High Resolution Electrical Characterization of III-V Materials and Devices

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of III-V Materials and Devices

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Abstract

The continuing shrinkage of semiconductor devices towards nanoscale features and increased functionality has prompted a strong need for high-resolution characterization tools capable of mapping the electrical properties with nanoscale lateral resolution. In this regard, scanning capacitance microscopy (SCM) scanning spreading resistance microscopy (SSRM) and Kelvin probe force microscopy (KPFM) have emerged as powerful techniques.

This thesis focuses on new applications of these techniques for the electrical characterization of III-V materials, devices and low-dimensional systems. One example is the investigation of GaAs/AlGaAs buried-heterostructure lasers with cross-sectional SCM. Several important issues have been addressed: nanoscale contrast related to local band structure, characterization of interfaces and evaluation of electrical properties of the regrown layers. These investigations demonstrate the ability of SCM for reliable, non-destructive and high resolution analysis of opto-electronic devices.

Applications of SCM and SSRM as potential in-line evaluation tool for III-V processing are demonstrated. In this scope, the first work deals with the characterization of ion beam implanted InP, a promising approach to achieve ultrashort carrier lifetimes. The changes in the local electrical properties of this material induced by annealing are tracked. SCM and SSRM measurements were crucial in identifying the local regions of different conductivity due to the non-uniform damage profiles. The results are correlated with those obtained by complementary structural, electrical and optical characterization.

The second work in this category establishes the utility of SCM for evaluating the impact of dry etching on the electrical properties of InP. The highly conductive nature of the near surface damaged layer and its subsequent recovery upon annealing is evidenced. A striking correlation between the SCM signal distributions and the ideality factors of macroscopic Schottky contact is observed.

The last part deals with the electrical characterization of low-dimensional systems using SCM, SSRM and KPFM. The challenging issues motivating this work are the detection and quantification of confined carriers, the determination of band-offsets and the determination of the spatial resolution of the technique employed. The ability of SCM, SSRM and KPFM to detect carriers accumulated in InGaAs/InP quantum wells (QWs) is demonstrated. In each of these techniques, the physical mechanisms underlying the contrast characteristic obtained at QWs are elucidated. The specific issues relating to the determination of the band-offsets are discussed. A new method to determine the “electrical” spatial resolution of SCM and SSRM is addressed using quantum well structures with varying inter-well spacings. Using commercial probes, sub-30 nm and sub-5 nm lateral resolution are determined for SCM and SSRM, respectively. The experimental conditions to perform high resolution measurements are identified.
To my family
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Technical Acronyms

1D/2D/3D: one/two/three dimensional
½FWHM: half of the full width at half maximum
AFM: atomic force microscopy
BH: buried heterostructures
BSE: back scattered electron
CAIBE: chemically assisted ion beam etching
CPD: contact potential difference
DSE: dopant selective etching
DSS: dopant selective staining
ECV: electrochemical voltage profiling
EFM: electrostatic force microscopy
FE: field emission
FET: field effect transistor
FM/AM: frequency/amplitude modulation
FWHM: full width at half maximum
HOPG: highly oriented polygraphite
HVPE: hydride vapor phase epitaxy
KPFM: Kelvin probe force microscopy
LPE: liquid phase epitaxy
MOS/MIS: metal-oxide/insulator-semiconductor
MOSFET: metal-oxide-semiconductor field effect transistor
MOVPE: metal organic vapor phase epitaxy
MQW: multi quantum well
QW: quantum well
RIBE: reactive ion beam etching
RMS: root mean square
RTA: rapid thermal annealing
RCA: resistance capacitance amplifier
SCH: separate confinement heterostructures
SCM: scanning capacitance microscopy
SCS: scanning capacitance spectroscopy
SE: secondary electron
SEM: scanning electron microscopy
SI: semi-insulating
SIMS: secondary ion mass spectroscopy
SPM: scanning probe microscopy
SRP: spreading resistance profiling
SSHM: scanning surface harmonic microscopy
SSRM: scanning spreading resistance microscopy
SSRS: scanning spreading resistance spectroscopy
STM: scanning tunneling microscopy
TEM: tunneling electron microscopy
TUNA: tunneling atomic force microscopy
UHF: ultra high frequency
UHR: ultra high resolution
UHV: ultra high vacuum
List of Symbols

$A^*$: Richardson constant
$a$: tip radius
$C$: tip-sample capacitance
$C_{\text{eff}}$: effective capacitance per unit area
$C_{\text{ins}}$: insulator capacitance per unit area
$C_{\text{tot}}$: effective insulator capacitance per unit area
$C_{\text{net}}$: tip/insulator/semiconductor net capacitance per unit area
$C_{\text{ox}}$: oxide capacitance per unit area
$C_{\text{sc}}$: semiconductor capacitance per unit area
$C_{\text{tot}}$: total capacitance per unit area
$d_{\text{ox}}$: oxide thickness
$d_{\text{ins}}$: insulator thickness
$D_{\text{D}}$: deep donor density per unit of energy (eV$^{-1}$m$^{-2}$)
$e$: magnitude of elementary charge (no unit)
$E$: electrostatic field
$E_g$: electronic bandgap
$E_s$: ground state
$E_n$: energy level of the state $n$ in an infinite potential well
$F$: electrostatic force
$F_{2\omega}$: second harmonic term of the electrostatic force
$F_{\text{dc}}$: constant term of the electrostatic force
$F_{\text{q}}$: first harmonic term of the electrostatic force
$h$: Planck’s constant
$h$: Planck’s constant divided by $2\pi$
$I$: current
$J$: current density
$J_{\text{sat}}$: reverse saturation current density
$k$: Boltzmann’s constant
$L$: width of the quantum well
$L_d$: Debye length
$m_e$: effective electron mass
$n$: density of electrons in the conduction band of the semiconductor or number of the energy level in a quantum well
$N_{\text{e}}$: effective density of states in the conduction band
$N_{\text{v}}$: effective density of states in the valence band
$N_d$: donor concentration
$n_i$: intrinsic electron concentration
$p$: density of holes in the valence band of the semiconductor or pressure in the UHV chamber
$q$: elementary charge
$r$: radius of the annular ring
$R$: total resistance
$R_C$: contact resistance
$R_S$: spreading resistance
$R_{tip}$: tip radius
$S$: tip-sample contact area
$T$: absolute temperature
$V$: voltage
$V_{ac}$: amplitude of the ac modulation
$V_{ap}$: applied voltage
$V_{bias}, V_{dc}$: dc bias
$V_{CPD}$: contact potential difference
$V_{0b}$: diffusion voltage at zero bias, i.e., built-in potential
$V_{fb}$: flatband voltage
$V_t$: potential drop across the insulator
$V_{out}$: SSRM output signal
$w$: depletion width beneath the tip
$x$: element's fraction
$\delta C$: differential capacitance
$\delta C_e$: differential capacitance relative to electrons
$\delta C_p$: differential capacitance relative to holes
$\delta V$: amplitude of the ac modulation voltage
$\Delta C_n$: capacitance variation in the $n$-doped region
$\Delta C_p$: capacitance variation in the $p$-doped region
$\Delta E_c$: confinement potential
$\Delta r$: width of the annular ring
$\Delta V_t$: potential drop due to the oxide
$\Delta V_{SS}$: potential drop due to surface states
$\Delta \Phi$: work function difference between the tip and the sample.
$\varepsilon_0$: permittivity of free space
$\varepsilon_r$: relative permittivity of the insulating layer
$\varepsilon_{ox}$: relative permittivity of the oxide
$\varepsilon_{sem}$: relative permittivity of the semiconductor
$\eta$: ideality factor
$\lambda$: wavelength
$\mu_e$: electron mobility
$\mu_h$: hole mobility
$\rho$: semiconductor's resistivity (SSRM) or charge density (Poisson equation)
$\sigma$: semiconductor's conductivity
$\phi$: effective barrier height
$\Phi_{sample}$: sample work function
$\Phi_{tip}$: tip work function
$\chi_{sample}$: sample electron affinity
$\omega_1$: first cantilever resonance frequency
$\omega_2$: second cantilever resonance frequency
$\Omega$: frequency of the ac modulation
Chapter 1

Introduction

In the last fifty years, semiconductor technology has progressively invaded our everyday life. The last decade of the 20th century has especially seen a spectacular growth of the semiconductor industry, significant enough to induce economical and social changes throughout the world, and opened the door to the age of information. The new challenges imposed by this new information technology (IT) business were set in terms of higher efficiency, density, speed, at lowest cost and dissipated power of the manufactured devices. This led naturally to a continuous shrinkage of the device dimensions. Competitive pressure due to high financial investments in the IT market also implies moving devices rapidly from research to development and manufacturing stages. This requires a tremendous amount of process learning, development and control. Metrology tools are therefore meant to be even more essential since they can provide rapid feedback to those developing the devices.

Major applications motivate the research in III-V materials and devices. Due to the rapid development of the radio and optical communication networks such as the Internet, III-V technology has emerged as a key player. The unique possibility for bandgap engineering and the wide variety of semiconductor materials in the III-V family make it the most appropriate to meet today’s requirements. Using the state-of-the-art of epitaxial growth techniques [1,2] and processing technology [3], fabrication of advanced optoelectronic devices keeps developing tremendously. Lasers with low threshold currents [4,5], novel concepts of optical devices using photonic crystals [6] or optoelectronic integrated circuits [7] are among today’s challenges. However, the fabrication of devices requires numerous tricky processing steps likely to influence carrier distributions in the device, thus their performance [8]. Electrical characterization of devices is therefore of primary importance. This includes, for instance, the control of orientation dependent dopant incorporation during regrowth on patterned substrates, the delineation of regrown interfaces or the evaluation of dopant diffusion [2,9]. Understanding and evaluating the effect of these processing steps on device performance is also a primary concern for successful optimization. For example, dry etching and ion beam implantation are essential routinely employed processes. However, it is well known that they can induce undesirable modifications of the electrical properties of the material. Therefore, evaluation of these processes is crucial to impart better understanding and control of the device performance.

Advances in epitaxial growth opened the door to the fabrication of low-dimensional systems such as quantum wells, quantum wires and quantum dots, providing new possibilities to tailor properties such as the density of states, the band structure and the carrier confinement. The rich variety of heterostructures formed by the different III-V materials (binary, ternary or quaternary) favored the development of these new structures.
and their use in optoelectronic devices. Carrier distribution is then ruled by quantum mechanics and appropriate two and three dimensional (2D and 3D) characterization methods with ultra-high-resolution (UHR) can be foreseen as necessary. On the one hand, such methods can be used to obtain important information on carrier distributions and band-offsets. On the other hand, standard low-dimensional systems can be used to assess the limits of the methods themselves, thus providing feedback for further developing the characterization techniques.

To summarize, high resolution characterization tools are indispensable for a successful development of semiconductor device technology. The key issues driving the development of such tools are the reduced device feature size and the increasing complexity and functionality of recent devices. New nanoscale electrical characterization methods have been developed, driven primarily by the Silicon Technology and its International Technology Roadmap for Semiconductors (ITRS) [10]. For example, for the actual 2003 node, it prescribed 5 nm spatial resolution for 2D and 3D dopant profiles with a concentration precision of ±5 %.

Numerous 2D electrical characterization methods (e.g., dopant/carrier profiling) are available today. They can be divided into three major categories: (i) 2D techniques which are based on widely used one dimensional (1D) techniques such as secondary ion mass spectroscopy [11-16], (ii) Electron microscopy-based techniques like field emission scanning electron microscopy (FE-SEM) [17] and electron holography [18] and (iii) scanning probe microscopy (SPM) based techniques [19]. SPM refers to all techniques that implement a mechanism for scanning a sharp probe tip across a sample surface at very low distance in order to obtain the topographical image and the physical properties of the surface with nano- or atomic scale resolution. The first techniques that used this concept were the scanning tunneling microscopy (STM) [20] and later the atomic force microscopy (AFM) [21]. The importance of SPM was recognized quite early when G. Binnig and H. Rohrer were awarded the Nobel Prize for their invention of the STM in 1982.

The evolution of the SPM concept into a tool suitable for industrial applications then led to the specialization and the development of new measurements [22]. Twenty years later, SPM based techniques have yielded high lateral resolution and high sensitivity together with a wide range of capabilities and applications. The ability to perform UHR topographical profiling has been augmented with new types of measurements: mechatronic, optical, thermal or electrical. The instrumentation allows the scanning process to be operated in a variety of environments – in vacuum, in air as well as in fluids; on any type of material – conductive or insulating, crystalline or amorphous, organic or inorganic. Specifically, electrical measurements have been shown to provide a wide amount of information such as electrical field strength, resistance and capacitance [23,24]. The simplicity of use combined with a short time measurement procedure has, in addition, demonstrated the ability of SPM to provide rapid feedback on device simulation, development and production. However, even if SPM based techniques are now commercially available and some are routinely used in the industry, much work remains to be done to develop 3D-physical models for quantitative analysis. Meanwhile, measurement methodology is still evolving and the capabilities of the techniques are being expanded – new types of materials, structures and devices are being investigated.
The main theme of this thesis work is the nanoscale electrical characterization of III-V materials, devices and low-dimensional structures. It focuses on three SPM techniques, namely scanning capacitance microscopy (SCM), scanning spreading resistance microscopy (SSRM) and Kelvin probe force microscopy (KPFM). The first demonstration of the SCM concept was shown by Matey & Blanc in 1985 [25]. The SCM technique is based on the local measurement of the equivalent differential capacitance of the Metal-Oxide-Semiconductor (MOS) system that is formed by contacting an oxidized semiconductor surface with a conductive AFM probe [23]. The first SSRM measurements were presented by Vandervorst et al. in 1992 [26]. This technique, initially called nano-spreading resistance profiling, allows one to measure current flowing from the tip into the sample at a fixed dc bias and image the variation of the sample resistivity or conductivity [24]. KPFM was proposed for the first time by Nonnenmacher et al. in 1991 [27], although the physical principles involved in the technique were first demonstrated by Thomson (aka Lord Kelvin) at the end of the 19th century [28]. The technique measures the contact potential difference between the conductive probe and the sample by nullifying the electrical field. Each technique has emerged as an appropriate tool for electrical characterization with high spatial resolution and high sensitivity. Today’s applications of these techniques for III-Vs include carrier profiling, delineation of pn junctions, heterostructures and interfaces, analysis of selective area epitaxy and regrowth on patterned substrates, and evaluation of processing (e.g., dry etching, implantation, diffusion).

This thesis is organized as follows: Chapter 2 gives an overview of techniques used for electrical characterization of semiconductor materials and devices, with an emphasis on SPM-based methods. This includes the three techniques used in the scope of this work, other SPM-based techniques and some non-SPM characterization techniques. Chapters 3, 4 and 5 describe the physical principles, the instrumentation and experimental procedure of SCM, SSRM and KPFM, respectively. They also present the different types of image contrasts obtained in several different contexts typically encountered in semiconductor structures and devices contrast relating to doping, pn junctions, depletion regions, heterojunctions and high resistivity materials. The following chapters deal with the main results obtained during the PhD work and represent a summary of the work reported in Papers B to I. In Chapter 6, a complete 2D electrical mapping of GaAs/AlGaAs buried-heterostructure (BH) lasers using cross-sectional SCM is presented. This includes the characterization of the mesa, the delineation of interfaces, the evaluation of the regrowth process in terms of dopant incorporation, the verification of SI properties and specific local band structure related contrast. Chapter 7 presents spatially resolved studies on ion beam process InP. These include SCM investigations on dry etched InP. Consistent results are obtained between SCM results and well established I-V measurements. Experimental observations confirm the formation of a highly doped near surface region after dry etching and the significant recovery of the electrical properties upon annealing. A highlight of this work is the correlation between the SCM intensity distributions and the ideality factors of macroscopic Au/InP Schottky contacts. Cross-sectional SSRM and SCM measurements were also performed on ion implanted InP. Damage removal mechanisms upon annealing are also investigated for various ion species at different annealing temperatures. SCM and SSRM results are successfully correlated with complementary measurements obtained with well-established electrical,
structural and optical characterization techniques. Finally, in Chapter 8, SCM, SSRM and KPFM investigations of InGaAs/InP (QWs) are presented. For each technique, it is shown that it is possible to detect the carriers accumulated in the wells. A consistent trend in the different SPM contrasts was observed as a function of the well width and the barrier doping level. Under specific conditions, it was shown that the depletion regions around the wells could also be resolved. The specificities of the characterized structure make it suitable to address the issue of the spatial resolution of SCM and SSRM. The spatial resolution is shown to be mainly limited by tip averaging effects when the dimension of the structure becomes similar to the tip radius. Significant differences are however observed between SCM and SSRM measurements. This is explained by the different physical measurement principles peculiar to each technique. Further understanding requires proper 3D modeling and simulation. In this scope, a 3D modeling of SCM measurements and the simulation results obtained on QW are presented at the end of the chapter. A guide to the papers included in this thesis (Chapter 9) and the conclusion (Chapter 10) summarizing the major accomplishments and proposing future work orientations finally conclude this thesis.

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References


Chapter 2

High resolution electrical characterization techniques: an overview

In this chapter, an overview of the different SPM-based techniques for electrical characterization of semiconductor structures and devices is presented. The basic concepts of SPM are first introduced and for each specific method, pointing out the main physical principles involved, the strengths and weaknesses. A description of other methods for doping profiling, namely secondary ion mass spectroscopy (SIMS), scanning electron microscopy (SEM) and transmission electron microscopy (TEM) is also given. A summary and a comparison of the different methods complete this overview.

![Diagram of SPM setup](image)

**Figure 2.1:** (a) Schematic of a SPM setup for electrical characterization. The topographical and electrical measurements are simultaneously recorded. (b) Scanning head of the Digital Instrument’s Nanoscope Dimension 3000 microscope mounted with the SCM module.
2.1. SPM based methods

2.1.a Principles

Fig. 2.1 (a) shows a schematic description of a typical SPM experimental set up and Fig. 2.1 (b) shows a picture of the head of the Digital Instrument’s Nanoscope Dimension 3000 microscope mounted with the SCM module. The scanning procedure is conventionally controlled by small voltages applied to high precision piezoelectric actuators to provide accurate nano-positioning.

Every SPM based technique implies a probe-tip scanning across a sample surface at a very small distance. In AFM based techniques, two major modes of scanning can be distinguished. In one case the tip-sample distance is kept constant by applying a constant force. This is accomplished by keeping the cantilever deflection constant by means of a feedback circuit. This serves to record the topography. This mode is called the contact mode and is commonly used in AFM, SCM and SSRM with a force range varying from 10 nN to 70 μN [1-4]. As the tip scans the sample surface, some relevant electric signals across the formed tip-sample system are recorded and provide a 2D-map of the electrical properties of the sample.

In the other scanning mode, the so-called non-contact mode, the cantilever is oscillated using an external signal. The cantilever is usually made oscillating at or close to its first mechanical resonance frequency. Topography is recorded by maintaining a constant force gradient using either the amplitude or phase as the feedback parameter. Electrical information (e.g., potential) is usually detected at the second resonance frequency of the cantilever, to improve the sensitivity and also to avoid interferences with the topography measurement. Due to the absence of contact, this mode is therefore gentler both for the tip and the sample. This mode is used for example in AFM, KPFM and electrostatic force microscopy (EFM). In both modes, the cantilever movement is optically detected by means of a laser and a photo-detector (see Fig. 2.1 (a)).

In order to perform high spatial resolution measurements, ultrasharp probes are required. Fig. 2.2 shows the SEM picture of an etched Si tip used in AFM; the typical tip radius is 10 nm. Each SPM technique, however, requires specific types of probes compatible with the physical parameter it intends to measure (see Appendix B). Obviously, the sharper the probe, the greater the resolution. Nonetheless, the obtainable spatial resolution depends on other parameters or conditions specific to the technique [5]. The “electrical” spatial resolution can vary from few angstroms (Å) to 100 nm depending on the SPM technique. The vertical resolution, i.e., the control sensitivity of the Z-piezoelectric is well below 1 nm.
2.1.b Scanning Capacitance Microscopy (SCM)

SCM measures the differential capacitance $dC/dV$ of a metal-insulator-semiconductor (MIS) structure formed by the contact between a conductive probe and an oxidized semiconductor surface [6]. The scanning operates in contact mode [7]. As in other capacitance-based characterization techniques, SCM is intrinsically sensitive to free carriers. Monotonic variations of the SCM signal are observed in a $10^{12}$ - $10^{18}$ cm$^{-2}$ doping dynamic range [6] and a sub-30 nm spatial resolution is typically obtained [8, Paper I].

