



Doctor of Philosophy in
Physics

INVESTIGATION OF STRUCTURAL, OPTICAL AND ELECTRICAL PROPERTIES OF MBE GROWN Si-Ge THIN FILMS

by

İsa ŞEKER

January 2015

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ABSTRACT

SiGe nanoislands have been grown by Molecular Beam Epitaxy (MBE) on Si (100) substrates with various layer designs and growth conditions. Multilayered thin films of these structures with different thicknesses were fabricated by changing the substrate temperature, Ge content, annealing temperature and annealing duration. Structural properties of the surfaces were examined by Reflection High Energy Electron Diffraction (RHEED), Grazing Angle X-Ray Diffraction (XRD), Atomic Force Microscopy (AFM) and Scanning Electron Microscopy (SEM). The optical investigation was acquired by Dispersive Raman Spectroscopy and the electrical characterization was performed by Semiconductor Analysis method. It has been observed that the surface morphology of the nanostructures can be tuned by changing their layer designs and growth parameters. The present work reports on the evolution of Ohmic, Semiconductor and Tunneling Diode type current-voltage (I-V) characteristics of the samples and the alteration of these electrical properties by LED illumination.

Closely stacked Ge rich nanoislands have shown Ohmic type I-V characteristics whereas intermixed SiGe nanoislands gained semiconducting type current flow mechanism. The tunnel diode type I-V conduction mechanism was observed in Si rich nanoislands which have isolated form. Fabrication of room temperature heterojunction SiGe tunnel diode structure with a peak-to-valley ratio of 1.45 at 20V without p-n doping is reported. It is also found that the tunnel diode structures respond to visible light excitation simultaneously switching between the tunneling and semiconducting type conduction mechanisms.

Keywords: Molecular Beam Epitaxy, SiGe Nanoisland, Heterojunction, Tunnel Diode

MBE İLE BÜYÜTÜLMÜŞ Si-Ge İNCE FİLMLERİN YAPISAL, OPTİK VE ELEKTRİKSEL ÖZELLİKLERİNİN İNCELENMESİ

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ÖZ

SiGe nanoadaları değişik katman tasarımı ve büyütme şartlarıyla Si (100) altlıklar üzerine Moleküler Demet Epitaksi (MBE) ile büyütülmüştür. Bu yapıların çok katmanlı ince filmleri altlık sıcaklığı, Ge içeriği, tavlama sıcaklığı ve süresi değiştirilerek farklı kalınlıklarda üretilmiştir. Yüzeylerin yapısal özellikleri Yüksek Enerjili Yansıyan Elektron Kırınımı (RHEED), Sıyran Açılı X-Işını Kırınımı (XRD), Atomik Kuvvet Mikroskopisi (AFM) ve Taramalı Elektron Mikroskopisi (SEM) ile incelenmiştir. Optik analiz Dağıtıcı Raman Spektroskopisiyle, Elektriksel karakterizasyon ise Yarıiletken Analiz tekniğiyle gerçekleştirilmiştir. Nanoyapıların yüzey biçimlerinin numunelerin katman tasarımları ve büyütme şartları ayarlanarak değiştirilebileceği gözlemlenmiştir. Bu çalışmada numunelerin Omik, yarıiletken ve tünel diyot tipi akım-gerilim (I-V) özelliklerinin ortaya çıkışı ve bu elektriksel özelliklerin LED aydınlatmasıyla değişimi rapor edilmiştir.

Sıkı istiflenmiş Ge içeriği yüksek nanoadalar Omik tip I-V özelliği gösterirken iç içe geçmiş SiGe nanoadaları yarıiletken tipi akım geçiş mekanizması göstermiştir. Si içeriği zengin nanoadalarda ise tünel diyot tipi I-V iletim mekanizması gözlemlenmiştir. 20V'ta oda sıcaklığında çalışan, 1.45 zirve/vadi oranlı heteroeklem SiGe tünel diyot yapısının p-n katkılanma olmaksızın elde edilmesi rapor edilmiştir. Ayrıca, tünel diyot yapılarının görünür ışıkla uyarılmaya tünelleme mekanizmasından yarıiletken tipi ilettime anlık geçiş yaparak tepki verdiği tespit edilmiştir.

Anahtar Kelimeler: Moleküler Demet Epitaksi, SiGe Nanoada, Heteroeklem, Tünel Diyodu

To my dear wife Aysun and daughters Dilara and Azra

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LIST OF SYMBOLS AND ABBREVIATIONS

SYMBOLS

R_a	Average roughness
k	Boltzmann constant
θ	Bragg's angle
K_α	Characteristic wavelength
μ	Chemical potential
q	Electric charge
v	Electron velocity
φ	Energy of desorption
P_∞	Equilibrium vapor pressure
$G(n)$	Gibbs free energy
α_∞	Intercept of the asymptote
d	Lattice spacing
ϕ	Spreading pressure
σ	Surface energy

ABBREVIATIONS

BE	Backscattered Electron
BiCMOS	Bipolar Complementary Metal Oxide Semiconductor
CCD	Charge-Coupled Device
CMOS	Complementary Metal Oxide Semiconductor
CVD	Chemical Vapor Deposition
DI	De-Ionized
EDS	Energy Dispersive Spectroscopy
EFM	Electric Force Microscopy
FEG	Field Emission Gun
FET	Field Effect Transistor
HBT	Hetero-Junction Bipolar Transistor
IC	Integrated Circuit
ITO	Indium-Tin-Oxide
LED	Light-Emitting Diode
LEEM	Low Energy Electron Microscopy
LFM	Lateral Force Microscopy
LPE	Liquid Phase Epitaxy
MC	Main Chamber
MFC	Mass Flow Control
MFM	Magnetic Force Microscopy
MOVPE	Metal-Organic Vapor Phase Epitaxy
NANOGEN	Nanoparticle Generation
NC	Nanocrystal
QCL	Quantum Cascade Laser
QDIP	Dot Infrared Photo Detector
RCA	Radio Corporation of America

RGA	Residual Gas Analyzer
RHEED	Reflection High Energy Electron Diffraction
SC	Standard Clean
SE	Secondary Electron
SEI	Secondary Electron Imaging
SIMS	Secondary Ion Mass Spectroscopy
STM	Scanning Tunneling Microscopy
UHV	Ultra High Vacuum
VLSI	Very Large Scale Integrated Circuit

CHAPTER 1

INTRODUCTION

1.1 MOTIVATION FOR STUDY

Nanotechnology revolutionized many areas of science and technology as it enables the manipulation of materials at the atomic or molecular level [1]. Nano-patterned structures of semiconductors like quantum wells and dots offer great promise for many applications in the fields of electronics, medicine, and energy. Heterostructures of these materials make it possible to tailor light-emitting devices using not only the direct bandgap semiconductors but also indirect bandgap materials [2]. As an example of these investigations, Silicon-Germanium (SiGe) heterojunction bipolar transistors (HBT) and bipolar complementary metal oxide semiconductors (BiCMOS) became the commercial fruits of this technology in many important circuit and system applications worldwide [3]. Although, SiGe HBTs are widely used in radio-frequency telecommunications and high-bandwidth instrumentation [4] in the cellular industry, the applications in optical networking and automotive collision avoidance radar systems are projected to represent future high growth areas for these systems [3].

Si is the most abundant, economic and widely used material in the semiconductors family [1,4]. As a result, its industrial processing technology has developed tremendously over the years [1]. In addition, it has superior structural properties including ease of processing and the maturity of the existing technology in the integrated circuits (IC) industry [5]. Hence, it is more advantageous to process it as a wafer by means of well established, very large scale integrated circuit (VLSI) fabrication techniques [1] with much lower cost compared to wafers such as GaAs, InP etc. [4]. Furthermore, Si has higher thermal conductivity than many III-V direct bandgap semiconductors like GaAs and AlAs [6]. However, the fact that Si has an

indirect bandgap and lower electron mobility are its disadvantages compared to its III-V counterparts [7]. There have been many efforts to address the challenges posed by Si such as the introduction of point defects, optical excitation of Raman lasers and rare earth dopants etc. [8].

Ge is an indirect bandgap material and it can perform like a direct one by means of band engineering through straining and doping effects [9] so, Ge leads to improvements in group-IV optoelectronic device applications. It is also compatible with Complementary Metal Oxide Semiconductor (CMOS) and provides stronger confinement than Si nanocrystals due to its larger radius and higher dielectric constant [10]. Ge, which can also function well as a photo-detector, is more compatible with Si than the III-V semiconductors due to their extremely large lattice mismatches [11].

III-V semiconductors such as GaAs have been used for optoelectronic devices since they are direct bandgap materials instead of Si and Ge with the indirect bandgaps of 1.12 eV and 0.67 eV respectively [5]. Despite their indirect bandgap nature, significant amount of research has been carried out to develop SiGe based optoelectronic devices [5].

It requires great effort to achieve high quality of Ge or $\text{Si}_{1-x}\text{Ge}_x$ hetero-epitaxial layers on Si substrates because of the large 4.18 % lattice mismatch relative to Si [12]. Since the good quality of Ge growth on Si demands high temperature process, novel deposition techniques must be developed in order to obtain a practical back-end fabrication process without impurity segregation and diffusion [11].

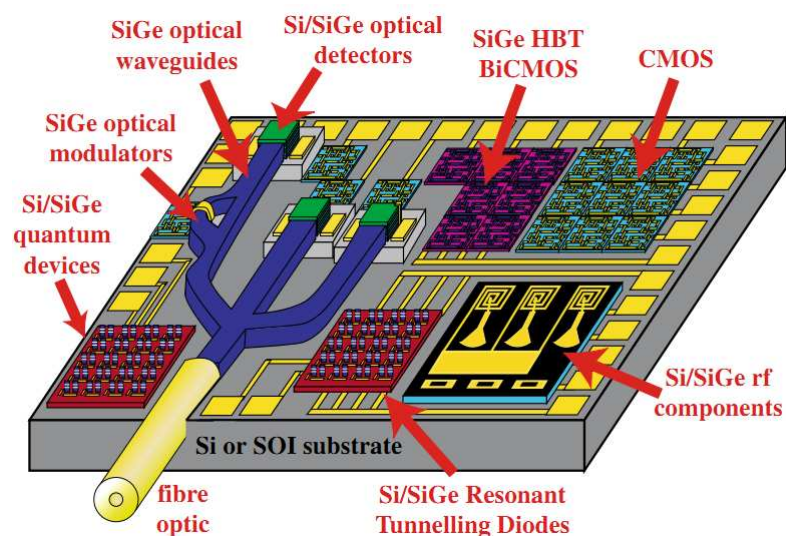


Figure 1.1 A Future schema of monolithic integration of SiGe devices on Si chip [13].

MBE grown SiGe hetero-structures such as nano islands have attracted great interest owing to their high-performance in hybrid photonics circuitry within CMOS platforms [14]. Monolithic integration of SiGe optoelectronic devices with CMOS that is depicted in Figure 1.1 can acquire lower cost, lighter weight and greater speed than separate chips performing distinct functions [15]. Higher speeds in telecommunications can be achieved using inter-chip optical interconnects which can overcome signal degradation, RC delay, electromagnetic interference, and high losses by power dissipation [16-18]. Moreover, the monolithic integration of electronic and optical components of SiGe on Si chip including optical interconnections is predicted to enhance the performance and functions of Si Very Large Scale Integrated circuits (VLSI) [19].

The device performance of the SiGe islands can be improved by differentiating the substrate growth temperature, Si buffer layer thickness, Ge content, Ge deposition rate, annealing time and annealing temperature. The field of hetero-epitaxy increased the diversity in substrate engineering which enhanced the fabrication of heterojunction devices in the last decades [4].

SiGe resonant tunnel diodes have possible applications in digital and high frequency devices in combination with high-speed field effect transistors [20,21]. Closely stacked and vertically aligned Ge islands that are grown in Stranski-Krastanov

mode on p-type Si substrates can be tailored as resonant p/i/p intraband tunneling diodes [22]. Channels with energetically deep thermalization layers are formed between the vertically aligned Ge islands and Si double barriers for holes in the valence band [22].

1.2 BACKGROUND OF SiGe HETEROEPITAXY

Molecular beam epitaxy (MBE) method was first introduced by J. R. Arthur and A. Y. Cho for the growth of GaAs structures in 1968 [23,24]. Subsequently, many other techniques such as chemical vapor deposition (CVD), liquid phase epitaxy (LPE) and metal-organic vapor phase epitaxy (MOVPE) were used on a variety of materials for epitaxial film growth [2]. By MBE method, thin films are grown in ultra-high vacuum medium and the system enables the efficient control for the formation of atomic monolayers. Furthermore, MBE makes the monitoring of the dynamics of the growth process possible with the implemented Reflection High Energy Electron Diffraction (RHEED) system.

In 1975, Kasper and his co-workers attempted to grow epitaxial $\text{Si}_{1-x}\text{Ge}_x$ alloys on Si substrates using MBE [25]. Since then, there was a great effort to grow such films on Si in order to build heterostructural devices [25]. These early attempts suffered from the poor surface morphology due to the high growth temperature of Si [26]. Bean and his co-workers could succeed growing a reasonable device quality strained $\text{Si}_{1-x}\text{Ge}_x$ crystal on Si in 1984 [26]. There have been many improvements in the growth of $\text{Si}_{1-x}\text{Ge}_x$ alloys and Si/ $\text{Si}_{1-x}\text{Ge}_x$ multilayered structures in years as shown in Table 1.1. For example, in 1988 Gary L. Patton *et.al* reported the first Si/Ge-based heterojunction bipolar transistor (HBT) grown by MBE [27]. Recently, by the commercialization of HBT many researchers have engaged in the development of Field Effect Transistors (FET) which has wide area of applications.

Table 1.1 Milestones in the development of SiGe strained layer epitaxy [3].

Year	Historical Event
1960	First epitaxially grown layer to be used in a transistor
1963	First investigation of high temperature Si epitaxy
1975	First growth of SiGe strained layers
1984	First growth of SiGe epitaxy by MBE
1985	First measurements of energy bandgap in SiGe strained layers
1988	First SiGe based HBT
1992	First SiGe HBT BiCMOS technology
1996	First SiGeC HBT
2001	First SiGe HBT with 200 GHz frequency

1.3 SiGe BASED NANOSTRUCTURES

1.3.1 Bandgap Engineering in SiGe

Introducing Ge into Si makes it a suitable candidate for bandgap engineering [28]. Figure 1.2 shows the bandgap versus lattice constant for several common semiconductors used for device fabrication.

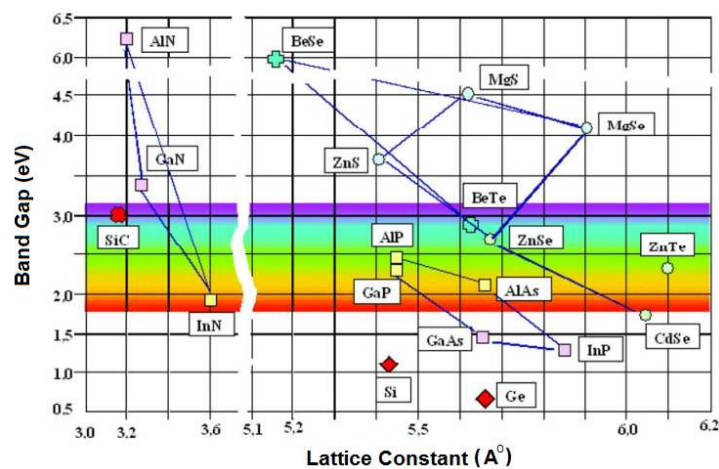


Figure 1.2 Bandgap versus lattice constant for various semiconductors [29].

It can be seen that Si and Ge operate in the infrared regime whereas GaP and AlAs act in the visible zone. The suitable manipulation of the bandgap determines the emission and/or absorption wavelengths of the optoelectronic devices like detectors, solar cells, light-emitting diodes (LED) and lasers [4].

1.3.2 Ge Nanocrystals (NC) and Quantum Dots

Structural, optical and electronic properties of the NCs are heavily influenced by the growth conditions [30,31]. Ge NC assemblies experience a tensile or compressive stress depending on the embedding matrix and their fabrication that can lead to the alteration of the band structure [31]. Thus, the result of enhancement in optical properties via strain makes Ge NCs a good candidate for optoelectronics and telecommunications applications [32]. For instance, these NCs, which are compatible with conventional CMOS processing techniques are good candidates as high-efficiency, low-cost, NC sensitized, thin film solar cells [1].

Ge quantum dot (QD) structures, which are also named as nanoislands are grown on Si substrates utilizing the concept of the self-organization that is based on the strained-layer hetero-epitaxy of semiconductor thin films [33]. These nanostructures can be fabricated without the requirement of lithography processes [34].

The utilization of the surfactants including boron, phosphorus, antimony, and carbon plays a significant role to exploit the surface properties of the substrate and growing layer [5,35]. Moreover, the surfactants also modify the surface strain field reducing the adatom surface mobility that results in the formation of smaller and more uniform islands [5,36]. Hence, the spatial localization of charge carriers in discrete energy states can be facilitated for quantum device applications such as light emitters or detectors [37].

1.4 HETEROEPITAXY DEVICE APPLICATIONS

SiGe heteroepitaxy devices can be used in high-density digital circuitry and sensor applications in a wide range of wavelengths between infrared and microwave regions [4]. Many devices in commercial use including solar cells and high-mobility

transistors and those which are still in research phase like quantum cascade lasers (QCL) require semiconductor heterojunctions [4]. A brief list of important device developments like Si metal oxide FETs (MOSFET), photo-detectors, waveguides and solar cell in history is given in Table 1.2.

Table 1.2 Improvement in SiGe FETs and other devices in years [3].

Year	Historical Event
1960	First Si MOSFET
1963	First Si CMOS
1986	First SiGe photodetector
1990	First SiGe waveguide
1992	First SiGe solar cell

It is clear that, despite the discovery of numerous novel materials, Si based technologies maintain its vitality for the IC industry.

1.4.1 Solar Cells

Among renewable energy technologies, solar energy is highly attractive in many aspects to respond the constantly increasing demand for energy. However, the challenge lies in producing the solar power more competitive compared to conventional technologies due to the constraints by materials and fabrication costs [1]. For instance, Si is widely used in conventional crystalline solar cells; however, it is vital that they need to be fabricated as in the form of thin films with organic components to make them cost-effective hybrid solutions for very large scale applications.

Nanostructure based photovoltaic devices could present high efficiency solutions since these structures promise better performance solar cells by broadening the spectrum of absorbed wavelengths [38]. Ge is a suitable material that is used in conjunction with III-V based systems monolithically to obtain high-efficiency multi-junction solar cells as it can work well in the infrared portion of the solar radiation

[5,39]. For example, Ge quantum dots can be incorporated in Si epitaxial solar cell that is shown in Figure 1.3 to improve long wavelength performance [40].

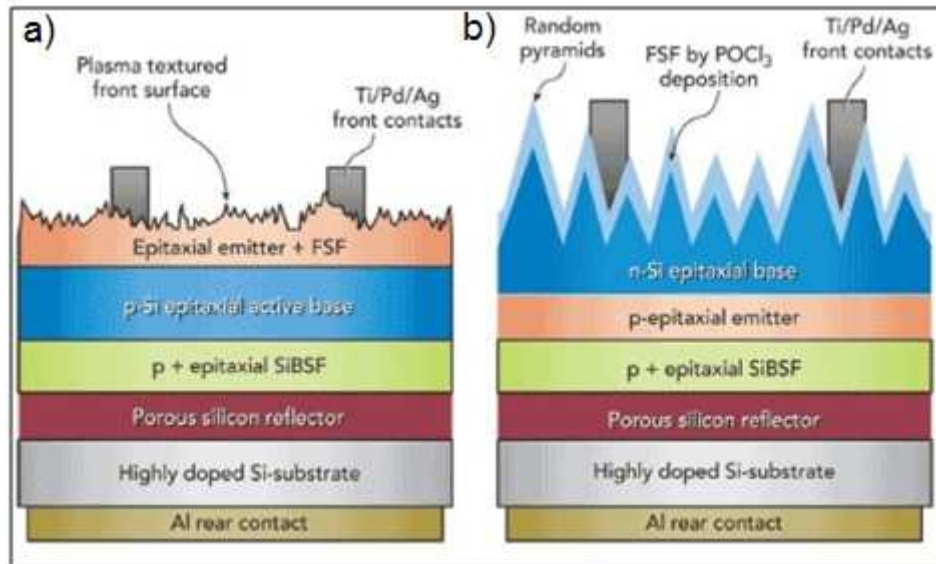


Figure 1.3 Epitaxial Si solar cell designs with a) plasma textured front surface, b) pyramid shape top layer [40].

Ge substrates of III-V active multi-junction solar cells can be replaced by epitaxial growth of Ge on cheaper Si substrates to make them suitable for terrestrial applications along with the more expensive space power solar panels.

1.4.2 Photonics, Lasers and Photodetectors

In recent years, incorporation of Ge in Si promises to achieve the electrically pumped lasing near optical telecommunication wavelengths and strained Ge attracts considerable interest in light emitting device applications [9]. Due to the lack of low bandgap semiconductors working in the mid- and far-infrared wavelengths regions efforts on these studies attract much attention [13]. Ge nanostructures can operate at room temperature owing to their low coupling of optical phonon scattering [13].

Ge quantum dot infrared photo detectors (QDIP) have been designed for detection of mid-infrared light and are also of great interest due to the advantages of small dark

current and high operating temperature [41]. Furthermore, SiGe quantum dot light emitting diodes (QD-LED) could be efficient candidate in near future in general solid state lighting as they consume very little power.

In the last decade, SiGe nanostructures attract also much attention as suitable neural interfaces. Both self-assembly and lithographic implementation of these nanostructures are biocompatible. Figure 1.4 represents the colorized electron image of a hippocampus neuron on a silicon chip with linear array of p-type buried-channel transistors after 8 days in culture.

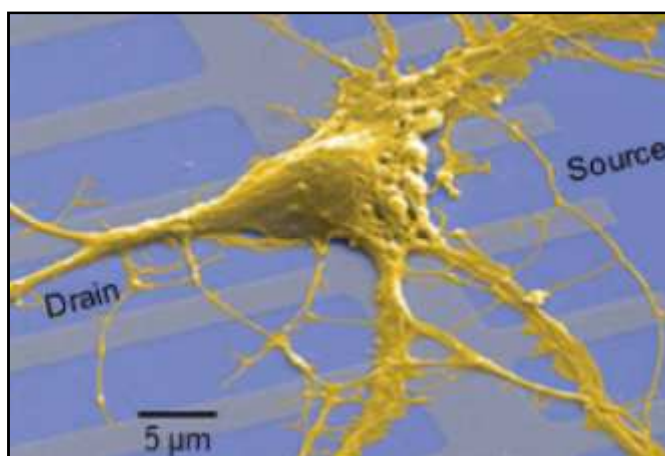


Figure 1.4 Colorized electron image of a hippocampus neuron on a silicon chip [42].

The stimulation of neuron cells that are seeded on SiGe heterostructures and transistors are being extensively studied [42] since they promise new solutions for the treatment of neural diseases.

