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Scanning tunneling luminescence on self-assembled InGaAs/GaAs quantum dots

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Scanning Tunneling Luminescence on Self-Assembled InGaAs/GaAs Quantum Dots

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Abstract

Carriers are injected from the tip of an STM into a n-i-p GaAs structure containing a layer of self-assembled InGaAs quantum dots in the intrinsic region. Under typical operational conditions, the number of holes in the dots largely exceeds the amount of electrons and is estimated to be of the order 10 to 20. The number of electrons is less than one per dot. Electrons are injected ballistically from the STM tip and relax mainly due to LO phonon scattering. Subsequent transport to the dots is by drift and diffusion. The relaxation rate is determined to be $3.5 \cdot 10^{13} / s$. Radiative recombination of the excitons formed in the dots results in a spectrum, which at 4.2 K consists of some 55 lines with linewidths below 1 meV. At 77 K the emission lines are broadened due to lifetime effects. A change in tip-sample voltage from 3 V to 5 V in general does not lead to significant changes in the spectrum. The incidentally observed emergence of peaks 10 meV to the low energy side can be ascribed to additional holes bound in the dots. Spatially resolved measurements indicate highly non-uniform transport of the carriers through the semiconductor and show that only a small fraction ($\sim 10 \%$) of the dots is optically active.
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In the last few decennia, interest in low-dimensional semiconductor physics has increased dramatically. This development is fed by the ongoing trend to fabricate ever smaller devices. Decreasing the dimensions of devices to a nanoscale level leads to confinement of charge carriers. From quantum mechanics it is well known that this confinement leads to quantised energy levels in the dimension in which the confinement is achieved. Fundamental research on the properties of these quantum structures is necessary to be able to produce devices with nanoscale dimensions. For example future solid-state opto-electronics is believed to require the specific properties of quantum dots. Concurrently, the development of new measurement techniques to probe nanostructures for quantum mechanical features has boomed. The possibility to perform spectroscopy on single nanostructures, combined with the existing interest in the objects under study, has led to a flourishing field of research on spectroscopic properties of quantum structures in semiconductor materials.

A structure in which the carriers are restricted in all three dimensions is called a quantum dot. To fabricate such a dot, different growth techniques can be used. One of the most common techniques is a self-organizing process called Stranski-Krastanow growth [1], which makes use of a relaxation mechanism of strained layers.

As a result of the 3D confinement, the energy levels present in quantum dots are very well defined. The lack of extra degrees of freedom for the charge carriers results in optical transitions with linewidths below 50 \( \mu eV \). As a result of these small linewidths, many transitions between different states can be resolved simultaneously. Therefore dots are good objects for studies on state filling and multi-particle states in quantum structures. Knowledge of these phenomena is necessary for the fabrication of for example more stable quantum dot lasers.
Chapter 1. Introduction

1.1 Project goal

Up to now, most studies on electronic properties of quantum dots have been done in photoluminescence (PL) experiments. In the field of quantum dot research, the scanning tunneling luminescence technique as presented in this report is a fairly new method. By using a scanning tunneling microscope (STM) to induce electro-luminescence in a dot, it should be possible to populate and probe energy levels of single dots. To enhance the quantum efficiency of the recombination process, experiments are preferably done at low temperatures.

Accordingly, the aim of this project is to use a low-temperature STM to fill self-assembled InGaAs/GaAs dots with an unequal amount of holes and electrons and study both the filling of the energy levels and the behavior of (charged) multi-particle states in single quantum dots. An additional aim is to study the carrier injection mechanism by performing position dependent measurements.

1.2 Outline report

This report discusses experiments performed with a scanning tunneling luminescence technique on a wafer containing a layer of self-assembled InGaAs/GaAs quantum dots. To be able to understand and interpret the results of these measurements, a short introduction to the physical properties of self-assembled quantum dots is given in the next chapter. The Stranski-Krastanow growth procedure is briefly explained. A short summary of the previous work done on multi-particle states in dots is also included in this chapter. Finally the structure of the sample and its band diagram at zero bias are presented.

Chapter three describes some theoretical concepts needed to understand the transport phenomena in semiconductor materials. Three different transport mechanisms are mentioned here.

In chapter four the principle of scanning tunneling luminescence is explained. First a short introduction is given to the scanning tunneling microscope. Both the tunnel process and the piezo control are described. Finally the use of the STM as a carrier injector is discussed and the band diagram of the sample at injection conditions is depicted.

The experimental setup is discussed in chapter five. For the spatially resolved measurements, a new experimental method is developed. This method is also presented here.

The results of the measurements are given in chapter six. First some general remarks about the spectrum are made. Estimates are made for both the amount of active dots in the spectrum and the mean amount of electrons and holes per dot. Temperature-, current- and voltage dependence of the spectrum are discussed. From the spatially resolved measurements some remarks are made on the spatial resolution of the STL technique and the apparent lack of symmetry in the injection mechanism.

Finally, some overall conclusions and recommendations for future work complete this report.
Chapter 2

Self-Assembled Quantum Dots

The density of states (DOS), i.e. the number of states per energy interval, changes as a result of carrier confinement. Going from no confinement in bulk semiconductors via confinement in one dimension in quantum wells and 2D confinement in wires to 3D or full confinement in dots, the density of states of the carriers changes from a continuous distribution to a $\delta$-function like behavior, as can be seen in figure 2.1.

