurements and thus they must be studied in detail.

**C.2. Modeling**

Both voltage and current measurements are of interest and obviously both are affected by noise; thus it is important to correctly model this phenomenon in order to obtain a lumped parameter model to facilitate the understanding of how noise can affect the measurements.

In electrical circuits noise can arise from the sum of internal and external factors; internally noise is essentially due to fluctuations arising from the discrete nature of electrical charges and other factors, while external noise sources could be caused by coupling with other devices or other phenomena that affect the normal circuit’s behavior. However in this study only internal noises are of interest since the measurement equipment is built to reject external ones.
C.2.1. Correlated and Uncorrelated Noise Sources

As stated previously it is usually needed to take into account a lot of different noise sources to obtain the total noise contribution in the system. Dealing with stochastic processes it is not possible in general to apply the superposition principle to calculate the exit of the system, since it is needed to pay attention also to the correlation between the different random processes. For example for two different sources it is possible to write that the power is

\[
P_{av} = \lim_{T \to \infty} \frac{1}{T} \int_{-T/2}^{T/2} [x_1(t) + x_2(t)]^2 dt = \]

\[
= \frac{1}{T} \left[ \int_{-T/2}^{T/2} x_1^2(t) dt + \int_{-T/2}^{T/2} x_2^2(t) dt + 2 \int_{-T/2}^{T/2} x_1(t)x_2(t) dt \right] = \] (C.10)

\[
= P_{av1} + P_{av2} + \lim_{T \to \infty} \frac{2}{T} \int_{-T/2}^{T/2} x_1(t)x_2(t) dt
\]

where the last term denotes the correlation between the two considered signals. Usually different devices in a circuit produce uncorrelated signals and thus the third term will be zero, e.g. the noise produced by a transistor have no correlation to that produced by a resistor.

C.2.2. Lumped Parameters Circuit for Noise

It is possible to consider the effect of noises and take into account the same, using equivalent circuits; in particular noise effects can be represented as a single voltage noise generator connected in series to the input generator. This is possible thanks to well known network theorems (Thèvenin and Norton theorems in particular).

The noise sources are taken into account via their RMS values. The equivalent input noise allows to make comparisons between the noise input level itself and the input signal.
amplitude giving indication on how small could the amplitude of the signal be without having too low and thus unacceptable SNR

![Equivalent circuit schematics](image)

Figure C.4 - Equivalent circuit schematics

However this model fails since it could be thought that for large source impedances it is possible to reduce the effect of the noise on the output; this is not true since noise is an intrinsic phenomenon of the system that cannot be eliminated. To obtain a more reliable model however it is possible to consider a current source connected in parallel to the noise voltage generator producing noise at the network input.
Figure C.5 - Equivalent circuit schematics considering the noise current input generator where, as in the previous example, the network is considered noiseless.

C.3. Types of Noises

C.3.1. Johnson-Nyquist Noise

Johnson-Nyquist noise, more commonly known as thermal noise, is a type of disturb that arises from random fluctuations in electrons' motion in a material. This type of noise is peculiar of conductors and a statistical physical derivation of the same is related to the fluctuation-dissipation theorem [25] that has more general applications (e.g. the brownian motion).

As the name suggests this disturb is related to the temperature of the material, in fact for temperatures different from the absolute zero the electrons of the material show random vibrations, these vibrations being proportional to the conductor's temperature. Thermal noise is present in any conductive part of a circuit (i.e. in any resistance) and limits the detection of signals under a certain amplitude.

The PSD of the thermal noise is then proportional to the temperature and, according to Nyquist formulation, it is possible to write that

\[ S_v(f) = 4k_BT \]  

\[ (C.11) \]
where \( k_B \) is the Boltzmann constant, \( T \) is the absolute temperature and \( R \) is the resistance of the conductor. So, at given values of temperature and resistance, the PSD of the thermal noise is constant over all the frequency range and so thermal noise can be considered a particular case of WGN.

It is then possible to model the system as a noiseless resistor with resistance value \( R \) in series with a voltage generator of value \( V_{n,RMS} = \sqrt{4k_BTR\Delta f} \). It is also possible to use the Norton equivalent circuit in which the current of the generator has a value of \( I_{n,RMS} = \sqrt{4k_BTR\Delta f}/R \).

M.O.S.F.E.T.s, and transistors in general, also exhibit thermal noise problems; in particular, since the most resistive part of a F.E.T. is the conductive channel, it will give the largest contribution to noise signal. Moreover the contacts of gate, source and drain introduce in the system thermal noise due to the finite contacts' resistivity. These effects are controllable: the former can be regulated by M.O.S.' transconductance, while the latter can be reduced optimizing the layout of the device.