1.5 DISSERTATION OVERVIEW

The present PhD study focuses on evaluating and validating the influence of growth conditions on structural, optical and electrical properties of SiGe nanoislands. For this purpose, a number of characterization methods are performed using Reflection High Energy Electron Diffraction (RHEED), Grazing Angle X-Ray Diffraction (XRD),

Atomic Force Microscopy (AFM), High Resolution Scanning Electron Microscopy (HR-SEM), Raman Spectroscopy and Semiconductor Analysis.

Chapter 2 presents the theoretical background of SiGe hetero-epitaxy. Basic terms and definitions of the topic will be mentioned with a comparative approach of statement between the concepts of crystal growth and epitaxy. In addition, the mathematical derivations of the three well-known growth modes of Frank Van der Merwe, Volmer-Weber, and Stranski-Krastanov were given and their properties were discussed in detail.

In chapter 3, basics of MBE technique, design of samples and the whole fabrication process were summarized. Furthermore, the technical details of the MBE system, starting from the insertion of the sample to main chamber until the end of the annealing process are explained. The layer growth conditions such as growth temperature and rate etc. have been shown in detail.

Chapter 4 outlines the characterization methods that are used for analysis of samples. All the characterization techniques of RHEED, XRD, AFM, SEM, Raman Microscopy and Semiconductor Analysis are explained and schematized. In addition, the technical details, working principles and the capabilities of the systems and devices are explained.

Chapter 5 details the study of structural, optical and electrical properties of the samples. Grazing Angle XRD graphs and images of AFM and SEM measurements are evaluated as structural characterization in a comprehensive and comparative manner. Meanwhile, the results of Raman Microscopy were discussed as optical analysis whereas I-V measurements were evaluated as electrical characterization.

In the conclusion chapter, the summary of the results of the dissertation is presented and a future work for a potential research is foresighted briefly.

CHAPTER 2

THEORY

2.1 BASIC CONCEPTS AND DEFINITIONS

The term epitaxy, which is derived from the Greek words epi - on and taxy -in order, refers to the oriented growth of a crystalline material on another single crystal surface [43]. One can propose that the structure and the energy of the epitaxial interface play a predominant role in determining the epitaxial orientation [44]. It is vital to compare the concepts of epitaxial growth and crystal growth, in other words, the growth of a single crystal film on the surface of the same material [45]. The main difference between epitaxial growth and crystal growth are due to the nature and strength of the chemical bonds of both substrate and deposit crystals [46]. The usual crystal growth takes place when both crystals do not differ simultaneously energetically and geometrically which means that the chemical potentials of the substrate μ_s and the deposit μ_d are equal [47]. On the contrary, epitaxial growth occurs only when the chemical potentials of the deposit and the substrate are different.

The term epitaxy can be categorized in two as homoepitaxy and heteroepitaxy depending on the type of the atomic species that are grown one top of the other one. Assume the case of deposition of intrinsic Si on B doped single crystalline Si [48]. Although nature and strength of the chemical bonds in both substrate and deposit crystals are identical to each other, the deposit film should be compressed to match the substrate due to the presence of the dopant in the substrate. Then the chemical potential of the strained deposit which is defined as strain energy per atom differs from that of the substrate crystal. Thus, chemical potentials $\mu_s \neq \mu_d$ and the difference is solely due to

the difference of the lattice parameters, and this case is called as homoepitaxy [44]. On the other hand, when the two materials differ geometrically, their bonds differ in strength; hence their chemical potentials become different. As a result, the phenomenon called heteroepitaxy arises.

The orientation of the epitaxial process depends on the structure of the crystal planes and the bonding of the substrate regardless of the process of growth itself. As a result, the same processes of adsorption and desorption, coalescence, surface diffusion of adatoms and incorporation into kink sites are valid in the case of vapor deposition as shown in Figure 2.1 [44,49].

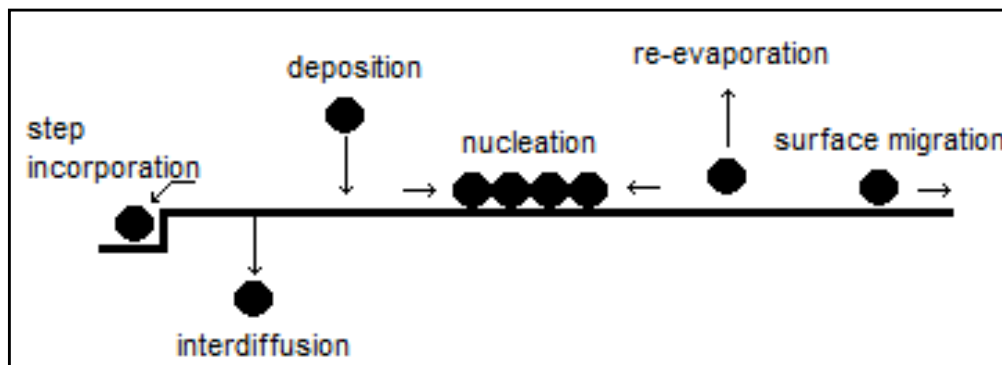


Figure 2.1 Schematic illustrations of major growth processes [49].

2.1.1 Epitaxial Growth

The film thickness of the deposit crystal changes the chemical potential of the overlayer $\mu(n)$ since the atoms of the deposit can be bound more loosely or more tightly to the substrate atoms [50,51]. In other words, chemical potential can be altered by straining the film periodically [52]. Misfit dislocations which are described as lattice distortions in the vicinity of the epitaxial interface constitute the main difference between epitaxial growth and usual crystal growth [53,54]. Figure 2.2 indicates the epitaxial Ge growth on Si in pseudomorphic and strain relieving in a misfit dislocation site.

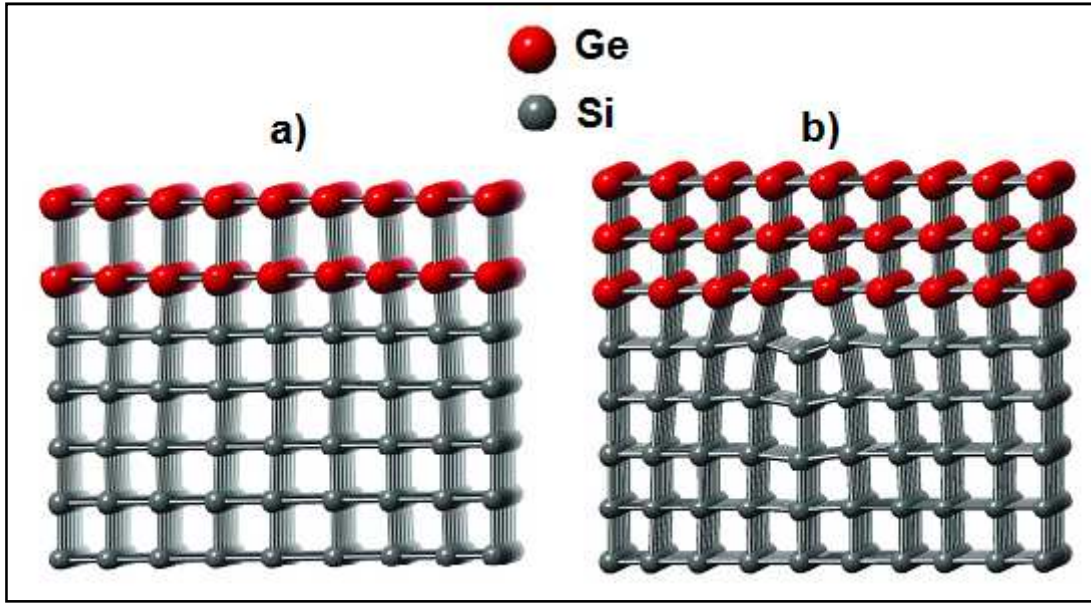


Figure 2.2 a) Pseudomorphic growth of Ge on Si, b) a misfit dislocation site [4].

This leads to the evolution of the three well-known epitaxial growth mechanisms: (i) Frank Van der Merwe mechanism or layer-by-layer growth (ii) Volmer-Weber mechanism or islanded growth and (iii) Stranski-Krastanov mechanism or layer-by-layer growth followed by formation of isolated 3D islands [55]. The equilibrium shape of the deposit crystal is determined in terms of surface energies [45].

The change in surface energy

$$\Delta\sigma = \sigma + \sigma_i - \sigma_s \quad (2.1)$$

shows a degree of the wetting. Incomplete wetting leads to the following equation;

$$\sigma_s < \sigma + \sigma_i \quad (2.2)$$

resulting in the film growth as 3D islands [44]. However, the complete wetting gives the equation of

$$\sigma_s > \sigma + \sigma_i \quad (2.3)$$

which favors the layer-by-layer growth when misfit is negligible, or Stranski-Krastanov mode at a perceptible misfit [46,47].

2.2 GENERAL TENDENCIES

The applicability of the growth modes can be more or less summarized in main tendencies for the formation of any kinds of chemical bonds [53]:

- i. The formation of isolated 3D islands is favored more than monolayer growth when the interracial bonding is weaker than the bonding with the deposit itself.
- ii. High substrate temperature (see Fig. 2.3) during the growth leads to 3D islanding both in Volmer-Weber and Stranski-Krastanov mechanisms. Besides, 3D crystallite size is directly influenced by the increase in substrate temperature.
- iii. Layer-like growth is favored in the case of the higher deposition rates.
- iv. The lattice misfit plays a predominant role in the formation of island-like growth.
- v. The crystallographic orientation of the substrate also plays an important role in determining the mechanism of growth. For instance, the more densely packed substrate planes result in greater layer-like growth in comparison with less densely packed planes.
- vi. The existence of surface-active surfactants such as B, P dopants favors the deposition of planar film growth rather than clustering.

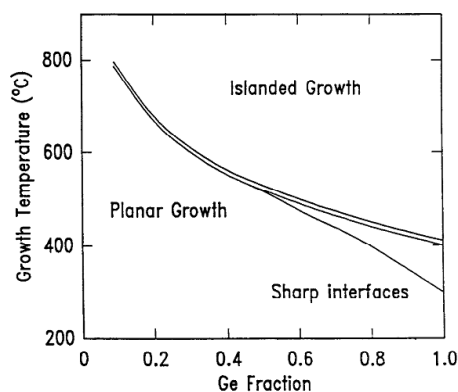


Figure 2.3 Effect of growth temperature and Ge content on growth type [53].

2.2.1 Wetting and Clustering

Gibbs free energy $G(n)$ is defined as the energy of a uniform adsorbed layer on a substrate that is a monotonically decreasing function of the amount n of atoms per unit

area [44,56]. Therefore, the chemical potential $\mu = \frac{dG}{dn}$ must be negative. At ($n = 0$), the interception with the G-axis represents the free energy of the bare substrate, the free surface energy, σ_{s0} of the interface substrate-vacuum. When the thickness of the adsorbate is big at large n values its properties approaches the properties of the bulk adsorbate. In other words, $G(n)$ tends asymptotically to the free energy of the bulk adsorbate. The asymptote equation becomes

$$G_{\text{asym}} = n\mu_{\infty} + \alpha_{\infty} \quad (2.4)$$

where α_{∞} is the intercept of the asymptote with the G-axis.

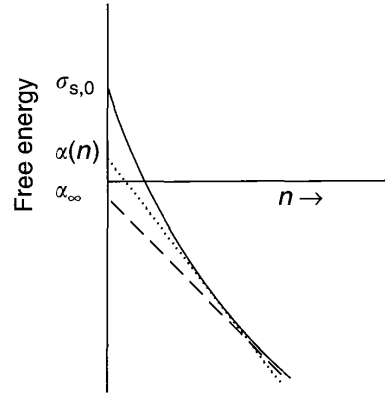


Figure 2.4 The Gibbs free energy graph of a uniform film per unit area versus number of atoms per unit area n [56].

Since G is free energy per unit area and α_{∞} is a constant, it means that α_{∞} is surface free energy. The system consists of a substrate and a thick adsorbate at large n that means there are two interface boundaries - substrate-adsorbate and adsorbate-vapor. In the case of $n = 0$ along the asymptote, the adsorbate becomes thinner leaving the energies of the phase boundaries. Therefore, α_{∞} is the addition the surface free energies of both interfaces, σ_i of substrate-adsorbate, and σ of adsorbate-vapor, or

$$\alpha_{\infty} = \sigma + \sigma_i. \quad (2.5)$$

First, consider the case when the free energy tends to the asymptote from above (Fig. 2.3). One can find

$$\alpha(n) = G(n) - n \frac{dG}{dn} \quad (2.6)$$

for any value of n drawing a tangent which has an intercept with the G -axis. Differentiating the above equation we obtain

$$\frac{d\alpha(n)}{dn} = -n \frac{d^2G}{dn^2} = -n \frac{d\mu}{dn} \quad (2.7)$$

or

$$d\alpha(n) = -nd\mu. \quad (2.8)$$

Compare this equation with the Gibbs adsorption isotherm [57]

$$d\sigma_s(n) = -nd\mu \quad (2.9)$$

that gives the change, $d\sigma_s(n)$, of the surface energy of the substrate which is defined with the adsorption of an amount n per unit area of an adsorbate with a chemical potential μ . The spreading pressure that is defined as $\phi = -d\sigma_s(n)$ represents the change in the surface energy [58]. The adsorption affects the interracial tension between the solid and the vapor and reduces σ_s by an amount equal to the spreading pressure ϕ of the film. It can be defined also as $\phi = \sigma_{s0} - \sigma_s(n)$ [59]. Comparing eqns. (2.8) and (2.9) gives the differential equation for $\alpha(n)$

$$d\alpha(n) = -\phi. \quad (2.10)$$

The integration gives

$$\alpha(n) = -\phi + C. \quad (2.11)$$

Using the initial condition $n = 0$, $\phi = 0$ and $\alpha(n) = \sigma_{s0}$ we find the value of the integration constant C . Then $C = \sigma_{s0}$ and

$$\alpha(n) = -\phi + \sigma_{s0} = \sigma_s. \quad (2.12)$$

Subtracting (2.12) from (2.5) gives

$$\alpha_{\infty} - \alpha(n) = \sigma + \sigma_i - \sigma_s, \quad (2.13)$$

where $\alpha_{\infty} - \alpha(n) < 0$ for all n values using the curve in Fig. 2.3.

This leads to the condition of $\Delta\sigma = \sigma + \sigma_i - \sigma_s < 0$ for complete wetting and satisfied for all n for the shape of $G(n)$ shown in Fig. 2.3. The curve in Fig. 2.4 should have positive curvature in order the free energy to tend to the asymptote from above. Therefore, the second derivative $\frac{d^2G}{dn^2} > 0$, similarly, $\frac{d\mu}{dn} > 0$ for all n [59, 60]. In other words, the conditions for the complete wetting $\Delta\sigma < 0$ and $\frac{d\mu}{dn} > 0$ are equivalent to each other [60]. It is clearly stated that the planar adsorbate is stable as long as $\frac{d\mu}{dn} > 0$.

The equilibrium vapor pressure of the planar adsorbate is always lower than that of the bulk adsorbate P_{∞} as $\mu = \mu_0 + kT \ln P$ and $\mu < \mu_{\infty}$. As a result, a stable planar film and an undersaturated vapor phase are in equilibrium with each other [47,51,58].

Consider the case when G approaches the asymptote from below, i.e. $\alpha_{\infty} > \alpha(n)$. In this case, the curve should have an inflection point to possess a negative curvature $\frac{d^2G}{dn^2} < 0$ beyond some point n (Fig. 2.5), or a negative curvature all the way (Fig. 2.6).

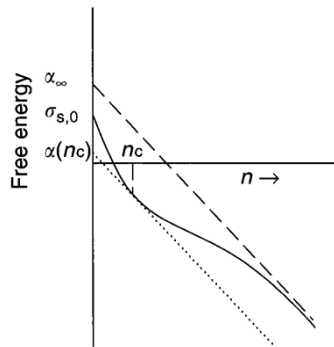


Figure 2.5 Gibbs free energy of a uniform film versus coverage n having an inflection point [56].

Consider first the curve with the inflection point. At a particular tangent n_c , $G(n)$ is parallel to the asymptote, i.e. the slope of the tangent is the value of the bulk chemical potential. At $n = n_c$, $\mu = \mu_\infty$ and $P = P_\infty$. The so called "wetting layer" is defined as the amount of material $n = n_c$ that wets the substrate completely. The asymptote represents a planar film beyond the coverage n_c values. The uniformly thick adsorbate stays stable for $0 < n < n_c$ values whereas it becomes unstable at $n > n_c$ since the free energy $G(n)$ is above the tangent. No planar film can grow when the $G(n)$ curve has a negative curvature all the way (Fig. 2.6). Draw a straight line beginning from the initial point σ_{s0} and being parallel to the asymptote. The line $G = \sigma_{s0} + n\mu_\infty$ stands for the free energy of a bulk adsorbate in the shape of a single droplet on the substrate. Since this line lies below $G(n)$ a bulk liquid rather than a planar film is thermodynamically favored from the very beginning of adsorption.

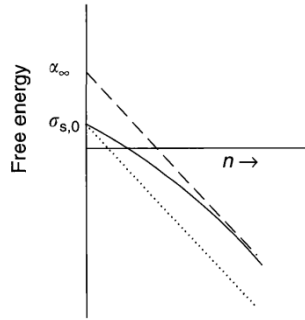


Figure 2.6 Gibbs free energy of a uniform film versus coverage n with a negative curvature from the very beginning of the adsorption [56].

As $\frac{d\mu}{dn} > 0$, the planar, uniformly thick adsorbate remains stable whereas the uniform adsorbate becomes unstable against clustering when $\frac{d\mu}{dn} < 0$. The $\frac{d\mu}{dn} < 0$ case can take place at the beginning of adsorption $n = 0$, or beyond some critical $n = n_c$ value. Differentiating the three curves in Figs. 2.3, 2.4 and 2.5, the three curves μ_n shown in Fig. 2.7 can be obtained.

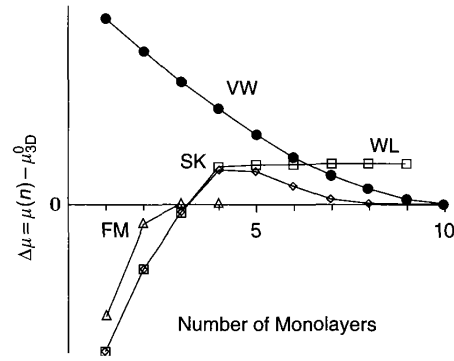


Figure 2.7 Dependence of the chemical potential on the film thickness for the three modes of growth [61].

WL represents the chemical potential of a hypothetical thicker wetting layer. The height of the plateau is strain energy per atom in the homogeneously strained wetting layer [61].

As a result, the three general classes of behavior defined by Dash [59] are observed: Frank van der Merwe, Stranski-Krastanov and Volmer-Weber mechanisms of growth that are described as (i) uniform deposits at all thicknesses, (ii) clustering after a thin uniform adsorbate, and (iii) clustering with no preadsorption, respectively as shown in Fig. 2.8.

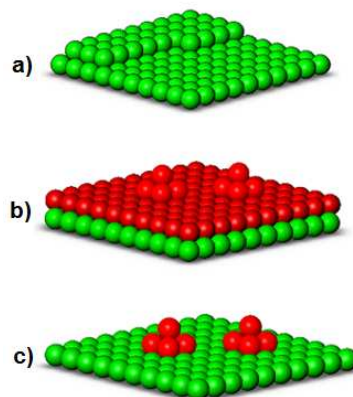


Figure 2.8 Three possible epitaxial growth mechanisms according to the classification of Bauer [55]: (a) Frank-van der Merwe layer-by-layer growth, (b) Stranski-Krastanov layer-by-layer growth followed by 3D islands (c) Volmer-Weber island growth [4,53].

CHAPTER 3

FABRICATION PROCESSES

3.1 BASICS OF MBE SYSTEM

3.1.1 Description of MBE Technique

MBE system enables the deposition of epitaxial layers on substrates with highly controllable parameters such as surface conditioning, growth rate, growth temperature, sample rotation, layer thickness etc. Two or more target elements can be evaporated for co-deposition by means of automated control of the e-beam or Knudsen cells.

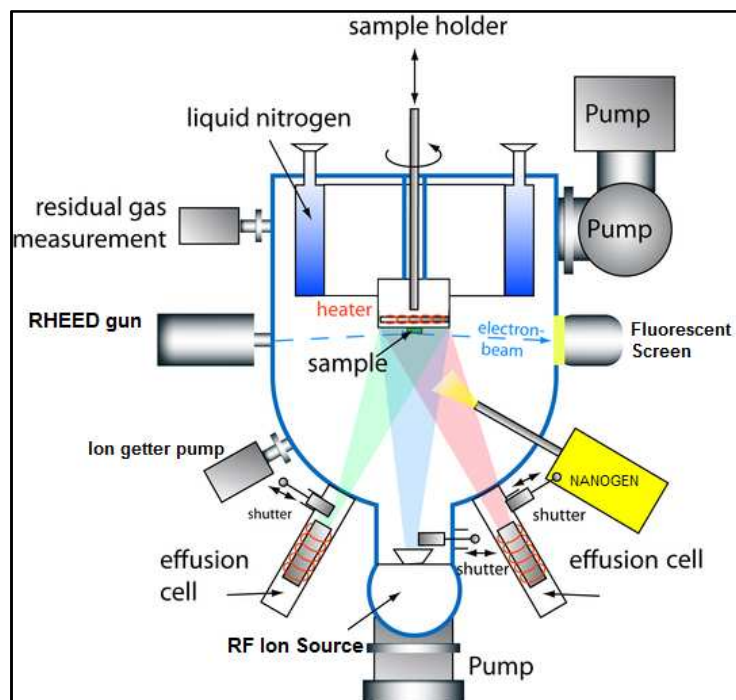


Figure 3.1 Basic components of MBE system [62].

3.1.2 MBE System Components

The components of the Mantis M600 Molecular Beam Epitaxy System (MBE) with a load-lock and main chamber (MC) units are shown in Figure 3.1. In addition, it is implemented with a sample holder, a STAIB instruments RHEED system, a Stanford Research Systems residual gas analyzer (RGA), an RF ion source, a nanoparticle generation (NANOGEN) unit and a Quartz Crystal Microbalance (QCM). All the system is connected to a computer via Rack unit enabling both manual and automated control. The automation software has almost all functions of the system that enable recipe driven processes.

3.1.2.1 Vacuum System and Load-Lock Unit

Load-lock unit is equipped with a magnetic transfer arm and substrate pre-heating section. It is pumped by a binary mechanism of a dry rotary pump and a turbo pump down to 10^{-8} mbar. On the other hand, MC pressure may be dropped down to 1×10^{-10} mbar using the dry rotary pump in conjunction with two turbo molecular pumps and an ion getter pump.