SCM has emerged as one of the most promising tools for 2D carrier profiling due to its high sensitivity to carriers and its high spatial resolution. However, the technique remains confronted by issues such as the tip-sample contact, sample surface preparation and the complex modeling for carrier quantification procedure [6].

2.1.c Scanning Spreading Resistance Microscopy (SSRM)

The SSRM technique inherits the ability to perform 2D carrier mapping from conventional spreading resistance profiling (SRP) [9] and AFM. It has a higher spatial resolution compared to SRP [10]. The essential principle of SSRM is similar to that of conventional SRP, i.e., a resistance measurement. Beyond a specific threshold force, the total resistance is dominated by the spreading resistance, which directly relates to the doping. The tip-sample forces (contact forces) are high, typically in the μN range. This implies the use of hard materials for the probes. (e.g., diamond coated).

Improvements in terms of reproducible tip properties, signal to noise ratio and spatial resolution [10, Paper I] can still be obtained. This implies a better control of the tip-sample contact and the impact of probe penetration [11].
2.1.d Kelvin Probe Force Microscopy (KPFM)

KPFM operates in non-contact mode and measures the contact potential. EFM is also based on similar principles. However, KPFM differs from EFM by an additional closed feedback loop which applies a voltage to nullify the electric field [12,13]. Prior to measurement, the work function of the probe-tip is determined. The KPFM signal then directly relates to the work function of the investigated sample. The measurement is therefore sensitive to both the free carrier density and the electron affinity. A sensitivity of 5 meV has been reported [14]. Spatial resolution is improved by performing the measurements for the contact potential in ultra high vacuum (UHV) [14].

With a very high sensitivity and a wide doping dynamic range, KPFM has therefore also emerged as a promising technique for nanoscale electrical characterization. However, significant improvements are still required specifically in terms of lateral resolution and simple surface preparation. Finally the requirements for UHV operating conditions hampers the use of the technique.

2.1.e Scanning Tunneling Microscopy (STM)

This technique measures the current tunneling between a conductive probe and the sample. The tunneling current itself is used for feedback to keep the tip-sample distance constant. Under specific experimental conditions such as UHV, STM has shown to yield the atomic resolution of various surfaces [5]. STM has been used to count the dopant atoms near the sample surface [15,16]. However, dopant concentration must be rather high and located in a very near surface region. The severe experimental conditions (UHV) and the requirement to prepare cross-sections in UHV hamper the practicability of STM for 2D dopant profiling.

2.1.f Tunneling Atomic Force Microscopy (TUNA)

In TUNA, the tunneling current between a conductive probe and a conductive sample through a thin highly resistive layer, usually the oxide of a semiconductor is measured. In this sense TUNA differs from STM and SSRM. The scan operates in low force contact mode and under normal ambient conditions. TUNA operates in the 100 fA-1 pA current range and its main application has been the characterization of oxides in terms of breakdown voltage and thickness [17,18]. However, attempts have been made to use TUNA for carrier profiling [19].

2.1.g Scanning Surface Harmonic Microscopy (SSHM)

SSHM uses an STM integrated into a tunable microwave resonator. The microwave field applied across the tunneling gap is shown to generate higher harmonics in the signal. SSMH measures the third harmonic at frequencies above 1 GHz [20]. Its inherent sensitivity to capacitance/voltage characteristics of semiconductors makes it also
a suitable technique for dopant profiling [21]. SSHM is still at an early stage. Further developments of the technique are expected to raise issues similar to those confronted with SCM.

2.1.h Dopant Selective Etching (DSE) or Staining (DSS)

In DSE, impurity sensitive solutions are used to selectively etch regions depending on the type and the concentration of dopants. The resulting topography is measured using AFM and is suitable for qualitative doping profile [22]. In DSS, a metallic element is selectively deposited via electrochemical reactions between the metal and the dopants using a metal-incorporated solution. Information on the doping is then obtained by imaging the structure using electron microscopy techniques. The poor understanding and control of the etching/staining processes and the lateral resolution limit their utility.

2.2 Conventional characterization techniques

2.2.a Secondary Ion Mass Spectrometry (SIMS)

Secondary ion mass spectrometry is a very powerful technique for analyzing impurities in solids. The technique is sensitive only to the dopant atoms and relies on removal of material by ion beam sputtering. In this sense, the technique is destructive. Passing through an energy filter and a mass spectrometer, ionized atoms sputtered out are analyzed. SIMS's high sensitivity (<10¹⁵ cm⁻³) and high depth resolution (1-5 nm) makes it one of the most powerful and versatile techniques for 1D dopant profiling. More recently, SIMS related techniques have demonstrated their abilities for 2D and 3D dopant mapping [23-26]. 2D-tomography SIMS and lateral SIMS are two such techniques. However, with these methods, the main drawbacks are the complexity of both measurements and analysis.

2.2.b Electron microscopy based techniques

Several techniques using electron microscopy can be used to perform 2D mapping of electrical properties. Field-emission scanning electron microscopy (FE-SEM) using secondary electrons (SE) and backscattered electrons (BSE) has shown to be suitable to extract compositional and doping differences in most popular semiconductor systems, specifically across pn junctions [27]. Sensitivity as low as 10¹⁶ cm⁻³ and sub-6 nm spatial resolution have been reported for favorable specimens [27,28]. These techniques are however qualitative and imply specific sample preparations.

Electron holography is a high resolution interferometric TEM method that has been shown to provide high resolution (sub-10 nm), high sensitivity mapping of the electrostatic potential in semiconductor devices [29,30]. Complex experimental
conditions (including sample preparation) inherent to TEM however make it seldom employed.

2.3 Summary and comparison

The main characteristics of the different characterization techniques discussed above are summarized in Table I. While each method has its own advantages and drawbacks, they differ in various aspects – measured parameter, level of complexity, requirement of special ambient, sample preparation procedure, quantification routine etc.

For nanoscale electrical characterization, the SPM techniques presented above appear to be the most promising tools. The large variety of physical parameters that can be measured with SPM-based techniques makes it possible to address issues encountered in semiconductor technology. In particular, the carrier profiling capability coupled with a high lateral resolution make SCM, SSRM and KPFM appropriate for electrical characterization of advanced devices. Crucial physical issues to which all SPM methods are compelled, for instance the tip/sample interactions, still require further understanding to improve their functionalities.

Table I: Intercomparison of the different techniques suitable for electrical characterization of semiconductor structures and devices. D: dopants, C: carriers.

<table>
<thead>
<tr>
<th>Technique</th>
<th>Resol. (nm)</th>
<th>Dynamic range (cm²)</th>
<th>D/ C</th>
<th>Quantification</th>
<th>Comments and problems</th>
</tr>
</thead>
<tbody>
<tr>
<td>STM-STS</td>
<td>1</td>
<td>10⁷ - 10⁹ cm²</td>
<td>D</td>
<td>Yes</td>
<td>Only junction delineation and dopant type identification</td>
</tr>
<tr>
<td>SCM</td>
<td>10</td>
<td>10⁵ - 10⁸ cm²</td>
<td>C</td>
<td>Yes</td>
<td>Complex quantification procedure</td>
</tr>
<tr>
<td>SSRM</td>
<td>5</td>
<td>10⁴ - 10⁷ cm²</td>
<td>C</td>
<td>Yes</td>
<td>High force range: surface destructive and hard probe requirements (diamond)</td>
</tr>
<tr>
<td>KPFM</td>
<td>100</td>
<td>10⁵ - 10⁸ cm²</td>
<td>C</td>
<td>Yes</td>
<td>Stray-fields limit resolution. Surface sensitive technique induces complex quantification procedure</td>
</tr>
<tr>
<td>C-AFM/ TUNA</td>
<td>10</td>
<td>10⁴ - 10⁷ cm²</td>
<td>C</td>
<td>Yes</td>
<td>New technique. Very sensitive to oxide properties</td>
</tr>
<tr>
<td>SSHEM</td>
<td>5</td>
<td>NA</td>
<td>C</td>
<td>No</td>
<td>No quantification procedure</td>
</tr>
<tr>
<td>DSE + AFS/STM</td>
<td>-</td>
<td>10⁴ - 10⁶ cm²</td>
<td>D</td>
<td>Limited</td>
<td>Poor reproducibility, quantification limited by etching control</td>
</tr>
<tr>
<td>DSE/DSS + SEM/TEM</td>
<td>-</td>
<td>10⁴ - 10⁶ cm²</td>
<td>D</td>
<td>Limited</td>
<td></td>
</tr>
<tr>
<td>Imaging SIMS</td>
<td>100</td>
<td>D</td>
<td>Yes</td>
<td>Resolution limited by target volume Special structures required</td>
<td></td>
</tr>
<tr>
<td>2D-SIMS</td>
<td>30</td>
<td>10⁴ - 10⁶ cm²</td>
<td>D</td>
<td>Yes</td>
<td>Complex sample preparation</td>
</tr>
<tr>
<td>Tomography SIMS</td>
<td>50</td>
<td>10⁴ - 10⁶ cm²</td>
<td>D</td>
<td>Yes</td>
<td>Only lateral dose measured</td>
</tr>
<tr>
<td>Lateral SIMS</td>
<td>5</td>
<td>D</td>
<td>Yes</td>
<td>Special structures required No device characterization</td>
<td></td>
</tr>
<tr>
<td>2D-SRP</td>
<td>100</td>
<td>10⁴ - 10⁶ cm²</td>
<td>C</td>
<td>Yes</td>
<td>Special structures required</td>
</tr>
<tr>
<td>FE-SEM</td>
<td>&lt;10</td>
<td>10⁴ - 10⁶ cm²</td>
<td>C</td>
<td>Limited</td>
<td>Surface sensitive</td>
</tr>
<tr>
<td>E-holography</td>
<td>1</td>
<td>10⁴ - 10⁶ cm²</td>
<td>C</td>
<td>Yes</td>
<td>Special sample preparation</td>
</tr>
</tbody>
</table>
References


Chapter 3

Scanning Capacitance Microscopy

3.1 Background

The first local C-V curve using SCM was performed on Si-based structures in 1989 [1]. Doping dynamic range and lateral resolution were significantly improved in the following years and, by 1997, SCM was shown to provide monotonic $dC/dV$ variations in a doping dynamic range from $10^{15}$ to $10^{20}$ cm$^{-3}$ with a spatial resolution of 25 nm [2-5]. SCM analysis of $pn$ junctions has been discussed in detail [6,7]. The suitability of scanning capacitance spectroscopy (SCS) for high resolution junction delineation was also demonstrated [8]. Full 2D carrier mapping of Si based semiconductor devices was accomplished by several groups [4,6,9-12]. In the meanwhile, the application of the SCM technique has been extended to other materials and devices. Some examples include SiC [13,14], InP-based [15,16], GaAs-based [Papers B and C] advanced optoelectronic devices and AlGaN/GaN based structures [17]. More recently, SCM has been applied to low dimensional structures such as QWs [Paper F]. In addition, methods to determine the spatial resolution of SCM have been proposed and demonstrated [18,Paper I]. Novel applications for SCM such as an in-line evaluation tool for III-V processing have also been demonstrated [Papers D and E]. The technique has been recently applied to characterize ferroelectric materials [19].

3.2 Description

3.2.a Physical principles

In SCM, a metal or a metal coated tip and the semiconductor sample form a metal-insulator-semiconductor (MIS) structure. The insulator part is achieved by covering the sample with a thin insulating layer, usually an oxide. Initially etched tungsten wires were used, later replaced by metal-coated Si AFM probe. The metal coating is typically CoCr or PtIr. TiN and TiW have recently shown interesting performances in terms of conductivity, sharpness and hardness [1,20,21]. An overview of the different tip-probes used is given in Appendix B.

In a 1D MIS structure, the total capacitance $C_{tot}$ of the system is a series combination of the insulator capacitance $C_i (=\varepsilon_{si}/d_i)$, and the semiconductor depletion-layer capacitance $C_{sd}$:

\[
\frac{1}{C_{tot}} = \frac{1}{C_i} + \frac{1}{C_{sd}} \quad \text{or} \quad C_{tot} = \frac{C_i C_{sd}}{C_i + C_{sd}} \quad \text{~F.cm}^{-2} \quad \text{ (Eq. 3.1)}
\]
$C_r$ is a parameter depending only on the thickness $d_e$ of the insulating layer and $\varepsilon_r$ its relative dielectric permittivity. It also corresponds to the maximum capacitance of the system [22]. $C_r$, however, is voltage and doping dependent. For a 1D system and an $n$-type semiconductor in depletion regime the total capacitance can be expressed as,

$$\frac{C_{nr}}{C_r} = \frac{1}{1 + \frac{2\varepsilon_r^2}{qN_d\varepsilon_r d_e^2} V} \quad \text{(Eq. 3.2)}$$

where $\varepsilon_{sr}$ is the dielectric permittivity of the semiconductor, $q$ the elementary charge, $N_d$ the donor concentration and $V$ the voltage, which differs from the applied voltage by an offset equal to $V_i + V_{ab}$. $V_i$ and $V_{ab}$ are the potential drop across the insulator and the built-in potential in the semiconductor, respectively. The results of the simulations showing the variations of the capacitance with the applied voltage for different doping levels are given in Fig. 3.1. Fig. 3.1 (a) shows the case of an ideal 1D $n$-type MIS system. The simulation shows the high frequency C-V curves, relevant for the high frequency used in the capacitance sensor in SCM. As seen in Fig. 3.1 (a) the capacitance variations are more pronounced for lower doping.

![Figure 3.1](image)

**Figure 3.1:** (a) Simulated C-V curves of a 1D MIS structure for different doping levels. $n$-InP is the semiconductor considered in the MIS structure. $1 \times 10^{15}$ cm$^{-3}$ (circle) and $1 \times 10^{16}$ cm$^{-3}$ (square) are the different doping levels. (b) Derivatives $dC/dV$ of the curves given in (a). (c) Simulated C-V curves of 3D MIS structure using the first order annular ring model. The tip radius and the oxide thickness were set to 10 and 3 nm, respectively. (d) Derivatives $dC/dV$ of the curves given in (c).

In SCM, for the typical tip-sample capacitor geometry, the capacitance is of the order of $10^{18}$ F, that is several orders of magnitude lower than the background/stay capacitance. Therefore, measuring the differential capacitance $dC/dV$ or $dC$ using a
lock-in technique is necessary, and requires a highly sensitive capacitance detection system. For a 1D MIS system the expression of the differential capacitance can be expressed using Eq. 3.2:

\[
\frac{d(C_{sd}/C_{s})}{dV} = \frac{1}{V} - \frac{1}{1 + \left(\frac{qN_d \sigma_d d_s^2}{2\varepsilon_0 V}\right)}
\]  

(Eq. 3.3)

Fig. 3.1 (b) shows the variations of the \(dC/dV\) signal for a 1D n-type MIS system for two different doping levels. The SCM \((dC/dV)\) signal decreases monotonically with the doping level. An analytic approximation would even state: \(SCM(dC/dV) \propto N_d^{-a}\), with \(a\) a negative number. However, it must be recognized that in the context of SPM, a fortiori SCM, the situation is inherently three-dimensional (3D). Fig 3.1 (c) and (d) show the variations of the capacitance and the differential capacitance respectively, of a 3D n-type MIS system simulated using the annular ring model first developed for SCM by C. C. Williams [23]. A brief description of the model is given in CH. 8. Despite the similar trend in the C-V and dC/dV-V curves, significant divergences can be observed between the 1D and the 3D models (see Fig. 3.1). Even though the annular ring model remains an approximation of the actual tip-sample capacitor, these observations stress the need for advanced modeling to quantify carrier density. In the 3D case, the C-V curves are stretched out and the peak in the dC/dV-V curves is significantly broadened and reduced in magnitude (Fig. 3.1) [23].

![Diagram](image)

**Figure 3.2:** Schematic of the SCM detection system. In the capacitance detection system, the solid lines correspond to the \(\Delta C\) or \(dC/dV\) mode, and the dashed lines correspond to the \(\Delta V\) or feedback-bias mode.
3.2.b Detection system

The SCM measurements were performed using a Digital Instrument’s (DI) Nanoscope III Dim 3000 microscope. The system was operated in contact mode AFM using PtIr coated Si tips. A schematic of the SCM system is given in Fig. 3.2. The tip-sample distance is maintained using an electronic feedback loop controlling the cantilever deflection. Topography and capacitance data are recorded simultaneously, the latter using a capacitance sensor. Very high sensitivity sensors are required to resolve capacitance variations as low as an attofarad \(10^{-18}\) F. The provided sensor for SCM in the DI’s system is capable of \(10^{-22}\) F/\(\sqrt{\text{Hz}}\) sensitivity [24,25]. The sensor is connected to the grounded tip while the sample is separately controlled using dc and low frequency ac biases.

Capacitance sensors operate at high frequencies for sake of better sensitivity. The basic elements of the RCA (resistance-capacitance-amplifier) sensor used in the system are presented in Fig. 3.3 [23]. An ultra high frequency (UHF) oscillator operating at 915 MHz is coupled to a detection circuitry via a resonant circuit. Variations in capacitance will induce variations in the resonant circuit, thus inducing variations in the amplitude of the resonator. The electric characteristic of the sensor and an appropriate tuning make capacitance variations to vary proportionally with the output signal [23]. Thus if the tip-sample capacitance is varied using an additional low frequency ac-voltage modulation, the amplitude of the resonator (output) will be also modulated at the same modulation frequency. The output signal is then amplified and detected using a lock-in-amplifier. In feedback-bias mode, an additional feedback loop is necessary to adjust the value of the output signal (Fig. 3.2).

![Figure 3.3: Schematic diagram of the RCA capacitance sensor used in the DI’s SCM module.](image-url)
Fig. 3.1 shows the C-V curves simulated for a n-type MIS structure, where the voltage is applied on the metal side. The capacitance is shown to decrease with negative applied bias. Using similar voltage convention, for a p-type structure the capacitance drop will occur with positive voltage. Therefore, for a p-type region the $dC/dV$ signal will be of opposite phase compared to that in the n-type region. However, in the system used here, only the rectified signal $|dC/dV|$ could be recorded. No distinction between p- and n-doped regions could therefore be made at first sight.

Using the dependence of the capacitance on the voltage, the differential capacitance $dC$ is recorded using an ac voltage (modulation) superimposed on the dc bias. The amplitude of the ac modulation is typically between 0.5 V and 2 V. To obtain maximum SCM ($dC/dV$) contrast, the dc bias should be tuned. The optimal experimental conditions correspond to having maximum $dC/dV$ signal with an operating dc bias in the vicinity of the flatband voltage [23]. However, this value is dependent on the investigated semiconductor, the carrier concentration and especially the properties of the insulating layer [26-28]. The full width of half maximum of the $dC/dV$ peak can be used as an indicator of the quality of the surface oxide and reflects the influence of the surface/interface states. This measurement mode in which the differential capacitance is recorded for a fixed ac-voltage modulation is called the $\Delta C$ or $dC/dV$ mode as opposed to the $\Delta V$ or the feedback-bias mode. In the feedback-bias mode, the ac voltage is adjusted by a feedback loop to keep $dC$ constant as the tip moves from one region to another. The $\Delta V$ mode is therefore complementary to the $\Delta C$ mode.

In SCM measurements, $\Delta C$ mode is the most commonly used mainly due to its simplicity since it does not require any additional electronic feedback loops. The other reason is to distinguish between p- and n-regions and to lower the electric field applied on the structure especially at pn junctions. On the other hand, the depletion volume beneath the tip varies with the doping and affects the spatial resolution. In contrast, in the $\Delta V$ mode the depletion volume beneath the tip can be held constant, of the order of the tip radius (~20-30 nm).