The dependence of the noise upon the temperature suggests that a method to reduce thermal noise related problems is to decrease the temperature of operation of the circuit, but it is not possible to decrease this parameter under a certain limit, also for a matter of cost of the required cooling equipment; it is then usually preferable to take into account the effects of the noise rather than reducing it.

**C.3.2. Flicker Noise**

Flicker noise is also referred to as 1/f noise, since it shows a PSD inversely proportional to frequency, or pink noise, since this power spectrum is like that of pink color in visible
spectrum. Since the spectrum of this signal is inversely proportional to the frequency it is possible to state that the interest in the study of the disturb is mainly related to its behavior at low frequencies, where the noise amplitude is higher.

![Pink noise PSD](image)

**Figure C.6 - Pink noise PSD**

This phenomenon is present in all electronic devices and it is due to different causes; it is believed that one of them, and in particular the one of interest in the conducted studies, is the trapping of carriers at interfaces between silicon and $SiO_2$ where it is possible to observe dangling bonds producing extra energy states. The carriers are randomly trapped and released and this mechanism produce a flicker noise in the current flowing through the structure related both to the carriers' mobility and number variations (this cause also another type of noise as described later in this section). However no definitive evidence has been reported on
what physical mechanism produce this type of noise and thus the causes are still object of debate.

The spectrum of the pink noise could be expressed in terms of current as [26]

\[ S_i(f) = k_0 \frac{1}{f} f^\beta \]

where \( k_0 \) is a constant, \( \beta \) is the current exponent and \( \lambda \) is the frequency exponent determining the behavior of the noise at the frequency variations and usually ranging from 0.7 to 1.3 (however it is often close to 1 at low frequencies and from that peculiarity the noise derive its name).

In terms of applied voltage another way to express the previous formula is

\[ S_v(f) = S_i(f) R_{\text{sample}}^2 = k_0 \frac{v_{\text{bias}}^2}{f^\lambda} \]

fixing \( \beta \) parameter to 2. The term \( k_1/f \) is smaller for single crystalline materials if compared with polycrystalline or amorphous ones.

Another important parameter is the corner frequency defined as the frequency at which the Johnson's noise equals the flicker noise.

### C.3.3. Shot Noise

Shot noise is due to the discrete nature of the carriers (that produces current pulses and not smooth current waveforms) and it arises particularly when the number of carriers in a structure is sufficiently small to produce significant uncertainties in the Poisson's distribution, describing the probability of a certain number of independent events to occur over a certain time period. Shot noise is then only a problem with small currents and it has a significant
component particularly over the corner frequency, since before it the predominant mechanism is the one related to pink noise.

The PSD of this type of noise can be written as

\[ S_i(f) = 2ql \]  \hspace{1cm} (C.14)

where \( q \) is the elementary electric charge and \( I \) is the average current in the structure. It could be noticed that in this representation the current spectrum of the shot noise is constant with the frequency and then the noise can be treated as a WGN.

![Figure C.7 - Representation of the frequency distribution of the different types of noise [27]](image)

C.3.4. Generation/Recombination Noise

The presence of defects in the crystal and in general of permitted states in the forbidden bandgap can create a generation/recombination mechanism due to the random trapping and releasing of the carriers in these trap sites. Related to that a type of noise, also called generation/recombination (GR) noise arises in the measurements.
Since both Si and SiGe are indirect bandgap materials the GR process follows the Shockley-Read-Hall (SRH) model in which, for energy conservation and crystal momentum conservation principles, a third element besides the hole and the electron is needed; this element is called phonon and the process occurs following four sub processes:

✓ *electron capture*: an electron from the conduction band is captured in an allowed empty state in the forbidden bandgap. The excess energy $E_c - E_t$ (with $E_c$ energy of the conduction band and $E_t$ energy of the trap) is transferred to the lattice via phononic emission;

✓ *hole capture*: one of the captured electrons is absorbed in the valence band, recombinating with a hole of the same band. A phonon with $E_t - E_v$ energy is generated;

✓ *electron emission*: a captured electron is reabsorbed by the conduction band. A phonon of energy $E_c - E_t$ is absorbed;

✓ *hole emission*: an electron from the valence band is captured, leaving a hole in the same band. A phonon of energy $E_t - E_v$ is absorbed.

![Schematic of the GR trap assisted process](image)

Figure C.8 - Schematic of the GR trap assisted process (the white dots represent holes, while the black ones electron) [5]
The phenomenon is characterized by a PSD given by a Cauchy-Lorentz distribution. Analytical formulas for the electrons distribution and the PSD are given by

\[ p_N(f) = 4\sigma^2 \frac{\tau}{1 + (2\pi f \tau)^2} \quad (C.15) \]

where \( \sigma^2 \) is the mean squared value of the variance of the distribution and \( \tau \) is the carriers lifetime; moreover

\[ S_{v,GR}(f) = \frac{K_{GR}}{1 + (2\pi f \tau)^2} \quad (C.16) \]

where \( K_{GR} \) is a parameter peculiar of the type of material.