Figure 2.1: Density of states from bulk semiconductors to dots.

This shape of the density of states causes the small linewidths of the optical transitions and makes dots extremely suitable for studies on fundamental properties of the energy level transitions. The dots under study here are self-assembled InGaAs dots embedded in GaAs.

2.1 Stranski-Krastanow growth

Quantum dots can be made in different ways. One can for example etch a quantum well to isolate dots [2]. Etching seems a simple technique, but has the disadvantage that the resulting dots usually contain many structural imperfections, especially at the side-walls. The most employed method is therefore based on a different concept: a strain induced
self-organization process which is known as Stranski-Krastanow growth. When crystalline materials with different lattice constants are grown on top of each other, the (thin) top layer will form small islands to release its strain energy. In the case of the InAs/GaAs combination (lattice mismatch ≈ 3.5%) under study here this principle works the following way (see figure 2.2).

On top of a GaAs substrate several monolayers of InAs are deposited. First a smooth but strained layer of In(Ga)As is formed. This layer is called the wetting layer. After a critical thickness of approximately 2 monolayers dependent on the growth conditions, islands of InGaAs will be formed. These islands are then covered with GaAs and a layer of self organized InGaAs quantum dots encapsulated in GaAs is the result of this procedure. In literature, the shape of these dots is still debated [3]. The InGaAs/GaAs dots used here most probably have a truncated pyramidal shape [4].

The Stranski-Krastanow method has some disadvantages for spectroscopy purposes. First, dots grown this way have a not so homogeneous size distribution. Therefore it is desirable to study single dots. Second, the typical area density of the dots is $10^{10}-10^{11}$ cm$^{-2}$ and these high densities result in interdot distances of 30-100 nm. As this distance is also the required spatial resolution in spectroscopic measurements, achieving single dot spectroscopy is not trivial. The STM with its atomic spatial resolution seems a promising tool for this purpose.
2.2 Luminescence from dots

2.2.1 Excitons and multi-exciton complexes

In a quantum dot, carriers are confined in a potential barrier in all three dimensions. This potential barrier arises from the difference in bandgap between the InGaAs dot material and the surrounding GaAs matrix. Quantisation in three dimensions occurs as soon as the dimensions of the InGaAs islands are smaller than the De Broglie wavelength of the particles. When both an electron and a hole are present in the dot, the Coulomb attraction between the opposite charged particles creates a binding between the two carriers. This bound state is called an exciton.

![Exciton formation diagram]

**Figure 2.3**: Schematic energy level diagram of InGaAs dots in GaAs. a) When an unbound electron and hole recombine, a photon can be emitted. b) Coulomb attraction between the charged particles results in formation of an exciton. The energy of the emitted photon is lowered by the exciton binding energy.

The most stable binding that can occur is the binding of a single electron to a single hole ($E_b \approx 10$ meV). Nevertheless, other more complex combinations are also possible. In the case of equal numbers of holes and electrons bound together we speak of multi-excitonic states, like bi- or tri-excitons in which respectively two electrons and two holes, or three electrons and three holes are bound together to form a single complex. These multi-exciton complexes have been under study since the early nineties but are still not fully understood. Because the binding energy of these complexes decreases with increasing number of carriers, higher order complexes are less stable and are therefore hard to detect. Besides these multi-exciton complexes, another option is the formation of charged exciton
complexes. This formation occurs when an unequal number of electrons and holes form a bound state in the dot. In a PL experiment, these charged excitons are not likely to occur, because the laser light induces electrons and holes pairwise. In such an experiment the probability for the formation of a charged exciton is therefore much lower than that of a complex with an equal amount of electrons and holes.

Excitons and exciton complexes can recombine in two different ways: radiatively and non-radiatively. Non-radiative recombination results in the emission of phonons, quanta of lattice vibration. Radiative recombination yields a photon. In principle, the energy of this photon equals the difference between the energy levels the individual carriers occupied before recombination.

2.2.2 Previous work

The observation of narrow emission lines from isolated quantum dots was first reported in 1994. Since then, amongst other techniques, cathodoluminescence (CL), photoluminescence (PL) and scanning near field optical microscopy (SNOM) experiments have been done to unravel the physics of quantum dots.

Temperature dependent measurements have shown that the optical linewidth is homogeneously broadened. The linear temperature dependence found at temperatures above 25 K can be described with an acoustic phonon scattering mechanism [5]. Up till now, the linewidth could not be resolved experimentally in the limit of low temperatures.

In the last few years, multiparticle effects in dots have been studied in some detail. PL measurements have shown that with increasing excitation power extra lines appear in the spectrum, which can be assigned to bi- or even tri-excitonic states [6]. Kamada et al. [7] studied disk shaped InGaAs dots and found a bi-exciton emission energy of approximately 5 meV below the single exciton ground state. This result is in reasonable agreement with the 3 meV shift to the low energy side observed in InGaAs/GaAs dots as reported by Chavez-Pirson et al. [8] and the binding energy of 3.1 meV found in In_{0.60}Ga_{0.40}As/GaAs dots by Kuther et al. [9].