3.1.2.2 Substrate Holder and Heating Controller

Substrate holder mechanism is located at the center of MC. It has variable size substrate stages ranging from 1cm to 10 cm. Once the substrate is positioned it can be rotated at a desired speed up to 80 rpm. Besides, the holder can be moved in the vertical direction up and down using linear position controller. Substrate heater system with four implemented halogen bulbs of 500 W each can raise the temperature of the substrate up to 1000 °C for flash heating (see inset picture of Fig. 3.2)

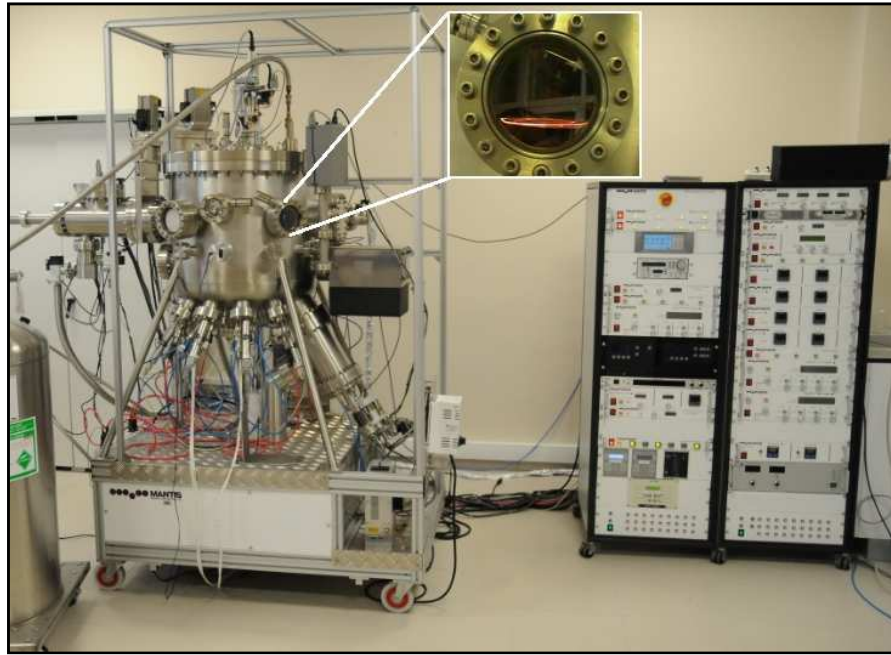


Figure 3.2 MANTIS M600 MBE system and substrate heating unit.

3.1.2.3 RF Ion Source

Radio frequency (RF) Ion Source system (see Fig. 3.3a) has a maximum power up to 400W and 50 mm wide plasma beam. It can operate at a wide range of beam energy in between 5-1000eV for surface conditioning and pre-treatment. Plasma is formed (Fig. 3.3b) when Mass Flow Control (MFC) system is arranged to an appropriate flow with desired high purity Ar, O₂, N₂ gases at about 1-20 standard cubic centimeters per minute (sccm).



Figure 3.3 a) RF Ion Source and b) Argon Plasma [63,64].

3.1.2.4 Reflection High Energy Electron Diffraction (RHEED) System

RHEED is a dynamical in-situ characterization system to study the kinetics of the growth processes at the surface of the sample. 10-15 keV energetic electrons are emitted by the gun (see Fig. 3.4a) to the surface of the sample with a glancing angle of 1° and the diffracted electrons form a RHEED pattern on the fluorescent screen (Fig. 3.4b).

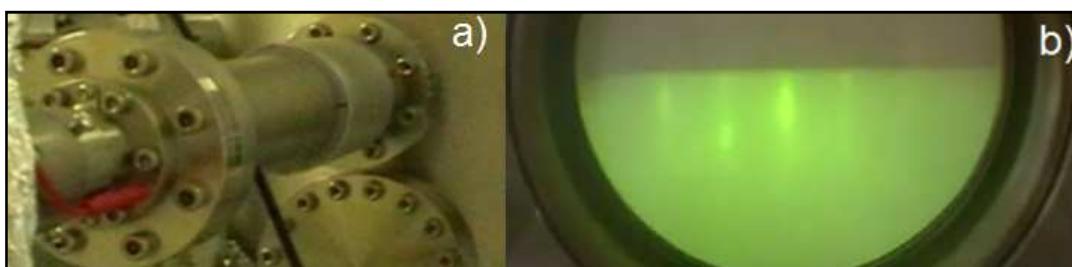


Figure 3.4 a) RHEED gun, b) Fluorescent screen.

RHEED patterns give the first evidence of the epitaxial growth since they are surface sensitive depending on the orientation of the substrate or grown layers.

3.1.2.5 Evaporation System

MC unit has four Knudsen cells (max 1700°C), four E-beam evaporator (max 3000°C) guns and a Carbon sublimation source. Knudsen cells are made of Al_2O_3 crucibles and they are filled with high purity (99.999%) Si and Ge source materials.

3.2 GROWTH CONDITIONS AND LAYER DESIGN

3.2.1 Description of Growth Conditions

A brief list of the ranges of the epitaxial layer growth parameters was depicted in Table 3.1. The growth parameters such as growth rate, annealing temperature etc. needs to be adjusted depending on the desired 2D or 3D growth mode. It can be clearly seen that the epitaxial growth is promoted at certain temperature ranges between 400°C and 850°C .

Table 3.1 Epitaxial SiGe thin film growth parameters [49].

	MC base pressure (mbar)	Substrate temp. (°C)	Growth rate (Å/s)	Ge content (%)	Annealing temp. (°C)	Annealing time (hr.)
Min-Max	10^{-11} - 10^{-8}	300-850	0.1-5	0-100	550-990	0.1-10
2D growth		300-550	2-5	0-25	550-600	0.1-0.5
3D growth		550-850	0.1-2	25-100	650-990	0.5-10

3.2.2 Epitaxial Layer Design

Eight pieces of samples were grown in accordance with the 3D growth mode mentioned in the Table 3.2. Four different group layer designs named as A, B, C and D are depicted in Figure 3.5.

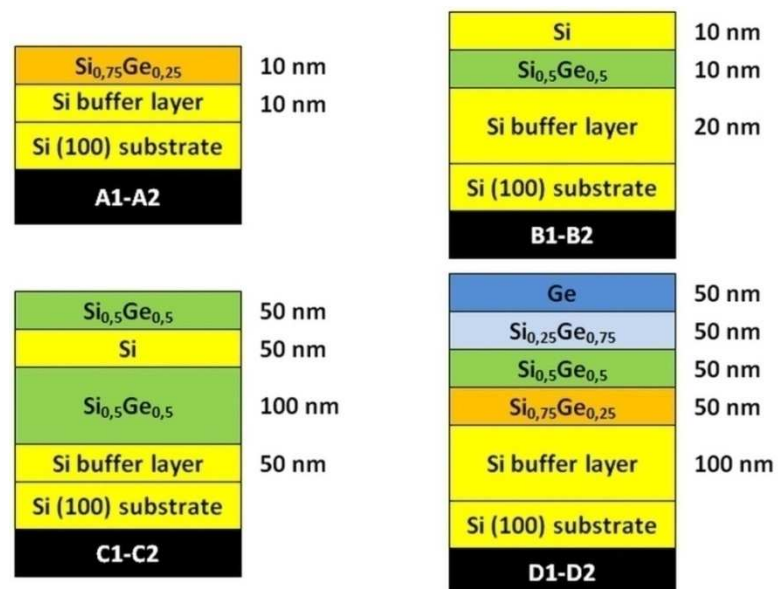


Figure 3.5 Schematic design of SiGe multilayers a) $\text{Si}_{0.75}\text{Ge}_{0.25}$ b) $\text{Si}/\text{Si}_{0.50}\text{Ge}_{0.50}$ c) $\text{Si}_{0.5}\text{Ge}_{0.5}/\text{Si}/\text{Si}_{0.5}\text{Ge}_{0.5}$ d) $\text{Ge}/\text{Si}_{0.25}\text{Ge}_{0.75}/\text{Si}_{0.50}\text{Ge}_{0.50}/\text{Si}_{0.75}\text{Ge}_{0.25}$.

The different colors indicate the particular Si-Ge content in the corresponding layer. Each group member was treated to one separate growth parameter as shown in the Table 3.2. Si (100) wafers were used as substrate for each group and a buffer layer of Si with various thicknesses was grown for the basement layer upon all samples.

Table 3.2 SiGe comparative growth conditions.

	GROWTH TEMP. (°C)	ANNEALING TEMP. (°C)	ANNEALING TIME (MIN.)	INTERSTEP ANNEALING? YES/NO
A1	550	850	5	N
A2	750	850	5	N
B1	750	750	5	N
B2	750	750	50	N
C1	750	750	30	N
C2	750	900	30	N
D1	750	850	30	Y
D2	750	850	30	N

Group A samples have been grown with a total thickness of 20 nm with 25% top Ge content by varying growth temperatures of 550°C and 750°C. For B1 and B2, layer thickness was 40 nm with a Si cap layer where annealing durations were 5 and 50 minutes respectively. Unlike group A and B, group C and D samples have been produced with 250 nm and 300 nm film thicknesses having 50% and 100% top Ge contents respectively. C1 and C2 were grown by varying annealing temperatures of 750°C and 900°C whereas D1 differs than D2 by the application of inter-step annealing process repeated after the growth of each layer.

3.3 SUBSTRATE AND MBE MAIN CHAMBER PREPARATION

In recent years, there have been many modifications in Radio Corporation of America (RCA) cleaning process and various new wet-cleaning techniques have been developed [65]. A series of rinsing steps is also added in between RCA cleaning processes and is performed by flowing ultra high purity de-ionized (DI) water [66].

Wafer drying is also another issue that must be carefully done in order to prevent recontamination [67]. This process must be done physically rather than leaving it to evaporate which could result in residues [68]. Forced pure N_2 spraying has proved to be a good technique for this purpose [69].

Finally, wet-cleaned wafers should be taken into the device chamber as quickly as possible [70]. If this is not possible they must be stored in glass desiccators rather than plastic containers since they could organically re-contaminate the wafers [71]. The desiccators must be stored in clean rooms and the wafers must be held with Teflon tweezers, not with metallic ones since they eventually result in metal contamination [69].

In addition, immersing the wafers into an ultrasonic bath during the standard clean (SC)-1 and SC-2 steps has proved to yield better results since it reduces the critical boundary layer between the wafer surface and the solution chemicals [68].

3.3.1 Wet Cleaning Process

P-type high resistivity (1-10 Ohm-cm) Si (100) wafer substrates with 2.5 cm x 2.5 cm dimensions were wet cleaned according to the RCA procedures with SC-1, 2 and diluted hydrofluoric acid (DHF) steps (see Fig. 3.6) [65]. First, Si substrates were immersed into a mixture of NH_4OH (25%), H_2O_2 (30%) and DI- H_2O in the ratio 1:1:5 for 5 min. Following this, wafers were immediately rinsed in a DI- H_2O bath for a further 3 min.



Figure 3.6 RCA cleaning chemicals in wet bench.

Second, the substrates were put in a mixture of HCl (37%), H₂O₂ (31%) and DI-H₂O in the ratio of 1:1:6 for 10 min. for the removal of the alkaline and trace-metals that inherited from SC-1. After this, the wafers were rinsed in the DI-H₂O bath for 3 min. to eliminate residual ions of the acid. Subsequently, substrates were dried by blowing pure N₂ gas.

Third, DHF solution that is a mixture of HF (40%) and DI-H₂O with ratio 1:20 was used to remove the native oxide from the wafer surface and for creating H-passivated, hydrophobic Si surface. After rinsing 30 seconds and drying with N₂ gun, they were immediately put into the load-lock chamber.

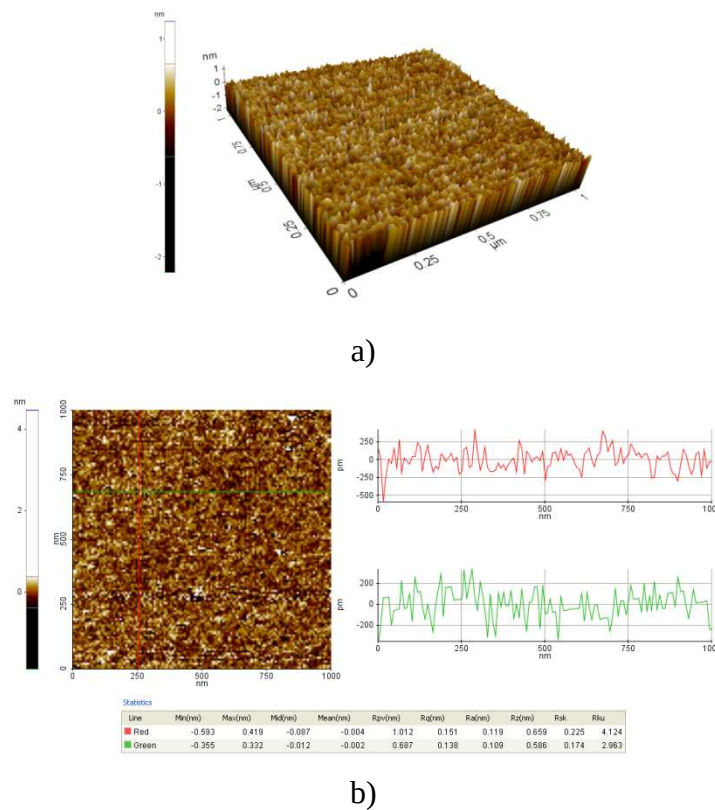


Figure 3.7 a) AFM surface topography and b) roughness analysis of a wet-cleaned Si wafer.

AFM surface topography image and roughness analysis of a Si wafer indicate that the surface having an average roughness value at about 0.11 nm is smooth and clean as shown in Figure 3.7.

3.3.2 Residual Gas Analyzing

First, substrate was positioned on the substrate holder by inserting the magnetic arm through the MC. After that, when the pressure is dropped to an ideal level in the ultra high vacuum (UHV) range, liquid Nitrogen was circulated around the chamber and RGA was employed to ensure the non-existence of unwanted gas species. RGA can also be used as a detective system for out-gassing due to the heating components. Figure 3.8a illustrates the cleanness of the MC with a base pressure of 3×10^{-10} mbar. At another case shown in Fig. 3.8b, MC has main atmospheric contaminant gases as indicated with atomic masses due to a leakage point around the system. In this case, vacuum leakage point was found using slightly pressurized Helium gas by simply spraying a gas gun to every possible leakage point like gaskets, joints etc. Once the leakage point was detected the problematic part was fixed or vacuum greased.

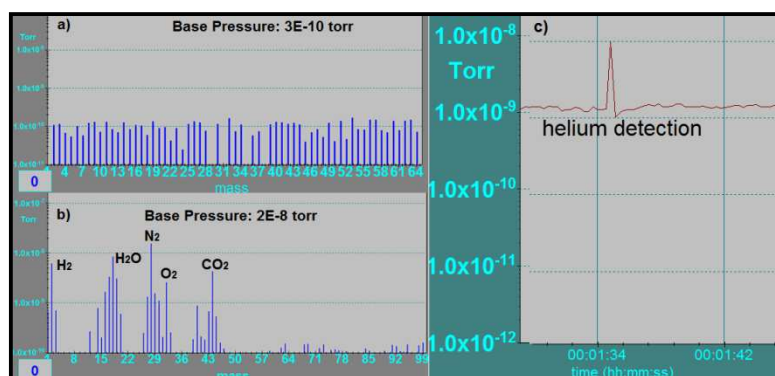


Figure 3.8 Residual gas analyzing of a) clean main chamber, b) a dirty chamber with atmospheric leakage c) detection of leak point by helium spraying.

3.3.3 RHEED in-Situ Surface Conditioning

RHEED pattern of the Si (100) substrate gives information about the surface condition before starting the film growth. It must be verified that the surface is clean before evaporation; otherwise the epitaxial film growth cannot be performed. For example, Figure 3.9 shows the various possibilities of the surface contamination that is inherent due to ex-situ or in-situ sources.

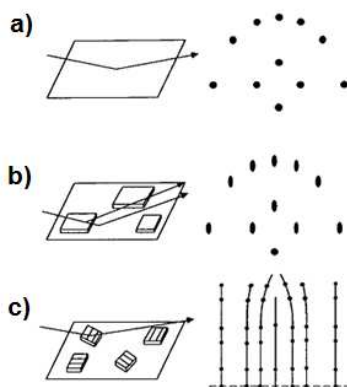


Figure 3.9 RHEED patterns of various surface structures, a) a clean smooth surface b) a surface with 2D defect extension c) 3D island formation [72].

The image shown in Figure 3.10a is the RHEED pattern of a wet-cleaned Si (100) wafer. The shape and the distribution of the streaks indicate that the surface has 2D extensions. Subsequently, the substrate was flash heated at 900 °C for 15 min. By this process, surface native oxide was removed and Si-H passivation is eliminated. As a result an atomically clean Si surface for epitaxial growth was obtained as depicted in the inset image of Fig 3.10a. RHEED analysis was performed at every stage of the growth process in order to ensure that the film was growing in 2D planar arrays of the terraces. On the other hand, the existence of the streaks (Fig. 3.10b) evidences the epitaxial growth of the evaporated Si on Si substrate that guaranties the pathway to the Ge co-deposition. Ge was also grown on Si epitaxially at the 2D regime, however, when the hut islands started to grow on the surface more than 10 or more monolayers (1 monolayer Ge= 1.5Å^o) the RHEED patterns disappeared.

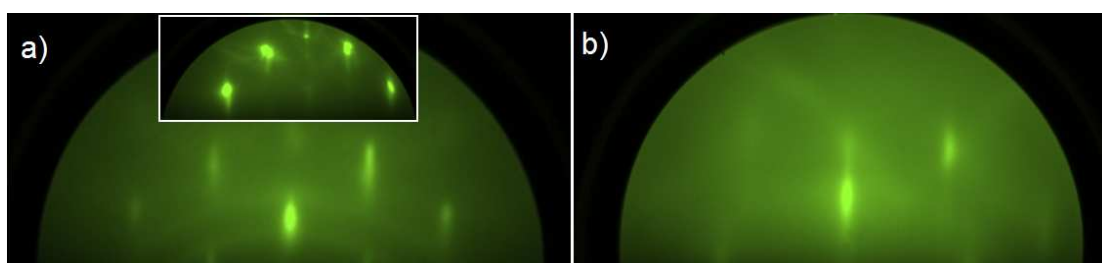
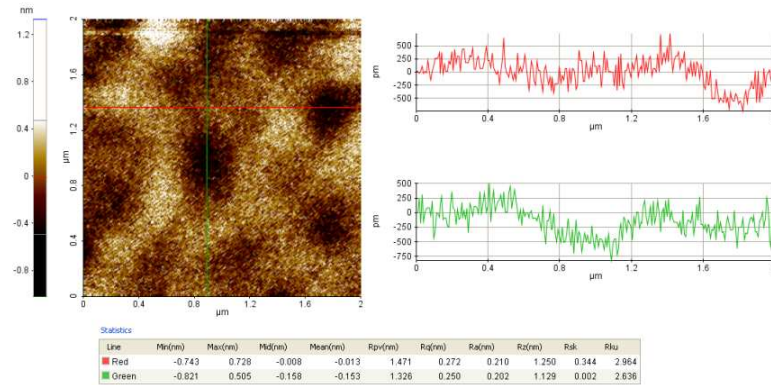


Figure 3.10 RHEED patterns of a) Si (100) wafer before growth at room temperature b) 100 nm Si grown on Si wafer at 750 °C.

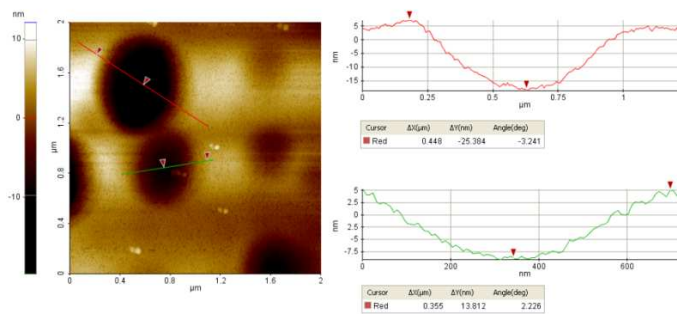
3.3.4 Dry Etching

In-situ dry etching with RF argon plasma was also performed at various energy values ranging between 20-400 eV. AFM image in figure 3.11a and 3.11b reveal the formation of etch pits of various diameters and depth profiles opened by 50 eV and 400 eV ion impacts respectively. It is clear that, there is a direct relationship between the energy of the plasma beam and the dimensions of the etch pits. As a result, RHEED patterns disappeared due to the excess energy of exposed Ar^+ ions.

It requires that the energy of the plasma beam should be reduced to 5-30 eV range in order to remove oxide without penetrating the surface atoms. Besides, a high temperature annealing process after etching enhances the reconstruction of the dimers. However, thermal methods such as flash heating are preferred more than non-thermal processes like plasma etching since etching eventually distorts the orientation of the dimers and result in the formation of dangling bonds.



a)



b)

Figure 3.11 a) AFM images of a) 50 eV, b) 400 eV plasma etched Si substrate.

3.4 DEPOSITION AND ANNEALING PROCESSES

Before deposition, the base pressure and substrate temperature were set as 3×10^{-10} mbar and 900 °C respectively. First, the samples were flash-heated at 900 °C for 15 minutes. Second, the four Si and Ge effusion cells which are located on both sides of the substrate were heated up to 1350°C-1400°C (see Fig. 3.12).

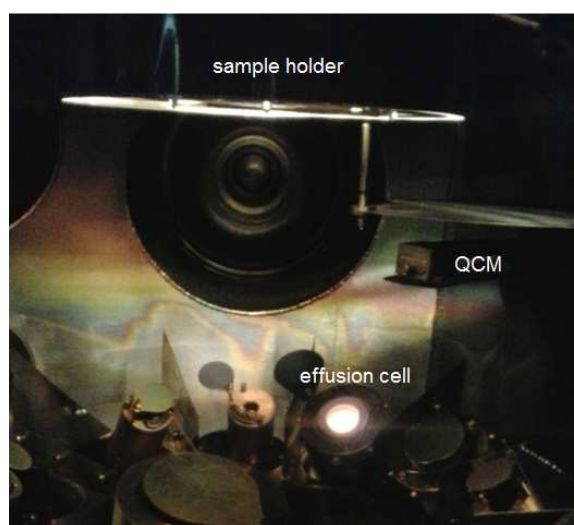


Figure 3.12 MBE MC during growth procedure.

The exact value of the growth rate is monitored with a piezoelectric QCM probe. Crystal parameters were adjusted entering the density and Z-factor of each element to the crystal monitor. A growth rate of 0.33 Å/s was adjusted by opening the effusion cell shutters of Si and Ge effusion cells independently. For homogenous coating, sample holder was rotated at 10 rpm. In addition, surface condition was properly checked by RHEED applying 10keV at a glancing angle around 1°. All the samples were post-annealed at 750-900 °C for 5-50 minutes. Growth procedures are controlled automatically by the previously written software procedures.

CHAPTER 4

CHARACTERIZATION TECHNIQUES

Various characterization techniques have been used to study the structural, optical and electrical properties of MBE grown SiGe nanoislands in the scope of this thesis. In-situ surface analysis was performed by Reflection High Energy Electron Diffraction (RHEED) System. The structural properties of the samples were characterized by Grazing Angle X-Ray Diffraction (XRD), High Resolution Scanning Electron Microscopy (HR-SEM) and Atomic Force Microscopy (AFM). The optical characterization of the samples was carried out with Raman Spectroscopy and the electrical characterization was performed by Semiconductor Analyzer system.

4.1 RHEED IN-SITU CHARACTERIZATION

Si (100) has three most common surface constructions that are named as Si (100) (1x1), (2x1) and c (4x2) as shown in the Figure 4.1.