The limitation in the sensitivity of the SCM detection system can be discussed in terms of noise. The capacitance sensor noise depends on the capacitance tuning parameters [23,29]. The surface induced noise includes the stationary and the non-stationary surface noises created by the variations of the oxide thickness and of the density of oxide traps, respectively [29].

3.2.4 Sample preparation

Except for specific cases, most SCM measurements are performed on cross-sectioned sample. The sample preparation depends however on the materials to be characterized. In the case of Si based structures, a routine procedure comprises of stacking, sawing, gluing and polishing steps [30]. The quality of the surface oxide can be controlled using different oxidation techniques such as wet oxidation, thermal oxidation or low temperature (300°C) heat treatment under ultraviolet radiation [28,30]. Additional beveling can also be performed [31]. In the case of most III-V materials, cross-sections are prepared by simple manual cleaving in air. Very smooth surfaces are commonly obtained without specific procedures as described in the case of Si. The native oxide that
is formed due to exposure to air usually serves as the thin insulator (typically 2 nm) between the metal-coated probe and the sample. While this approach is quite appropriate for obtaining qualitative information, quantitative analysis remains rather difficult mainly due to the poor quality of the native oxide. A more practical approach has been to use internal doping reference layers.

Samples are fixed on a conductive stand and then mounted on the sample holder. The epoxy used for fixing the sample is conductive (Ag), in order to contact the sample with the potential of the chuck. The epoxy must be carefully deposited to contact all the layers of the structure in order to avoid artifacts induced by local floating potentials.

Freshly cleaved cross-sections properly contacted are the basic requirements to be fulfilled in the SCM measurements.

### 3.3 Contrast

![Contrast Diagram](image)

**Figure 3.4:** (a) Cross-sectional SCM ($dC/dP$) image and corresponding averaged profile of an $n$-InP staircase structure. The doping levels of the layers ranged from $5 \times 10^{17}$ to $4 \times 10^{18}$ cm$^{-3}$ (substrate (dashed line)). The $ac$ and $dc$ bias were 2 V and 0 V, respectively. The doping profile of the sample obtained by ECV is also shown. (b) Experimental SCM calibration curve built from the SCM and ECV profiles given in (a).
The SCM contrasts described in this section refer to SCM measurements in the 
\(dC/dV\) mode. The contrast in the feedback-bias mode is inverted compared to the \(dC/dV\) 
mode. A summary of the different types of contrasts typically observed with SCM is 
given in Appendix A.

3.3.1 Doping contrast

From section 3.2.1, we saw that the SCM \((dC/dV)\) signal is expected to 
monotonically decrease with the doping level. Fig. 3.4 (a) shows the experimental data 
obtained on a 1D InP structure, made of seven 500 nm \(n\)-doped epilayers grown by 
metal-organic vapor-phase epitaxy (MOVPE) on a highly \(n\)-doped substrate. The 
provided doping range was within \(5 \times 10^{12} - 5 \times 10^{18} \text{cm}^{-3}\), covering a larger part of 
the doping dynamic range of the SCM system. Fig. 3.4 (a) shows the SCM image and the 
 correspondingly averaged profile. The measured \(dC/dV\) signal consistently decreases as the 
doping increases.

The independently obtained carrier density profile from electro-chemical voltage 
profiling (ECV) is also shown in Fig. 3.4 (a). ECV is a 1D carrier profiling technique 
based on C-V measurements [32,33]. The technique measures the capacitance of a 
Schottky-like contact, where the metal contact is replaced by an electrolyte (e.g., an 
ammonium tartrate solution is used for InP and GaAs). A potential barrier whose height 
depends on the redox potential is produced within the semiconductor, accompanied by a 
depletion region. The C-V behavior is similar to that of a Schottky contact. A progressive 
etching of the sample together with C-V measurements yields the carrier profile as a 
function of the depth from the sample surface. The ECV profile of the given InP 
staircase structure clearly shows the seven layers. This complementary technique was used to build 
the experimental doping calibration curve for SCM and is shown in Fig. 3.4 (b). 
Although the monotonic decrease of the \(dC/dV\) signal with the carrier density is clearly 
seen, the shape of the curve is significantly non-linear. Such a calibration curve can then 
be used as a reference for quantification of carrier density in other structures.

For both SCM and ECV, and in general all capacitance measurement based 
methods, the spatial resolution is fundamentally limited by the Debye length:

\[
L_D = \left( \frac{\varepsilon_e k T}{q^2 n} \right)^{1/2} \quad (\text{Eq. 3.4})
\]

with \(\varepsilon_e\), the dielectric permittivity of the semiconductor, \(k\) the Boltzmann’s constant ,
\(T\) the absolute temperature and \(n\) the carrier concentration. Thus, in SCM for low doped 
regions the resolution is limited both by the larger depletion volume under the tip and the 
larger Debye length.

3.3.2 pn junctions

Fig. 3.5 shows the energy band diagram, the dopants and the carrier distributions 
of a \(pn\) junction. The simulation was performed using 1D Poisson solver (SimWindows).
The position of the metallurgical and electrical junctions is indicated on the figure. The
different doping levels in the $p$- and $n$-regions imply that the metallurgical and the electrical junction do not coincide, as it is generally the case in devices. SCM is a powerful tool for $p n$ junction delineation [6]. Due to its inherent sensitivity to carriers, SCM provides information on the position of the electrical junction. This ability makes it therefore even more attractive for the mapping of electrical properties of semiconductor devices. The signature of the $p n$ junction in $dC/dV$ mode is that the signal crosses the zero signal line, thus easily observed on recorded images and line scans. Strictly speaking, the position of the SCM zero-signal is the position of the electrical junction [23].

![Diagram](image)

**Figure 3.5:** Simulated energy band diagram, dopant and carrier profiles of an InP $p n$ junction using 1D Poisson solver (SimWindows). The doping in the $p$- and $n$-doped regions was set to $1\times10^{18}$ and $1\times10^{17}$ cm$^{-3}$, respectively. The dashed arrows indicate the position of the electrical and metallurgical junctions.

As previously mentioned, the $dC/dV$ signal exhibits a $180^\circ$ phase difference between $p$- and $n$-doped regions. When scanning across a $p n$ junction, the $dC/dV$ signal changes sign. At the location where the signal crosses the zero line, the condition $dC=0$ can only be satisfied if $\delta C = \delta C_p + \delta C_n = 0$. This implies that $\delta C_n = -\delta C_p$, i.e., $p=n=n_0$, and confirms that the location of the zero signal corresponds to the position of the electrical junction. In the case where only rectified signal ($|dC/dV|$) is recorded, the contrast still exhibits a zero signal indicating the position of the electrical junction while the neighboring $p$- and $n$-doped regions both show positive signals (see for example,
Fig. 3.6). The lateral width of this low signal region corresponds to the depletion width of the \textit{p-n} junction. The analysis of the depletion width can also be an indirect tool to extract carrier concentration if the doping of one side is known. The gradual decrease of the carrier concentrations at the \textit{p-n} junction may (depending on the resolution and the biasing) induce additional peaks in the SCM (\(dC/dV\)) contrast.

\begin{figure}[h]
\centering
\includegraphics[width=0.5\textwidth]{fig36}
\caption{Representative SCM (\(dC/dV\)) profile of an \textit{n-p-n} GaAs structure (see Paper A). The dc and ac biases were 0V and 2V, respectively. The \textit{n}, \textit{p} and \textit{n}+ layers were doped to \(6\times10^{16}\), \(7\times10^{16}\) and \(4\times10^{19}\) cm\(^{-3}\), respectively.}
\end{figure}

For doping levels in the range \(10^{15}\) cm\(^{-3}\) - \(10^{19}\) cm\(^{-3}\), the width of the depletion region can vary significantly and affect the resolution in the location of the electrical junction. In the case of \textit{p-n} junctions involving lowly doped materials, the depletion region is wide. When the tip is in this region, the probing volume underneath the tip increases far beyond the diameter of the tip. The SCM zero-signal does not correspond to a single position but to a wide region including the electrical junction. In the case of \textit{p-n} junctions involving highly doped materials, the depletion width is very small and tip-averaging effect prevents any clear zero-signal from being observed, thus any electrical junction from being clearly located. In the case of \textit{p-n} junction involving intermediate doping levels, the electrical junction should correspond to the minimum intensity in the \(dC/dV\) contrast. This situation is however ideal and does not take into account the influence of the dc bias on the \(dC/dV\) contrast. If the operating dc-bias (for \(dC/dV\) mode) differs for the \textit{n} and the \textit{p}-side of the junction – that is across the junction there is a built-in potential typically of the order of the bandgap – the choice of an appropriate intermediate “dc-voltage” must be set for imaging \textit{p-n} junctions [6,34]. Fig. 3.6 shows the \(dC/dV\) profile across a GaAs \textit{p-n} junction with a 0V dc bias applied to the sample. A spatial motion of the zero-signal region is observed with the operating voltage, from the \textit{p}-side to the \textit{n}-side. This screens the spatial motion of the electrical junction with bias (see Paper A). To locate more accurately the position of the electrical junction, scanning capacitance spectroscopy may be used [8,35]. Although this method is promising, it is complicated in terms of measurement procedure and is not extensively used yet.
3.3.c Low dimensional systems

Fig. 3.7 shows the simulated energy band diagram, the dopant and the carrier distributions of an \( n \)-doped InGaAs/InP heterojunction. The simulation was performed using a 1D Poisson/Schrödinger solver (SimWindows). The band-offset difference between InGaAs and InP induces space charge regions at the interface, electron accumulation in InGaAs and electron depletion in InP. The relatively large extent of the depletion regions makes them most likely to be observed in the SCM contrast. The typical \( dC/dV \) contrast is therefore a characteristic peak in the \( dC/dV \) signal (lower carrier concentration). Fig. 3.8 shows the typical \( dC/dV \) contrast observed across an InGaAs/InP heterojunction. The variation of the carrier concentration is rather abrupt at the interface and the electrons are confined in a potential well. The \( dC/dV \) contrast at the interface varies steeply.

![Simulated energy band diagram and concentration profile](image.png)

**Figure 3.7:** Simulated energy band diagram (conduction band edge) and electron concentration profile of a lattice matched \( \text{In}_{0.53}\text{Ga}_{0.47}\text{As}/\text{InP} \) heterojunction and of a 10 nm quantum well using 1D Poisson/Schrödinger solver (SimWindows). The doping in the \( n-\text{InP} \) and \( n-\text{InGaAs} \) bulk layers was \( 1 \times 10^{16} \) and \( 1 \times 10^{17} \text{ cm}^{-2} \), respectively. The InGaAs QW was undoped and 240 meV was chosen as the conduction band offset between InP and InGaAs [36].
The understanding of the SCM contrast observed in quantum wells (QWs) follows the same kind of analysis as the one described for a single heterojunction. Fig. 3.8 also shows the typical \( dC/dV \) contrast observed across a 10 nm InGaAs/InP QW. The dip corresponds to the carriers accumulated in the well (see Paper F). Due to the sensitivity of SCM to carriers, the width of the dips will however be limited by the spatial extent of the ground state wave-function of the QW [37,38]. However, the discrepancies resulting from this specificity are hardly distinguishable due to other averaging effects in SCM such as the tip size and the fringe fields [Paper F]. A complete analysis of these effects will be presented in CH. 8.

Figure 3.8: SCM \( (dC/dV) \) profile of an \( \text{In}_{0.53}\text{Ga}_{0.47}\text{As}/\text{InP} \) heterojunction and a 10 nm quantum well. The \( n\)-InGaAs and \( n\)-InP bulk regions were doped \( 1\times10^{16} \) and \( 1\times10^{17} \) cm\(^{-2} \), respectively. The InGaAs QW is undoped. The dc and ac biases were 0 V and 2 V, respectively.

3.3.d Highly resistive materials

Despite the doping dynamic range being restricted to about \( 10^{15} \)–\( 10^{10} \) cm\(^{-3} \), SCM can also be used to qualitatively characterize materials with very low carrier concentration (intrinsic levels). Such materials can be fabricated in several ways, for instance by high-energy ion implantation or by incorporating certain deep level impurities during growth [16, Papers C and E]. The purpose is to have a sufficiently high density of deep levels in the bandgap to trap the free carriers. This turns the semiconductor into a semi-insulating (SI) material [39]. In such materials, the volume probed underneath the tip becomes extremely large (typically on the order of the millimeter). Under the depletion approximation, Eq. 3.1 becomes

\[
C = C_0 = \frac{\varepsilon_0 \varepsilon_w}{w} \quad \text{(Eq. 3.5a)}
\]

with \[
\frac{dw}{dV} = \frac{w}{2V} \quad \text{(Eq. 3.5b)}
\]
\[
\frac{dC}{dV} \propto \frac{1}{w}
\]  
(Eq. 3.5c)

with \( w \) the depletion width beneath the tip. Upon any given ac modulation, the capacitance variations remain negligible and the \( dC/dV \) signal therefore exhibits uniform zero signal. This behavior has been observed in Paper B. To differentiate SI from semiconducting regions with carrier density in excess of \( 10^{17}\) cm\(^{-3}\), the variation of the \( dC/dV \) signal with the amplitude of the ac modulation voltage (\( \delta V \)) can be used. The \( dC/dV \) signal increases with \( \delta V \). For doped regions, a few volt increase in the ac amplitude is enough to observe a significant increase in the \( dC/dV \) signal. In the case of SI materials, the \( dC/dV \) signal remains zero irrespective of the applied bias. This method is used to verify SI properties in any desired location of the SI-layer (see \textbf{CHL 6} and Paper C). SCM instrumentation may be implemented for the characterization of deep levels present in low densities, example of which would include having the capability to perform temperature dependent SCM measurements (including analyzing capacitance transients).
References


27
[30] DI’s Support Note No. 224, Rev. C.
Chapter 4

Scanning Spreading Resistance Microscopy

4.1 Background

The early 1D SSRM profiles demonstrate a doping dynamic range between $10^{14}$ cm$^{-3}$ and $10^{16}$ cm$^{-3}$ and a resolution around 50 nm [1]. The resolution, at that time, was limited by the high tip-sample contact forces, resulting in large contact radii. The characterization of $p-n$ junctions and of the diffusion of implanted species were then successfully accomplished together with the 2D carrier mapping of Si MOSFET [2]. The ability of SSRM to characterize SiC with a similar doping dynamic range as for Si has been demonstrated [3]. 2D characterization of III-V devices with SSRM has also been reported [4]. Using the inherent penetration of the probe into the samples, a method for 3D carrier profiling has also been proposed [5]. In the scope of this thesis, the characterization of ion-implanted InP with cross-sectional SSRM has been performed [Paper E]. Carrier profiling in low dimensional structures such as InGaAs/InP QWs was also recently reported [Paper G]. Together with recent improvements of probes [6,7], sub-5 nm spatial resolution in SSRM was demonstrated [6, Paper I]. Furthermore, scanning spreading resistance spectroscopy (SSRS) providing local I-V spectra was also developed and the application of SSRS to enhance 2D carrier calibration procedure was proposed [8]. Local I-V measurements have also been used to investigate the properties of the tip-sample contact in the case of some III-V semiconductors [9]. Simulation and modeling on tip-sample contacts for quantitative carrier profiling have also been addressed [10].

4.2 Description

4.2.a Physical principles

In SSRM, the spreading resistance is measured by recording a current flowing into the sample at a given dc voltage. The current is concentrated just below the scanning tip and spreads out radially from this region. Hence the name scanning spreading resistance microscopy. This technique uses sufficiently high contact forces, typically 1 - 50 µN, to penetrate the surface oxide layer and to form an electrical contact with the semiconductor underneath. The force is controlled by setting the deflection of the cantilever and hard probes are required to resist tip-wear induced by surface bulldozing [11]. This effect is shown in Fig. 4.1. A scan under typical SSRM conditions was preliminary performed on a $2\times2 \mu m^2$ window, then expanded to a $4\times4 \mu m^2$ window and
the topography was recorded using contact mode AFM. Significant topography variations can be observed ($\Delta z=1.5$ nm).

The electrical contact implies the need of conductive probes. The metal-coated and diamond coated Si AFM probes have been typically employed. Metals used for coating are for example TiN or TiW while diamond coating is usually heavily $p$-doped. The choice is so as to have the electrical contact between the probe and the sample as ohmic as possible. Significant attention is today devoted to the fabrication of probes and the optimization of their capabilities. Diamond coated probes and full diamond probes are among the latest developments in this domain [6] (See Appendix B).

![Figure 4.1: Topography after one SSRM measurement across a 2×2 μm² region of a n-InP sample showing the formation of a 1.5 nm deep crater. The deflection set point was 0.7 V.](image)

The total resistance of the tip-sample system can be seen as a series combination of the contact resistance $R_C$ and the spreading resistance $R_s$:

$$R = R_C + R_s$$  \hspace{1cm} (Eq. 4.1)

To be more rigorous, Eq. 4.1 should have also included other resistances such as the resistance of the sample and the back contact resistance. However, these resistances are usually very low compared to $R_C$ and $R_s$ and are neglected.

$R_C$ first depends on the nature of the point contact. As previously noted the small area and the 3D properties of the contact complicate the modeling. However, if the contact is not purely ohmic, $R_C$ is usually modeled by taking into account the same conduction mechanisms as the ones involved in ideal Schottky contacts. The intrinsic parameters of the contact then depend on the type of semiconductor materials and/or the probes. The sample surface and the tip geometry will also significantly affect critically the contact parameters, for instance the barrier height [9,12]. The structural modifications
of the sample at the point contact induced by the high contact force may also have to be taken into account. \( R_C \) is both voltage and doping dependent and should decrease with the applied voltage or the doping level. The quantitative modeling of the contact is a crucial issue and is still today under investigation.

In an ideal situation the tip-sample contact is ohmic and the spreading resistance dominates the other resistances. The spreading resistance \( R_s \) is governed by the ratio of the bulk resistivity \( \rho \) and the contact area \( S \) [13]. For a nonindenting cylindrical contact with a planar circular interface and a highly conductive probe, the spreading resistance of a semi-infinite sample is [13]:

\[
R_s = \frac{\rho}{4a} \quad \text{and} \quad S = 4a \quad \text{(Eq. 4.2a)}
\]

with \( a \) the radius of the tip. For a hemispherical indenting contact of radius \( a \), the spreading resistance becomes [13]:

\[
R_s = \frac{\rho}{2\pi \cdot a} \quad \text{(Eq. 4.2b)}
\]

Today’s literature on SSRM most often refers only to Eq. 4.2a for conversion of resistance data into doping. The resistivity is given by:

\[
\frac{1}{\rho} = \sigma = gn\mu_n \quad \text{(n-type)} \quad \text{(Eq. 4.3)}
\]

\[
\frac{1}{\rho} = \sigma = gp\mu_p \quad \text{(p-type)} \quad \text{(Eq. 4.3)}
\]

where \( \sigma \) is the conductivity; \( \mu_n \) and \( \mu_p \) are the electron and hole mobilities, respectively; \( n \) and \( p \) are the electron and hole concentrations. \( R_s \) is consequently expected to be inversely proportional to the carrier concentration. Taking \( n\text{-InP} \) as an example, for a doping range of \( 10^{12} \text{ to } 10^{19} \text{ cm}^{-3} \), \( R_s \) varies from \( 10^8 \) to \( 5 \times 10^5 \Omega \).

**Figure 4.2:** I-V curves obtained with SSRM on a \( 4 \times 10^{14} \text{ cm}^{-3} \) \( n^-\text{InP} \) sample using different probes. The dc voltage was applied to the sample and ranged between -1.5 (forward) and +3 V (reverse).
The development of SSRM probes was driven by the need of having an ohmic contact between the tip and Si, and ohmic behavior was experimentally observed [7,8,14]. This was explained by the local formation of a β-tin phase of Si under the tip [15]. Departures from this behavior were shown to be due to other factors, for example surface states [16]. With similar probes on III-V materials, the contact resistance contributes significantly to the overall resistance measured [10,12,Paper G]. The total resistance should then vary from a Schottky-like behavior at low dc bias to an ohmic like behavior at high dc bias (forward). Fig. 4.2 shows typical I-V curves obtained for different probes on $n^+$ InP.