Besides the occurrence of uncharged multi-exciton states, luminescence spectra from dots have also revealed features that can be ascribed to charged exciton complexes [10]. The first to report the observation of photoluminescence from both positive and negative charge states of a single dot were Regelman et al. [11]. They found that negative charging reduces the emission energy relative to the neutral case, and positive charging increases it. The explanation given in this paper is that the hole wavefunction is more spatially localized than the electron wavefunction. As the binding energy $E_b(\alpha, \beta)$ ($\alpha, \beta = e$ or $h$) is proportional to $< \psi_\alpha | \frac{1}{r} | \psi_\beta >$, the inequality $| E_b(\text{hh}) | > | E_b(\text{eh}) | > | E_b(\text{ee}) |$ holds. As $E_b(\text{hh})$ and $E_b(\text{ee})$ are negative and $E_b(\text{eh})$ is positive, the binding energy is increased (i.e. more negative) with an increasing number of holes and decreased with an increasing number of electrons.
2.3 Sample design

2.3.1 Structure and characterisation

The sample design is taken from Markmann et al. [12] and is depicted in figure 2.4. The InGaAs quantum dots are grown on the (100) GaAs plane in the intrinsic region of a p-i-n structure. Holes are injected from the p-doped layer below the intrinsic GaAs when a positive bias is applied to the surface. The sample is capped with an n-doped layer. The high doping level in this layer is mainly meant to decrease the Schottky barrier between the STM tip and the sample. The resulting thin barrier should make it possible to inject electrons with a low excess energy through the surface band bending region. A low energy is preferred to improve the spatial resolution. Transmission electron microscopy measurements on similar samples showed a dot density of approximately $10^{10}/cm^2$ [13].

Room temperature PL spectrum

A room temperature PL spectrum taken at the university of Sheffield, where the sample is grown, shows that the GaAs and InGaAs peaks occur at wavelengths well below the quantum dot luminescence features (see figure 2.5). The dots emit light around 1000 nm (1240 meV), whereas the GaAs and InGaAs wetting layer peak are found to be at 876.3 nm.
2. Self-Assembled Quantum Dots

PL spectrum M2247 at room temperature (University of Sheffield)

12/03/2001
wavelength 633.0 nm
power 16.2 mW
grating 300 l/mm
slit width 0.200 mm

PL spectrum M2247 at room temperature (TU/e)

29/05/2002
wavelength 532 nm
power 250 mW
grating 100 l/mm
slit width 0.100 mm

Figure 2.5: Two PL spectra on wafer M2247 at room temperature, taken by different institutes. a) The GaAs peak is visible at 876.3 nm. To the right, at 920 nm the InGaAs wetting layer is visible. The dots are seen around 1000 nm. b) In the spectrum we measured, only the GaAs peak can be seen. The position of this GaAs peak is determined at 873 nm.

and 920 nm respectively. However, the PL spectrum taken at the TU/e shows a slightly different result: even at 10 times higher excitation power, only the GaAs peak is observed. The position of this peak is in both cases approximately the same. The reason that neither the InGaAs wetting layer nor the dots are visible in this spectrum, is probably because they are outside the sensitive range of the used CCD camera.

The peak positions at room temperature can not be used to predict the position of the quantum dot lines in the low temperature measurements. As the experiments are done at liquid nitrogen and liquid helium temperatures, the spectra obtained in the STL experiments are blueshifted with respect to these PL spectra. A PL spectrum taken at 4.2 K is given in chapter 6 for comparison.

2.3.2 p-i-n diodes

As the quantum dots under study are embedded in a p-i-n structure, some remarks have to be made about the luminescence properties of p-i-n diodes [14]. Usually, p-i-n- diodes are used as photodetectors. This means that light is shone on the diode (operated in reverse bias for this purpose), electrons and holes are created and the applied electric field collects the carriers. A photocurrent is the result. This photocurrent can be expressed as

\[ I_L = eA \int_0^{x_d} G_L(z)dz, \]  

(2.1)
2.3. Sample design

with \( A \) the area, \( z_d \) the depletion width and \( G_L \) the carrier generation rate. In this equation, the only factor that depends on the applied bias is the depletion width \( z_d \), which is given by the equation

\[
z_d = \sqrt{\frac{2\varepsilon_r(\varphi_i - V_{surf})}{q^2 N_d}}, \tag{2.2}
\]

with \( \varphi_i \) the built-in potential and \( N_d \) the donor concentration in the n-doped layer.