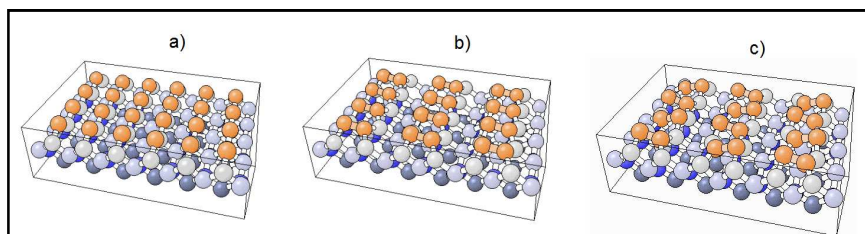


Figure 4.1 Most common Si (100) constructions, a) Unreconstructed Si (100)-(1x1) surface b) Reconstructed Si (100)-(2x1) surface c) Si (100)-c (4x2) reconstruction [73].

Si (100) (1x1) structure is generally observed on ideal bulk-terminated surfaces [73]. Si (100) (2x1) topmost layer is reconstructed by the covalent bonding of the adjacent atoms and these atoms form dimer pairs [74]. On the other hand, Si (100) c (4x2) buckling takes place at lower temperatures at around 140 K where Si (100) (2x1) reconstruction is more common at typical growth temperatures at about 900 K [75]. This is explained by the 1eV energy excess of 2x1 unbuckled reconstruction than buckled c (4x2) since the heat absorption make the dimers switch their orientation [75].

RHEED in-situ characterization is an efficient technique used to monitor the kinetics of the growth processes at the surface of the samples. It is so sensitive to the surface undulations that even the pits of 1-2 nm depth can result them to disappear. The RHEED gun sends the 10-15keV electron beam bent by a magnetic or electrostatic deflector on the sample with a glancing angle at around 1° - 4° on the substrate [74]. When RHEED electrons are incident on the surface of the sample as in Figure 4.2a they are diffracted by the atoms of the reciprocal lattice (Fig. 4.2b-c) and form streaks (Fig. 4.2d) on the Laue ring which is surrounded by Ewald sphere. Finally, the shapes of the fingerprint patterns falling on a fluorescence screen that consists of a phosphor covered, indium–tin-oxide (ITO) coated pyrex disk [76] are evaluated and the growth conditions are rearranged accordingly.

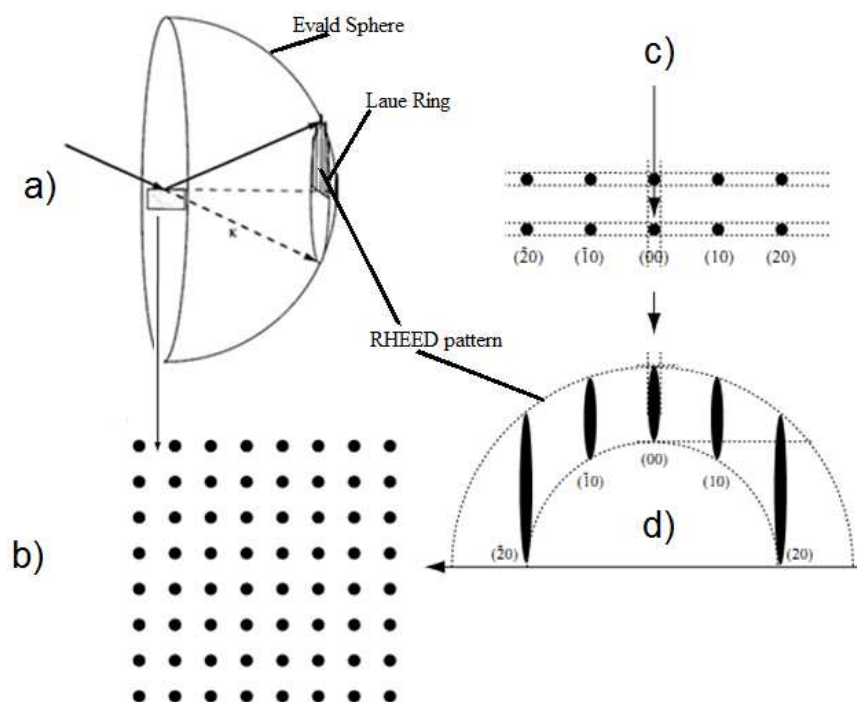


Figure 4.2 a) Ewald sphere construction of a Laue ring, b) 2D array of lattice points c) Reciprocal lattice, d) RHEED construction of streaks [72, 74].

RHEED can also be used in combination with other surface probing techniques like Scanning Tunneling Microscopy (STM) [74], Low Energy Electron Microscopy (LEEM), X-Ray Photoelectron Spectroscopy (XPS), and Secondary Ion Mass Spectroscopy (SIMS) etc. Energies greater than 10keV are used for dynamical analysis whereas it is possible to glance with higher incident angles to maintain surface sensitivity at lower energies like 3-5eV [74]. By this way, step edges were passed over and multiple scattering between terraces become negligible.

4.2 X-RAY DIFFRACTION (XRD)

XRD is a non-destructive technique that is used to characterize the crystalline properties, composition and strain state of materials [4]. X-rays that are emitted from an X-Ray source interact elastically with the specimen. Subsequently, the diffracted characteristic beams at certain Bragg angles from the crystalline surface atoms are counted by a detector (see Fig. 4.3).

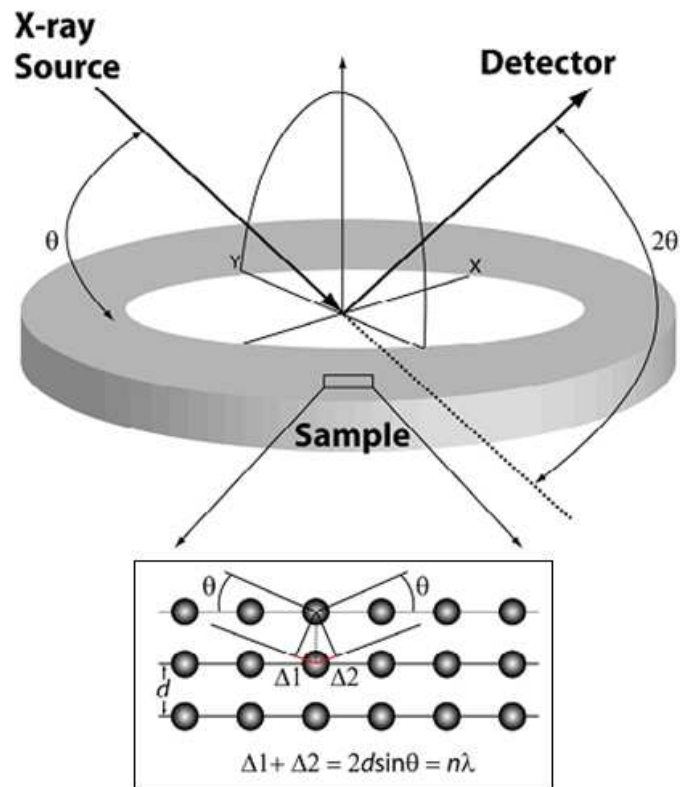


Figure 4.3 Schematic representation of the XRD geometry. The inset image shows the Bragg's diffraction and equation using the X-Ray path differences Δ_1 and Δ_2 [4].

The relationship between the incident Bragg's angle θ , the wavelength of the X-Ray beam λ and the lattice spacing of the material d is calculated with the so called Bragg's equation $n\lambda = 2d \sin \theta$ where n is an integer (see inset image of Fig. 4.3).

The X-Ray source used for diffraction is Cu K_α radiation with 0.154 nm wavelength (λ) which is utilized for determining the inter-atomic distances between 0.15 to 0.4 nm for most materials [4].

XRD technique has a wide variety of application area for numerous material types such as powder, thin film, liquid etc [77]. In addition, it enables thin film thickness analysis, density-roughness determination, phase identification and particle pore size analysis along with crystal peaks detection. Users can also utilize the different orientation measurements of crystal planes such as in-plane, asymmetric and out of-plane for the characterization of ultra thin films (see Fig. 4.4).

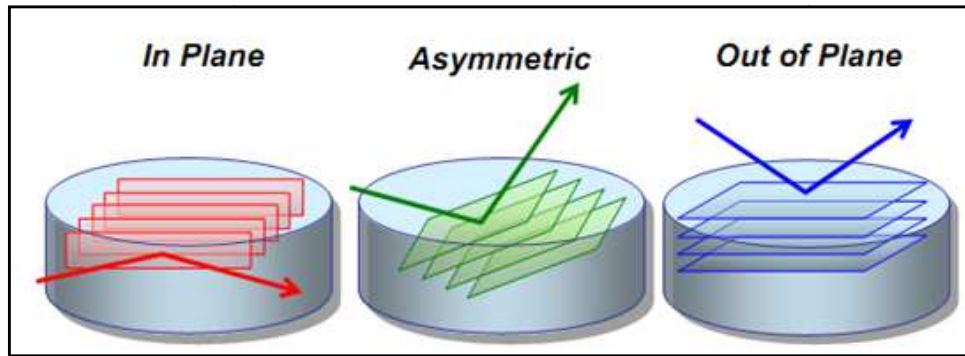


Figure 4.4 Crystal planes at various orientations [78].

Furthermore, Grazing Angle XRD method enables the out of-plane measurement of thin films with various thicknesses as shown in Fig. 4.5. The optimum grazing incidence is found by varying the angle between 1-5 degrees and observing the intensity increase of the desired peaks.

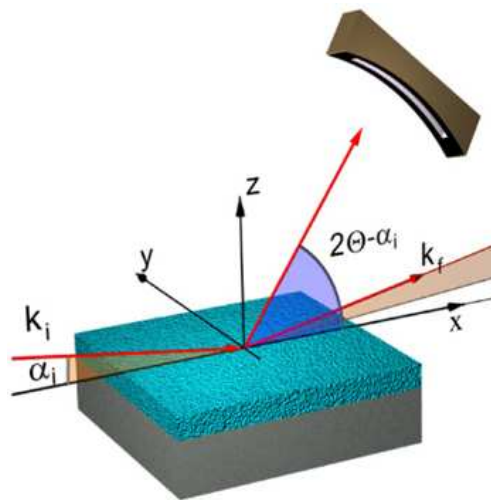


Figure 4.5 Grazing angle beam diffraction from a thin film surface [79].

XRD intensity peaks may broaden due to residual strain, defects, and amorphization segments existing on the surface of substrate [4]. Despite these complications, XRD allows a relatively quick analysis that is widely used in materials characterization.

4.3 ATOMIC FORCE MICROSCOPY (AFM)

AFM is a high-resolution type physical scanning probe method used to image the topography across the sample surface. As illustrated in Figure 4.6, the AFM probe consists of a cantilever with a sharp tip that is used to scan the sample surface. The typical cantilever is made of Si or Si_3N_4 that has a conical tip of $10\text{ }\mu\text{m}$ cone basement with a tip radius of curvature of 10 to 20 nm [1, 80].

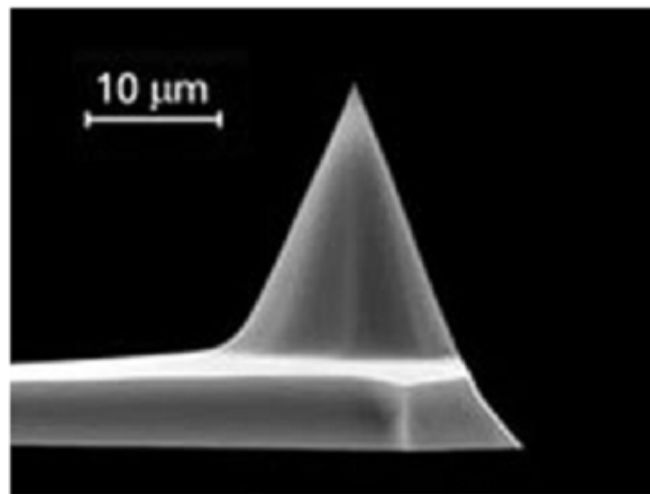


Figure 4.6 SEM image of uncoated Si probe tip [80].

The tip scans a determined surface area line by line using piezoelectric crystals and records the probe-surface interaction as a function of position. Meanwhile, the cantilever and tip are deflected up and down due to topography. As the tip moves on the sample surface, a laser beam that is reflected from the backside of the cantilever is monitored by a position sensitive photo detector as shown in Figure 4.7. These position changes are converted into height measurements that correspond to surface features.

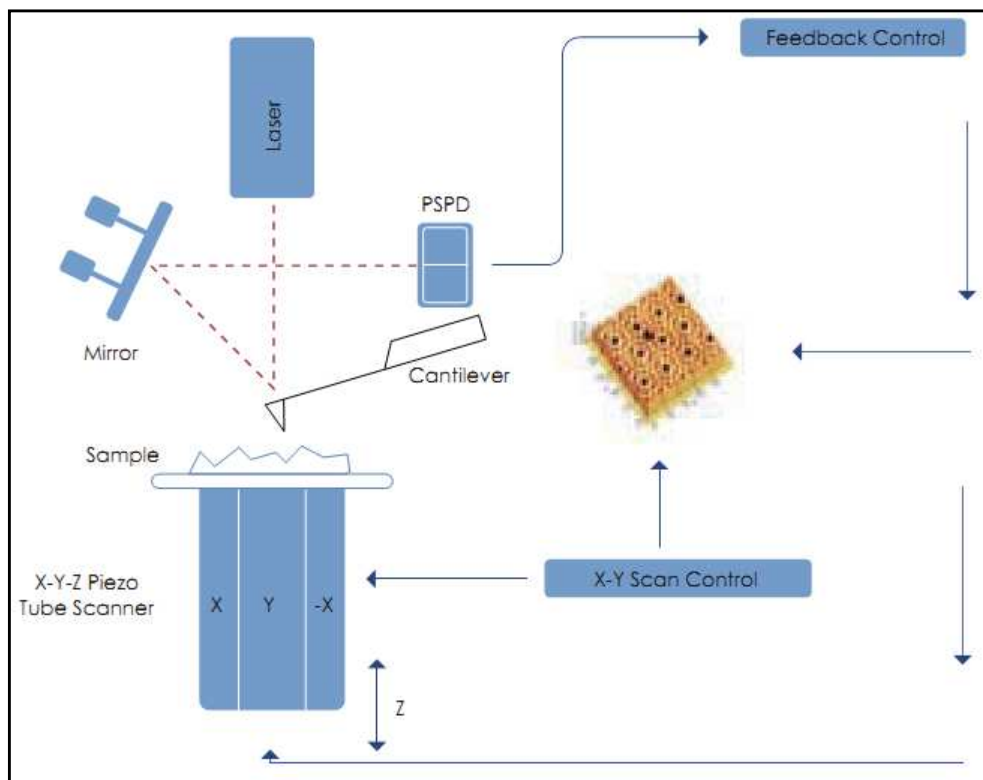
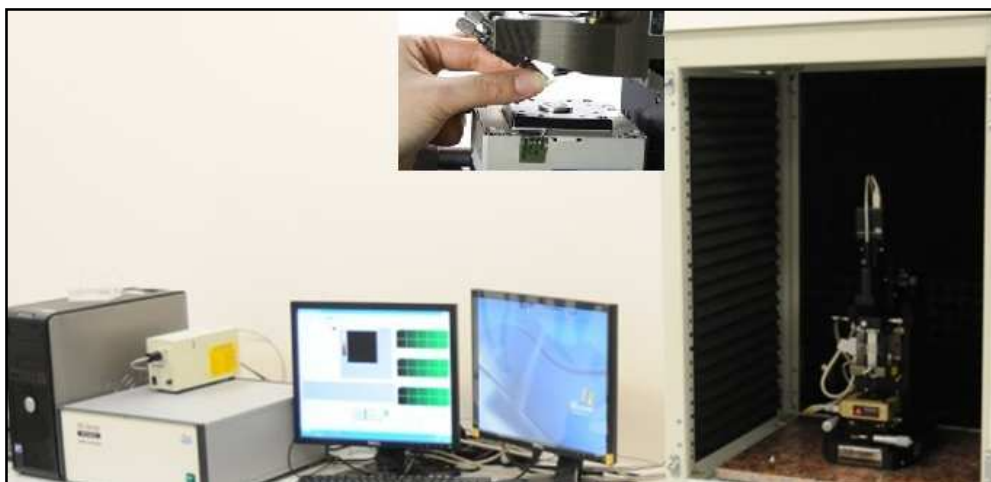


Figure 4.7 Primary system components of AFM [81].

The AFM system consists of a mechanical and sound vibration isolated main probe station, power control and light bank units (see Fig. 4.8). It is operated by a data acquisition program and the images are processed by graphics software.



1. Figure 4.8 AFM device components.

AFM system has many types of measuring and patterning modes like Lateral Force Microscopy (LFM), Electric Force Microscopy (EFM), Magnetic Force Microscopy (MFM), and Nanolithography etc. It is commonly operated in three modes; contact, non-contact and tapping modes. The tip is altered (inset image of Fig. 4.8) depending on the desired mode and renewed after a few ten times usage for maintaining the quality image acquisition.

In contact mode, cantilever is moved back and forth dragging the tip across the sample surface. It gives high quality images for hard materials. However, the tip can be damaged or contaminated by the surface features during scanning which may result in a decrease in the measurement accuracy.

In non-contact mode, the tip is brought into close proximity of the sample surface. As a result, the tip is deflected by Van der Waals interaction forces between the sample and the tip. Although this method is not as accurate as contact mode, it doesn't suffer from the problems encountered in that method.

Tapping mode is based on the oscillation of the cantilever up and down at its resonance frequency by means of a small piezoelectric element mounted in the AFM tip holder [1]. It is counted as a gentler method than conventional contact AFM since the cantilever is not dragged over the sample surface. Consequently, this method enables to work with soft, adhesive or fragile samples that are difficult to image as they can easily be damaged by contact mode.

4.4 SCANNING ELECTRON MICROSCOPY (SEM)

SEM is based on focusing and scanning of high energy electron beams onto the sample surface resulting in a magnified image of the sample. The use of electrons in SEM instead of photons as in optical microscopy for scanning is more advantageous since the electron wavelength is much smaller than the photon wavelength. Hence, magnification and resolution is much greater in electron microscopy compared to optical microscopy [82].

Assume that, v is the electron velocity and V is accelerating voltage where the electron wavelength is λ_e . It follows that,

$$\lambda_e = \frac{h}{mv} = \frac{h}{\sqrt{2qmV}} = \frac{1.22}{\sqrt{V}} = 0.012 \text{ nm}$$

for $V = 10.000\text{V}$ which is much smaller than 400 nm-700 nm visible light wavelength [82].

In a conventional SEM, electrons are emitted from a tungsten electron gun thermionically. Tungsten electron guns were preferred since they had the highest melting point with low vapor pressure among all metals. However, Lanthanum Hexaboride (LaB_6) cathodes replaced Tungsten guns due to their better performance and subsequently the Field Emission Guns (FEG) became commonly utilized as FEGs have higher brightness and longer life. Among FEGs, Schottky-Emission gun (see Fig. 4.9) utilizes the Schottky effect with an applied high electric field to a heated cathode of a Zr/W emitter which is a tungsten single crystal coated with ZrO [1].

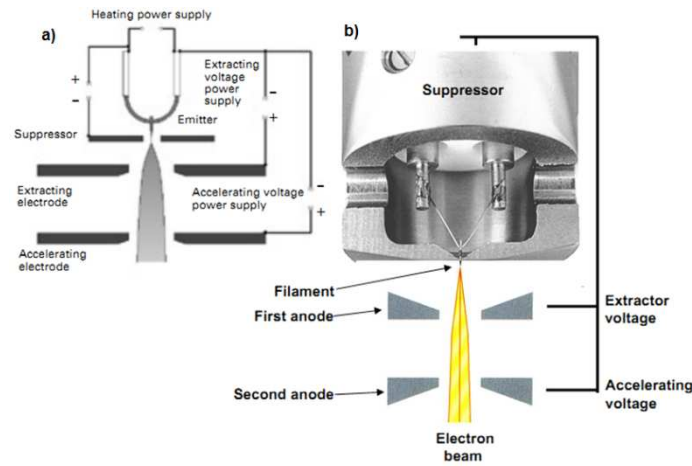


Fig 4.9 Construction of the Schottky Emission a) schema and b) gun [83, 84].

It greatly reduces the work function enabling a large emission current at a relatively low cathode temperature about 1800K.

When an electron beam scans the sample surface, various signals are generated due to the interactions between the electron beam and the surface atoms. These are classified as secondary electrons (SE), backscattered electrons (BE), X-Rays, Cathode luminescence emitted light and Auger electrons (Fig. 4.10) [83]. SE are generated by inelastic collisions whereas BE are produced by elastic collisions.

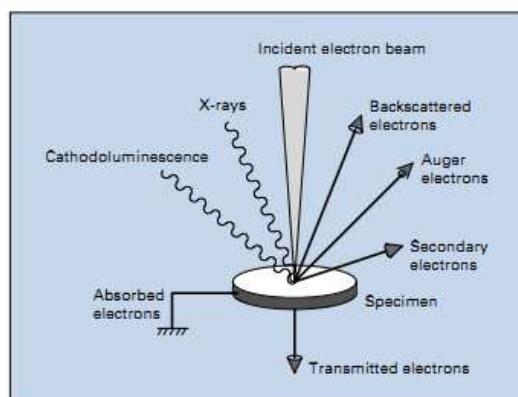


Figure 4.10 Emissions of various electromagnetic waves from a specimen [83].

The generation volume of the signals increases as e-beam acceleration voltage increases. Most incident electrons penetrate into the sample and then absorbed for low-Z samples. On the contrary, a large portion of the incident electrons are backscattered in high-Z samples like heavy metals.

Secondary electron imaging (SEI) is the most common SEM mode, that can produce very high-resolution images of samples even at about 1-5 nm in size. On the other hand, BE mode can provide different brightness and contrast on different element segments of the sample as its signal is strongly related to the atomic number Z of the sample.

It is important that non-conductive samples (generally with low- Z value) are coated with a conductive material like Au or Pt in order to eliminate surface charging. In addition, the resolution can be increased by decreasing the accelerating voltage to ensure that the number of primary electrons are roughly equal to the number of SE and BE.

Figure 4.11 depicts the basic system and construction schema of SEM system. It contains SE and BE imaging detectors, X-Ray detector of Energy Dispersive Spectroscopy (EDS) and Electron Beam Lithography ports. It can perform magnifications ranging from 10 to 1,000,000 times with 1.2 nm resolution at 30keV.

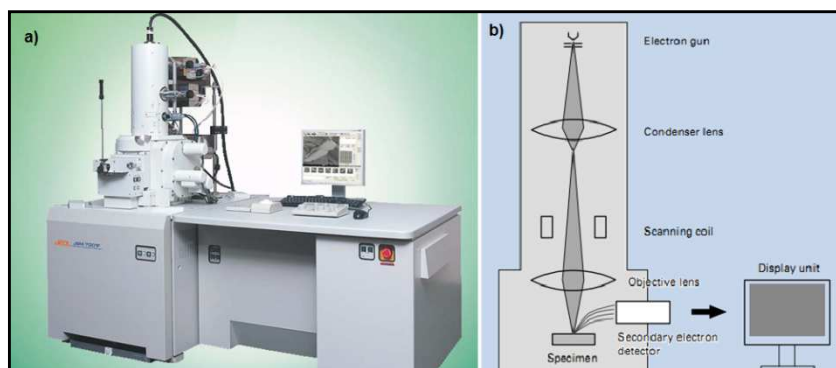


Figure 4.11 a) Basic system photo and b) construction schema of SEM [83, 85].