Irrespective of the roles of $R_0$ and $R_c$, the measured resistance is evidently expected to decrease with the carrier density. However, because of the significant influence of the contact resistance on III-V materials, the measured resistance will exhibit a non-linear dependence on the doping.

Quantitative modeling of the tip-sample system requires experimental results with high reproducibility in terms of the measured current at a given biasing condition. This requirement is not easy to meet with a system operating in air and with the existing knowledge about the properties of the tip-sample contact. As in SCM, the quantitative mapping of electrical properties remains a question of comparison between standard reference samples and structures. In the case of ohmic contacts, the resistance profile can be simply obtained from the measured current using Ohm’s law, $V = R \cdot I$. However, this is not the case for Schottky contacts. The resistance is then voltage dependent and accurate resistance profile can only be obtained using the slope of the I-V curve at a given voltage value: $R = \frac{\partial V}{\partial I}$. This procedure implies that the I-V curve should also be recorded at every probing point of the sample. This is tedious, time consuming, and relatively unnecessary since no resistance value can directly be used without an established model for $R_c$. It is moreover in contradiction with SSRM functionality as a rapid characterization tool. Therefore, in this thesis, the validity of the Ohm’s law is taken as a fair approximation to construct the resistance profile. Qualitatively, the trend should not be modified using this approximation; quantitatively, discrepancies will occur and will be specifically discussed when necessary.

**Figure 4.3:** Schematic of the SSRM detection system.
4.2.2 Detection system

The SSRM measurements were performed using a Digital Instrument’s (DI) Nanoscope III Dim 3100 microscope and was operated in contact mode. The probes used were (primarily) B-doped diamond-coated Si tips [17]. The contact forces in SSRM are significantly higher than in SCM and require stiffer cantilevers. A schematic of the SSRM system is given in Fig. 4.3. Topography and spreading current are recorded simultaneously. The current is measured using a sensitive logarithmic current amplifier. The output signal providing the resistance data is given in Volt, whereby a change of 1 V corresponds to a change in the resistance of one order of magnitude. The relations between the output voltage and the input current are:

\[
V_{\text{out}} = \pm \log(I * 10^n / I_{\text{bias}}) \quad \text{if } V_{\text{bias}} > 0 \quad \text{(Eq. 4.4a)}
\]

\[
V_{\text{out}} = -\log(I * 10^n / I_{\text{bias}}) \quad \text{if } V_{\text{bias}} < 0 \quad \text{(Eq. 4.4b)}
\]

These relations are clarified on Fig. 4.4. Typically, a 0 V output signal corresponds to a resistance of 1 MΩ.

The dc bias voltage is applied to the sample while the conductive tip is grounded. For positive dc biases electrons flow from the tip to the sample and for negative dc biases electrons flow from the sample to the tip. Due to the Schottky-like contact between diamond-coated tip and III-V materials (e.g., InP, GaAs), asymmetric behavior for positive and negative voltages will be observed at any point in any structure or device (see Fig. 4.2). For n-doped semiconductors, we will then refer to the positive (sample) bias as the reverse bias, and the negative (sample) bias as the forward bias. For p-doped semiconductors, this will be the contrary.

![Figure 4.4: Transfer curve of the SSRM Logarithmic Current Amplifier](image)

Unlike in SCM, in SSRM a single line scan is often not directly used due to the relatively low signal to noise ratio and requires averaging over a large number of line scans. First, noise obviously occurs at the limit of sensitivity of the current detection system (typically on the order of the nA). This noise is however negligible compared for
example to the scanning (scratching) induced noise. With the motion of the probe across the sample surface, every measurement point is obtained by creating a new tip-sample contact. The so-formed contact introduces uncertainties that are difficult to control. As previously mentioned, the type of materials, the range of forces and the tip geometry affect the properties of the contact. In addition, the scratching of the surface with the material either removed or lying on the surface or sticking to the tip, and the possible progressive degradation of the tip could also contribute to such noise. Other possible reasons are the poor back contacts, a rough surface or the local distribution of oxide traps/surface states [16]. Some of the above effects can also induce artifacts. Thus an awareness of the possible reasons that affect the tip-sample contact is essential for correct interpretation of the SSRM data.

4.2.c Sample preparation

The sample preparation in SSRM is similar to that used in SCM (see section 3.2.e). The requirements in terms of back contacts and surface roughness are similar to that in SCM, except that the quality of the surface oxide is relatively of lesser importance. To enhance lateral resolution, low-angle surface beveling is also used [14,15] as in conventional SRP [18,19]. In the case of III-V materials, manual cleaving in air provides atomically flat cross-sections and is usually done just before the measurement. The surface native oxide is very thin (<2 nm), thus the forces required for penetration can be very low. The effective contact area can also be very small improving the lateral resolution.

Compared to SCM, it is more critical in SSRM that all the layers in the structure are properly contacted. The back contact must also be formed upon a large area in order to reduce the back contact resistance. These considerations have been taken into account in every SSRM measurement presented in this thesis work.

4.3 Contrast

In this section the different contrasts typically observed with SSRM are presented. A summary is given in Appendix A.

4.3.a Doping contrast

As shown in Eq. 4.2a and 4.3, the spreading resistance is expected to decrease with the doping level. Fig. 4.5 (a) shows the SSRM measurements obtained on the n-InP doping staircase structure (see section 3.3.a). Fig. 4.5 (a) shows a representative SSRM image (current) and the corresponding SSRM resistance profile obtained from an averaging procedure performed on the scan lines of the image. A monotonic dependence of the SSRM signal (resistance profile) with doping is clearly observed in the figure: the SSRM resistance increases as the doping decreases.
Figure 4.5: (a) Cross-sectional SSRM image (current) and averaged resistance profile of an $n$-InP doping staircase structure. The doping levels of the layers range between $5 \cdot 10^{15}$ and $5 \cdot 10^{18}$ cm$^{-3}$ (substrate). The dc bias was set to 1 V. The doping profile obtained by ECV on the structure is also shown. (b) Solid line: experimental SSRM calibration curve built from the SSRM and ECV profiles given in (a).

The experimental data obtained by ECV, previously presented in Fig. 3.4 (a) are also included in Fig. 4.5 (a). A comparison of the dependence of the SSRM resistance with the doping ranges confirms the divergence of the SSRM signal from the ideal model presented before (see Eqs. 4.2 and 4.3). While the ECV profile shows the variation of carrier density over three orders of magnitude, the SSRM resistance only varies over two orders of magnitude. These results indicate that both the contact and the spreading resistance have to be considered.

The ECV data are also used to build the experimental calibration curve as shown in Fig. 4.5 (b). This calibration curve shows the variations of the SSRM resistance with the carrier density. The monotonic decrease of the resistance with doping is clearly visible. However, it differs significantly from the $-1$ slope linear dependence that could have been expected from the ideal model. The deviations can be explained, in this case, in terms of the different current transport mechanisms in a Schottky-like structure – the decrease of the contact resistance with the doping level is expected be non-linear and more pronounced for higher doping levels. This can explain the shape of the calibration curve for low doping levels.

Due to the sensitivity of SSRM to carriers, the resistance profile at the doping steps in the structure appears smooth. This effect would be more pronounced for lowly doped layers due to the larger Debye length (see Eq. 3.4 in section 3.3.1). As seen from
Eq. 4.2, the lateral extent from which the current is spreading in the sample only depends on the dimension and the geometry of the probe and is therefore insensitive to the doping level and the voltage applied. In this aspect, SSRM differs from SCM (dC/dV mode). The lateral resolution of SSRM thus remains mainly a matter of probes. This issue is discussed later in CH. 8.

4.3.b pn junctions

SSRM has proven to be a suitable technique for investigating pn junctions [16]. Like SCM, SSRM is a carrier sensitive characterization technique and will therefore provide information about the electrical junction rather than the metallurgical junction.

From Fig. 4.5, in the ideal situation (i.e., an ohmic contact) the SSRM signature of a pn junction is expected to be a peak, with the location of the maximum corresponding to the electrical junction (n=p=n), and with a lateral extent corresponding to the depletion region. As mentioned before, in SSRM on III-V materials, the contact resistance cannot be neglected in the contrast analysis. The I-V curves shown in Fig. 4.2 confirm the asymmetrical behavior between forward and reverse biases, with large resistance variations. For a given dc bias, p- and n-doped regions will therefore exhibit significantly different resistance value and can be easily distinguished.

Fig. 4.6 shows the averaged SSRM resistance profiles, obtained for 1 V forward and reverse bias on the n+p+n GaAs structure presented in section 3.3.b (Paper A). The SSRM resistance is shown to decrease between two to three orders of magnitude from reverse to forward bias. As a simple approach, the crossing point between the two curves can be attributed to the approximate location of the electrical junction and the spatial extent of the resistance step to the width of the depletion region. The n+p+n GaAs structure has two pn junctions. The spatial extent of the resistance steps in Fig. 4.6 is larger at the np junction than at the n+p junction, consistent with the expected depletion region widths.

At the np junction, the resistance variations for opposite dc-bias exhibit symmetrical behavior implying the two curves to cross one another in the middle of the resistance step. This is in good agreement with a symmetrically doped junction predicting the electrical junction to be located in the middle of the depletion region. Similarly, the depletion region of the n+p junction is seen to be shifted into the p-doped region while the crossing-point is shifted towards the n+ doped region. These observations are qualitatively consistent with the results presented in Fig. 3.5 (section 3.3.b). This simple approach however does not take into account the asymmetry of the I-V curves between two equally doped n- and p-doped regions mainly due to non-identical tip-sample contacts. Moreover this does not consider the difference between electron and hole mobilities, which also affects the actual resistance. The good consistency observed between experimental results (Fig. 4.6) and the 1D Poisson simulation (Fig. 3.5), makes therefore this simple approach attractive for pn junction delineation even if some refinements remain to be done.

The pn junction can also be characterized using I-V curves recorded at different locations across the junction (see Paper A). In the vicinity of the electrical junction, the I-V curve exhibits high resistance behavior for both positive and negative biases.
Information about a possible lateral movement of the junction upon dc bias may also be obtained. However, this procedure needs to be investigated further. Better control of the tip-sample contact and the understanding of the structural modifications underneath the probe may in addition be required.

![Graph showing SSRM profiles](image)

**Figure 4.6:** Representative averaged SSRM profiles of an $n$-$p$-$n'$ GaAs structure. Dc biases of the two profiles were -1 V (black) and +1 V (gray). The location of the depletions regions and the electrical junctions are indicated. The dc bias was applied to the sample.

### 4.3.c Low dimensional systems

Fig. 4.7 shows the SSRM resistance profile obtained for both forward and reverse bias on a structure including an $n$-doped InGaAs/InP heterojunction and a 10 nm QW. The 1D Poisson/Schrödinger simulation of this structure including band diagram variations so as the dopant and the carrier distributions have been presented in Fig. 3.7 (section 3.3.c).

At the heterojunction, both reverse and forward bias resistance profiles show significant differences between InP and InGaAs regions. Irrespective of the polarity of the applied bias, the heterojunction is characterized by an abrupt decrease of the resistance from InP to InGaAs. This can be first attributed to the different contact resistance between InGaAs and InP, especially to the difference between the barrier heights at the contacts due to the lower InGaAs bandgap.
The drop in the resistance is even more pronounced when carriers are accumulated in InGaAs, like in QWs. The signature of the QW in SSRM is therefore a narrow dip. Like for SCM, the space charge regions in the vicinity of the heterojunction or the well can also be resolved under specific biasing conditions. In the depletion regions, the SSRM resistance is expected to increase. Conversely, in the case of carrier accumulation, the resistance is expected to decrease. These two behaviors are usually observed for low forward bias as shown in Fig. 4.7 (black line). The width of the resistance peaks observed in InP, neighboring the well or the heterojunction, provides additional information about the depletion widths. For reverse bias, a decrease in the resistance is instead observed and is attributed to tunneling electrons flowing from the tip into the well (see Paper G). This behavior can also be observed at heterojunctions. Due to this tunneling mechanism, the resistance dip at the QW appears broader, and the contrast at the depletion regions is inverted.

As previously discussed in the case of SCM (cf. 3.3.c), due to discontinuous variations of carrier concentrations across a hetero-interface, the shape of the resistance profile may not be affected by the Debye length. Much more abrupt resistance variations are therefore observed at heterojunctions compared to homojunctions (see for example Fig. 4.5 (a)). The width of the resistance dips observed at QWs is primarily limited by the geometrical tip-averaging effects and fundamentally by the spatial extent of the electron wave function. A complete analysis of the SSRM contrast in QWs and the related issues will be addressed in CH. 8.
4.3.d Highly resistive materials

Like SCM, SSRM can also be used to characterize structures with carrier density out of its doping dynamic range \(10^{12} - 10^{15} \text{ cm}^{-3}\). Fig. 4.8 shows the SSRM image obtained on cross-sectioned \(n\)-InP implanted by Ga\(^+\). Using reverse bias, a dip in the contrast is locally observed in the InP epilayer consistent with the locally high resistance induced by the high concentration of deep levels created at the expected implantation depth. SSRM characterization of the structure can therefore be used for implantation depth control and lateral extent delineation. A better understanding and description of the involved physical mechanisms is possible using SSRM, but requires further measurements such as an evaluation of the damage removal process after annealing and the contrast comparison using different implanted species. Results are presented in Paper E, and will be discussed briefly in CH. 7.

Figure 4.8: Cross-sectional SSRM image (current) of a Ga\(^+\) implanted InP. The dark line in the epilayer roughly 1 \(\mu\)m from the cleaved edge corresponds to the maximum implantation depth.
References

Chapter 5

Kelvin Probe Force Microscopy

5.1 Background

The application of KPFM for device characterization was first demonstrated on Si-based structures [1,2]. These investigations included Si staircase structures and pn junctions, and projected a doping dynamic range between $10^{15}$ cm$^{-3}$ and $10^{19}$ cm$^{-3}$, i.e., similar to SCM and SSRM. The preliminary sensitivity of 15 meV for the surface potential measurements was then improved to 5 meV [3]. Recently developed microscopic surface photovoltage spectroscopy showed promising information in terms of short minority carrier diffusion length [4,5]. KPFM has been also applied on III-V and other materials. Some examples include chalcopyrite based micro-scale solar cells [6], GaSb laser diodes [9], GaAs/AlGaAs [10] and GaAs/AlAs multi quantum wells (MQWs) [11]. InAlAs/InGaAs [11] and InP/InGaAsP laser diodes [12]. Results on GaAs pn junctions were also reported, while better contrast was then obtained operating in UHV [7,8]. The ability of KPFM to detect isolated features as narrow as 5 nm was demonstrated [Paper H]. However, large tip averaging effect due to long distance probe sample electrostatic interaction is shown to significantly hamper high spatial resolution measurements. Modeling and simulation of KPFM measurements have been addressed [8].

5.2 Description

5.2.a Physical principles

KPFM operates in non-contact AFM mode and images the contact potential differences ($\Delta \Phi$) between the sample ($\Phi_{\text{sample}}$) and the conductive tip ($\Phi_{\text{tip}}$) upon a given applied voltage. It is important to stress that contrary to SCM and SSRM, KPFM only senses surface properties. The probes are the same as in SCM, typically a PtIr, or AuCr coated etched Si tip, with an effective tip radius of about 25 nm [13,14]. The choice of the metal coating is usually motivated by its work function in order to avoid large voltages to be applied on the tip. Further information concerning the probe related issues in KPFM, is presented in Appendix B.

Fig. 5.1 shows schematically the physical principles involved in KPFM. When the conductive tip is brought in proximity of the sample surface, the two systems (probe-tip and sample) equalize their Fermi levels and, due to their different work functions, the two surfaces become equally but oppositely charged. Biasing one system with respect to the other can be used to find the work function or contact potential difference (CPD) between
the two surfaces. This is directly obtained by measuring the dc voltage $V_B$, required to reach the flat band condition, i.e., the condition wherein the electric field between the two systems vanishes.

$$V_B = \text{CPD} = \frac{\Delta \Phi}{q} = \frac{\Phi_{\text{Sample}} - \Phi_{\text{Tip}}}{q}$$  \hspace{1cm} \text{(Eq. 5.1a)}$$

For the semiconductor samples the work function depends on the doping level as follows:

$$\Phi_{\text{Sample}} = \chi_{\text{Sample}} + \frac{kT}{e} \ln \left( \frac{N_C}{n} \right) \quad \text{\textit{(n-doped)}} \hspace{1cm} \text{(Eq. 5.1b)}$$

$$\Phi_{\text{Sample}} = \chi_{\text{Sample}} + E_s + \frac{kT}{e} \ln \left( \frac{p}{N_v} \right) \quad \text{\textit{(p-doped)}} \hspace{1cm} \text{(Eq. 5.1c)}$$

where $E_s$ is the bangap, $\chi_{\text{Sample}}$ the electron affinity; $e$ the magnitude of the elementary charge (no unit), and $N_C$ and $N_v$ the effective density of states in the conduction and valence band of the semiconductor, respectively; and $n$ and $p$ the electron and hole concentration, respectively. The work function of the selected tip is first measured using a calibration sample having a known work function, e.g., highly oriented polygraphite (HOPG) ($\Phi_{\text{HOPG}} = 4.5 \text{ eV}$). By measuring the local CPD, information on the work function of that region of the sample underneath the tip can be obtained, and thereby the doping level can be determined.

![Diagram](image)

**Figure 5.1:** Physical principles involved in KPFM measurements. 1- No contact, open circuit. 2- Fermi levels aligned, surfaces oppositely charged. 3- Backing voltage $V_B$, the electrical field vanishes, $|V_B| = |\Delta \Phi|/q$.

The nullifying procedure is characteristic of KPFM, and can be controlled either mechanically (force control) or electrically (field control). The latter is preferably used for small tip-sample distances to minimize the risks of tip crushing onto the surface. The tip-sample system can be seen as a simple capacitor. Non-contact mode is a useful scanning procedure that induces dynamical variation of the capacitance, i.e., by modulating the tip-sample separation distance. When a voltage is applied between a probe and a sample put in proximity, the force $F$ due to the electrical field $E$ can be written as:

$$F = q \cdot E = \frac{1}{2} \frac{dC}{dV} V^2$$  \hspace{1cm} \text{(Eq. 5.2a)}$$
where $C$ is the tip-sample capacitance, $z$ the direction normal to the sample surface and $V$ the applied voltage. $\nu$ is a sum of the CPD, the dc ($V_{dc}$) and ac ($V_{ac}$) voltages. Eq. 5.2a becomes:

$$F = -\frac{1}{2} \frac{\partial C}{\partial z} \left[ \text{CPD} + V_{dc} + V_{ac} \sin(\Omega t) \right]^2$$  \hspace{1cm} (Eq. 5.2b)

with $\Omega$ the frequency of the ac voltage modulation. Referring to this frequency, the electrostatic force can be decomposed into three terms:

$$F = F_{dc} + F_{\Omega} + F_{3\Omega}$$  \hspace{1cm} (Eq. 5.3)

$$F_{dc} = -\frac{\partial C}{\partial z} \left( \frac{1}{2} \left( V_{dc} - \frac{\Delta \Phi}{q} \right)^2 + \frac{V_{ac}^2}{4} \right)$$  \hspace{1cm} (Eq. 5.3a)

$$F_{\Omega} = -\frac{\partial C}{\partial z} \left( V_{dc} - \frac{\Delta \Phi}{q} \right) V_{ac} \sin(\Omega t)$$  \hspace{1cm} (Eq. 5.3b)

$$F_{3\Omega} = -\frac{\partial C}{\partial z} \frac{V_{ac}^2}{4} \cos(2\Omega t)$$  \hspace{1cm} (Eq. 5.3c)

where $\Delta \Phi/q$ is the CPD between the sample and the tip (see Eq. 5.1a), $F_{dc}$ corresponds to a static deflection of the cantilever and is rather difficult to detect. $F_{\Omega}$ is directly proportional to $|V_{dc} \Delta \Phi/q|$. Hence for an appropriate dc bias, $F_{\Omega}$ can be nullified. This gives the ability to determine the CPD. Since both $F_{2\Omega}$ and $F_{3\Omega}$ are positive and depend on $V_{ac}$, low ac amplitude is usually required to minimize the electrostatic contribution to the topographic signal [15].