In a normal light emitting diode (LED) the photon current (number of photons per second) as a function of the current \( I \) is given by

\[
I_{ph} = \eta_{tot} \cdot \frac{I}{e}. \tag{2.3}
\]

Here \( \eta_{tot} \) is the total quantum efficiency of the recombination process. At very high injection currents, the device starts to heat up. As a result, the radiative recombination efficiency decreases and the light output starts to saturate. But until this effect occurs, the amount of photons emitted is linear dependent on the current. No explicit statement on the bias dependence of \( \eta_{tot} \) is found in literature.
Chapter 3

Carrier Transport

The STL setup is operated such that electrons are injected from the tip and holes from the back contact. Therefore the hole transport mechanism is drift directed by the applied electric field. Three different mechanisms play a role in the transport of the electrons to the quantum dots after leaving the STM tip and tunneling through the vacuum barrier between tip and sample. In first instance the electrons will travel ballistically through the sample. However, before being able to be trapped by the dot, they will have to relax to the bottom of the conduction band. After this relaxation process, drift and diffusion [15] of the electrons can eventually drive them towards the dot and there the recombination and resulting electro-luminescence process can occur.

3.1 Ballistic electrons

When we assume only injection into a single conduction band and no injection into the valence band, a straightforward method can be used to calculate the scattering rate. If we assume the scattering rate $\Gamma_s$ to be independent of energy, then the number of unscattered electrons $n_b$ as a function of depth $z$ is given by [16]

$$\frac{dn_b(E, k)}{dz} = -\frac{n_b(E, k)\Gamma_s}{v(E, k)}.$$ (3.1)

Here $v$ is the group velocity of the electrons, which in lowest order depends on the bias at the sample surface via

$$v = \sqrt{\frac{2e(V_{surf} - V_0)}{m^*}},$$ (3.2)

with $V_0$ the barrier height and $m^*$ the effective mass of the conduction band. From these equations, the equality

$$n_b(E, k) = \exp \left[ -\frac{\sqrt{m^*/2} \Gamma_s z}{\sqrt{e(V_{surf} - V_0)}} \right].$$ (3.3)
follows directly. The scattering rate can now be determined from the luminescence intensity $L$ of the GaAs peak via

$$L(V) \propto I_t \left( \exp \left[ -\frac{\sqrt{m^*/2}}{\Gamma_s z_0} \right] \right),$$  \hspace{1cm} (3.4)

with $I_t$ the tunnel current. As the carriers that are scattered in the intrinsic region are assumed to recombine in the dots, GaAs luminescence only occurs in the p-doped layer below the intrinsic region. Therefore $z_0$ in equation 3.4 is the position of the p-doped GaAs layer. A measure for the typical distance of the ballistic path is the mean free path of the electrons in the GaAs material, which can be calculated from equations 3.4 and 3.2:

$$\lambda_{mfp} = \frac{v}{\Gamma_s}.$$  \hspace{1cm} (3.5)

### 3.2 Relaxation mechanisms

Relaxation of the ballistic electrons to the conduction band occurs through one or more scattering events. Electrons scatter mainly from ionized impurities, other electrons or phonons. Electron-electron scattering is not considered here, because the total momentum of the carriers is not changed by collisions between them.

Ionized impurity scattering originates from the charged donors or acceptors left behind after releasing electrons or holes. As the electrons are injected through a severely n-doped layer, scattering from donors will take place here. However, because the mass of the scattered electrons is much smaller than the mass of the ionized donors, the energy loss of the electrons will be small and relaxation will hardly occur as a result of this process. Almost all of the scattering events therefore take place in the large intrinsic region (since the n-doped top layer is much thinner) and are caused by phonon scattering.

Phonons are lattice vibrations, which induce electric and magnetic fields due to the motion of the ions in the lattice. These fields scatter the electrons passing them. Four different types of phonons can be distinguished: transverse and longitudinal acoustic (TA and LA) and transverse and longitudinal optic (TO and LO) phonons. The influence of transverse phonons is negligible due to symmetry considerations [17] and as LA phonons do not carry away much energy (typically < 5 meV), LO phonons are the dominant scattering centers. The corresponding scattering time is approximately $10^{-13}$ s [14].

### 3.3 Drift

Drift of carriers is driven by an external electric field resulting from the applied bias. The drift current $J_{\text{drift},n}$ in its simplest form is given by

$$J_{\text{drift},n} = e n \mu_n E.$$  \hspace{1cm} (3.6)
3.4. Diffusion

The resulting drift velocity is then given by the relation

\[ v_d = \mu_n E, \]  

(3.7)

with \( \mu_n \) the mobility of the electrons, in GaAs about \( 0.85 \frac{m^2}{V\cdot s} \) at room temperature. Mobility is a measure of the ease of carrier motion within the semiconductor. As the amount of scattering decreases with decreasing temperature, the mobility of the carriers will increase with decreasing temperature.

3.4 Diffusion

Carriers migrate through the semiconductor as a result of their random thermal motion. When a concentration gradient is present, a net current will result, which will eventually tend to produce a uniform distribution. For the electrons, this so-called diffusion current is given by

\[ J_{\text{diff},n} = e D_n \nabla n, \]

(3.8)

with \( D_n \) the diffusion coefficient (defined as the product of the mean free path and the thermal velocity of the electrons) and \( \nabla n \) the electron concentration gradient. The temperature dependence of the diffusion process is incorporated in the temperature dependence of the thermal velocity and thus the diffusion coefficient. In equation 3.9 the corresponding diffusion length is given:

\[ L_n = \sqrt{D_n \tau_n}, \]

(3.9)

with \( \tau_n \) the recombination time.