4.5 RAMAN SPECTROSCOPY

When a sample is exposed to laser beam most of the radiation is scattered back with the same wavelength. This process is known as Rayleigh scattering. However, a small portion of the incident light (a few photons out of millions) is scattered at a longer wavelength from the original laser wavelength [86]. Relaxation of a molecule from the virtual state to the first excited vibrational level results in a Stokes-Raman shift (Fig. 4.12) [86].

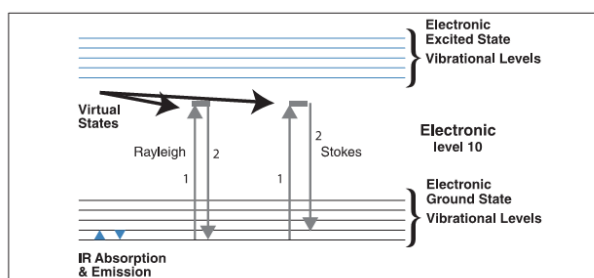


Figure 4.12 Energy level diagram of a molecule [86].

In addition, anti-Stokes Raman scatter is observed when the Raman process initiates from the excited vibrational level to the ground state with higher energy and shorter wavelength [86].

Raman spectrometers can be categorized as Dispersive Raman and Infrared Raman. Both techniques have advantages in specific types of analysis. Dispersive Raman is more efficient in symmetric bonds whereas Infrared Raman is more sensitive to functional groups and polar bonds.

Raman technique is now widely used in many applications since it can be applied non-destructively in many areas such as Chemistry, Biology Pharmacology and Forensic Science etc.

Dispersive Raman utilizes visible laser radiation at typical laser wavelengths of 532 nm, 780 nm and others. Fluorescence interference which is an obstacle for Raman spectra can be corrected with appropriate software and other strategies [86].

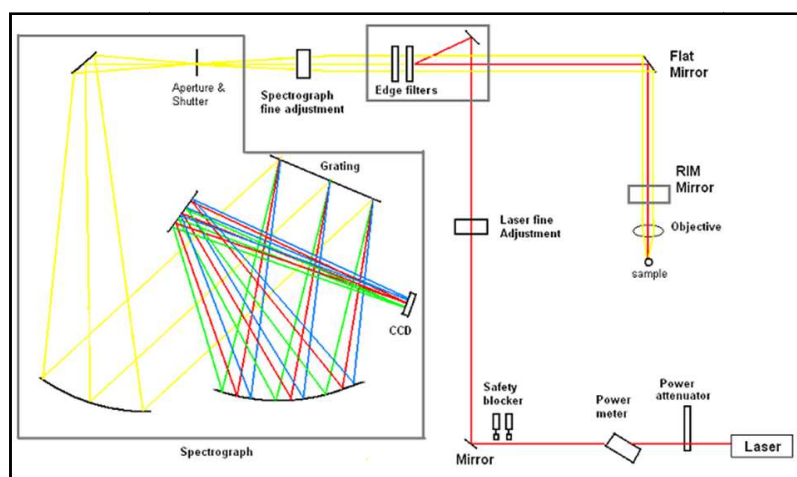


Figure 4.13 Diagram of laser pathway in Dispersive Raman [87].

In Dispersive Raman a diffraction grating splits the scattered beam into its constituent wavelengths as shown in the optical path and diagram of the Figure 4.13 [87]. The splitted beams are directed upon a charge-coupled device (CCD) detector.

Raman technique enables both qualitative and quantitative information about the sample with library searching and data manipulation. In addition, it has many advantages in analysis since it requires minimal sample preparation and measurement can be done directly through glass tubes. The Dispersive Raman System (see Fig. 4.14) can perform micron level analysis with 1 μm spatial resolution as it has a microscope unit and software controlled motorized stage [88].



Figure 4.14 Dispersive Raman Spectroscopy system [88].

4.6 SEMICONDUCTOR ANALYSIS

Semiconductor analyzer with four probe stations is designed as a total system for electrical characterization of devices, materials and semiconductor processes (Fig 4.15).

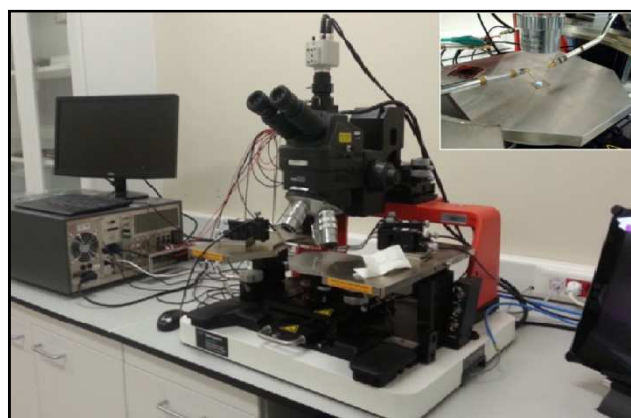


Figure 4.15 Four Probe Semiconductor Analyzer system.

Many types of measurements including DC Current-Voltage (I-V), AC Impedance, Capacitance-Voltage (C-V) etc. can be performed with high precision. DC source-measure units can supply voltage or current and do measurements from 0.1fA to 1A and from 1 μ V to 210V [89]. Pulse I-V measurements which is used for electrical characterization of semiconductor surfaces enables high speed analysis without the problems of self-heating which can affect measurement response [89].

In I-V measurements, samples are fixed on the table of the probe station and the conductor needles are brought into close proximity of the surface of the sample. Meanwhile, needle tips are viewed by the microscope screen to ensure that they are in contact with the surface. Besides, care should be taken in order the surface of the specimen not be scratched (see inset figure of Fig.4.15). Figure 4.16 illustrates a common four probe measurement model for the thin film surfaces which is indicated in red. Accordingly, the current flow is kept within the grown film since the depletion region at the interface of the substrate and the film acts as an insulator [90].

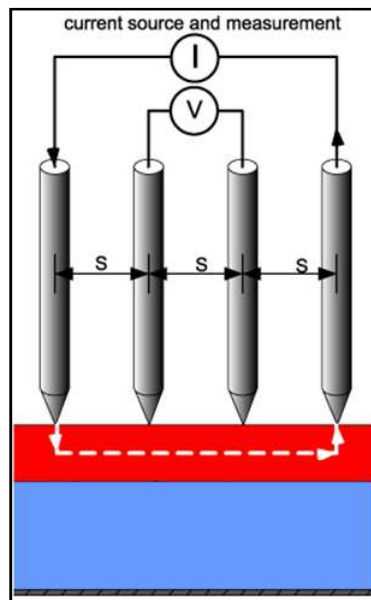


Figure 4.16 Representation of the four probe I-V measurement method [90].

Following this, forward and bias voltage range and current steps are adjusted and the measurements are carried out in dark and illuminated ambient.

CHAPTER 5

STRUCTURAL, OPTICAL AND ELECTRICAL CHARACTERIZATION

In the scope of PhD thesis research, all the samples were intensively analyzed by structural, optical and electrical characterization methods using XRD, AFM, SEM, Raman Spectroscopy and Semiconductor Analysis. Grazing angle XRD measurements were carried out by XRD Smart Lab Rigaku with Cu target wavelength $\lambda=1.54 \text{ \AA}$ at a glancing angle of 5° . AFM topography images were obtained by Park Systems XE-100E Scanning Probe Microscope. SEM images were taken by Field Emission JEOL SEM 7001F with 10-20 kV acceleration voltages in secondary electron imaging (SEI) mode. Raman Spectroscopy measurements were held by Thermo Scientific DXR Raman Spectroscopy system with a 532 nm laser excitation at 3 mW laser output power. As a final investigation, Semiconducting Analysis was performed with Keithley 4200-SCS Model Four Probe Semiconductor Analyzing System.

5.1 CHARACTERIZATION OF GROUP A SAMPLES

5.1.1 Structural Characterization of SiGe Nanoislands

Grazing angle XRD spectra of Si (100) substrate was acquired with X-ray beams that skim over the surface at 5° incidence. Si (311) and Si (222) diffraction peaks at 56.6° and 60.3° were obtained respectively. It can easily be seen in Figure 5.1 that Si (311) peak is in a broadened shape where Si (222) peak is a sharp one

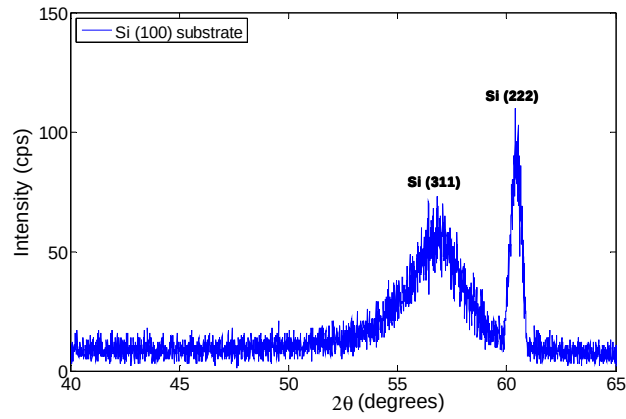


Figure 5.1 Grazing angle XRD pattern of Si (100) substrate.

5.1.1.1 Grazing Incidence XRD Scan Results

Figure 5.2 indicates the grazing angle XRD Si (220) peaks of group A samples along with the Si (311) and Si (222) inherent substrate peaks. Si (220) peaks of A1 and A2 were observed at 44.2° . The sample A2, which had been grown at 200°C higher substrate temperature than A1 has a slightly greater crystalline Si peak value at the indicated angle. Similarly, Si (311) and Si (222) substrate peaks of A2 are greater than that of A1. The unusual shape of A2 peak may be due to the strain developed at the Si-Ge interfaces [91]. A significant amount of diffraction angle shift is observed between the Si (222) peaks of the samples.

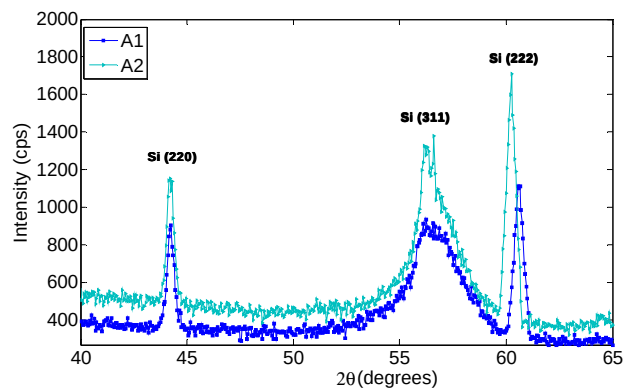


Figure 5.2 Grazing angle XRD spectra of group A samples.

In Figure 5.3, a magnified view of Si (222) peaks of A1 and A2 which are located at 60.60° and 60.25° are shown respectively. The lattice spacing values were calculated with the Bragg's diffraction formula $d = n\lambda/2\sin\theta$ for all samples. It is noteworthy that the shift in calculated lattice spacing values of $1.526 \text{ \AA}$ and $1.534 \text{ \AA}$ (see inset table of Figure 5.3) can be due to the presence of the strain induced between the substrate and the grown layer [92]. In addition, the higher growth temperature of A2 than A1 may have led to more closely packaging of Si nanocrystals within the matrix resulting in the sharpening of the related peak [93]. The absence of Ge peaks in both samples can be explained by low Ge content (25%) at cap layer.

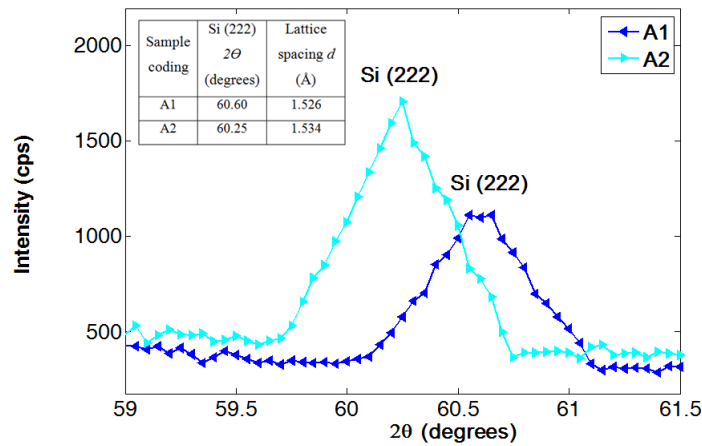


Figure 5.3 Si (222) XRD peak shifts of group A samples in magnified view.

5.1.1.2 AFM Topography and Roughness Analysis

AFM measurements give detailed information about the size, shape and distribution of the nano features of the samples. Measurements were carried out in contact AFM mode as it enables the acquisition of sharper surface topography images.

Figure 5.4 depicts the inclined view AFM topography images of group A samples. It is apparent that A1 has a uniform size, shape and distribution of closely stacked islands where A2 is more heterogeneous in these aspects. Nano island features of A1 are in the shape of dome and those in A2 are of pyramid-like shape with a random distribution.

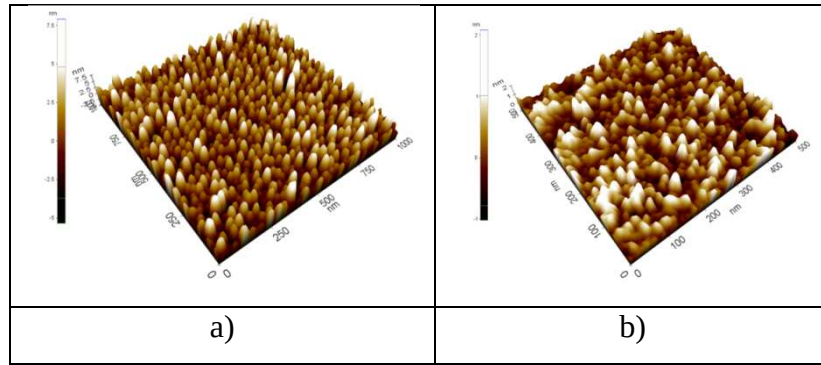


Figure 5.4 a) Inclined view AFM topography images of the samples a) A1 and b) A2.

Top view AFM images and length histograms of the samples A1 and A2 reveal the grain size distribution of both samples as illustrated in Figure 5.5. Accordingly, diameters of nanoislands in A1 and A2 vary between 20-50 nm and 10-45 nm in average respectively.

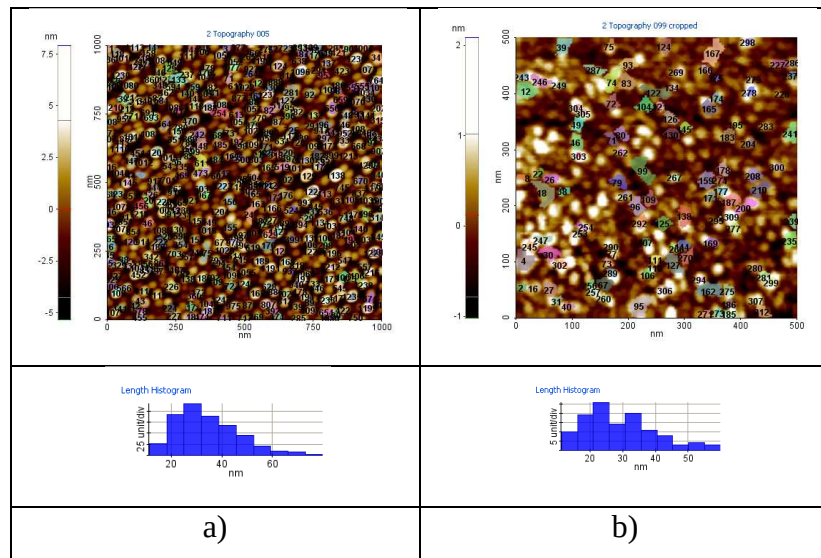
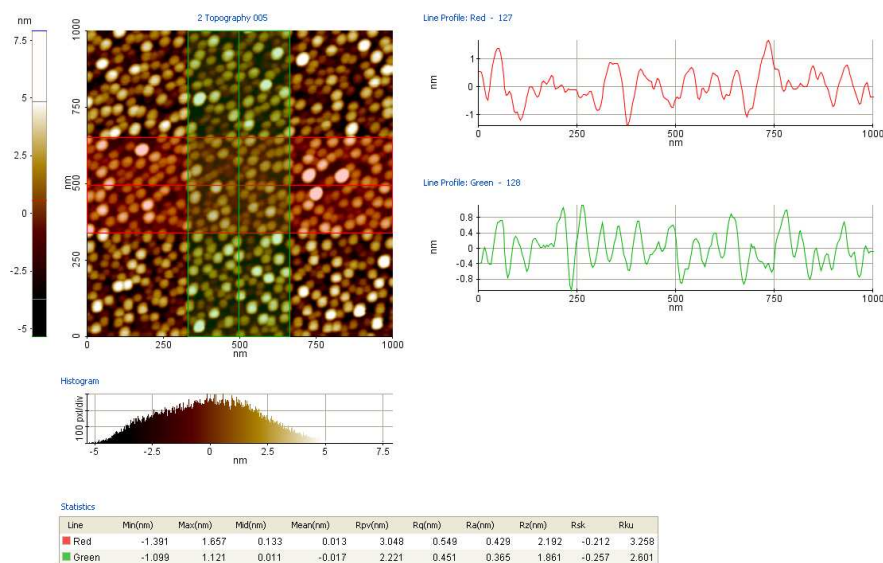


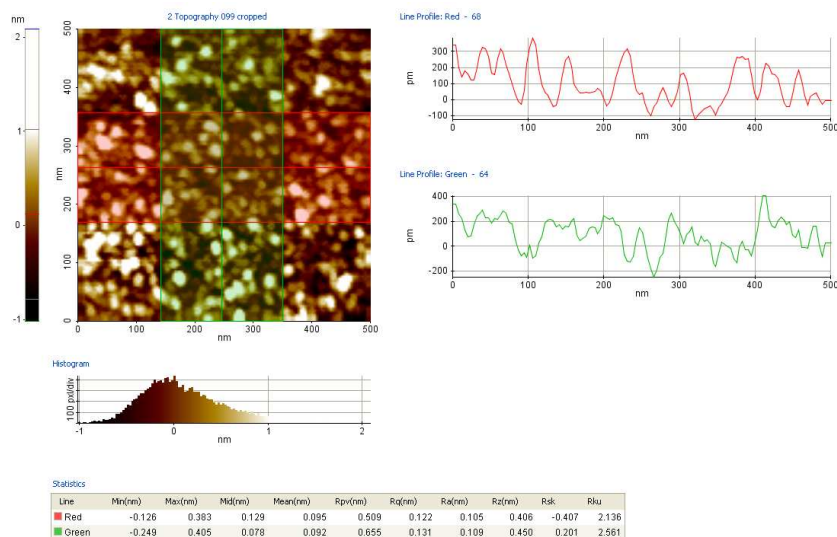
Figure 5.5 Top view AFM images and length histograms of the samples a) A1 and b) A2.

Average roughness (R_a) analysis of both samples is carried out through the selected horizontal and vertical area segments that are shown in red and green as depicted in Figure 5.6. The related figures also contain line profiles, height histograms and detailed statistical data about the samples. It is remarkable that the R_a values of A1

vary between 0.365 nm and 0.429 nm. On the other hand, this value is in between 0.105 nm and 0.109 nm for A2. It is clear that A1 has an almost four times greater value of R_a compared to A2. R_a discrepancy between the samples is a direct result of the large vacancies formed in A2 due to the nucleation that took place because of 200°C higher growth temperature of A2 than A1.



a)



b)

Figure 5.6 AFM area roughness analysis of the samples a) A1 and b) A2.

5.1.1.3 SEM Analysis

HR-SEM images of all samples were taken at suitable magnification values between 4.000X and 500.000X under 15-20 kV acceleration voltages. Samples were mounted on the specimen holder by conductive Carbon tape, and wafer sides were contacted to the holder by conductive Silver paste. Au Coating at about 7-10 nm was preferred for Si rich samples in order to prevent charging of the samples. Measurements were performed in SEI mode. Semi-tilted images were acquired at 23° inclination and the cross-sectional imaging was performed at almost 88° from the normal to the surface.

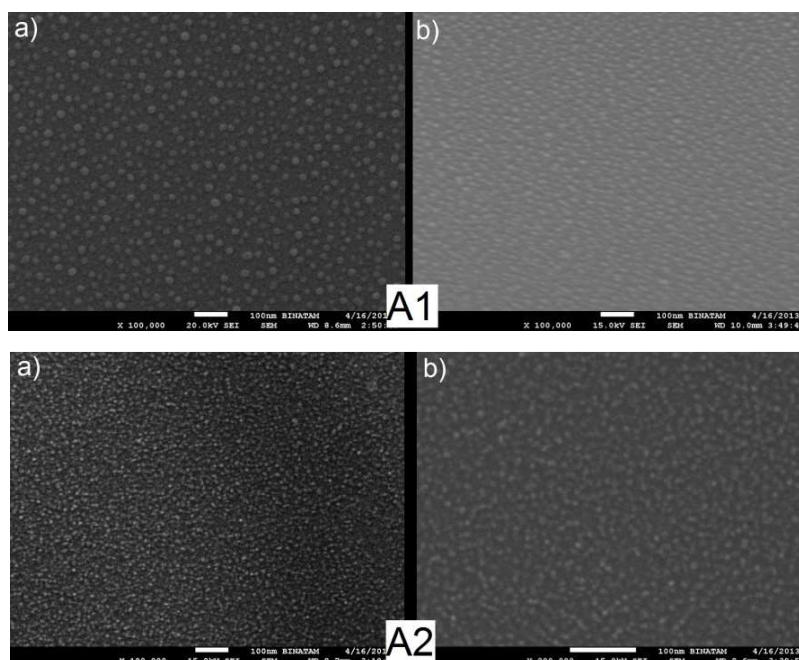


Figure 5.7 Top and inclined view SEM images of group A samples.

In Figure 5.7, top and inclined view of SEM images of A1 and A2 are shown. It is apparent that A1 has island formation with a homogeneous distribution. Similarly, A2 has also a homogeneous islanded form at relatively smaller sizes. It can be inferred from the top and inclined views of both samples that A1 have droplet type islands at low height whereas those in A2 seem to have gained closely stacked nanodot form. It can be stated that 200°C higher growth temperature that is applied to A2 resulted in greater nucleation than A1 which was grown at 550°C.

5.1.2 Optical Characterization of SiGe Nanoislands

5.1.2.1 Raman Analysis

Figure 5.8 illustrates the dispersive Raman spectra of group A samples. Accordingly, Si-Si and Ge-Ge phonon vibration peaks [94] are observed at 518 cm^{-1} and 301 cm^{-1} respectively. It is apparent that the strong Si-Si signal, which is two orders of magnitude greater than Ge-Ge peak is originated by Si substrate. On the other hand, Ge-Ge signal is very weak due to the fact that top layer has low Ge content. There observed no sign of Si-Ge vibration peak that arises at about 400 cm^{-1} due to the lack of SiGe matrixes. It is remarkable that despite the difference in growth temperature, Raman spectra of both A1 and A2 are perfectly identical. Hence, it can be stated that the shape, size and distribution differences of the nanoislands in both samples did not have a significant effect on the phonon vibrations of the symmetric bonds.