The topography is simultaneously recorded using non-contact mode AFM principles using the first resonant frequency $\omega_1$ of the cantilever ($f_1=\omega_1/2\pi$ is typically in the range 50-70 kHz). The CPD variation is measured independently from the topography using the second resonant frequency $\omega_2$ of the cantilever. The frequency of the ac voltage is set to $\Omega=\omega_2$. This is motivated by a better sensitivity in the detection of the zero $F_{\Omega}$ while operating at a resonance frequency. The sensitivity of the measurement can be improved by reducing the tip-sample distance. Of late, KPFM measurements are therefore most often performed in UHV where tip-sample distances as low as 2-3 nm can be commonly achieved.

From Eq. 5.1 the measured CPD is expected to decrease as $\ln(n)$ where $n$ is the carrier density. Although a monotonic trend of the signal with the doping level is usually observed, discrepancies can occur mainly due to the 3D geometry of the probe-sample system and to the electrical properties of the characterized surfaces. Modeling and simulation of these two complex issues are required to perform reliable quantitative carrier profiling. The impact of the electrical interaction between the probe and the sample and the influence of the surface states density on the KPFM signal will be discussed further in this thesis.

### 5.2.2 Detection system

The KPFM measurements were performed using a modified UHV-AFM (Omicron Inc.) set-up operating at $p \leq 10^{-10}$ mbar. PtIr metal-coated etched Si tips were used. A schematic of the KPFM system is given in Fig. 5.2.
Two modes can be distinguished to detect tip-sample electrostatic forces. The frequency modulation (FM) mode [16] measures the frequency shift at an appropriate frequency, and is proportional to the gradient of the electrostatic force. The amplitude modulation (AM) mode [16] measures the amplitude of the cantilever oscillation at a proper frequency and is proportional to the electrostatic force itself. In FM-mode mainly the apex of the tip contributes to the measured signal due to the detection of the force gradient. However, the poor sensitivity of this mode imposes the use of large ac-voltage amplitudes. Conversely, a much stronger contribution of the tip shape was determined for AM detection, affecting the contrast of the measured contact potential difference, while very low ac-voltage amplitude was sufficient to yield good sensitivity. The low contribution of the tip implies better lateral resolution compared to AM-mode. FM-mode was therefore applied to measure the topography.

Figure 5.2: Schematic of the KPFM/EFM detection system. In the potential detection system, the solid lines correspond to the KPFM measurements (Kelvin controller on) and the dashed lines correspond to the EFM measurements (Kelvin controller off) [15].

From Eq. 5.3, it can be seen that a large ac-amplitude increases the contribution of $F_{20}$ and $F_{40}$ in the electrostatic force, which may first contribute to the topography signal but also generates tip induced band bending at the surface. For these reasons, AM detection mode was used to record CPD data. For topography imaging, the frequency shift of the first resonance frequency ($f_1 = \omega_1/2\pi \approx 65$ kHz) was set in a range between 10 and 40 Hz at constant oscillation amplitude and corresponds to a tip-sample separation between 1 and 3 nm. The CPD was measured using a lock-in technique and the ac modulation frequency was tuned to the second cantilever resonance ($f_2 = \omega_2/2\pi \approx 350$ kHz). This requires ac amplitudes of typically 100-200 mV.

The detection at the second frequency was performed by isolating electronically the signal from the rest of the circuit by means of a high-pass filter. This procedure was performed before the tip approach during which the ac modulation is turned off to avoid any electrostatic contribution. The force signal was then compensated using an integrated
Kelvin controller applying a dc voltage to the sample, thus giving the CPD. Photovoltage could also be measured by illuminating the sample for different light powers [4,5]. Such measurements were not performed in this thesis. All KPFM measurements were performed under “dark” conditions.

Recent experimental results have shown that CPD variations as small as 5 meV can be detected [13]. This obtainable sensitivity depends on the amplitude background noise observed in the line scan recorded at the lowest scan rate. This noise is lowered using phase adjustments during the calibration procedure described above. Low cantilever damping can also contribute to the noise but is easily minimized using a high frequency shift.

KPFM measurements can be influenced by several physical factors or experimental conditions. KPFM is first very sensitive to the tip-sample distance. Rough topography, resulting for instance from bad quality cleaving, induces significant parasitic variations in the output signal irrespective of the optimization of the feedback loop gains. The tip averaging effect caused either by the geometry of the tip or the long-range electrostatic interaction between the probe and the sample also significantly affects the KPFM contrast. The former is more pronounced for large tip-sample distance. The latter could involve the whole cantilever and increases dramatically close to cleaved edges. Finally, due to its inherent sensitivity to surface properties, KPFM is extremely sensitive to surface states. The main evidence of the influence of surface states is generally a reversal of doping contrast and a CPD range narrower than expected. This issue will be discussed further in this chapter.

5.2.c Sample preparation

In addition to the ability to scan closer to the sample surface, one of the main advantages of the UHV conditions is the possibility to investigate uncontaminated surfaces. In the case of cross-sectional investigations, this requires cleaving the sample in situ. However, this procedure is seldom employed due to the complex manipulation of samples in UHV and the low yield [13,14]. Thus, conventional air-cleaving procedure prior to introduction into UHV can also be motivated from the point of improved spatial resolution. The sample preparation methods presented for SCM can be used. With manually cleaved InP- and GaAs-based structures it was possible to bring the tip very close to the sample surface. However, the remnant native oxide makes the KPFM measurements qualitative except under special circumstances (see Paper H).

The cross-sectioned samples were contacted using UHV-compatible conductive silver epoxy. Sample and tip transfer into and from the UHV chamber were via an intermediate low vacuum chamber (10^{-7} to 10^{-8} mbar). Finally, to avoid potential shielding by surface water [17], the sample was annealed for one hour above 100°C in UHV prior to measurements.
5.3 Contrast

In this section the different contrasts typically observed with KPFM are presented. A summary is given in Appendix A.

![Cross-sectional KPFM picture and corresponding averaged potential (CPD) profile of an n-GaAs doping staircase structure. The doping levels of the layers ranged between $5 \times 10^{17}$ and $5 \times 10^{19}$ cm$^{-3}$ (substrate). The doping profile obtained by ECV on the structure is also shown. (b) Comparison curve between KPFM and ECV measurements. The discrepancies are attributed to the presence of surface states and oxide charges.](image)

Figure 5.3: (a) Cross-sectional KPFM picture and corresponding averaged potential (CPD) profile of an n-GaAs doping staircase structure. The doping levels of the layers ranged between $5 \times 10^{17}$ and $5 \times 10^{19}$ cm$^{-3}$ (substrate). The doping profile obtained by ECV on the structure is also shown. (b) Comparison curve between KPFM and ECV measurements. The discrepancies are attributed to the presence of surface states and oxide charges.

5.3.1 Doping contrast

The measured CPD is expected to decrease as ln(n) (see Eq. 5.1). Fig. 5.3 (a) shows the variations of the KPFM signal recorded on an n-GaAs structure grown by MOVPE on an n+ doped substrate. The doping levels are in the range $10^{15}$ - $10^{18}$ cm$^{-3}$. The structure was not optimized to have a well-defined doping staircase. Nevertheless, ECV measurements provide the necessary information – the doping in the structure. Fig. 5.3 (a) shows a typical KPFM image of the structure and the averaged CPD profile. The experimental data obtained by ECV are also presented in Fig. 5.3 (a). The different lowly doped steps in the KPFM profile are barely distinguishable. And as seen in Fig. 5.3 (b), as a function of the doping, the measured KPFM data differs significantly from the ideal dependence (dashed line). Similar observation was made for the InP
doping staircase structure presented in section 3.3.a. These discrepancies are due to the presence of the surface native oxide and thus surface/interface states. High density of surface states can pin the Fermi level. In this situation the dc voltage applied to nullify the electrostatic field will not drastically vary for different doping levels. As qualitatively described later in section 5.3.d, this behavior is dominant at lower doping levels. This explains the shape of the curve in Fig. 5.3 (a).

5.3.b pn junctions

Due to its inherent sensitivity to the work function variations, KPFM is a priori a suitable technique for characterization of pn junctions. The KPFM contrast basically gives the CPD difference across the junction. This is expected to correspond to the built-in potential of the junction, typically of the order of the energy gap.

![Graph](image)

**Figure 5.4:** Representative averaged CPD profile of an n-p-n' GaAs structure. The location of the depletion regions is indicated. The ac modulation amplitude was set to 0.1 V.

Fig. 5.4 shows the averaged CPD profile across the n+-p-n GaAs structure (see sections 3.3.b and 4.3.b). Qualitatively, the measured data are consistent with Eq. 5.1: the CPD is lower for the n+ substrate and higher for the p-doped region. However, the net CPD variation across the junction is much lower than the built-in potential due to the surface native oxide.

The KPFM profile clearly shows the two pn junctions. The drops (see Fig. 5.4) in the CPD profile correspond to the depletion regions. As expected, the drop in the CPD profile is more abrupt at the n+p junction. However, the lateral extent of these regions remains much higher than the simulated widths. This is due to a cantilever averaging effect. Clear evidence of this effect is the strongly decreasing KPFM signal for the n-doped layer closer to the edge.
5.3.c **Low dimensional systems**

Fig. 5.5 (a) shows the variations of the KPFM signal obtained on the structure presented in sections 3.3.c and 4.3.c consisting of an InGaAs/InP heterojunction and a 10 nm QW. Fig. 5.5 (b) shows the simulated potential (variation of the vacuum level) of the structure obtained from 1D Poisson/Schrödinger simulation. The trends in the experimental data are in excellent agreement with the simulation.

At the heterojunction, the CPD drops from the InGaAs to the InP layer. This is primarily due to the different electron affinities for the two materials. From such data the band offsets between the two materials can in principle be directly be extracted, but it clearly requires a UHV cleaved surface.

A characteristic peak in the CPD is observed at the location of the quantum well. The magnitude of the contrast peak is due to the electron affinity difference and the carriers accumulated in the well. The width of the contrast peak includes the nominal well width and the depletion regions in the barriers. These aspects are discussed separately in **CH. 8**.

![Figure 5.5: (a) Representative averaged CPD profile of an In\textsubscript{1.15}Ga\textsubscript{0.85}As/InP heterojunction and a 10 nm quantum well. The n-InGaAs and n-InP bulk regions were doped 1×10\textsuperscript{10} and 1×10\textsuperscript{17} cm\textsuperscript{-3}, respectively. The InGaAs QW is undoped. (b) Simulated vacuum level of the structure obtained using 1D Poisson/Schrödinger solver (SimWindows). 240 meV was chosen as the conduction band offset between InGaAs and InP.](image-url)
5.3.d Surface states

Due to its inherent sensitivity to surface properties, KPFM has been used to characterize surface states, i.e., their position in the bandgap and their density [13]. The main evidence of the presence of surface states is a typical contrast reversal in the CPD profile. Fig. 5.6 shows a schematic diagram of the potential variations in a 1D tip-sample system, with and without surface states. Without surface states, the potential profiles for two different doping levels do not cross each other, while the contrary occurs with surface states, thus inducing contrast reversal. A simple 1D model was also analytically proposed to support the given qualitative analysis. For an n-doped sample it follows:

\[ \Delta V = \Delta V_I + \Delta V_H \]  
\[ \Delta V_I = \frac{qd}{\varepsilon \varepsilon_\text{ox}} \sqrt{\frac{2 \varepsilon_\text{ox} \cdot N_d}{q}} \left( \varepsilon \Delta \Phi - q\Delta V \right) \]  
\[ \Delta V_H = \frac{qd}{\varepsilon \varepsilon_\text{ox}} D_\text{S} \left( kT \cdot \ln \left( \frac{N_C}{N_d} \right) + \varepsilon \Delta \Phi - q\Delta V \right) \]  
and \[ \Delta \Phi = \Phi_\text{tip} - \Phi_\text{sample} \]

with \( \Delta V_I \) corresponding to the potential drop due to the oxide and \( \Delta V_H \) corresponding to potential drop due to the surface states. \( \varepsilon_\text{ox} \) and \( \varepsilon_\text{se} \) are the permittivity of the native oxide and of the semiconductor, respectively, \( d \) the tip-sample distance, \( N_d \) the shallow donor density, \( D_\text{S} \) the deep donors density per unit of energy at the oxide-semiconductor interface, and \( N_C \), the effective density of states in the conduction band. Consistent with experiments, the model predicts the reversal contrast to occur in lowerly doped regions. The surface states were assumed to be donor-like and continuously distributed in the bandgap.

![Figure 5.6: Schematic band diagram showing the potential drop between the tip and the n-doped sample for two different doping levels. In dashed line, the potential drop in the oxide without oxide/interface states (no reversal contrast). In solid line, the potential drop includes the oxide/interfaces states influence (reversal contrast).](image)

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References

Chapter 6

Nanoscale characterization of GaAs-based buried-heterostructure lasers

6.1 Introduction

The need to obtain relevant information on the nanoscale in the large variety of semiconductor devices (lasers, transistors...) in conjunction with many different materials (Si, SiGe, III-V...) has made SPM based characterization tools very important. SPM techniques such as SCM and SSRM have already demonstrated their capabilities for nanoscale analysis of semiconductor devices. Si-based metal-oxide-semiconductor (MOS) transistors have been extensively investigated by both SCM [1-3] and SSRM [4-6]. SCM has been used for the characterization of III-V-based semiconductor devices, for instance InP- and GaAs- heterojunction bipolar transistors [7]. AlGaN/GaN field effect transistor (FET) [8] and edge emitting lasers [9-11]. In this thesis, we focused on the investigation of GaAs/AlGaAs buried heterostructure (BH) lasers by SCM, and the results are reported in Papers B and C.

6.2 Description and fabrication

In the simplest terms, a semiconductor laser can be seen as a p-i-n structure placed inside an optical cavity defined by parallel cleaved facets. The forward biasing of the p-i-n structure injects carriers into a thin active layer where they recombine and emit photons, i.e., generation of light. Typically, in such laser devices, the light is emitted through the cleaved facets. Hence the name edge-emitting lasers. The optical cavity provides the necessary optical feedback to obtain lasing conditions. The optically active region is usually one or several QWs located in the intrinsic layer between the p- and n-layers (also called claddings). The choice of the wavelength range of the laser depends on the bandgap $E_g$ of the QW:

$$\lambda = \frac{1.24}{E_g} \mu \text{m} \quad \text{with} \ E_g \ \text{in eV} \quad \text{(Eq. 6.1)}$$

Lasers are made from direct bandgap materials (e.g., several of III-V compound semiconductors). The choice of the operating wavelength dictates which specific material combination can be used. For example, for the 1300-1600 nm range, the commonly used materials are InP and GaInAsP [12]. Similarly for the 700-1000 nm range, GaAs, AlGaAs and InGaAs have been used [13].

The semiconductor lasers are designed to laterally confine the current and the optical mode in the mesa to get high efficiency and single mode operation. The mesa itself is formed using lithography and etching. Vertical optical and electrical
confinements are commonly improved providing additional layers called separate confinement heterostructures (SCH), between the active region and the claddings. In buried-heterostructure lasers, a high bandgap material is then epitaxially regrown around the mesa. The lateral optical confinement is obtained using a material with lower refractive index. Electrical confinement can be obtained either using higher bandgap materials with high resistivity (semi-insulating materials), or by pn junctions [9]. This thesis work deals specifically with BH-lasers with electrical confinement obtained by the former approach.

A semi-insulating (SI) material is a non-intrinsic semiconductor but has its free carrier concentrations at near intrinsic levels, thereby causing high resistivity, typically of the order of \(10^8\Omega\ cm\) [14]. Semi-insulating materials are commonly achieved by introducing deep level impurities whose energies lie close to the middle of the bandgap. The choice of the SI material is also dependent on whether it can be grown lattice matched to the (laser) substrate material. The epitaxial method of regrowth employed can be metal-organic vapor-phase epitaxy (MOVPE), hydride vapor-phase epitaxy (HVPE) or liquid-phase epitaxy (LPE) [9]. HVPE is especially suitable due to its high growth rate and the planarizing nature of the regrowth process. Thick regrown layers are required for high speed operation, typically implying low device capacitance. Finally, a good thermal conductivity of the burying layer can help in the dissipation of the heat generated during device operation. The formation of ohmic contacts on the \(p\)- and \(n\)-side of the device completes the fabrication.

Spatially resolved electrical characterization of BH-lasers is motivated by crucial issues such as the good planarization of the device and the quality of the electrical confinement. The latter implies specifically the local characterization of various hetero-interfaces involving the mesa and the regrown layers and the verification of the SI properties at different location of the regrown layers. These issues strongly motivate the use of electrical characterization techniques such as SCM. In this work, cross-sectional SCM was used to characterize GaAs/AlGaAs BH-lasers regrown with two different materials, GaAs and lattice matched GaInP (see Paper C). The two types of regrown layers were \(n\)-type and Fe-doped up to \(~10^{17}\)\(\text{cm}^{-3}\). Fe is a deep acceptor in both GaAs and GaInP with its energy level lying in the vicinity of the midgaps. The GaInP regrowth was carried out using HVPE: \(\text{Ga}_{0.51}\text{In}_{0.49}\text{P}\) lattice matched to GaAs was chosen for its higher bandgap (1.9 eV) and lower refractive index compared \(\text{Al}_{x}\text{Ga}_{1-x}\text{As}\) (\(x=0.38\)) [15]. High resistivity was obtained by incorporation of Fe (density \(~10^{18}\)\(\text{cm}^{-3}\)) during the HVPE regrowth [13,15]. In addition, this material is aluminum-free, therefore eliminating any oxidation related problems. The mesa was about 5 \(\mu\text{m}\) wide and 6 \(\mu\text{m}\) tall. The claddings were doped to about \(10^{17}\)\(\text{cm}^{-3}\); the SCHs and the active layer were undoped.

### 6.3 SCM contrast

Fig 6.1 (a) shows a SCM \((dC/dV)\) image of a cross-sectional GaAs/AlGaAs BH-laser regrown by SI GaInP:Fe. The layers in the mesa and the \(n^+\) GaAs substrate are clearly distinguished. The contrast for the \(n^+\) GaAs substrate is darker than for the \(n\)-AlGaAs cladding, consistent with its higher carrier density. The regrown layer appears
dark (zero signal). A dark contrast is also observed between the claddings and corresponds to the depleted waveguide including the SCHs and the QW. The bright region between the substrate and the n-cladding corresponds to the low doped buffer layer. An extension of this bright contrast is observed beyond the location of the mesa, between the substrate and the regrown layer. This observation indicates an incomplete etching of the mesa. The n-doped GaInP arm grown above the SI GaInP:Fe layer is also visible. Characteristic bright contrast lines are observed at the interfaces, especially between the n-doped layers and SI GaInP:Fe. The contrast lines lie inside the n-doped layers and their spatial extent is in good agreement with the width of the space charge region obtained from 1D Poisson simulation (typically 300-400 nm). The SCM (dC/dV) peak is much narrower at the interface involving p-doped region, consistent with the lower spatial extent of the depletion region in the p-cladding (~100 nm). These observations confirm the existence of the space charge region as the origin of the SCM (dC/dV) peak contrast.

![Diagram](image)

**Figure 6.1:** (a) Cross-sectional SCM (dC/dV) image of the GaAs/AlGaAs BH-lasers regrown with SI GaInP:Fe (4×10^{17} cm^{-3}). The dc and ac biases were 0 and 1 V, respectively. (b) ac bias dependence of the SCM (dC/dV) signal along the line indicated on the image. The bias was varied from 0.5 V to 2 V in steps of 0.5 V. The dashed circle in (a) highlights the specific contrast feature at the interface between the waveguide and the regrown layer.