Under equilibrium conditions, the drift and diffusion currents exactly cancel and the diffusion coefficient can be related to the mobility by the Einstein relationship (for the derivation of this relation, see appendix A)

\[ \frac{D_n}{\mu_n} = \frac{kT}{e}. \]

(3.10)
Chapter 4

Scanning Tunneling Luminescence

4.1 Scanning Tunneling Microscope (STM)

Since its invention in the early eighties, the STM has extensively proven its worth for fundamental research projects. The basic principle behind the microscope is the ability of electrons to tunnel through a region where classically no current is possible. Quantum mechanics describe that a current starts flowing between two conducting objects as soon as their carrier wavefunctions overlap. This overlap and thus the tunnel current increases exponentially with decreasing distance according to

\[ I \propto \exp(-2\kappa z), \quad \kappa = \frac{(2m_0\bar{\phi})^{1/2}}{\hbar}. \]  

(4.1)

Here \( m_0 \) is the free electron mass and \( \bar{\phi} \) is the average tunnel barrier height.

![STM tip with XYZ piezo elements](image)

Figure 4.1: STM tip with XYZ piezo elements. A voltage is applied between the STM tip and the back contact of the sample.

To achieve a controlled tunnel process, the most important part of an STM is a very sharp tip that can be moved at sub Ångström precision in the horizontal and at picometer
Chapter 4. Scanning Tunneling Luminescence

precision in the vertical directions by piezo elements (see figure 4.1). When the tip is positioned at a distance of several Ångströms from the surface of a conducting sample and a bias is applied between the two, a tunnel current will flow between the tip and the sample. At low temperatures where $k_B T$ is smaller than the required energy resolution and at constant tip-sample distance, the tunnel current can be approximated by the Bardeen formula given in equation 4.2. This equation is merely a convolution of the DOS of the sample $\rho_S(E)$ and that of the tip $\rho_T(E)$:

$$I \propto \int_0^{eV} \rho_S(E_F - eV + \epsilon)\rho_T(E_F + \epsilon)d\epsilon. \quad (4.2)$$

Since the current depends exponentially on the distance between the tip and the sample, as is given in equation 4.1, a topographic image of the surface can be made by keeping the tunnel current constant. In normal operation, a feedback loop keeps the tunnel current constant by permanently correcting the voltage on the z-piezo as the tip moves across the surface. The voltage that has to be applied to the z-piezo is a measure of the height difference of the sample surface. This way topographic images at atomic scale resolution can be made.

4.2 STM as carrier injector

Making topographic images is one possible application for an STM, but since electrons tunnel from the tip to the sample, it can just as well be regarded as a carrier injector. Usually quantum dots are studied by focussing a laser spot onto the sample. This laser light creates electrons and holes. The emitted light resulting from the recombination of these particles is detected. This technique is called photoluminescence and has been employed with great results in the last few decades. However, the laser spots used can not be focussed to areas smaller than $\frac{\lambda}{2}$ (typically tenths of microns) as a result of diffraction effects. Since this area is larger than the interdot distance, the technique requires masks or mesa’s to be able to look at single dots. As an STM has a well defined injection spot, which is reflected by the atomic resolution of the tunnel process, it should be possible to perform single dot spectroscopy with an STM. Another advantage of the scanning tunneling luminescence (STL) method is that it is very easy to fill the dots with unequal amounts of holes and electrons and by doing so create charged exciton complexes.

4.2.1 Bias effects

As is explained in section 2.3, the dots under study are embedded in a p-i-n structure. To be able to inject carriers into the quantum dot layer, the structure is used under forward bias. When a bias is applied, the band diagram as depicted in figure 2.4 changes. In figure 4.2, three different regimes can be distinguished:

1. At zero bias, the valence band lies entirely below the Fermi level. The hole states in the dots are not occupied.
4.2. STM as carrier injector

Figure 4.2: Change in band diagram as a function of applied voltage. At 0.64V ($V_{\text{inv}}$ at 4.2K), the valence band is bent above the Fermi level and an accumulation layer is formed. At 0.87V ($V_{\text{fill}}$ at 4.2K), the valence band at the dots is pulled above the Fermi level. As a result, holes are injected into the dot from the substrate. In the experiments, the maximum tip bias is 5V. The calculated band diagram at this tip potential shows a Schottky barrier height of approximately 3V.

2. Applying a forward bias to the p-i-n structure pulls both the conduction band and the valence band upwards. At a certain voltage $V_{\text{acc}}$ the surface valence band passes the Fermi level. As a result, an accumulation layer is formed. This accumulation layer effectively screens most of the potential above $V_{\text{acc}}$ for the deeper layers and causes an exponential shape of the conduction and valence band near the sample surface.

3. Increasing the voltage even further, at a critical voltage $V_{\text{fill}}$ the valence band in the dots is also pulled above the Fermi level. As soon as this happens, the hole states
in the dot become occupied. The amount of holes in the dot is determined by the applied voltage, as this voltage determines how far the band is pulled above $E_F$.

The energy of the injected electrons is influenced by a change in voltage, because their kinetic energy equals the difference between the potential energy at the tip and the conduction band energy. Both factors depend on the applied bias.

The electron injection rate equals the tunnel current.