Figure C.9 - Cauchy-Lorentz distribution [27]
Appendix D. Theory of Oxygen Absorption at Si/SiGe Interfaces

First of all from the mechanical quantum theory of chemical bonding two different types of adsorption arise: physisorption and chemisorption. These two mechanisms are related to the binding energy between the structure's atoms and the adsorbed molecule or atom.

D.1. Physisorption

Physisorption, also known as physical absorption, is an absorption process in which the electronic structure of the molecule or atom undergoes to a slightly perturbation. The fundamental interacting force to be considered in the process is the van der Waals force; in the process correlated charge fluctuations generate an attractive force between two bonding partners inducing dipoles.

To model non-reactive molecules physisorption on a solid surface a measurement by an oscillator can be executed. In fact this case can be compared to the simple harmonic one dimensional motion of an electron. The location of the atom is at a certain distance $z$ outside the solid surface. The main factor influencing the van der Waals forces between the surface and the atom is the time dependent interaction between the valence electrons and the nucleus with their images. In fact an image-charge attraction can describe the van der Waals attraction as the screening effect of the solid substrate [28].
Since the binding energy in a van der Waals' interaction is low the physisorbed atoms are placed at large distance from the surface and they can move in a plane parallel to the substrate. Usually physisorption arises at low temperatures since at room temperature (with a potential energy of approximately 25 meV) it is impossible to have the bonding, as shown in Figure D.1.

![Figure D.1. Calculated physisorption potentials for He atoms outside Ag, Cu, Au and Li surfaces](image)

**D.2. Chemisorption**

Chemisorption, also called, chemical adsorption, is a process in which covalent or ionic bonds are formed and at the same time new molecular orbitals are formed, having, thus, a significant perturbation of the electronic structure of the molecule or atom.

Chemisorption can be defined by considering the simple adsorption system shown in Figure D.2. This system is composed of a transition metal with an energetically sharp, partially-filled d-band, and a molecule with a partially-filled molecular orbital M. Approaching a molecule to the metal surface a covalent bonding can arise between the
partially-filled orbitals (M and d) which is followed by chemisorption. At the same time rehybridisation and formation of new Md orbitals occur. The strongest bonding arising from a such a process happens in the case of overlapping between occupied and unoccupied orbitals. Transferring an electron into the Lowest Unoccupied Molecular Orbital (LUMO) and donating an electron from the Highest Occupied Molecular Orbital (HOMO) into an empty substrate state explain this phenomenon.

![Figure D.2. Simple model of covalent chemisorption bonding between a molecule and a transition metal with partially filled d bands](image)

Chemisorption potential is defined as a function of the distance z between the atom or molecule and the surface and it is characterized by a short equilibrium separation $z_0$ [1÷3] Å and a high binding energy $E_B$ in the eV range.
Rapid adsorption happens when the clean metal surface is exposed to $H_2$; the process will be followed by the dissociation of the molecules producing atomic species H bonded to the surface. Approaching to the surface from a large distance $z$ shows physisorption behaviour for potential which is decreasing until potential minimum $z_0$. Approaching closer to the surface causes a rapid increase in the potential energy due to overlapping between the molecule’s electronic shell and the metal states.

**D.3. Adsorption of Oxygen and Water on Silicon Surface**

$O_2$ and $H_2O$ make permanent dipole moments at the surface and adsorption bonding takes place due to the charge transfers between the substrate and the adsorbed atom or molecule. The interaction between parallel dipoles is repulsive [28].

Depending on the coverage of exposing gas and other parameters, different phenomena may occur. As shown in Figure D.4, there are two kinds of 2D distributions that can take place.
on the surface: a random one for a diluted phase or an ordered one in which the adsorbed atoms assume a crystalline configuration and tend to form islands on the surface.

Figure D.4. 2D adsorbate phases

Temperature variations can cause one state to change into the other. Besides the temperature other two parameters play a main role in the considered transitions, and thus three parameters have to be taken into account:

✓ temperature;

✓ pressure;

✓ density.

When oxygen reacts with the silicon surface, the work function increases and the electron affinity too. This implies that all of these interactions could be considered as chemisorptions. Due to the annealing, physisorbed oxygen evaporates while chemisorbed atoms or molecules do not. This means that the physisorbed adsorbates can move on the surface at low temperatures while at high temperatures just chemisorbed atoms exist on the surface. The physisorption and chemisorption of oxygen on a silicon surface can take place in different configurations and direction of bonding related to the energy of atoms or molecules [29].
Figure D.5. Various physisorbed and chemisorbed configurations of oxygen on a silicon substrate [29]
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