### 6.4 Analysis of the regrown layer

The quality of the regrown interfaces is an important issue that can drastically affect device performance. In the case of SI regrown layers, the density of incorporated deep impurities may vary due to the orientation dependent growth. Fig. 6.1 (b) shows the SCM signal obtained for different ac biases between 0.5 and 2 V, approximately along the line indicated on the image (Fig. 6.1 (a)). While the SCM (dC/dV) signal monotonically increases in the cladding and the substrate, it exhibits a uniformly zero intensity in the regrown layer, irrespective of the applied ac bias. These observations strongly suggest sufficient incorporation of active Fe in GaInP, i.e., the semi-insulating nature of the regrown layer at different locations close to and far away from the mesa.
The shape of the contour of the $n$-GaInP layer grown above the GaInP:Fe layer (see Fig. 6.1 (a)) indicates the effective planarization of the structure using HVPE regrowth method. Independent current-voltage (I-V) measurements [16] and especially the good performances of these devices [17,18] support the SCM analysis. Although the SI nature of the regrown layer can be clearly verified, no quantification of active Fe concentration can be directly ascertained from the SCM measurements.

For sake of comparison, identical structures regrown with GaAs:Fe were also characterized (Paper C). In this case, the SCM contrasts are significantly different from that observed for GaInP, showing high intensity signals in the regrown layers. This indicates first that the regrown layers are not semi-insulating but semiconducting. The very high intensity contrast suggests in addition low carrier density. The zero SCM signal at the interface with the $n$-cladding is typical of a $p/n$ junction and therefore suggests shallow $p$-doping in the regrown layers. A possible explanation lies in the influence of EL2 defects in the regrown layer [19-21]. The EL2 defect is a deep donor in GaAs and the incorporation of deep impurities like Fe stimulates the formation of EL2 levels [22]. For a certain density of Fe, EL2 and Fe can compensate one another and in a narrow range of Fe concentration the resistivity of the structure can be lowered. More detailed studies are required to confirm this hypothesis.

![Figure 6.2: (a) Cross-sectional SCM (feedback-bias) image showing the contrast spot at the interface between the AlGaAs active region and the Si:GaInP:Fe ($4\times10^{18}$ cm$^{-3}$). The dc bias was 0 V. (b) Conduction band profile across the mesa and along the relevant interface of the sample. The data were extracted from 2D Poisson simulation (LASTIP program) [18]. The Fermi level in the GaInP:Fe is pinned close to the Fe acceptor level ($E_{\text{Fe}}=1.14$ eV). The dashed circle in (b) highlights the dip responsible for the special contrast feature shown in (a).](image)

### 6.5 Nanoscale contrast due to local band-bending

Papers B and C analyze a special feature in the SCM ($dC/dV$) signal observed at the interface between the Si GaInP:Fe and the depleted waveguide, i.e., a bright contrast spot about 200 nm wide and located in the regrown region (dashed circled in Fig. 6.1 (a)). The same region is shown on Fig. 6.2 (a) but the image was obtained with the
feedback-bias mode. Note that the contrast is inverted compared to the SCM \((dC/dV)\) image (Fig. 6.1 (a)). The absence of any corresponding feature in the topography image confirmed the electrical origin for this contrast spot.

The high intensity SCM \((dC/dV)\) signal indicates locally higher conductivity (Paper B), i.e., high carrier density compared to the surrounding layers. This is unexpected since the region is located between a depleted waveguide and a semi-insulating layer. The variations of the SCM \((dC/dV)\) signal with ac bias amplitude indicates that the carriers probed at the spot location could come from the \(n\)-doped cladding (Paper C). The variations of the SCM \((dC/dV)\) signal with the dc bias corroborates this interpretation of the contrast: the lateral displacement of the contrast induced by the dc bias is observed at the interface involving the \(p\)-cladding, while the contrast spot and the interface involving the \(n\)-cladding are motionless — confirming the \(n\)-type behavior of the spot.

A 2D Poisson simulation [18] supported these observations as shown in Fig. 6.2 (b). The Fermi level pinning in the SI regrown layer induces specific band-bending at the interface with the AlGaAs layers, resulting in an abrupt and large variation of the conduction band level between the regrown layer and the \(n\)-cladding or the \(n\)-side barrier – typically \(-1\) eV (see Fig. 2 (b)). Much lower variations were observed at the interface involving the \(p\)-cladding or the \(p\)-type barrier (see Paper C). These observations are consistent with the typical SCM \((dC/dV)\) contrasts observed at the different interfaces in the structure (see section 6.3) and confirm the interfacial nature of the spot contrast.
References

Chapter 7

Spatially resolved studies
of ion beam processed InP

7.1 Introduction

Another application of SPM-based electrical characterization techniques has been
developed in the last ten years – the evaluation of semiconductor processing. This can
include various processes such as dry etching, ion implantation, thermal annealing, etc.
Since they have a direct bearing on the performance of the devices, continuous
evaluation, understanding and improvement of these processes constitute a natural
requirement. In this scope, SCM has already proven to be a powerful evaluation tool [1-
4]. This thesis demonstrates the application of SCM and SSRM for spatially resolved
analysis of ion beam processed InP. More specifically, two processes, namely dry etching
and ion-implantation are studied. The application of SCM to evaluate dry etching induced
damage is presented (Paper D). This is followed by the analysis of ion implanted InP,
using SCM and SSRM (Paper E).

7.2 Dry etching on InP

In the fabrication of most optoelectronic devices, the transfer of a lithographically
defined pattern into the semiconductor is often accomplished by wet chemical and/or dry
(plasma) etching. Dry etching employs a gas plasma to provide reactive ions, inert ions,
radicals or a combination of these. The main advantages of dry etching compared to wet
etching are good process control, compatibility with an all-vacuum processing, low
process temperatures and especially its capability to perform anisotropic etching.
Therefore, dry etching has proven to be a powerful and a key technique in processing
technology, and its field of application has been abundantly reported in literature [5]. A
major drawback of dry etching is that the etch induced damage is likely to degrade device
performance.

Two types of etch mechanisms can be considered: the physical etching component so-called ion sputtering and the chemical etching component in which reactive ion gas ions or radicals react with the sample forming volatile etch products.
There are several different dry etching technologies available, e.g., ion milling,
chemically assisted ion beam etching (CAIBE) and reactive ion beam etching (RIBE)
[6,7]. These are the techniques used in this thesis. All of them use a remote plasma and
ions are extracted and accelerated towards the target (sample). In RIBE, both reactive and
inert ions are in the plasma. In CAIBE, only the inert gas is in the plasma while the
reactive gas is introduced close to the surface of the sample. The evaluation of dry
etching induced damage on \textit{n}-InP presented in this thesis refers to different processes and chemistries: Ar or N$_2$ milling, Ar/CH$_4$/H$_2$ CAIBE, N$_2$/CH$_4$/H$_2$ CAIBE, TMA RIBE and Ar/TMA RIBE.

Due to its high sensitivity to doping variations and its high lateral resolution, SCM appears to be the appropriate technique to perform local electrical characterization of etch induced damage. Moreover, the low forces involved in the AFM contact mode make SCM a good candidate as a non-destructive and in-line process evaluation tool, therefore more appropriate than SSRM. SCM measurements were performed on low doped \textit{n}-InP samples. The evaluation of the damage removal upon annealing was also characterized. Furthermore, differences between the etching processes were analyzed using the measured distributions of the SCM intensities. The ability of SCM to perform simultaneous mapping of electrical and topographical properties provides complementary information about the surface morphology.

The SCM results are correlated to I-V measurements of macroscopic metal-semiconductor contacts. For this purpose, macroscopic Au dots were evaporated on the processed sample surface forming Au/InP contacts. For forward bias, the current-voltage relationship predicted by the thermionic-emission theory can be expressed as follows:

\[
J = J_0 \exp(qV/\eta kT) \left[ 1 - \exp\left( -qV/kT \right) \right] \quad \text{(Eq. 7.1a)}
\]

\[
J_s = A' T^2 \exp(-q\phi_s/kT) \quad \text{(Eq. 7.1b)}
\]

where $\phi_s$ is the effective (macroscopic) barrier height, $\eta$ the ideality factor, $A'$ the Richardson constant and $J_0$ the reverse saturation current density [8].

![Figure 7.1: Overall qualitative trend of the SCM signal \((dC/dV)\) for different processed samples. The displayed data is obtained in one sequence, positioning the SCM-tip on each sample thus limiting influence of external parameters (humidity, tip, etc.)](image)

Fig. 7.1 shows the variations of the SCM \((dC/dV)\) signal for three different processes. The intensities correspond to the mean value measured over a large number of measurement instances. Low SCM \((dC/dV)\) signal is observed for as-etched structures, indicating high coping in the near surface region after etching. These observations are made irrespective of the etch process and therefore suggest a similar nature of the etch induced damage. This is attributed to the stoichiometric imbalance due to preferential
removal of phosphorus [9]. These results are consistent with the observation that Au contacts on as-etched surfaces show ohmic behavior (Paper D).

The damage removal process was investigated by annealing the samples for 10 min at 650°C under PH3 atmosphere. Fig. 7.1 shows the increase of the SCM \( (dC/dV) \) signal for the three processed samples after annealing. SCM \( (dC/dV) \) intensities are nearly as high as the one observed for the reference sample, which indicates significant recovery of the initial electrical properties. This must correspond to a reduction of the surface charge density induced by the etching processes therefore indicating damage removal upon annealing. Consistently, the macroscopic Au contacts on the annealed samples show Schottky barrier heights around 0.5 eV, similar to that measured on the unprocessed sample. The measured barrier heights and the ideality factors are given in Table II.

**Table II:** Characterization of etch-damage removal process upon annealing. The barrier height \( \Phi_h \) and the ideality factor \( \eta \) were obtained from I-V measurements. The SCM \( (dC/dV) \) distributions were obtained on 3×3 µm² regions, using 512×256 data points. The SCM distribution peaks were then normalized. The FWHMs correspond to the full widths at half maximum of the normalized SCM distribution peaks.

<table>
<thead>
<tr>
<th>Etch Process/Sample</th>
<th>( \Phi_h ) (barrier height) (eV)</th>
<th>( \eta ) (ideality factor)</th>
<th>SCM-FWHM (a.u.)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Reference</td>
<td>0.50</td>
<td>1.01</td>
<td>0.020</td>
</tr>
<tr>
<td>Ar/TMA RIBE</td>
<td>0.49</td>
<td>1.01</td>
<td>0.025</td>
</tr>
<tr>
<td>TMA RIBE</td>
<td>0.47</td>
<td>1.13</td>
<td>0.038</td>
</tr>
<tr>
<td>N2 milling</td>
<td>0.42</td>
<td>1.17</td>
<td>0.071</td>
</tr>
<tr>
<td>N2/CH4/H2, CAIBE</td>
<td>0.51</td>
<td>1.25</td>
<td>0.111</td>
</tr>
</tbody>
</table>

A better evaluation of the damage removal process is possible by investigating the distribution of the SCM signal. The ideality factors \( \eta \) is an empirical quantity used as an indicator of the quality of the Schottky contact. Among other mechanisms [8,10], barrier height inhomogeneities are an important factor and result in ideality factors being larger than unity. With dry etched and annealed InP surfaces, there is a strong likelihood that the near-surface properties (doping and stoichiometry) are not locally homogeneous [11]. Therefore the ideality factor is an additional important parameter to evaluate etch-damage removal. Due to the reasons mentioned above, one would also expect local variations in the SCM signal distributions. Indeed as detailed in Paper D and on Table II, the full widths at half maximum (FWHM) of the SCM signal distributions do show a correlation with the (macroscopic) ideality factors.

Fig. 7.2 (a) and (b) show the topography images acquired for a representative process prior to and after annealing, respectively. These images highlight how the initial structural damage (randomly spiky morphology) induced by dry etching is transformed after annealing into a surface with irregular step-like features. Very low RMS roughness, typically 0.2 to 0.3 nm, is commonly observed in both as-etched and annealed samples, and suggests that the structural damage removal process only involves a thin near surface region. The step-like features observed for annealed samples are clearly irregular and differ significantly from the regular monolayer steps observed on as-grown InP (001) surfaces (Fig. 7.2. (c)) [12]. Finally, the comparison between topography and capacitance
images simultaneously acquired does not show any specific correlation. This could be due to the fact that the electrical damage is more deeply inlaid into the sample (10 to 20 nm).

Figure 7.2: Local variations of topography. (a) as-etched InP surface; etch process: Ar/TMA RIBE. (b) corresponding surface after annealing. (c) as-grown (001) InP surface. The scan size for all the images was 3×3 μm². Topographical variations are below 2 nm.

7.3 Ion implanted InP

Cross-sectional SCM and SSRM are used to track locally the modifications of the electrical properties of ion implanted InP as a function of the annealing conditions. Ion implanted InP is an attractive material for devices such as photodetectors, exhibiting ultrashort carrier lifetimes, good mobilities and high resistivities [13,14]. All the SCM and SSRM analysis were part of a wider scope aimed at evaluating and correlating the structural, electrical and optical properties of the implanted materials by means of various complementary characterization techniques. The use of cross-sectional SCM and SSRM was first motivated by their ability to perform depth profiles of the electrical properties. Their high lateral resolution was then shown to be useful to delineate local regions with different electrical properties. Correlations with structural data obtained with X-ray diffractometry and cross-section transmission electron microscope were finally shown to provide further information on damage removal mechanisms upon annealing (Paper E).

A 2 μm thick undoped InP epilayer grown on n⁺ InP substrate was implanted with Ga⁺, As⁺, P⁺ or In⁺ with a dose of 10¹⁶ cm⁻². The implantation energy corresponding to each ion was specifically chosen to induce maximum damage lying approximately 1 μm from the surface. Rapid thermal annealing (RTA) of the sample was carried out at 400, 500 and 600°C for a duration of 30 s.
Figure 7.3: (a) SCM ($dC/dV$) and (b) SSRM profiles obtained on cleaved cross-sections of the Ga$^+$ implanted InP annealed at 600°C. $R_1$, $R_2$, $R_3$ and $R_4$ correspond to the regions of different conductivity in the epilayer.

For the sample annealed at 400°C, zero SCM ($dC/dV$) signal and high spreading resistance are commonly observed in the whole epilayer. Interestingly, the SSRM profile exhibited a small resistance peak at about 1 µm from the cleaved edge in good agreement with the expected maximum implantation depth (Paper E). Both SCM and SSRM results indicate very low carrier densities in the epilayer due to implantation induced defects. These results are in addition consistent with the Hall measurements performed on these structures.

As the annealing temperature increases, highly non-uniform contrasts are observed. This is illustrated in Fig. 7.3 (a) and (b) showing the SCM and SSRM profiles, respectively, obtained on a Ga$^+$ implanted sample annealed at 600°C. In Fig. 7.3 (a), two SCM dips (regions $R_3$ and $R_4$) appear in the epilayer indicating a progressive recovery of the properties in the epilayer. This suggests, in addition, a migration of the implanted defects towards the surface and the epi/substrate interface, together with a progressive domination of the shallow donors created by the implantation process [14]. The SCM profile shows a relative maximum around 1 µm from the surface (region $R_0$) where the defect density remains higher than in the surroundings. Similar observations are made in Fig. 7.3 (b). The SSRM profiles exhibit a high resistance peak around 1 µm from the cleaved edge (region $R_0$) in good agreement with high defect density. The low resistances in the surroundings (regions $R_2$ and $R_4$) exhibits resistance levels of the same order than the $n^+$ substrate after annealing at 600°C confirming significant reduction of defect density and creation of excess carriers (shallow donors) [14]. The decrease of the SSRM and SCM peaks at the epi/substrate interface with the annealing temperature also indicates a progressive recovery in this region. This is however not the case in the near surface region (< 400 nm; $R_1$ on Fig. 7.3) where uniform zero SCM ($dC/dV$) signal and high spreading resistance are continuously observed irrespective of the annealing temperature.

Paper E confirms good consistency between these results and those obtained from both optical and structural investigations. Moreover, the SSRM profiles showing the presence of high conductivity channels framed by highly resistive regions helped to understand the range of low resistance values obtained from Hall measurements.
The influence of the implanted ion species has also been investigated. The overall trend showing progressive expansion of the conductive regions with the annealing temperature is also observed in As⁺ and P⁺ implanted samples (see Paper E). The spreading resistance values in the conductive channels are in addition in good agreement with what is observed in Hall measurements, confirming higher resistance in the Ga⁺ implanted samples. Significant differences are however observed with the different implants at the maximum implantation depth. The SSRM peak is for example observed in As⁺ implanted sample but missing in the P⁺ implanted one. This is understood in conjunction with the structural and optical analysis in terms of scattering and trapping properties that differ with the nature of the implant. In the case of In⁺⁺ implanted sample, only a narrow but highly conductive region is observed close to the epi/substrate interface. These results are again in good agreement with the other structural and electrical measurements.
References

Chapter 8

Characterization of InGaAs/InP quantum wells

8.1 Introduction

The development of optoelectronics has generated tremendous interests for low dimensional systems, i.e., quantum wells (QWs), quantum wires and quantum dots. This chapter focuses on the characterization of lattice matched InP/In_{0.53}Ga_{0.47}As QWs. The well widths were 5, 10 and 20 nm. The barriers were $n$-doped at different levels and the wells undoped. Detailed information on the investigated samples is available in Papers F and G. The capability of SCM, SSRM and KPFM to detect and evaluate the density of electrons accumulated in the wells as well as to resolve the depletion regions in the surrounding barriers is demonstrated. Specific trends in the SPM signals are also observed for different well widths or barrier dopings. The SCM, SSRM and KPFM results are reported in Paper F, Paper G and Paper H, respectively. These measurements were also used to evaluate the performances of the techniques, especially in terms of lateral resolution. The “electrical” spatial resolution in SCM and SSRM was addressed using QWs and is reported in Paper I. The behavior of the SCM signal across a QW was simulated using the annular ring model. The results qualitatively reproduce the experimental observations. A brief description of the model and a presentation of the results finally conclude this chapter.

![Figure 8.1: Cross-sectional SCM profiles of a 20 nm InP/InGaAs QW structure for different dc biases. The $n$-InP barriers were doped to $1 \times 10^{18}$ cm$^{-3}$.](image)
8.2 Image contrast

8.2.a SCM

Fig. 8.1 shows representative SCM \( (dC/dV) \) line scans obtained with different dc biases on a 20 nm QW framed by InP barriers (doping \( 1 \times 10^{16} \text{cm}^{-3} \)). A clear dip is observed at the location of the QWs indicating a higher carrier density in the well than in the surrounding barriers, consistent with the accumulation of electrons in the well. The full width at half maximum (FWHM) of the dip is wider than the well width. This indicates significant tip-averaging effect, induced by the geometry of the tip and the presence of fringe fields. Under appropriate bias, the depletion region in the vicinity of the well can also be resolved, as shown in Fig. 8.1. The SCM signal intensity difference between the two line scans is explained in terms of shifts of the operating point in the C-V curves. The width of the depletion regions remains in good agreement with the 1D Poisson/Schrödinger simulation.

![SSRM Resistance vs Distance](image)

**Figure 8.2:** Cross-sectional SSRM averaged profiles of a 20 nm InP/InGaAs QW structure for different bias polarity. Gray line: forward bias. Black line: reverse bias. The \( n \)-InP barriers were doped to \( 1 \times 10^{16} \text{cm}^{-3} \).

8.2.b SSRM

Fig. 8.2 shows two representative SSRM averaged profiles obtained in forward and reverse bias across the same QW structure as in Fig. 8.1. A resistance dip is also observed at the location of the well, consistent with an accumulation of carriers. Compared to Fig. 8.1, the dip is more pronounced and its FWHM is much narrower. This indicates better spatial resolution in SSRM than in SCM. In this case, only the effective
tip-sample contact area contributes to the averaging effect (see CH. 4). Using low
deflection setpoint (i.e., low tip-sample forces) and under low forward bias, characteristic
dips are shown to exhibit a FWHM comparable to the effective well width, even for the
narrowest wells (Papers G and I). The depletion regions surrounding the well are shown
to be resolved for forward bias and for low reverse bias. This observation was discussed
earlier in CH. 4, section 4.3.c in terms of current transport mechanisms.

![Graph](image)

**Figure 8.3:** Cross-sectional KPFM profile of a 20 nm InP/InGaAs QW. The n-InP barriers were doped
to $1 \times 10^{17}$ cm$^{-2}$.