The potential at each point inside the semiconductor can be calculated from the electric field $E$ or the carrier density $\rho(z)$ by Poisson's equation

$$\frac{\partial^2 V}{\partial z^2} = \frac{\partial E}{\partial z} = -\frac{\rho(z)}{\varepsilon_0 \varepsilon_r}. \tag{4.3}$$

For the p-i-n structure under study, $V_{\text{acc}}$ and $V_{\text{fill}}$ are calculated with a computer program called HETMOD. This program calculates a self consistent solution to Poisson's equation for a given temperature, doping concentration and applied bias. Also the influence of the surface accumulation layer and the depletion regions are incorporated in the program. The calculations yield the following values:

<table>
<thead>
<tr>
<th>(T)</th>
<th>4.2 K</th>
<th>77 K</th>
</tr>
</thead>
<tbody>
<tr>
<td>$V_{\text{acc}}$</td>
<td>0.64V</td>
<td>0.60V</td>
</tr>
<tr>
<td>$V_{\text{fill}}$</td>
<td>0.87V</td>
<td>0.80V</td>
</tr>
</tbody>
</table>
Chapter 5

Experimental Setup

5.1 STM and cryostat

To inject carriers into the quantum dots, a home-built low temperature STM is used [18], which is depicted in figure 5.1. For the course approach of the tip to the sample in the $z$ direction, a differential screw is used. As soon as a tunnel current starts to flow, the tip-sample distance is further controlled electronically by a computer control unit. The measured tunnel current is compared to the preset value and the difference is led to the feedback system, which adjusts the voltage on the $z$ piezo if necessary. Movement in the $x$-$y$ plane is controlled by applying a voltage to the $x$-$y$ piezo element.

Because the tunnel current is very sensitive to external vibrations, several arrangements are used to isolate the STM from these vibrations. First, the STM itself is designed to be very stiff. Second, the STM is inserted in a cryostat that is held by a metal frame standing on four air legs. And third, the long stick that holds the STM is an effective low-pass filter.

The STM is contained in a stainless steel tube which is filled with high-purity helium contact gas. The helium serves different purposes, such as preventing unpassivated samples from oxidizing during experiments and providing a thermal coupling to the main bath. The used cryostat is a superinsulated system with an insert separated from the main bath by a vacuum shield. The STM is mounted in the insert and the main bath is filled with liquid nitrogen or helium. In figure 5.1 a schematic diagram of the setup is given.

5.2 Optical detection

Because the cryostat used also contains a superconducting magnet and the STM is inserted in the bore of this, there is no direct line of sight from the outside to the tip-sample region. Therefore light collection is done with optical fibers, which are aimed at the tunneling contact. The fibers have a core diameter of 600 $\mu$m and a numerical aperture of 0.37. Focussing is done by positioning the fibers by hand, but since the area of the fiber is the largest limiting factor in the light collection, slight misalignments cause no significant loss
Chapter 5. Experimental Setup

in light collection. A 30 cm monochromator disperses the light on a nitrogen cooled silicon CCD camera with an enhanced sensitivity in the near infrared. WinSpec software is used to measure and analyze the optical spectra.

5.3 Sample preparation

To prevent the samples from oxidizing during mounting, the following passivation procedure is used:

1. Etch for 30 s in a 1:10 diluted HCl solution.

2. Dip for 2 min in NaS₂ solution for sulfur passivation.
3. Flush with distilled water and blow dry with nitrogen.

4. Anneal 10 min at 400 °C under protective atmosphere of nitrogen.

After this passivation procedure, the samples are stable against oxidation for at least several weeks. However, the passivation procedure forms a surface layer with unknown electronic properties. To exclude the effects of this surface layer, samples 2247E through 2247K have not been passivated. Prior to the measurements the oxide layer on these samples has been etched away by a 5-second etch in 1:10 diluted HCl, and the time between this cleaning procedure and the actual mounting in the STM was kept short (maximum 30 minutes), so only a thin oxide layer is formed.

5.4 Tip preparation

As one of the main reasons for using the STM in this research project is its extreme spatial resolution, it is essential to use tips with a well-defined tip geometry. Furthermore, the fiber alignment requires tips with a narrow top angle to prevent blocking of the emitted light by the tip. Here we use platinum tips etched from 0.15 mm wire according to the procedure proposed by Libioulle et al. [19].

**Preparation**

Prior to etching, the following preparations have to be done:

- Ultrasone cleaning of the platina wire in alcohol.
- Prepare a fresh solution of 7 gr CaCl₂·H₂O in 40 ml 1:1 H₂O/acetone solution.
- Prepare a 1:10 solution of H₂SO₄ and water.

**Etching process**

After these preparations, the tips are made in an electrochemical etching procedure. This procedure consists of the following four steps, which are depicted and further explained in figure 5.2:

1. Coarse electrochemical etch in cooled CaCl₂·H₂O/acetone solution.
2. Anneal in Bunsen flame.
3. Micropolish in diluted H₂SO₄.
4. De-oxidize in H₂SO₄.