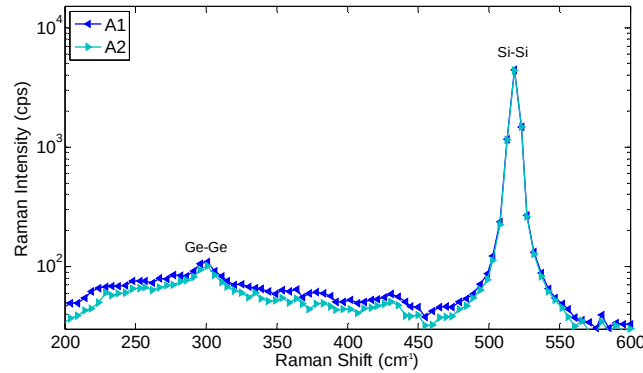


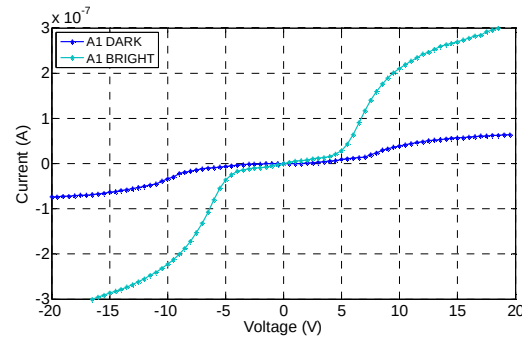
Figure 5.8 Raman spectra of group A samples.

5.1.3 Electrical Characterization of SiGe Nanoislands

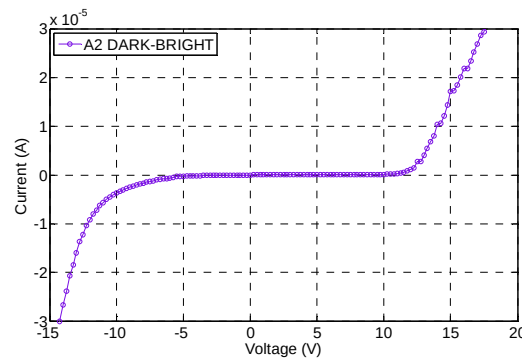
5.1.3.1 I-V Characteristics Measurements

Figure 5.9 illustrates the I-V characteristics of group A samples between -20 V and $+20\text{ V}$. Measurements were carried out in dark medium and under LED illumination for both A1 and A2. A quite low value of current flow at 10^{-7} A range was measured through A1, where it was observed at 10^{-5} A values in A2. Moreover, I-V

characteristics of the samples differ pretty much from each other. For dark measurements, A1 gained tunnel diode type I-V curve [95]; however, A2 got almost the form of a reverse diode with a positive breakdown voltage at around 12 V. When both samples were illuminated by a 22 W white LED source, the current gain of A1 significantly increased maintaining the tunnel diode type I-V. However, no sign of change in the I-V curve of A2 was observed by illumination. The tunnel diode type conduction mechanism of A1 can be explained by the existence of vacancies between the nanoislands that enable the quantum tunneling phenomenon [95]. On the contrary, the closely stacked nanoisland structure of A2 leads to semiconducting type diode characteristics by the high intermixing of SiGe domains with various alloy composition. Furthermore, despite the fact that the presence of illumination favors the current gain, it retards the acquisition of negative differential resistance [96] lowering the energy gap between the valence band and conduction band.



a)



b)

Figure 5.9 Dark & Bright I-V measurements of the samples a) A1 and b) A2.

5.2 CHARACTERIZATION OF GROUP B SAMPLES

5.2.1 Structural Characterization of SiGe Nanoislands

5.2.1.1 Grazing Incidence XRD Scan Results

Figure 5.10 depicts the grazing angle XRD Si (220) peaks of group B samples at 44.2° including the Si (311) and Si (222) substrate peaks. B1 and B2, which had been annealed at 750°C for 5 and 50 minutes respectively, have almost identical Si (220) peak intensities. Moreover, these peak intensities are quite smaller than that of substrate peaks that indicate the formation of poor Si crystalline structure with amorphized segments [91]. Poor Si crystal formation may be originated due to direct growth of Si on $\text{Si}_{0.50}\text{Ge}_{0.50}$ layer as it would result in the formation of a great percentage of misfit dislocations and protrusions. Furthermore, since the cap layers of both samples consist totally of Si, the various annealing duration did not have a significant effect on the crystallinity of the samples. Si (222) substrate peaks of both samples have a slight angle shift and difference in intensity.

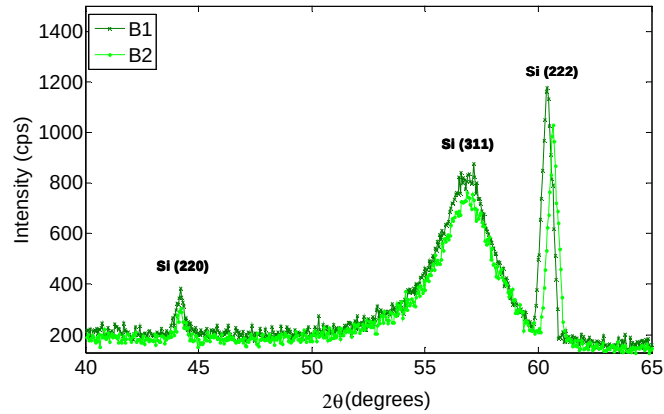


Figure 5.10 Grazing angle XRD spectra of group B samples.

A magnified view of Si (222) peaks of B1 and B2 which are located at 60.40° and 60.65° respectively are illustrated in Figure 5.11. The lattice spacing values of the samples that were calculated with the Bragg's diffraction formula $d = n\lambda/2\sin\theta$ are

1.531 Å and 1.525 Å (see inset table of Figure 5.11) respectively. It is apparent that a slight shift is formed between the related peaks and this can be explained by the presence of the strain [92] developed at the related Si (222) orientation. In addition, 45 min. longer annealing temperature of B2 than B1 may have resulted in the relief of the compressive strain. Hence, B2 gained slightly greater lattice spacing than B1 by the formation of excess misfit dislocations.

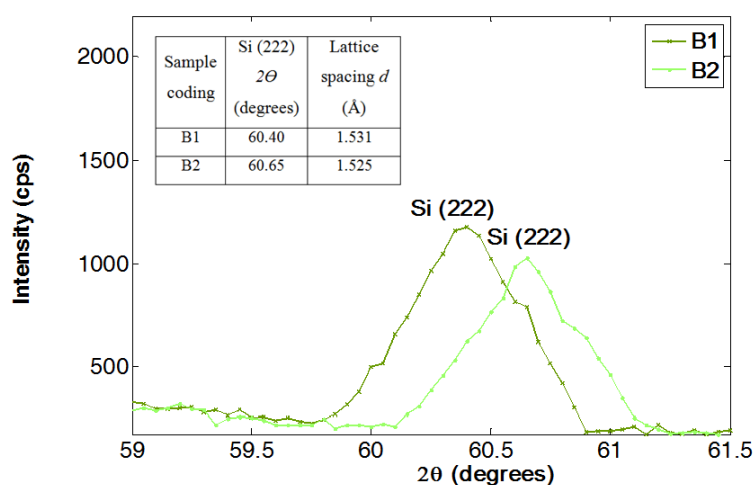


Figure 5.11 Si (222) XRD peak shifts of group B samples in magnified view.

5.2.1.2 AFM Topography and Roughness Analysis

In Figure 5.12, inclined view AFM topography images of group B samples are illustrated. It is evident that B2 has a more uniform size, shape and distribution of nanoislands than B1 which has a more heterogeneous structure in the above mentioned aspects. B1 has nanofeatures in the shape of very shallow pits and heights with random distribution, which resemble pre-nucleation phase formations. On the other hand, B2 gained a more islanded form having similar pit zones with B1. The relatively more islanded form of B2 compared to more planar B1 seem to be a result of the excess annealing duration that is applied to B2.

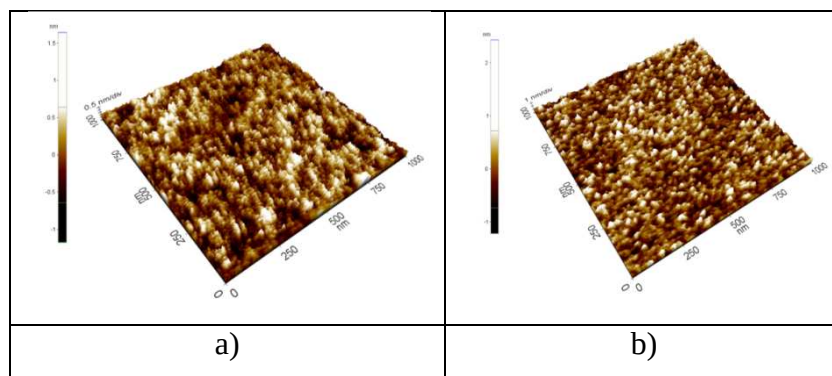


Figure 5.12 Inclined view AFM topography images of the samples a) B1 and b) B2.

Top view AFM images and length histograms of group B samples depict the feature size distribution of the samples as illustrated in Figure 5.13. Accordingly, lengths of nanofeatures of B1 and B2 vary between 30-60 nm and 10-40 nm in average respectively.

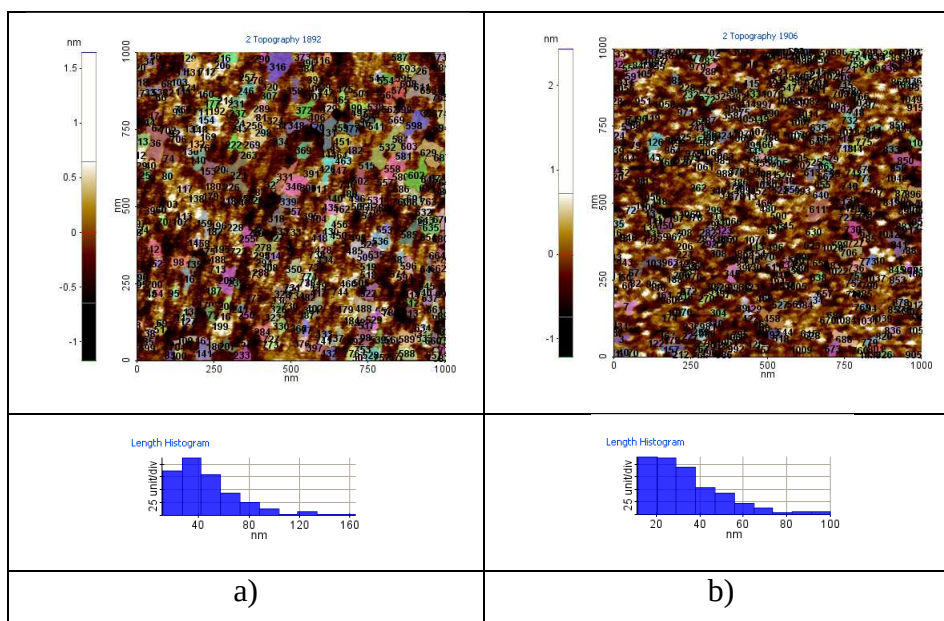
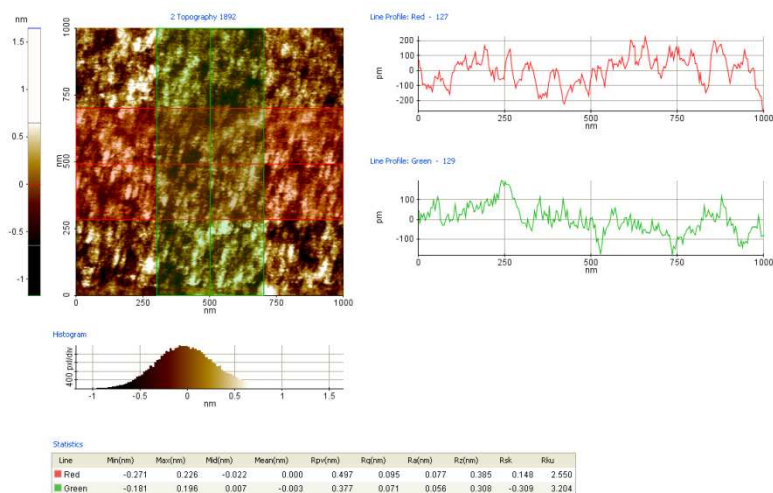


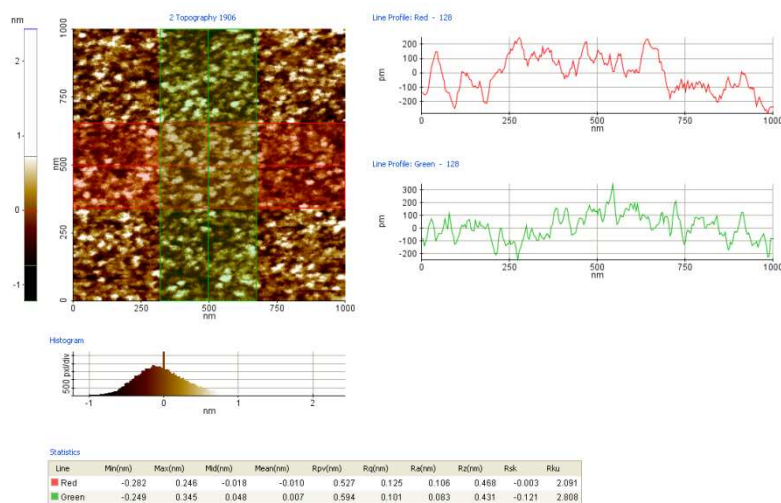
Figure 5.13 Top view AFM images and length histograms of the samples a) B1 and b) B2.

R_a analysis of the samples is performed in the area segments that are shown in red and green as depicted in Figure 5.14. It is noteworthy that the R_a values of B1 vary

between 0.056 nm and 0.077 nm which indicate that the surface is quite planar. On the other hand, this value is between 0.083 nm and 0.106 nm for B2 as it is in more islanded form.



a)



b)

Figure 5.14 AFM area roughness analysis of the samples a) B1 and b) B2.

5.2.1.3 SEM Analysis

B1 and B2 gained a channeled cluster form that is common in pre-islanding stage of growth process due to the excess Si on the surface as demonstrated in Figure 5.15. The 500.000X magnification images of both samples indicate that B2, which had been

annealed ten times longer than B1 gained slightly more granular structure compared to B1. In addition, partial island formation is also observed in both samples when viewed through the inclined images. It is also important that the obtained film thicknesses as indicated in the cross sectional views match with the layer design thickness values.

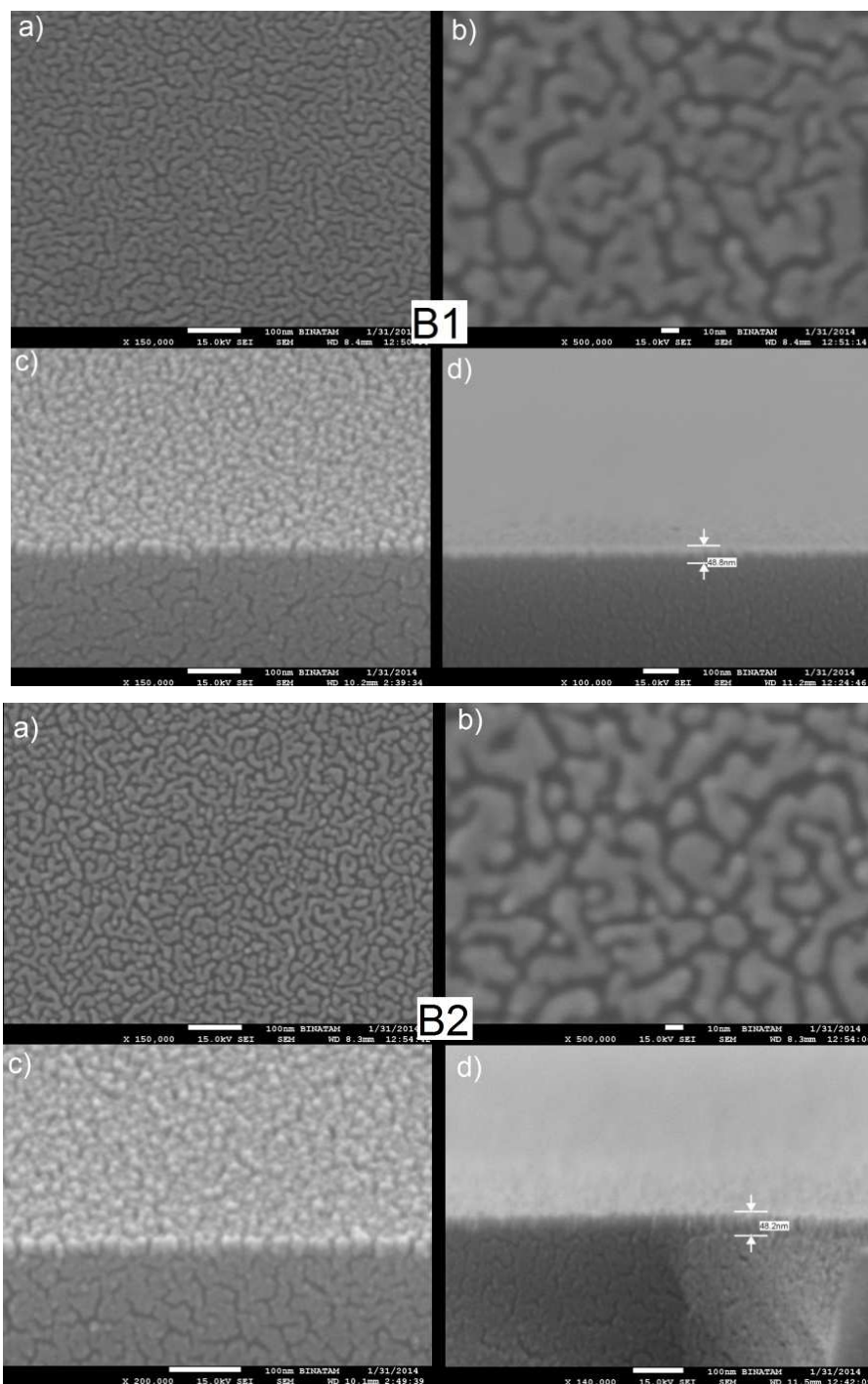


Figure 5.15 Top and inclined view SEM images of group B samples.

5.2.2 Optical Characterization of SiGe Nanoislands

5.2.2.1 Raman Analysis

Figure 5.16 demonstrates the dispersive Raman spectra of group B samples. Si-Si phonon vibration peaks of both B1 and B2 are observed at 518 cm^{-1} where Ge-Ge peaks of B1 and B2 are located at 296 cm^{-1} and 301 cm^{-1} respectively. The strong Si-Si signal, which is one order of magnitude greater than Ge-Ge peak is originated by Si substrate. On the other hand, Ge-Ge signal is very faint due to the fact that top layers have low Ge content. No Si-Ge vibration peak is observed at about 400 cm^{-1} due to the lack of SiGe matrixes. The slight shift of Ge-Ge wavenumber of B2 from 296 cm^{-1} to 301 cm^{-1} can be explained by the strain induced via extra 45 minutes of annealing of B2 than B1. It is also noteworthy that Ge-Ge peak of B1 is broader than that of B2 since B1 is annealed only 5 minutes.

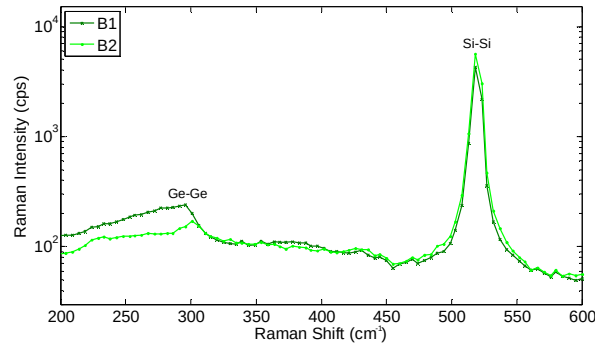


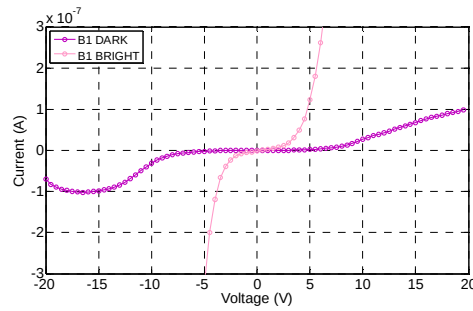
Figure 5.16 Raman spectra of group B samples.

5.2.3 Electrical Characterization of SiGe Nanoislands

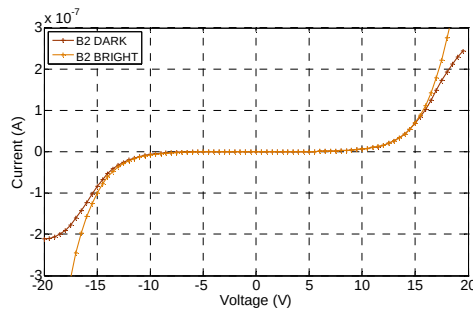
5.2.3.1 I-V Characteristics Measurements

I-V characteristics of group B samples between -20 V and $+20\text{ V}$ are depicted in Figure 5.17. Measurements were done in dark medium and under LED illumination for both B1 and B2. A quite low value of current flow at 10^{-7} A range was measured through the surfaces of both samples. I-V characteristics of the samples have shown

similar attitudes in dark and under illumination. For dark measurements, B1 gained tunnel diode type I-V curve for negative voltage values where under illumination its I-V characteristics shifted to semiconducting type [95] mechanism with an abrupt increase in current value. Similarly, B2 got the form of a tunnel diode with a relatively broader curvature of negative differential resistance in dark medium. Besides, under illumination, its tunnel type I-V mechanism turned to semiconducting one with a slight gain of current. A peak-to-valley ratio [97] of 1.45 at 20V in negative differential resistance zone, which indicates the degree of tunneling, is obtained for B1. The tunnel diode type conduction mechanism of both B1 and B2 can be explained by the existence of vacancies between the channeled type surface topography that enable the quantum tunneling phenomenon. Furthermore, the abrupt change of current value in B1 by illumination may be originated due to the longevity of the pre-islanded segments which enable the fast overlapping of the band gaps in between the channels. However, the more channeled and isolated topography of B2 due to extra duration of annealing resulted in almost conservation of the band gap structure and current gain despite the illumination.



a)



b)

Figure 5.17 Dark & Bright I-V measurements of the samples a) B1 and b) B2.