### 8.2.c KPFM

Fig. 8.3 shows a representative CPD line scan. A peak is observed at the location
of the quantum well. As discussed in CH. 5, section 5.3.c, the origin of the peak in the
KPFM signal must be first related to the carriers accumulated in the well. From the
magnitude of the peak, information such as the electron affinity difference and therefore
band offsets can be extracted. The determination of band offsets however requires full
understanding of the influence of perturbing effects on the surface potential, namely, the
surface states and the tip averaging effect. By scanning very close to the surface, the peak
lowering induced by tip averaging remains very small. The fair consistency observed
between 1D Poisson/Schrödinger simulation and the experimental results in terms of
peak magnitude suggested in addition similar contribution of the surface states in the
wells and the barriers (see Paper H). Finally, compared to SCM and SSRM, here the
KPFM signal (peak) includes the depletion regions. Good consistency is nevertheless
observed in the spatial extent of the experimental and simulated potential peak (Paper H).
8.2.4 Dependence on the well width and barrier doping

In the case of an infinite potential well, the discrete set of energy levels can be expressed as:

$$E_n^s = n^2 \frac{\pi^2 \hbar^2}{2m_e L^2}$$

(Eq. 8.1a)

with \(n\) an integer, \(m_e\) the effective mass of electrons and \(L\) the width of the potential well [1]. For a well with a finite potential, the calculation of the energy levels is more complex and cannot be analytically expressed [1]. However, the solution for the first energy level \((E_1, \text{ground state})\) can be analytically approximated as follows:

$$E = E_1 \left( \sqrt{ \frac{E_1}{\Delta E_C} } + \sqrt{ \frac{1}{4C^2} } \right)$$

with \(C = 1.293\)

(Eq. 8.1b) [1]

where \(\Delta E_C\) is the confinement potential and corresponds to the conduction band offset between the two materials forming the well.

![Diagram](image)

**Figure 8.4:** (a) Schematic energy diagram of a finite InGaAs/lnP potential well showing the confinement potential \(\Delta E_C\) and the energy level of the ground state \(E_1\), respectively. (b) Simulated variations of the first energy level (ground state) of a InGaAs/lnP quantum well with the well width for two different situations using Eq. 8.1a and b. 240 meV corresponds to the conduction band offset between InP and InGaAs [2].

Fig. 8.4 (b) illustrates how the position of the ground state \(E_1\) varies with the QW width. As observed in Fig. 8.4 (b), for wider wells, the ground state is closer to the bottom of the well. For a given barrier doping, the position of the level relative to the Fermi level will therefore decrease and, a fortiori, the electron sheet (2D) density will increase with the width of the well. The built-in potential and the lateral extent of the depletion regions will also vary for different well widths.
The 1D Poisson/Schrödinger simulation is used to compare the results obtained from the SPM profiles for different well widths and barrier dopings. The simulation predicts a higher built-in potential and a larger depletion regions for wider wells. The simulated values of the 2D and 3D electron densities for 10 and 20 nm wells are given in Table III.

The SCM results showed an increase of the spatial extent of the depletion region with the well width, consistently with the simulation (see Paper F). However, the dip magnitudes are more pronounced for the wider well suggesting higher carrier density, in contradiction with the simulation (see Table III). This discrepancy is due to tip averaging effects and evidently its effect is more pronounced for narrower wells. The averaging effect was initially evaluated by deriving the widening factor as the ratio between the FWHM of the SCM dip and the effective well width. The widening factors logically decrease with the well width.

With SSRM, the depletion regions will also consistently expand with the width of the well (see Paper G). The dip profile at the well location is also more pronounced for wider wells again suggesting the presence of tip averaging effect. The widening factors are however much smaller than in SCM indicating lower averaging effect, i.e., higher spatial resolution. This issue will be discussed later on in section 8.3.

The characteristic peaks in the KPFM contrast are also consistent with the simulation, exhibiting higher magnitude for wider QWs, consistent with higher sheet density in wider well (see Paper H). The FWHM of the peaks also logically increases with the width of the well. Compared to SCM and SSRM, a meaningful widening factor remains difficult to estimate due to the embedment of the depletion region related signal in the overall peak contrast; that is, the spatial resolution issue cannot be directly addressed with these structures.

Table III: Carrier densities in QWs extracted from $dC/dV$ line scans and resistance profiles for 10 and 20 nm QWs. For both SCM and SSRM, the carrier densities were extracted from the InP calibration curves constructed with internal reference layers. Possible electron mobility differences in the QWs [3,4] were not taken into account in SSRM calibration curves. 2D (sheet) and 3D carrier (peak) concentrations obtained from 1D Poisson/Schrödinger simulation (SimWindows solver) are also given. Conduction band offset was set to 240 meV [2].

<table>
<thead>
<tr>
<th>Techniques</th>
<th>Barrier doping $1\times10^{10}$ cm$^{-2}$</th>
<th>Barrier doping $1\times10^{10}$ cm$^{-3}$</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>10 nm</td>
<td>20 nm</td>
</tr>
<tr>
<td>Converted SCM</td>
<td>1.2-10$^7$ cm$^2$</td>
<td>3.4-10$^{10}$ cm$^2$</td>
</tr>
<tr>
<td></td>
<td>2.3-10$^{10}$ cm$^3$</td>
<td>4.5-10$^{10}$ cm$^3$</td>
</tr>
<tr>
<td>Converted SSRM</td>
<td>5.4-10$^7$ cm$^2$</td>
<td>1.2-10$^{10}$ cm$^2$</td>
</tr>
<tr>
<td></td>
<td>2.3-10$^{10}$ cm$^3$</td>
<td>6.7-10$^{10}$ cm$^3$</td>
</tr>
<tr>
<td>3D density (simulation)</td>
<td>1.2-10$^8$ cm$^2$</td>
<td>6.8-10$^{10}$ cm$^2$</td>
</tr>
<tr>
<td></td>
<td>3.6-10$^{10}$ cm$^3$</td>
<td>2.2-10$^{10}$ cm$^3$</td>
</tr>
<tr>
<td>2D density (simulation)</td>
<td>6.2-10$^{10}$ cm$^2$</td>
<td>6.8-10$^{10}$ cm$^2$</td>
</tr>
<tr>
<td></td>
<td>1.8-10$^{10}$ cm$^3$</td>
<td>2.2-10$^{10}$ cm$^3$</td>
</tr>
</tbody>
</table>

For a given well, the difference of doping in the barriers will also result in different accumulated electron densities, lateral extents of the depletion regions and built-in potentials. This trend is more clearly expressed both in SCM and SSRM (See Paper F and G, respectively). However, the order of magnitude of the peak electron densities in the QWs estimated from SSRM data is in better agreement with simulation compared to SCM values (see Table III). This is reasonable due to the much better spatial resolution with SSRM compared to SCM.
At this stage, the main issue is to perform the quantification of the carrier density in the wells from SCM and SSRM data. With this, one can in principle determine the band offsets. This requires specific 3D modeling for both SCM and SSRM, taking the tip shape into account. A more practical approach is experimental but requires reference layers of the appropriate material in the structure to construct meaningful calibration curves [Paper G].

8.3 Electrical spatial resolution

One of the main advantages of SPM techniques is the high spatial resolution. This part presents a method to determine spatial resolution using QWs. Applying this method to SSRM and SCM using commercially available probes, the spatial resolution was determined. A spatial resolution of sub-5 nm for SSRM and of sub-30 nm for SCM was obtained. The results are published in Paper I.

As mentioned earlier, the first limitation on spatial resolution is how sharp the probe-tip is. In SCM and in SSRM, the probed volume depends on the applied voltages. A low ac bias and a dc bias near flat-band condition are required to enhance resolution in SCM. In SSRM, it implies low dc biases together with low tip-sample forces. In SCM, the shape and the radius of the tip are the major parameters. The contribution of the fringe fields can be reduced using high aspect ratio probes. Tip radii comparable to the imaged feature size will then induce geometrical tip averaging effects. A representative SEM image of a commercial SCM probe (PtIr, metal-coated etched Si AFM probe) is shown in Fig. 8.5 (a). In SSRM, the force, the probe penetration depth and the tip radius and material are the main parameters that affect the resolution. The best resolution was obtained using diamond-coated probes (highly resistive to wear) and low range of force, i.e., low penetration depth and low contact dimensions. A representative SEM image of a commercial SSRM probe (diamond-coated etched Si AFM probe) is shown in Fig. 8.5 (b). Further information about the probes is given in Appendix B.

![Figure 8.5: (a) SEM image of a commercial PtIr metal-coated tip used for SCM measurements. (b) SEM image of a commercial boron doped diamond coated used for SSRM measurements.](image)

For addressing the resolution issue, suitable structures with abrupt variations of carrier density are required. Spatial resolution of SCM [5] and of SSRM [6] has been recently measured by imaging Si-SiO₂ step junctions. In III-V technology,
heterojunctions and quantum wells are potential candidates. In such systems, the fundamental limitation is therefore the spatial extent of the ground-state electron wave-function. The spatial resolution of the two techniques was evaluated using InGaAs/InP QW structures with varying interwell spacings. Structures had either 10 or 5 nm wide QWs and consisted of five sets. Each set had two wells, but different interwell spacings. The interwell spacing varied from 200 to 10 nm. Further description of the samples is given in Paper I. SCM and SSRM measurements were performed using commercial probes from NanoSensors GmbH (see Fig. 8.5) [7]. A first estimate for the spatial resolution was obtained from the smallest distance for which the two wells can be distinguished.

For interwell spacings larger than 50 nm, the two SSRM dips of a given set are sufficiently apart to avoid electrical interaction between the wells. The dips can be considered as the signature of clear independent wells and will be used as the reference signature. An overlapping of the dip contrasts occurs when reaching an interwell spacing of 20 nm. Irrespective of the well width, two dips could however be clearly observed in the set with 10 nm spacing. These results therefore indicated a sub-15 nm SSRM spatial resolution. In the case of SCM, appreciable overlap occurs for interwell spacings of about 100 nm and less. This concerns the depletion regions in the barriers. Irrespective of the well width, two dips in the SCM profile can still be distinguished for a spacing of 50 nm. However, only a single dip is observed in the 5 nm QW sample for the 20 nm interwell spacing set. This observation indicates therefore a sub-30 nm spatial resolution.

![SSRM profile](image)

**Figure 8.6:** SSRM profile obtained on two 5 nm QWs spaced 50 nm apart. The FWHM of the dips are indicated in the figure. The dc bias was -0.5 V.

In the case of QWs, with a peak carrier profile, the SSRM spatial resolution was then refined by measuring half of the full-width-at-half-maximum (½FWHM) of the characteristic dips. Since an overlap was previously observed between the dips in the narrowest sets, investigations were performed on wells with significant separation. Fig. 8.6 shows the SSRM profile obtained on two adjacent 5 nm QWs. The measured ½FWHMs remain between 4 and 4.5 nm, therefore indicating a sub-5 nm spatial resolution. An alternative way is to use Si/SiO2 step junctions [5,6]. The primary advantage with the present QW structure is the ease of sample preparation. Thus, it can
also be used for routine calibration of probes and in probe development. Finally, one notes that other material systems can also be used. Delta-doped structures can also be suitable provided that the extent of dopant diffusion is either negligible or at least independently known [8].

8.4 Annular ring model for 3D SCM simulation

In section 8.2.4, the quantification procedure of carrier densities emphasized the limitations of 1D modeling and stressed the need for 3D modeling and simulations for each SPM technique. 3D simulation of the KPFM signal, taking into account the long distance range electrostatic interactions between the probe and the sample surface, has been reported [9]. Modeling for quantitative carrier profiling using SSRM has also been proposed [10]. There are also several reports on SCM modeling [11-13]. This part focuses on SCM, using the so-called annular ring model [11,14,15]. A brief description of the model is first given and the preliminary results obtained for the QWs are presented.

![3D schematic of the annular ring first order modeling.](image)

The model derives the capacitance of the tip-sample structure for a given voltage. On this purpose, the sample surface plane is segmented into narrow annular regions surrounding the tip-oxide contact point. Hence the name, the annular ring model. The tip is represented as a metallic sphere embedded in a medium of uniform dielectric constant above the sample surface. The presence of the thin oxide layer at the sample surface is also taken into account. Fig. 8.7 shows a schematic description of the model.

The first step of the model consists in the determination of the effective insulator capacitance between the tip and each annular region. At this stage, the influence of the oxide is neglected. Using the method of images [16], the electric field distribution and the effective capacitance \( C_{\text{ew}} \) are calculated for each annular region. The effective insulator capacitance \( C_{\text{ew}} \) is then determined including the capacitance \( C_{\text{ox}} \) of the thin oxide layer.

\[
\frac{1}{C_{\text{ew}}(r)} = \frac{1}{C_{\text{er}}(r)} - \frac{1}{C_{\text{ox}}(r)}
\]  

(Eq. 8.3a)
$C_{\text{net}}$ is a fair approximation of the exact insulator capacitance. The capacitance of the semiconductor for different regimes is then calculated for each annulus using standard analytic 1D MOS band-bending model (see section 3.2.a) [17]. The tip-insulator-semiconductor capacitance ($C_{\text{net}}$) is obtained for each annulus by adding the effective insulator and the semiconductor capacitances:

$$
\frac{1}{C_{\text{net}}(r)} = \frac{1}{C_{\text{ins}}(r)} + \frac{1}{C_{\text{sc}}(r)} \quad (\text{Eq. 8.4})
$$

The total capacitance ($C_{\text{tot}}$) is finally calculated by summing over all the net capacitance contributions from each annular region. The annular regions are in parallel, therefore:

$$
C_{\text{tot}} = \sum r C_{\text{net}}(r) \quad \text{with } r=0, \Delta r, 2\Delta r, \ldots, r_{\text{max}} \quad (\text{Eq. 8.5})
$$

This completes the presentation of the first-order modeling, i.e., considering uniform carrier density in the semiconductor (see for example CH. 3, section 3.2).

![3D SCM simulation profiles on two InP/InGaAs QWs.](image)

**Figure 8.8:** 3D SCM simulation profiles using the second order annular ring model on two InP/InGaAs QWs. The wells were 10 and 20 nm wide. The $n$-InP barrier doping was $10^{17}$ cm$^{-3}$. (a) 10 nm tip radius, (b) 20 nm tip radius. The dc bias, the ac bias and the oxide thickness were 0.4 V, 0.5 V and 2 nm, respectively. The width of the annular $\Delta r$ and the number of rings were 1 nm and 90, respectively. The conduction band offset between InP and InGaAs was set to 240 meV.

The second-order modeling simulates structures with doping gradient. On this purpose, the annular rings used in the first-order model are sub-segmented into small regions, breaking the cylindrical symmetry in the direction of the doping variations. Fig. 8.8 shows the simulation results using second-order model. The wells are respectively 10 and 20 nm wide and the doping level in the $n$-InP barriers is $1 \times 10^{17}$ cm$^{-3}$. Clear dips in the SCM ($dC/dV$) signal are obtained at the wells. Especially, the widths of the dips are much wider than the effective well width in good agreement with what was experimentally observed (see Fig. 8.1 and Paper F). This widening effect is also consistently more pronounced for narrow wells. The depletion regions around the QWs are also resolved. The influence of the simulation parameters on the SCM profile is illustrated taking the tip radius as an example. The dips are logically less pronounced for a wider tip radius. This confirms tip geometry as the origin of the widening effect experimentally observed. At the present stage, the simulation must be regarded as a good and qualitative predictive tool.
References

Chapter 9

Guide to the papers

PAPER A: Comparative investigation of GaAs pn junctions by scanning probe microscopy: SCM, SSRM and KPFM

In this paper, a cross-sectioned n-p-n- GaAs structure is investigated using SSRM, SCM and KPFM. SSRM is shown to provide bulk information. Variations in the bias polarity are used to delineate pn junctions. The depletion widths are in excellent agreement with the expected values. Quantitative measurements (doping) are however prevented by the contribution of the tip-sample contact resistance to the overall SSRM signal. SCM measurements also show consistent results, although the observed contrast remains qualitative. The depletion regions generating locally zero SCM (dC/dV) signal are clearly observed. The delineation of the pn junction is then performed using the dependence of the SCM signal on ac and dc biases. Quantitative information is however limited by the contribution of the surface states to the SCM signal. KPFM is mainly sensitive to surface properties and in this case to surface state densities. Good qualitative contrast is however observed and the depletion regions are shown to coincide with what was observed in SSRM. A comparison of the techniques finally stresses the complementary aspect of the techniques.

Contribution of the author: SCM and SSRM measurements and analysis, participation in writing.

PAPER B: Characterization of GaAs/AlGaAs laser mesas regrown with semi-insulating GaInP by scanning capacitance microscopy

This paper demonstrates 2D nanoscale mapping of the electrical properties of a GaAs/AlGaAs BH-laser regrown with semi-insulating GaInP:Fe using cross-sectional SCM. This includes the delineation of regrown interfaces and the electrical nature of the regrown layers. Bias variations are used to verify the semi-insulating properties of the regrown GaInP:Fe, suggesting in addition uniform Fe incorporation close to and far away from the mesa. The paper finally investigates a special nanoscale contrast feature observed at the interface between the mesa waveguide and the regrown layer. The origin of this contrast is shown to arise from local band-bending at the interface.

Contribution of the author: measurements, analysis, writing.
PAPER C: Spatially resolved electrical characterization of GaAs-based buried heterostructure lasers

In this paper, cross-sectional SCM is applied to characterize GaAs/AlGaAs BH-lasers. Like in Paper B, the major issues addressed are the delineation of regrown interfaces and the electrical nature of the regrown layers. A comparative study is made for different regrown layers. While semi-insulating properties are verified for GaInP:Fe regrowth, the GaAs:Fe regrown layer is shown to exhibit a semiconducting behavior. The influence of the electrical properties of the regrown layers on the carrier distributions at the interface is then discussed. Finally, the nanoscale contrast feature described in Paper B is further analyzed, using bias dependent measurements. The Fermi level pinning in the Si regrown layer and the resulting specific band-bending at the interface with the active region are shown to be the main parameters responsible for this contrast feature.

Contribution of the author: measurements, analysis, writing.

PAPER D: Electrical characterization of dry etched InP surfaces with nanoscale lateral resolution

This paper demonstrates a novel application of SCM, i.e., as a nanoscale characterization method for evaluation of electrical damage induced by dry etching. The measurements are performed for different types of dry etching (milling, RIBE, CAIBE) and the damage removal by annealing is also investigated. The samples are made of InP. Consistent with independently performed conventional I-V measurements on macroscopic Au/InP contacts, the SCM measurements indicate the creation of a highly doped near surface region after etching and significant recovery of the electrical properties upon annealing (650°C under PH3 atmosphere). The nature of damage is explained in terms of stoichiometric imbalance in the near surface region. The damage recovery is further evaluated using distributions of the SCM signal intensities. A striking observation is the correlation between the ideality factors of macroscopic Schottky contacts and the FHWM of the SCM distributions. The origin of the greater-than-unity ideality factors is attributed to barrier inhomogeneities and is supported by the SCM results.

Contribution of the author: measurements, analysis, writing.

PAPER E: Structural, electrical and optical analysis of ion implanted semi-insulating InP

In this paper, n-InP has been implanted with P⁺, As⁺, Ga⁺ and In⁺ ions and the structural, electrical and optical properties as a function of annealing temperature are analyzed using various characterization techniques. The cross-sectional SSRM and SCM measurements and analysis were critical in tracking the local electrical properties in the structure. Consistent results are observed between the SCM and SSRM measurements showing a progressive evolution of the electrical properties with the annealing temperature. Specific behavior is observed at the maximum implantation depth indicating remnant defects even
after rapid thermal annealing at 600°C. Highly conductive regions also indicate significant defect migration towards the cleaved edge and the substrate. Specific trends are observed with the implanted species.

*This work was accomplished in collaboration with the Australian National University, Canberra, Australia.*

**Contribution of the author:** SCM and SSRM measurements and analysis, writing of the corresponding sections.

**PAPER F: Electrical characterization of InGaAs/InP quantum wells by scanning capacitance microscopy**

This paper focuses on the investigations performed on InGaAs/InP QWs using cross-sectional SCM. The ability of the technique to detect carriers accumulated in the wells is first demonstrated. Characteristic dips in the SCM ($dC/dV$) signal are observed at the position of the wells, consistent with a higher carrier density in the well. Under proper biasing, it is shown that the depletion regions in the surrounding barriers can also be resolved. These results are qualitatively consistent with 1D Poisson/Schrödinger simulation. The tip-averaging effect is shown to significantly influence the SCM profile thus affecting the lateral resolution. The influence of the tip radius is especially evaluated.