This procedure results in tips with a curvature radius of about 50 nm [20], which are inert to oxidation.
Chapter 5. Experimental Setup

1. Coarse electrochemical etching in CaCl₂ solution
   a. cool CaCl₂ solution prior to and during etching \((T \pm 0-10^\circ C)\)
   b. apply 20V at 40Hz to carbon electrode and tip
c. dip tip in solution until \(I = 100\ mA\)
d. etch until \(I = 30\ mA\)

2. Anneal
   anneal etched tip 15 seconds in the blue part of a Bunsen flame to smoothe the surface of the tip and anneal out dislocations.

3. Micropolishing in H₂SO₄ solution
   a. ground carbon electrode
   b. apply 16 \(\mu s\) 15V positive pulses at 4kHz for 15 s.

4. De-oxidation in H₂SO₄ solution
   apply -1.1V DC to tip until \(I \leq 30\ \mu A\)

Figure 5.2: The four-step etching process used to make STM tips.
5.5 Spatially resolved measurements (SRM)

A new method has been employed to perform spatially resolved STL measurements. Instead of positioning the STM at a certain location on the sample, taking a spectrum and repositioning the tip manually, the tip is set to scan the surface. During this scan, the current is kept constant and at multiple positions along the scan line a spectrum is taken. This technique is fully automated: a macro has been written (see Appendix B) for the nanostructuring mode of the STM control unit.

As soon as the tip reaches the pre-defined location, a trigger is given to the shutter of the monochromator and a spectrum is taken. This process is repeated several times until the predefined scan area has been covered. With this technique, luminescence maps of the sample can be made. This makes it possible to study the evolution of the spectrum as a function of the position on the sample and gives information on the resolution of the STM injection mechanism.

In the first attempt to perform these measurements, the tip scanned across the surface at constant speed with the current set to its experimental value of typically 1 to 10 nA. The tip-sample distance at this value of the tunnel current is about 5 Å. However, the luminescence maps showed photons of the same wavelength to be emitted across the whole scanning range of approximately (600-800 nm)². Because of the size fluctuations of the dots, we don't expect lines of one wavelength to emerge from different dots at different positions in the sample. Therefore the impression is given that the tip is pinned to a certain spot on the surface and the piezo is only wobbling the tip around the center of this spot. To prevent this from happening, the technique has been changed: in between measurements a small current I_{scan} of 0.1 to 0.01 nA is used and the current is restored to its experimental value I_{tun} only at measurement positions (see figure 5.3). This method also has the advantage that the tip is less likely to crash into surface irregularities.

![Diagram](image)

Figure 5.3: Improved SRM measurement method.
Another problem we encountered in our first measurements was that although the scan speed is very low (maximum 10 nm/s), the tip was not fixed at a certain position while a spectrum was taken. The first solution was to decrease the distance the tip travels during measurements by setting the integration time for the spectra at a short interval of 2 seconds. However, to increase the signal to noise ratio, longer integration times are preferred. To achieve this, a delay has been introduced in the macro. This delay stops the tip at the predefined positions. The integration time for the spectrum is then only limited by the built-in time delay.
Chapter 6

Results

6.1 General remarks

6.1.1 4.2 K PL spectrum

As the STL experiments are done at temperatures well below room temperature (77 K and 4.2 K), a PL spectrum is taken at 4.2 K for comparison. In this spectrum, the GaAs peak is blueshifted over 40 nm with respect to the room temperature spectrum (see figure 2.5) to 830 nm. The quantum dot luminescence is visible around 975 nm (1272 meV).

6.1.2 STL spectrum

Unless explicitly mentioned, all the spectra are taken with a 600 lines per millimeter grating, a slit width of 10 μm and an integration time of 60 s. For the spectra at 4.2 K, the monochromator is set at two different wavelengths for 30 s. The two resulting 30 s spectra are glued together to cover the whole wavelength range over which the dots are luminescing. Prior to all measurements, a background light signal from the setup is collected, which is subtracted from the measured intensity profiles. In an STL spectrum taken at 4.2 K, typically some 55 lines can be distinguished. When we compare the position of these lines to the PL spectrum (see figure 6.1), we conclude that the lines are indeed emerging from the quantum dots. The observed linewidth of these lines is approximately 1 meV, which is the resolution of the used monochromator.

Filling the dots

From the measured spectra, we can make an estimate of the number of electrons in each dot. When we integrate the total intensity of the spectrum, this yields values of $5 \cdot 10^4$ to $5 \cdot 10^5$ counts per 30 s at tunnel currents between 1 and 10 nA. Division by the integration time gives the number of detected electron-hole recombinations to be between $1.67 \cdot 10^3$ and $1.67 \cdot 10^4$ per second. The number of electrons that can be detected is limited by the cross section and alignment of the fibers. The fiber area is $\pi \cdot 0.3^2 mm^2$ and each fiber is
Chapter 6. Results

Figure 6.1: PL spectrum and STL spectrum at 4.2 K. In the PL spectrum, the GaAs peak is visible at 830 nm (1494 meV). The quantum dots are seen to luminesce at wavelengths around 975 nm (1272 meV). The InGaAs wetting layer is again absent here. An STL spectrum taken at a tip voltage of 4 V and a tunnel current of 5 nA shows lines to appear in a region between 1180 and 1420 meV. Comparison of the STL to the PL spectrum shows that the STL lines originate from the quantum dots.