5.3 CHARACTERIZATION OF GROUP C SAMPLES

5.3.1 Structural Characterization of SiGe Nanoislands

5.3.1.1 Grazing Incidence XRD Scan Results

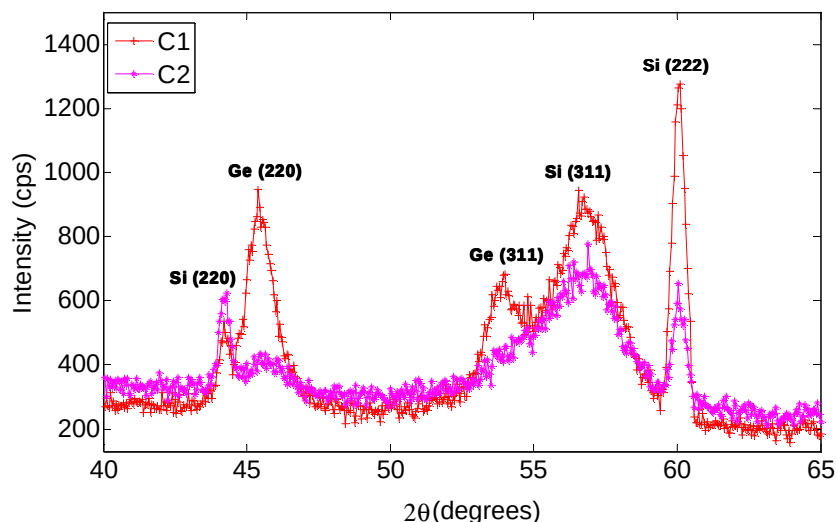


Figure 5.18 Grazing angle XRD spectra of group C samples.

In figure 5.18, grazing angle XRD Si (220) peaks and substrate peaks of group C samples are shown together with the Ge (220) and Ge (311) peaks located at 45.4° and 53.8° respectively. Si (311) and Si (222) substrate peaks of C1 are greater than that of C2 where Si (220) peak of C2 is slightly greater than related C1 peak. The observation of substrate peaks despite the growth of relatively thick Si and $\text{Si}_{0.50}\text{Ge}_{0.50}$ layers on Si can be an evidence of epitaxial growth in both samples. The sample C1, which had been annealed at 750°C for 30 minutes, has sharp Ge (220) and Ge (311) peaks. On the other hand, C2 with 900°C annealing temperature has a very faint Ge (220) peak and no Ge (311) peak at all. The reason why Ge peak intensities disappeared in C2 can be explained by over mixing of Ge crystals domains with Si layers below due to the excess annealing temperature. It is noteworthy that Si (222) substrate peaks of both samples have no diffraction angle shift.

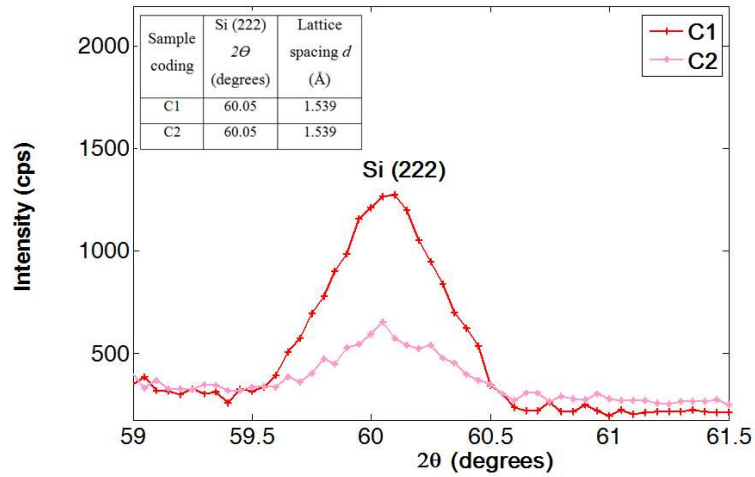


Figure 5.19 Si (222) XRD peak shifts of group C samples in magnified view.

A magnified view of the related peaks of C1 and C2 which are located both at 60.05° are depicted in Figure 5.19. The lattice spacing values of the samples are calculated as $1.539 \text{ \AA}$ (see inset table of Figure 5.19). It is possible that the formation of epitaxial SiGe matrixes [94] were promoted by the induction of equal amount of strain energy per cubic cell. As a result, the lattice spacing of both samples match well although their peak intensities are quite different from each other.

5.3.1.2 AFM Topography and Roughness Analysis

Inclined view AFM topography images of group C samples as illustrated in Figure 5.20 indicate a moderately uniform size, shape and distribution of nanoislands of C1 and C2. Both samples contain surface features like clustered islands and relatively deep pits along with the more dominant and homogeneous nanoislanded structure. It is also noticeable that nanoislands in C2 are highly intermixed due to their excess annealing temperature of 900°C where in C1 islands seem to be more isolated since they are almost left as-grown with an annealing temperature of 750°C .

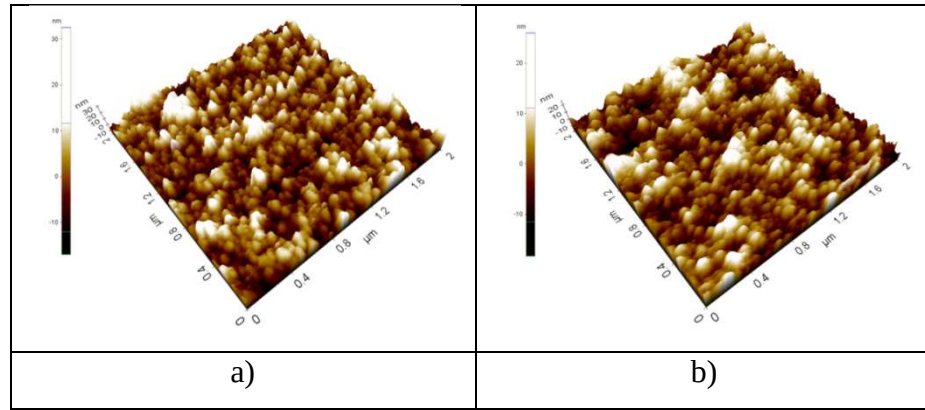


Figure 5.20 Inclined view AFM topography images of the samples a) C1 and b) C2.

Feature size distributions of the group C samples are illustrated by the top view AFM images and length histograms in Figure 5.21. Accordingly, length of nanofeatures of C1 and C2 vary between 40-120 nm and 30-120 nm in average respectively.

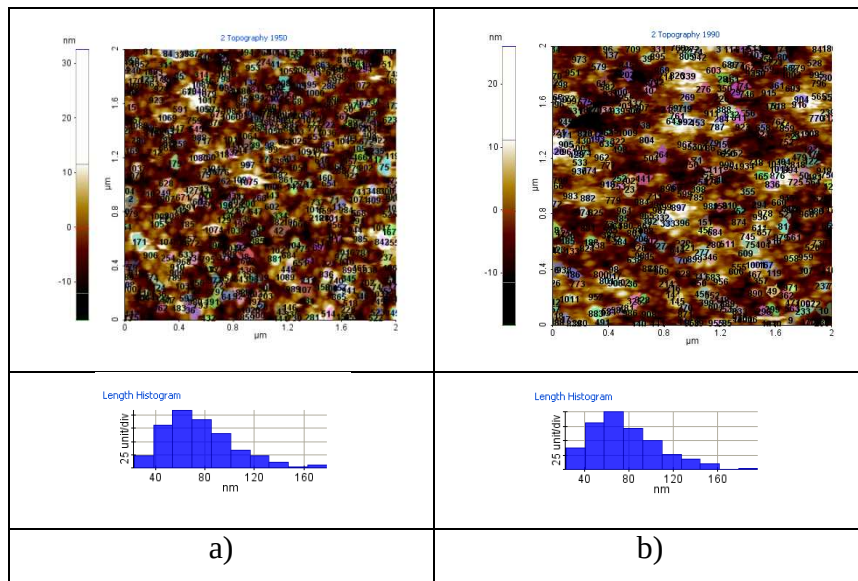
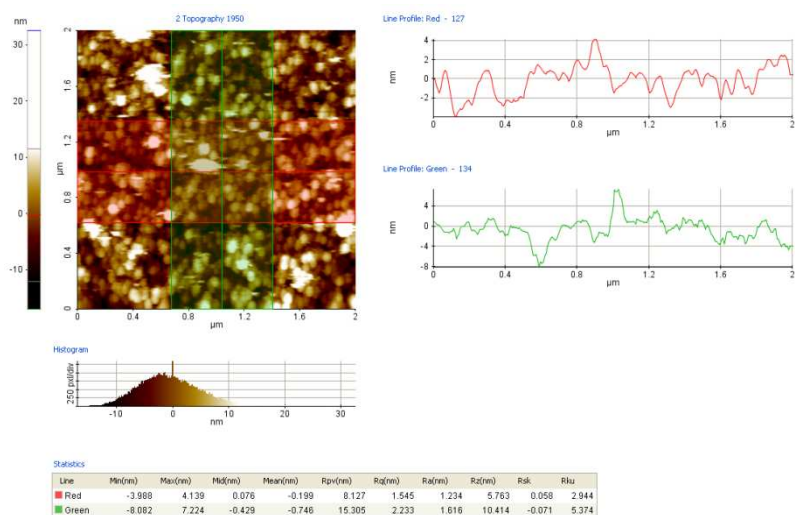


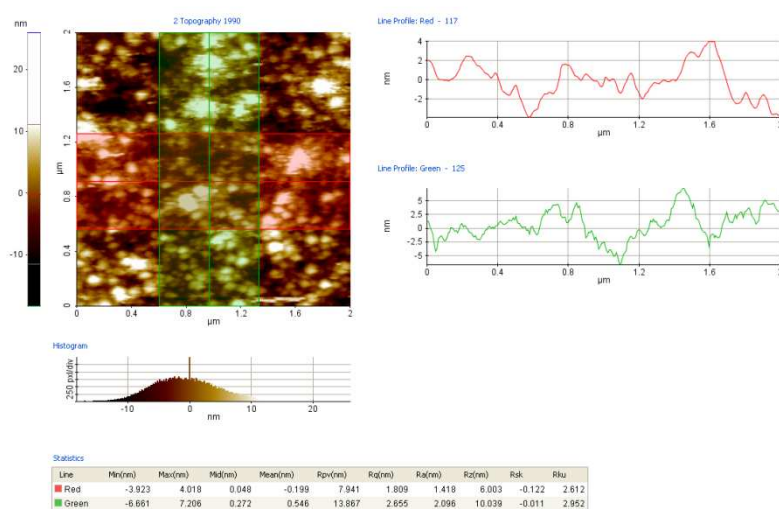
Figure 5.21 Top view AFM images and length histograms of the samples a) C1 and b) C2.

R_a analysis of the samples that is performed in red and green area segments as are shown in Figure 5.22. R_a values of C1 vary between 1.234 nm and 1.616 nm where

these values are between 1.418 nm and 2.096 nm for C2. So, it can clearly be stated that both samples have quite similar R_a values and surface topography as it can be seen in the top view images.



a)



b)

Figure 5.22 AFM area roughness analysis of the samples a) C1 and b) C2.

5.3.1.3 SEM Analysis

C1 and C2, as illustrated in top view SEM images in Figure 5.23 have a uniform distribution of islands with high intermixing ratio. As it can be seen in 75.000X and

100.000X magnification inclined view images of C1 and C2 respectively, C1 has relatively smaller islands that are extremely closely stacked where those in C2 have greater sizes in the shape of more isolated buds due to the extreme annealing temperature of 900°C.

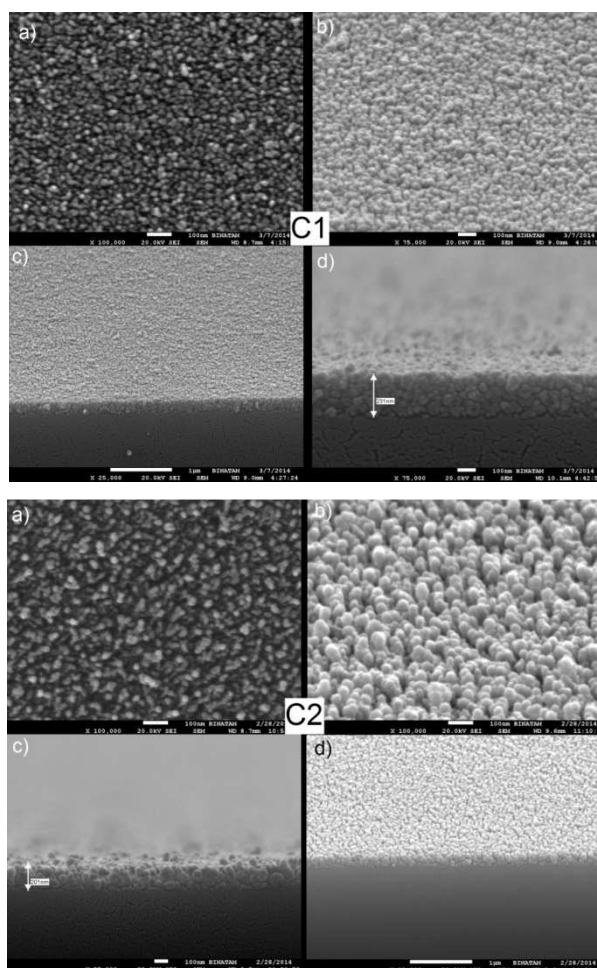


Figure 5.23 Top and inclined view SEM images of group C samples.

5.3.2 Optical Characterization of SiGe Nanoislands

5.3.2.1 Raman Analysis

Dispersive Raman spectra of group C samples are illustrated in Figure 5.24. Si-Si and Ge-Ge phonon vibration peaks are observed at 518 cm^{-1} and 291 cm^{-1} where Si-Ge peaks of C1 and C2 are located at 392 cm^{-1} and 397 cm^{-1} respectively. C1 has strong Ge-

Ge, moderate Si-Ge and weak Si-Si Raman signal. However, C2 has almost ten times stronger Si-Si signal than C1. The reason why both samples have the related Si-Si, Ge-Ge and Si-Ge Raman active optical phonons can be explained by the existence of epitaxial $\text{Si}_{0.5}\text{Ge}_{0.5}$ top layer obtained by adequate temperatures of both growth and annealing processes. Furthermore, peak intensity ratios of Si-Ge to Ge-Ge optical phonon vibrations of C1 and C2, which show the degree of intermixing and heteroepitaxy are calculated as 0.29 and 0.33 respectively [94]. It is also noticeable that the slight shoulder that is observed at 455 cm^{-1} in both samples may have emerged since the layers are grown extremely thicker compared to a few atomic layers.

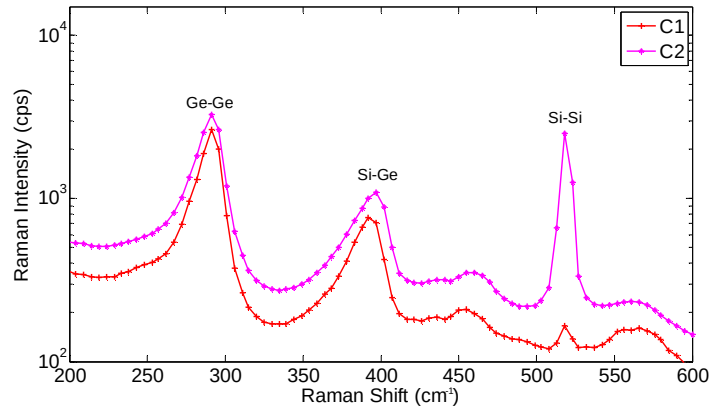


Figure 5.24 Raman spectra of group C samples.

5.3.3 Electrical Characterization of SiGe Nanoislands

5.3.3.1 I-V Characteristics Measurements

I-V characteristics of C1 and C2 between $\pm 13\text{ V}$ and $\pm 20\text{ V}$ are depicted respectively in Figure 5.25. Measurements that were done in dark and under illumination gave the current values in the range of 10^{-3} A for C1 and 10^{-5} A for C2. I-V characteristics of C1 has shown Ohmic type conduction mechanism where the curve in C2 got the shape of a semiconducting one both in dark and under illumination. The large discrepancy between the electrical properties of the semiconductor surfaces of C1 and C2 lies in the surface topography of these samples. Extremely intermixed islanded

structure of C1 than C2 having more isolated domes with vacancies enables the passage of higher currents on the surface. Besides, rich Ge content also enhances the current flow reducing the energy barrier between the valence band and the conduction band of both samples.

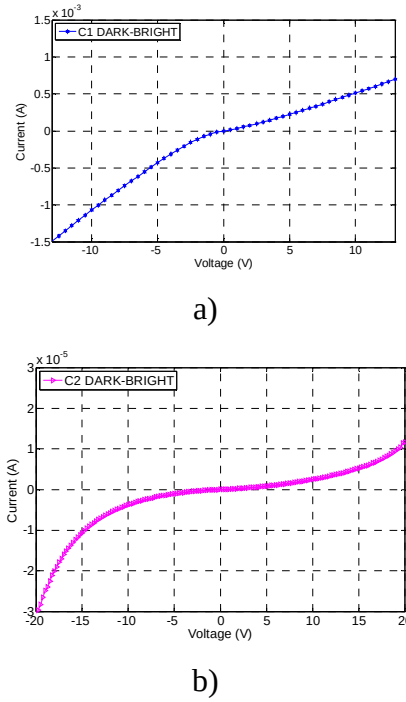


Figure 5.25 Dark & Bright I-V measurements of the samples a) C1 and b) C2.

5.4 CHARACTERIZATION OF GROUP D SAMPLES

5.4.1 Structural Characterization of SiGe Nanoislands

5.4.1.1 Grazing Incidence XRD Scan Results

Figure 5.26 depicts the grazing angle XRD spectra of Si (220) peaks and substrate peaks of group D samples together with the Ge (220) and Ge (311) peaks located at 45.4° and 53.8° respectively. Si (220) peak of D1 is fairly greater than that of D2 where Si (311) and Si (222) substrate peaks of both samples are almost of the same magnitude except that D1 gained an unusual shape [91] due to the strain between the SiGe interfaces. In addition, the presence of strong crystallization of Si rich Si (220) domains

may be explained by the repeated inter-step annealing process of D1. Both D1 and D2 have strong Ge (220) and Ge (311) peaks due to the rich Ge content at the cap layers along with the high temperature growth and annealing processes of both samples at 750°C and 850°C respectively. It is remarkable that Si (222) substrate peaks of both D1 and D2 have relatively large lattice spacing shift.

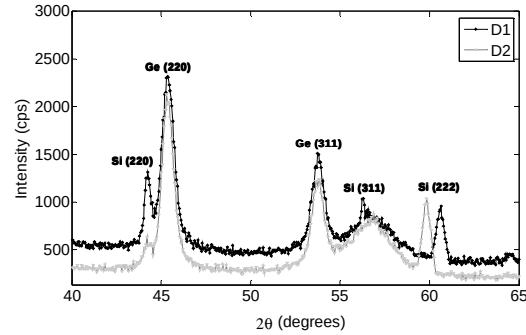


Figure 5.26 Grazing angle XRD spectra of group D samples.

The related peaks of D1 and D2 which are located at 60.65° and 59.85° respectively are depicted in Figure 5.27 in a magnified view. The lattice spacing values of the samples are given as 1.525 Å and 1.544 Å respectively (see inset table of Figure 5.17). Although the shape and intensities of Si (222) peaks are almost identical the lattice spacing of D1 and D2 has a large discrepancy due to the compressive strain induced in D2 and misfit dislocation formation in D1.

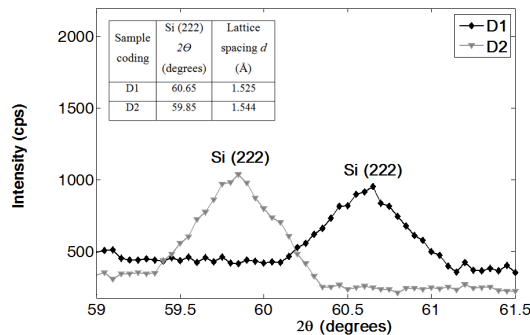


Figure 5.27 Si (222) XRD peak shifts of group D samples in magnified view.

5.4.1.2 AFM Topography and Roughness Analysis

In Figure 5.28, inclined view AFM topography images of group D samples illustrate the large discrepancy in size, shape and distribution of nanoislands of D1 and D2. D1, which was grown with the repeated annealing process of its individual layers at 850°C for 30 minutes, gained an enormously big islanded structure which is in the isolated dome shape. Besides, these nanodomains can be categorized roughly as small, medium and large sized ones according to their sizes. It can be stated that, the inter-step annealing process applied to D1 resulted in a dramatic nucleation with the presence of rich Ge content. On the other hand, D2, which was only post-annealed at the same temperature with D1, gained nanoislanded structure at moderate sizes compared to D1. In addition, nanoislands in D2 are highly intermixed with the local pits and clustered islands which are not observed in D1 at all.

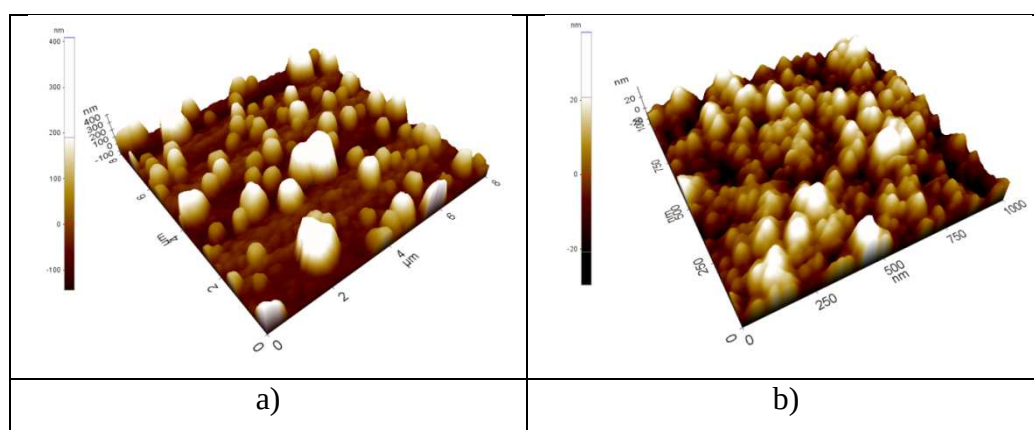


Figure 5.28 Inclined view AFM topography images of the samples a) D1 and b) D2.

Nanoisland size distributions of the group D samples are depicted by the top view AFM images and length histograms in Figure 5.29. Accordingly, diameters of nanoislands of D1 and D2 vary between 200-600 nm and 25-75 nm in average respectively.