**Contribution of the author:** measurements, analysis, writing

**PAPER G: Probing carriers in two-dimensional systems with high spatial resolution by scanning spreading resistance microscopy**

In this paper, carrier profiling in InGaAs/InP QWs is performed using cross-sectional SSRM. It is demonstrated that SSRM is capable of profiling electrons in QWs with high spatial resolution. Characteristic dips are observed at the location of the well consistent with carrier accumulation. Using appropriate internal reference layers, the carrier density in the wells could be estimated. The results are in good agreement with 1D Poisson/Schrödinger simulation. In the vicinity of the QWs, special bias dependent contrast is observed and explained in terms of different current transport mechanisms. Under specific bias, the depletion regions in the barriers adjacent to the well could be resolved. Using low forward bias the FWHM of the resistance dips are in good agreement with the geometric QW width, indicating very low tip averaging effect.

**Contribution of the author:** measurements, analysis, writing
PAPER H: Electrical characterization of InGaAs/InP quantum wells by Kelvin probe force microscopy

This paper deals with the characterization of InGaAs/InP QWs by cross-sectional KPFM performed under UHV. The work was motivated by the ability of KPFM to perform local surface potential measurements, thus to determine band-offsets. Clear peaks in the KPFM signal (contact potential) are observed at the position of the wells. The origin of the peaks clearly demonstrates the sensitivity of the technique to carriers accumulated in the wells. QWs as narrow as 5 nm could be detected. Good consistency was observed in terms of peak magnitudes and widths between the experimental results and 1D Poisson/Schrödinger simulation. The influence of both tip averaging effect and the presence of surface/interface states is also discussed in the paper.

This work was accomplished in collaboration with the Hahn-Meitner Institute (HMI), Berlin, Germany in the scope of the 5th Framework European Research Training Network project HERCULAS. The KPFM measurements were performed by the candidate at HMI during a three month stay.

Contribution of the author: measurements, analysis, writing

PAPER I: Determination of spatial resolution in AFM-based electrical characterization techniques using quantum well structures

This paper proposes a new procedure to determine the “electrical” spatial resolution of AFM-based electrical characterization methods. This procedure is based on the characterization of QWs and is applied in this paper to SCM and SSRM. Sub-5 nm and sub-30 nm spatial resolution are reported for SSRM and SCM, respectively. The influence of parameters such as the tip-sample bias and tip averaging effects is discussed. The appropriate measurement conditions for performing high resolution measurements are then highlighted. The measurements were performed using commercial probes. The influence of probe-related parameters on the spatial resolution is discussed in terms of tip radius and reproducibility of the tip-sample contacts and indicates that better resolution can be obtained with improved probes. Finally, it is proposed that such a procedure can also be applied for probe evaluation and development.

Contribution of the author: measurements, interpretation, writing
Chapter 10

Conclusion

10.1 Accomplished work

The recent developments of novel scanning probe techniques used in this thesis have been mainly motivated by the urgent need for 2D mapping of the electrical properties of semiconductor devices with high spatial resolution. Due to their nanoscale spatial resolution, their high sensitivity across a wide doping range and their versatility, SCM, SSRM and KPFM have been recognized to be suitable techniques. While the Si technology was the main driving force in the development of these techniques, SCM, SSRM and KPFM are increasingly being employed in III-V research and development. One recognizes however that further understanding and improvements can still be obtained with these techniques irrespective of whether it is III-V or Si technology. Specific issues related to these methods are: (i) ability to perform nanoscale electrical characterization of advanced semiconductor devices; reliability, reproducibility and quantification of the measurements, (ii) spatial resolution of the techniques and (iii) exploration of new application areas. This thesis addressed all these major issues using III-V based materials and devices, investigating in the mean time issues specific to III-V materials and devices technology.

The ability of SCM to perform nanoscale electrical characterization of advanced optoelectronic devices was demonstrated using edge-emitting GaAs-based BH-lasers. The inherent sensitivity of the technique to carriers rather than dopants, the specific signature obtained across different structures (p-n junction, heterojunctions...) and the bias dependent contrast allowed SCM to perform detailed electrical mapping of the devices and provided relevant information likely to play an important role in device development. Crucial issues such as the evaluation of the electrical confinement of the mesa have been successfully addressed through the characterization of the regrowth process, i.e., the characterization of the regrown interfaces and the verification of semi-insulating properties in the regrown layers. Finally, the presence of local band-bending have also been revealed and characterized. The results were compiled in Papers B and C.

Two main issues must be considered to perform quantitative SPM measurements. The first one is the measurement accuracy and reproducibility. The present investigations (Papers F and H) confirm high sensitivity of SCM and KPFM to the surface properties of the sample (surface states and oxide charges). Formation of high quality surface oxides and/or surface passivation methods for III-V materials are interesting issues to be considered in the future. In SSRM, the control of the tip-sample contact was confirmed to be the key factor for reproducible measurements, specifically in terms of contact diameter and tip load (see Paper I). The second important consideration for quantification is 3D modeling and simulation for each method. The application of the annular ring model to understand the SCM measurements on QW structures represents a step in this direction.
The versatility of SCM, SSRM and KPFM was further demonstrated addressing typical III-V materials related issues. Concerning material processing, ion beam induced damage in InP and the mechanism of damage recovery upon annealing were successfully characterized by SCM and SSRM (see Papers D and E). The added value in this study is the acquisition of “local” electrical properties of the modified materials. The investigations towards new type of carrier confinements were made by characterizing InP/InGaAs QWs with SCM, SSRM and KPFM (Papers F, G and H). The detection of carriers accumulated in the well was demonstrated and the potential of these methods for determining band-offsets was highlighted. A new method of determination of “electrical” spatial resolution in SCM, SSRM and related techniques was proposed and is based on structures with carrier confinement (see Paper I). The reliability of the method has been demonstrated for SCM and SSRM. Sub-5 and sub-30 nm lateral resolution were determined for SSRM and SCM, respectively, using commercial probes. Furthermore, the investigations provide insights on the experimental conditions necessary for high resolution measurements.

Finally, the detailed studies using the three different techniques allowed us to perform fruitful comparisons between SCM, SSRM and KPFM, pointing out the advantages and limitations of each method and thereby evidencing major directions for future work.

10.2 Future work

In terms of device characterization, new structures involving new materials and lower dimensions will always be challenging issues for SCM, SSRM and KPFM. Quantitative nanoscale mapping of the electrical properties appears as the next and ultimate step to turn these techniques into standard characterization tools. Having such a capability on a 5-100 nm length scale and in one or two dimensions may be sufficient for several applications. Pushing the resolution limits further down is a stiff challenge involving both developments of new probes and improvements on the detection system. One foresees such effort to be very important since mapping information on 2D may not be sufficient for 3D nanoscale devices, examples of which include semiconductor nanowires, carbon nanotubes, etc.

The characterization of ion beam induced damage demonstrated the suitability of SCM and SSRM to evaluate III-V based processing technology. Similar investigations on 3D micro-structures (for example mesas, trenches, etc.) constitute a challenging task, probably requiring new probe designs.

The characterization of low-dimensional systems opened the doors to address new issues. If the detection of carriers confined in the wells was demonstrated, much remains to be done to quantify carrier distributions. Higher spatial resolution using sharper probes may enhance the quality of the measurements and provide further information. Similar investigation on other low-dimensional systems such as quantum wires, quantum dots or more elaborated structures is an obvious near future objective. Ultimately, specific materials properties such band offsets are expected to be derived from the measurements.

Finally, the maturity acquired within the past ten years or so makes these techniques suitable to cope with a wider application range. Characterization of new functional materials may deserve particular attention. Meanwhile, although not obvious at
present, the feasibility of these or related electrical methods for addressing issues in biology is probably also worthwhile to be explored.
Appendix A

Measurement techniques: summary and comparison

Table A-I summarizes the main characteristics of the different SPM techniques for the electrical characterization of semiconductor devices. This includes the modes of scanning, the types of probes required and the measured quantities. The techniques can then be classified in three main categories depending on the measured quantity: current (SSRM, TUNA, STM), capacitance (SCM) and potential (KPFM). In this thesis, one technique of each category (SCM, SSRM and KPFM) has been described (Ch. 3-5). Table A-II summarizes the different types of contrast observed in these three different techniques.

Table A-I: Summary of the different scanning probe microscopy techniques which can be used for 2D carrier profiling. The “feedback” refers to the measured parameter to keep the tip-sample distance constant.

<table>
<thead>
<tr>
<th>Technique</th>
<th>Feedback</th>
<th>Probe tip</th>
<th>Measured quantity</th>
</tr>
</thead>
<tbody>
<tr>
<td>Scanning tunneling microscopy/spectroscopy (STM/STS)</td>
<td>Current</td>
<td>Metallic needle</td>
<td>Tunneling current I-V</td>
</tr>
<tr>
<td>Scanning capacitance microscopy/spectroscopy (SCM/SCS)</td>
<td>Force</td>
<td>Metal coated Si tip</td>
<td>Differential capacitance C-V</td>
</tr>
<tr>
<td>Scanning spreading resistance microscopy/spectroscopy (SSRM/SSRS)</td>
<td>Force</td>
<td>Diamond coated Si tip</td>
<td>Spreading resistance I-V</td>
</tr>
<tr>
<td>Kelvin probe force microscopy (KPFM)</td>
<td>Force gradient</td>
<td>Metal coated Si tip</td>
<td>Surface contact potential</td>
</tr>
<tr>
<td>Conductive/ tunneling atomic force microscopy (C-AFM/TUNA)</td>
<td>Force</td>
<td>Metal coated Si tip</td>
<td>Current I-V</td>
</tr>
<tr>
<td>Scanning surface harmonic microscopy (SSHM)</td>
<td>Current</td>
<td>Metallic needle or microwave cavity</td>
<td>Differential capacitance</td>
</tr>
<tr>
<td>Doping sensitive etching/staining and atomic force microscopy (DSE/DSS+AFM)</td>
<td>Force or force gradient</td>
<td>Sharp Si or SiN, tip</td>
<td>Topography after chemical etching/staining</td>
</tr>
</tbody>
</table>
Table A-II: Summary and intercomparison of SCM, SSRM and KPFM techniques in terms of physical principles, detection system and contrast in various contexts encountered in semiconductor structures and devices.

<table>
<thead>
<tr>
<th>Physical principles</th>
<th>SCM</th>
<th>SSRM</th>
<th>KPFM</th>
</tr>
</thead>
<tbody>
<tr>
<td>Measured quantity</td>
<td>Differential capacitance</td>
<td>Spreading Resistance</td>
<td>Contact potential</td>
</tr>
<tr>
<td>Tip-sample system</td>
<td>MOS contact</td>
<td>Schottky contact</td>
<td>Electrostatic interaction</td>
</tr>
<tr>
<td>Tip dc-biasing</td>
<td>Positive: accumulation (n) inversion (p) Negative: inversion (n) accumulation (p)</td>
<td>Positive: forward (n) reverse (p) Negative: reverse (n) forward (p)</td>
<td>Equal to the CPD (nullify the electric field)</td>
</tr>
<tr>
<td>Default bias</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Detection system</td>
<td>dC/dV (open loop)</td>
<td>Resistance (open loop)</td>
<td>CPD (closed loop)</td>
</tr>
<tr>
<td>Modes</td>
<td>Default mode</td>
<td>Default mode</td>
<td>Default mode</td>
</tr>
<tr>
<td>Tip-sample distance control</td>
<td>Cantilever deflection</td>
<td>Cantilever deflection</td>
<td>Frequency or amplitude shift</td>
</tr>
<tr>
<td>Contrast in default mode and bias</td>
<td>n-doping</td>
<td>high doping: lower signal</td>
<td>low doping: higher signal</td>
</tr>
<tr>
<td>Doping profile</td>
<td>p and n-doping</td>
<td>High doping: low signal</td>
<td>Low doping: high signal</td>
</tr>
<tr>
<td>Inverse variation with doping</td>
<td>p-doping</td>
<td>high doping: higher signal</td>
<td>low doping: lower signal</td>
</tr>
<tr>
<td>p-n junctions</td>
<td>Opposite Schottky characteristic between p- and n-regions</td>
<td>Large resistance difference</td>
<td>Large CP difference</td>
</tr>
<tr>
<td>Zero signal</td>
<td>n-doping: CP-Eg</td>
<td>p-doping: CP-Eg</td>
<td>A(CP)-Eg</td>
</tr>
<tr>
<td>Doping steps</td>
<td>n-doping: CP-Eg</td>
<td>p-doping: CP-Eg</td>
<td>Large CP difference</td>
</tr>
<tr>
<td>Heterojunctions</td>
<td>Smooth (Debye length and tip averaging effect)</td>
<td>Abrupt (tip-averaging effect)</td>
<td>Abrupt (tip-averaging effect)</td>
</tr>
<tr>
<td>QWs</td>
<td>Non abrupt (tip-averaging effect)</td>
<td>Abrupt (geometry of the tip apex)</td>
<td>Abrupt (tip-averaging effect)</td>
</tr>
<tr>
<td>Wide dip</td>
<td>QW location: low signal (carrier accumulation)</td>
<td>QW location: low signal (carrier accumulation)</td>
<td>QW location: high signal (built-in potential)</td>
</tr>
<tr>
<td>Narrow dip</td>
<td>Depletion regions: high signal</td>
<td>Depletion regions: high signal</td>
<td>Depletion region: high signal</td>
</tr>
<tr>
<td>Deep levels with</td>
<td>Zero signal</td>
<td>Zero signal</td>
<td>Zero signal</td>
</tr>
<tr>
<td>Fermi level pinning (Si, surface states)</td>
<td>No variation with ac bias (stretched C-V curves)</td>
<td>No variation with dc bias (no spreading current)</td>
<td>No signal variations CP-Eg</td>
</tr>
</tbody>
</table>

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Appendix B

Probe technology

B.1 Probe design

The recent breakthrough of AUM-based techniques has tremendously encouraged the development of a wide variety of probes. Probes typically consist of a sharp tip attached to a cantilever spring. During scanning across the sample, the cantilever deflects due to the force acting between the tip at the extremity of the cantilever and the sample. The magnitude of the cantilever deflection depends on the cantilever spring constant \( C \). Typically, \( C \) varies between 0.01 and 50 N/m in most applications. For a rectangular cantilever the cantilever spring constant \( C \) and the resonance frequency \( f_0 \) are expressed as:

\[
C = \frac{E}{4} \frac{w \cdot t^3}{l^3} \quad \text{(Eq. B-1)}
\]

\[
f_0 = 0.162 \sqrt{\frac{E}{\rho}} \frac{t}{l^2} \quad \text{(Eq. B-2)}
\]

where \( E \) is the Young’s modulus, \( \rho \) the density and \( t \), \( w \) and \( l \) the thickness, width and length, respectively, of the cantilever [1]. The resonance frequency should be high to avoid excitation of vibrations by ambient noise and to allow sufficiently high scan rates. Typically \( f_0 \) lies between 10 to 500 kHz. A high ratio of \( f_0/C \) can be achieved by simply reducing the size of the cantilever. Typically, the cantilever length lies between 80 and 500 \( \mu \)m, the width between 20 and 50 \( \mu \)m and the thickness between 0.5 and 5 \( \mu \)m [2].

B.2 Fabrication processes

Microfabrication utilizes well-known process technology together with additional etching techniques. The cantilevers can be fabricated in two different ways. The first one uses thin films deposited onto a patterned silicon substrate. The cantilever is defined by lithography and etched into the substrate by wet or dry etching, followed by selective removal of the silicon underneath the cantilever [1]. In the second fabrication method, the cantilever is micromachined out of the bulk silicon. The cantilever is patterned by lithography and released by a selective etching process [1,3]. The critical factors are the control of the cantilever thickness and the spatial resolution of the lithography process.

The tip is the most crucial part of the probe. High resolution characterization of patterned surfaces requires a well-defined tip with known cone/pyramid angle and sharp tip apex. Tips can be either prepared using structured silicon substrate as a mold or etched out of the silicon wafer. In the first case, a pit is formed in the silicon substrate and the tip material is deposited in the resulting mold. The mold is then removed by wet etching. Improvements in the tip shape can be obtained depositing additional film into the pit prior to the molding process [4]. In the second case, the fabrication procedure starts with the
deposition of a circular mask. The mask is then undercut until it is completely under etched using anisotropic etchant. The etching is stopped and a silicon cone/pyramid remains [1].

Finally, as the cantilever is too small to handle, it is connected with a stiff macroscopic silicon holder. The design of the holder is usually incorporated in the mask layout. The holder is large enough to provide for mounting the probe in the microscope. The tip, cantilever and holder can then be made out of one single material. This avoids any strain associated with thin film deposition, has a lower mechanical fatigue and provides high mechanical quality factors (Q) [1].

### B.3 Some specific properties of the probes

The well-established probe fabrication processes described above provide today a large variety of probes, in terms of geometry, material and coating suitable to various SPM techniques [2]. The main characteristics of the commercial probes used in SCM, SSRM, KPFM and AFM measurements are listed in Table B-1.

<table>
<thead>
<tr>
<th>Technical data</th>
<th>Tapping mode AFM</th>
<th>Contact mode SCM Non-contact mode KPFM</th>
<th>Contact mode SSRM</th>
</tr>
</thead>
<tbody>
<tr>
<td>Thickness</td>
<td>3.0- 5.0 μm</td>
<td>2.0- 4.0 μm</td>
<td>3.0- 5.0 μm</td>
</tr>
<tr>
<td>Mean Width</td>
<td>22.5-37.5 μm</td>
<td>20-35 μm</td>
<td>22.5- 37.5 μm</td>
</tr>
<tr>
<td>Length</td>
<td>115-135 μm</td>
<td>215-235 μm</td>
<td>115-135 μm</td>
</tr>
<tr>
<td>Force Constant</td>
<td>10-130 N/m</td>
<td>0.5- 9.5 N/m</td>
<td>10-130 N/m</td>
</tr>
<tr>
<td>Resonance Frequency</td>
<td>204- 497 kHz</td>
<td>45- 115 kHz</td>
<td>204- 497 kHz</td>
</tr>
<tr>
<td>Tip coating</td>
<td>–</td>
<td>PtIr, or CoCr</td>
<td>p-doped diamond</td>
</tr>
</tbody>
</table>

The AFM measurements were performed using a n+ doped etched Si probe. The probe type combines high operation stability, high sensitivity, high Q and fast scanning ability. The typical tip radius is lower than 10 nm. The high doping of the tip is used to avoid charging. A representative SEM image of a tapping-AFM probe is given in CH. 2, Fig. 2.2. Probes with specific geometry (high aspect ratio, etc.) are also available for other specific dimension metrology purposes.

The SCM and KPFM measurements were performed using an AFM probe metal-coated with PtIr. The metal coating increases the conductivity of the tip and allows electrical contact. The coating is an approximately 25 nm thick layer of PtIr [2]. The back side of the cantilever is also metal-coated and enhances the reflectivity of the laser beam by a factor of 2 or so and prevents light from interfering within the cantilever. The natural bending of the cantilever lies below 2°. The relatively low spring constant of the cantilever makes it suitable for both contact mode (SCM) and non-contact mode (KPFM) operations. A representative SEM image of the probe is given in CH. 8, Fig. 8.5 (a). Full metal probes can also be used for SCM and KPFM [5].

The SSRM measurements were performed using an AFM probe coated with p+ doped diamond. This probe combines high stability, hardness and electrical conductivity required for this type of measurements. The thickness of the diamond coating is around 100 nm. The nature of the contact however depends on the contact
force. To improve the reflectivity of the laser beam, the detector side of the cantilever is covered with an aluminum coating. A representative SEM image of the probe is given in CHR. 8, Fig. 8.5 (b). One limitation of this probe lies in the polycrystalline nature of the coating. The manufacturing of full diamond probes appears as promising candidate for the future generations of SSRM probes [6,7].

References