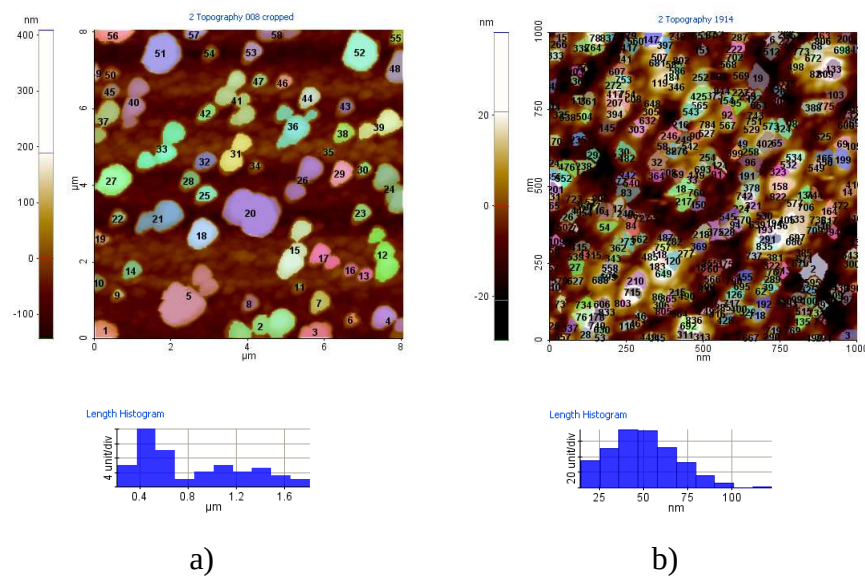
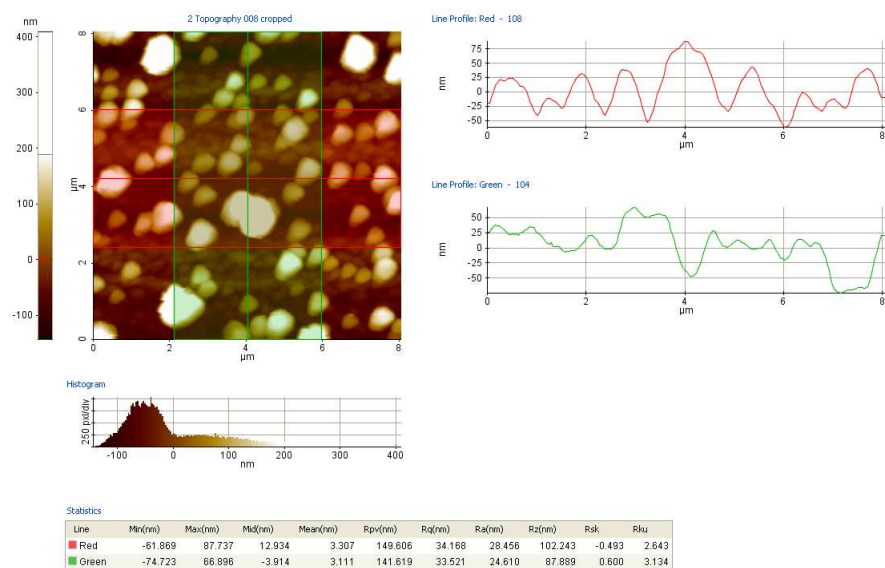
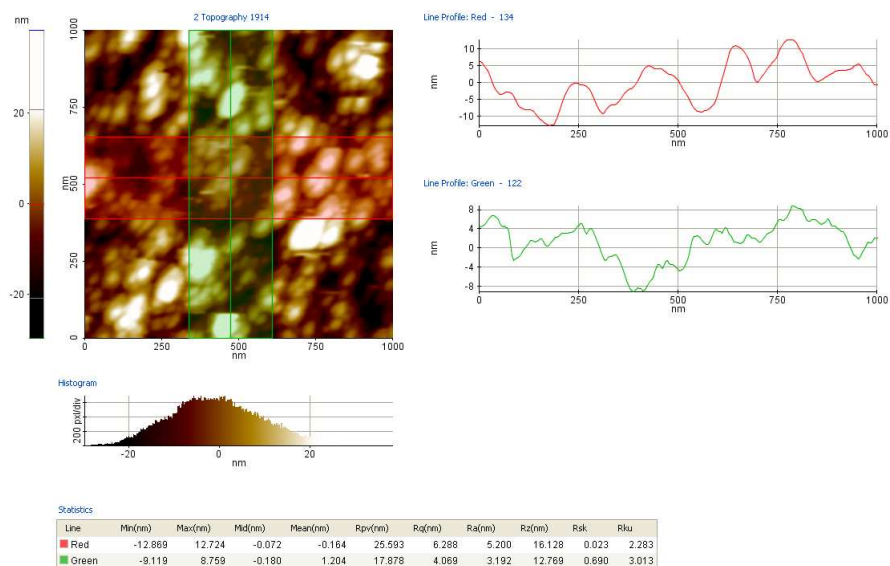


Figure 5.29 Top view AFM images and length histograms of the samples a) D1 and b) D2.

In Figure 5.30, R_a analysis of the samples that is performed in red and green area segments as are illustrated. R_a values of D1 vary between 24.610 nm and 28.456 nm where these values are between 3.192 nm and 5.200 nm for D2. So, it is apparent that D1 has almost seven times larger R_a value than D2 although there observed large vacancies on the surface of D1.



a)



b)

Figure 5.30 AFM area roughness analysis of the samples a) D1 and b) D2.

5.4.1.3 SEM Analysis

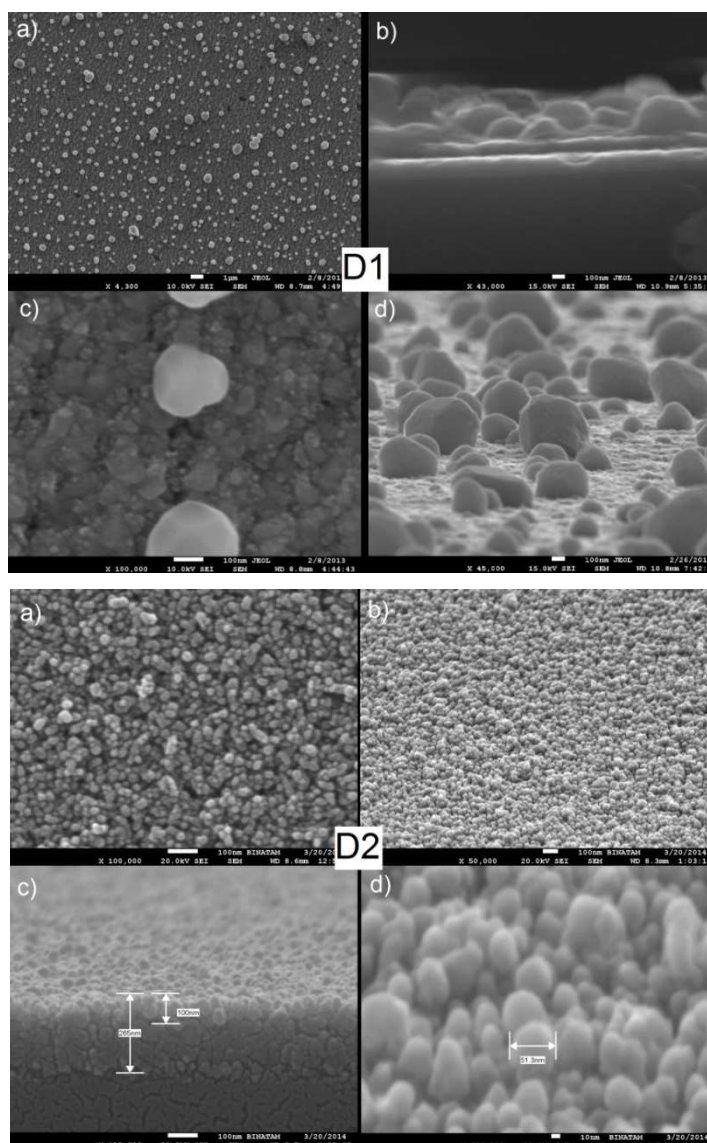


Figure 5.31 Top and inclined view SEM images of group D samples.

It is outstanding that crystalline Ge rich islands of D1 have sizes at about 100-400 nm and are about one order of magnitude greater than that of D2 which has 20-50 nm diameter islands. Stranski-Krastanov mode of island growth is clearly apparent in the cross sectional image of D1 in Figure 5.31 as 2D film growth is followed by 3D island formation. The inter-step annealing process performed in D1 resulted in a dramatic Ge nucleation plainly-different than D2. Ge crystal facets [93] of D1 islands with various

hkl orientations can be seen well in inclined image of D1. D2, on the other hand has quite homogeneous island distribution as seen in its top and inclined view images.

Figure 5.32 indicates the EDS spectra and the detailed quantitative analysis result of the sample D1. Ge/Si ratios of all samples were not shown here as it is not totally clear that whether the Si X-Ray signals are emitted from the substrate or the deposit. D1 seem to have 53.3% Ge and 46.7% Si content. However, it is predicted that the actual percentage of Ge should be quite larger than the measured value since the part of X-Rays collected are emitted from the substrate atoms due to the high penetration depth of incident electron beams.

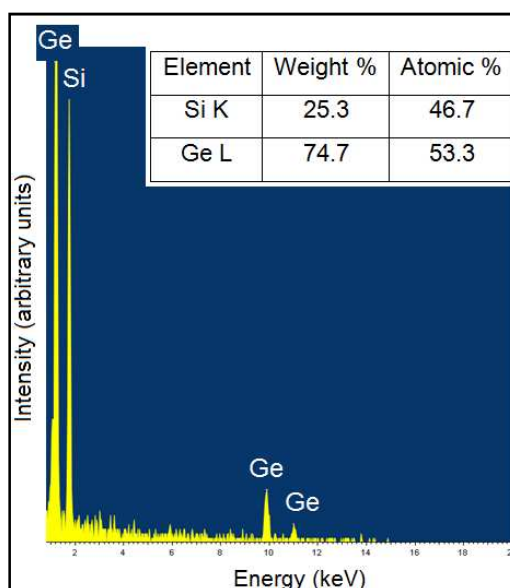


Figure 5.32 EDS spectra of sample D1.

5.4.2 Optical Characterization of SiGe Nanoislands

5.4.2.1 Raman Analysis

Dispersive Raman spectra of group D samples are depicted in Figure 5.33. There observed no substrate Si-Si vibration peaks at around 518 cm^{-1} , instead, bulk Si-Si vibration peaks that are located at 566 cm^{-1} arose in both samples. On the other hand, strong Ge-Ge phonon vibration peaks are detected at 296 cm^{-1} where no evidence of Si-

Ge vibration is apparent in both D1 and D2. It is possible that the substrate Si-Si vibration peak could not be obtained since the Raman signal could not penetrate the relatively thicker film as it has a total thickness at around 300 nm. As a result, the expected strong Si-Si vibration turned out to be a bulk Si-Si signal since it was surpassed and shifted by the thick Ge rich layers. In addition, the reason why D2 has almost twice the intensity of D1 in Ge-Ge vibration can be explained by the fact that, D2 has closely stacked nanoislands where nanodomes in D1 have more isolated form with large vacancies.

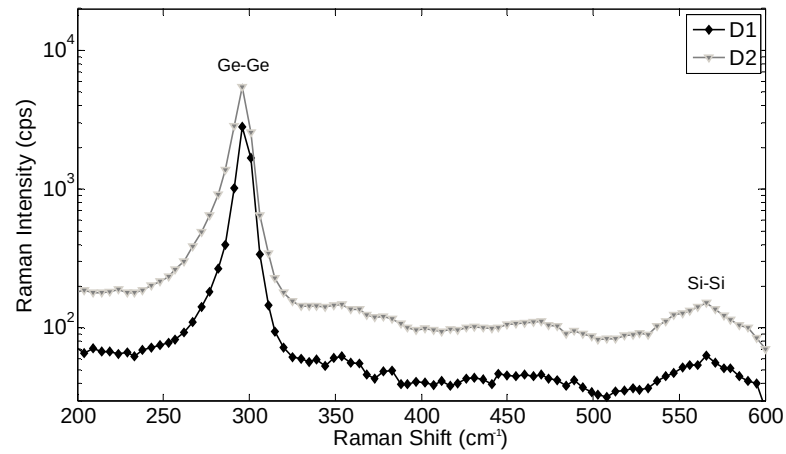


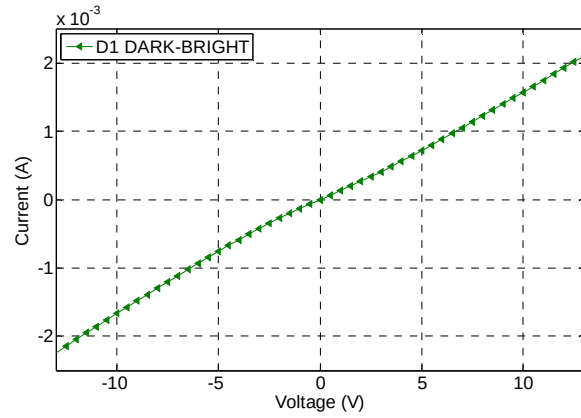
Figure 5.33 Raman spectra of group D samples.

5.4.3 Electrical Characterization of SiGe Nanoislands

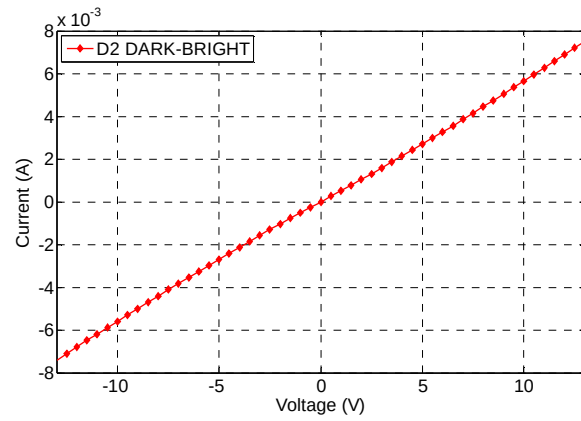
5.4.3.1 I-V Characteristics Measurements

I-V characteristics of D1 and D2 between ± 13 V are depicted in Figure 5.34. Measurements that were done in dark and under illumination gave the current values in the range of 10^{-3} A for both D1 and D2. I-V characteristics of D1 and D2 have shown Ohmic type conduction mechanism where D2 gained almost four times greater current gain than D1 in dark and under illumination. The slight discrepancy between the current gain of D1 and D2 may be explained by the difference of the surface topography of these samples due to the extreme inter-step annealing of D1 that resulted in dramatic islanding with large vacancies. More intermixed islanded structure of D2 than D1

enables the passage of higher currents on the surface. In addition, rich Ge content also promotes the current flow reducing the energy barrier between the valence and conduction bands of both samples.



a)



b)

Figure 5.34 Dark & Bright I-V measurements of the samples a) D1 and b) D2.

CHAPTER 6

CONCLUSIONS

6.1 SUMMARY OF RESULTS

The purpose of the current study was to determine the effect of layer design and growth conditions on structural, optical and electrical properties of MBE grown SiGe nanoislands. The four layer designs with various contents of pure Si, Ge and SiGe mixture at different composition percentages enabled a comparative approach to understand the behavior of the materials system. In addition, the efficient use of various growth conditions between the sample group members and among the groups yielded a well overall understanding of the kinetics of the MBE growth mechanism.

First, Grazing angle XRD method provided a broad analysis of the crystalline properties and lattice parameter shifts of the samples. The coexistence of Si (220) and Si (311) peaks along with Ge (220) and Ge (311) peaks prove the formation of nano sized crystalline SiGe alloys. Besides, utilization of Si (222) peak intensities reveals the strain [91] induced between the substrate and the growth thin film. It is also noticeable that Ge content and the effect of growth and annealing temperatures played a predominant role in determining the strength and shape of the related peaks. Furthermore, grazing angle XRD characterization of these samples with comparative growth conditions and layer design approach is unique to this study which enabled an overall contribution to the literature different than the previous studies [92,93].

AFM technique, on the other hand, made it possible to examine the surface topography, island size distribution and average roughness analysis of the samples. Accordingly, various types of surface structures such as hut islands, sharp edged

clusters, and nucleated superdomes depending on the layer design and growth conditions. It is a remarkable finding that there observed a strong relationship between the surface topography and the electrical conduction type of the samples.

In addition to AFM, SEM analysis of the samples also unveiled the structural properties of surface structures in more detail. The images taken at different magnifications and orientations provided detailed information about the shape, size and distribution of the nanostructures. Formation of various types of SiGe nanostructures like Ge rich nanodomes that are common in literature was observed. However, formation of unusual shapes like hump type topography or channeled type islanding made it difficult to understand the nature of the nanostructures. Fortunately, it became possible to explain the formation of these nanostructures utilizing a complementary set of characterization techniques which is particular to this research all together.

Dispersive Raman spectroscopy spectra of the samples evidence the formation of Si-Si, Ge-Ge and Si-Ge phonon vibrations. It is vital that the formation of the epitaxial SiGe matrix and the degree of intermixing can efficiently be determined using this method. The ratio of Si-Ge to Ge-Ge intensity, which is calculated as 0.33 for the maximum value, is counted as a moderate value of SiGe intermixing in the literature [94].

I-V measurements that were taken from the surfaces of the thin films in dark and under illumination enabled to understand the electrical properties of the samples. It is clear that SiGe nanostructures gained different I-V characteristics depending on the composition and morphology of the sample surfaces.

Closely stacked Ge rich nanoislands have shown Ohmic type I-V characteristics where those which have moderately intermixed SiGe composition gained semiconducting type current flow mechanism [95]. On the other hand, the tunnel diode type I-V conduction mechanism [96,97] was observed in the Si rich islands which have isolated form of island distribution. Besides, presence of illumination during I-V measurements resulted in a significant amount of change in the conduction type of the samples.

There have been significant findings in the electrical properties of the samples that are special to this study: First, fabrication of room temperature SiGe tunnel diode structure with a peak-to-valley ratio of 1.45 at 20V without p-n doping is. Second, it is found that the tunnel diode structures respond to visible light excitation simultaneously switching between the tunneling and semiconducting type conduction mechanisms.

Under normal circumstances, tunnel diodes are built by the growth of highly doped p-n layers one top of each other. However, in our case, the tunneling diode mechanism is obtained by bringing the various compositions of intrinsic SiGe domains to an adequate proximity to form heterojunctions. Self-assembly growth of nanostructures without p-n doping to tailor tunneling devices is more advantageous than the common methods as they require the strict control of the doping concentration of each layer. However, it requires much effort to construct these devices as they suffer from low peak-to-valley ratios, which should be at least 3 at lower voltages for room temperature [97].

In summary, SiGe nanoislands may be utilized to construct oscillators [98] in the terahertz operation zone since their composition, shape, size and distribution can efficiently be tuned using MBE growth method.

6.2 FUTURE RESEARCH

SiGe nanostructures are commercially used as SiGe HBTs [99]. Many device application of these hetero-structures such as photo detectors [100], resonant tunneling diodes [98] etc. are commonly studied. However, these systems which can monolithically be integrated on Si VLSIs have great potential to become more common in optical telecommunication wavelengths.

SiGe quantum wells, dots and cascade structures are intensively studied and can enable the fabrication of light emitting diodes in solid state lighting [97]. In addition, visible or infrared laser applications of these materials like quantum cascade lasers (QCL) which are still in their research phases are good candidates in photonics to

compete with III-V systems [101]. Hence, it is vital that these structures are examined intensely in order to be utilized in nanoelectronic device applications.

For further research, the lithographic patterning of these self-assembly SiGe nanostructures is envisaged. Combination of these two methods (lithography and self-assembly) may lead to improvements in device performance via more controlled band-engineering [102]. The efficiency of thin film solar cells [40] can be increased by incorporating the epitaxial SiGe layers within the device matrix as they could be used as heterojunctions. Finally, implementation of neuron cells [103] on bio-compatible nano-patterned SiGe structures and devices like FETs are projected among the further studies that could be possible in near future.

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APPENDIX A

DECLARATION STATEMENT FOR THE ORIGINALITY OF THE THESIS, FURTHER STUDIES AND PUBLICATIONS FROM THESIS WORK

A.1 DECLARATION STATEMENT FOR THE ORIGINALITY OF THE THESIS

I hereby declare that this thesis comprises my original work. No material in this thesis has been previously published and written by another person, except where due reference is made in the text of the thesis. I further declare that this thesis contains no material which has been submitted for a degree or diploma or other qualifications at any other university.

Signature:

Date: January 05, 2015

A.2 FURTHER STUDIES

Further studies related to this thesis work can be done in the following areas:

- a) Combination of the lithographic patterning and self-assembly growth methods for SiGe nano-electronic device construction.
- b) The incorporation of epitaxial SiGe layers within the thin film solar cells matrix to improve device efficiency.

A.3 PUBLICATIONS FROM THESIS WORK

Article Submission

- Şeker, İ., Ünal, B., Yanık, S., Bakış, Y., Karakız, M., “Influence of growth conditions on structural, optical and electrical properties of MBE grown SiGe nanoislands”, *Applied Surface Science*, December 19, 2014.

Posters

- Şeker, İ., Ünal, B., Bakış, Y., Karakız, M., Yanık, S., “Influence of Layer Design and Growth Conditions on the Structural, Optical and Electrical Properties of Epitaxial SiGe Quantum Dots”, *30th European Conference on Surface Science*, Antalya-Turkey, September 2014.

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EDUCATION

Ph. D., Physics, Fatih University, Istanbul, Turkey.

Dissertation: “Investigation of Structural, Optical and Electrical Properties of MBE Grown Si-Ge Thin Films”

M.S., Physics, Fatih University, Istanbul, Turkey, September 2009

Thesis: “Dielectric Properties of Polymeric Nanocomposites of Complex and Cobalt Ferrites”

B.S., Physics Education, Middle East Technical University, Ankara, Turkey, August 2004

PROFESSIONAL EXPERIENCE

- Specialist, Bionano Technology R&D Center, Fatih University, Istanbul, Turkey, (2013-)
- Research Assistant, Physics Department, Fatih University, Istanbul, Turkey, (2008 - 2013)
- Physics Teacher, International High School, St. Petersburg, Russian Federation (2005 –2007)

AWARDS AND HONORS

- Received Scientific Research Project Scholarship, Fatih Univ. Council 2012
- Awarded National Astronomy Olympiad Award, International High School 2006
- Received Undergraduate English Proficiency Scholarship, Middle East Technical University 1999-2003.

PUBLICATIONS

Article Submission

- Şeker, İ., Ünal, B., Yanık, S., Bakış, Y., Karakız, M., “Influence of growth conditions on structural, optical and electrical properties of MBE grown SiGe nanoislands”, *Applied Surface Science*, December 19, 2014.

Posters

- Şeker, İ., Ünal, B., Bakış, Y., Karakız, M., Yanık, S., “Influence of Layer Design and Growth Conditions on the Structural, Optical and Electrical Properties of Epitaxial SiGe Quantum Dots”, *30th European Conference on Surface Science*, Antalya-Turkey, September 2014.
- Köseoğlu, Y., Şeker I., Okutan, M., San, S. E., Akdoğan, N., “Magnetic and Dielectric properties of $\text{Ni}_{0.5}\text{Zn}_{0.4}\text{Cu}_{0.1}\text{Fe}_2\text{O}_4$ and CoFe_2O_4 Nanocomposites Incorporated with PMMA Polymeric Matrix”, *International Conference on composites and nanocomposites*, Kerala-India, January 2011.

RESEARCH PROJECTS

- “Fabrication of Si/SiGe thin films by Molecular Beam Epitaxy method, their Characterization and Quantum Cascade Laser Application”, Fatih University Research Grant, Project Member: Ünal, B., Researchers: Şeker, İ., (April 2012 – May 2013), Budget: 12,000 TL.

TECHNICAL SKILLS

- Project management experience in both industrial and academic environment.
- Excellent team work abilities and guidance for an interactive academic environment.
- Skilled in structural characterization and imaging software of XRD, AFM, SEM and EDS systems.
- Experience in MBE UHV mechanical and electrical system maintenance and repair.
- Experience in word-processing, presentation and data analysis
- Proficient in MATLAB SIMULINK software used in graph plotting.