positioned at a distance \( h \) of approximately 1.5 mm from the tip apex, so only a factor of \( \frac{\pi \cdot 0.3^2}{4 \pi \cdot 1.5^2} = 0.01 \) is detected. Furthermore the light originating from the semiconductor is refracted on the GaAs/Helium interface as a result of the difference in refractive index between GaAs and the Helium contact gas (\( n_{GaAs} \simeq 3.63 \) and \( n_{He} = 1.000035 \)). Therefore the radius \( r_{GaAs} \) of the emitted light cone inside the semiconductor that is detected is

\[
r_{GaAs} = h \cdot \tan \left( \arcsin \left( \frac{n_{He} \cdot \sin(\arctan \frac{r_{fiber}}{h})}{n_{GaAs}} \right) \right) = 8.1 \cdot 10^{-2} \text{ mm.} \quad (6.1)
\]

When we assume the detector efficiency to be about 0.2, the total detection efficiency is determined at \( 0.2 \cdot 0.01 \cdot (\frac{8.1 \cdot 10^{-2}}{0.3})^2 = 1.5 \cdot 10^{-4} \). Therefore the number of emitted photons is estimated between \( 1.1 \cdot 10^7 / \text{s} \) and \( 1.1 \cdot 10^8 / \text{s} \). With a recombination time of \( 10^{-9} \text{s} \) \[14\] the upper limit for the total number of radiative electrons is thus estimated at 0.1. From this calculation, we expect the dots to be occupied by one electron at a time. From the estimated number of emitted photons we calculate that \( 1.7 \cdot 10^{-3} \) photons are emitted per injected electron. An estimate for the number of holes per dot can be made from band diagram calculations with the HETMOD program. This program calculates a self-consistent solution to Poissons equation for a given temperature, doping concentration and applied bias. Also the influence of the surface accumulation layer is incorporated in the program. The carrier density in the dot layer is calculated at both 4.2 K and 77 K as a function of the bias applied to the sample surface. The results are presented in figure...
6.1. General remarks

Figure 6.2: Calculated hole density in the quantum dot layer as a function of applied bias. On the bottom horizontal axis, the voltage applied at the surface of the sample is given. This voltage is the tip bias minus the voltage drop across the vacuum gap. For comparison, the corresponding tip bias at a tip-sample distance of 5 Å and $T = 4.2$ K is given at the top axis. The right axis shows the maximum number of holes per dot at a dot density of $10^{10}$/cm$^2$ under the assumption that all the holes in the InGaAs layer are captured by the dots. In the actual sample, the holes can also be present in the wetting layer.
Chapter 6. Results

6.2. In this figure it can be seen that at 4.2 K, more holes will occupy the dots than at 77 K. The reason for this lies in the number of charge in the accumulation layer: at 77 K, more acceptors are ionized and thus more carriers are present in the accumulation layer. This charged surface layer screens the externally applied potential. The more charge in the layer, the higher the screening effect. As a result, the electric field in the dot layer is smaller at higher temperatures and from Poisson's equation (4.3) it follows that less charge is present in the dots.

The voltage drop across the vacuum gap can be calculated from conservation of the displacement field combined with electric field calculations inside the semiconductor. When we estimate the tip-sample distance to be 5 Å, a tip voltage between 3.5 V and 5 V results in a surface bias between 1.5 V and 2 V at 4.2 K. At the elevated temperature of 77 K, the surface bias for the same tip voltages is calculated to be between 1.4 V and 1.8 V. This procedure can only be applied if no charged surface states are present. The presence of these states is ignored in all calculations.

Injection radius calculations

![Injection radius calculations graph](image)

Figure 6.3: Calculated bands in 3D 5 V tip bias. The tip is positioned at 5 Å from the surface of the sample. The donor and acceptor concentrations are chosen to be equal at $1 \cdot 10^{15} \text{cm}^{-3}$. In a) the bands are shown in 3D, with the position of the sample surface deduced from figure b). The potential drop on the surface of the semiconductor in lateral direction is depicted in c).

To be able to eventually assign the lines in the spectrum to particular transitions in the dots, the first question that has to be answered is how many dots are visible in the spectrum. To determine whether the distance the electrons can travel in the sample or
6.2. Temperature dependence and phonons

the area where the holes are accumulated is the limiting factor for the dimensions of the probed region, several calculations have been made.

For the electrons, the injection area in lateral direction is limited by their diffusion length. Measured values for the diffusion length in GaAs/AlGaAs systems indicate typical length scales of some 200 to 250 nm (e.g. [22] and [29]). For the holes, the injection area is determined by the distance where the valence band at the dots is pulled above the Fermi level. 3D band diagram calculations have been done to determine this distance. The 3D program calculates the potential drop inside a semiconductor material at a given tip-sample bias. The voltage drop across the barrier is calculated from displacement field conservation. However, as in this program it is not possible to define layers with different doping concentrations, the results of these calculations can only be regarded as qualitative estimates and no quantitative conclusions can be drawn from the results. In figure 6.3 a calculated band diagram at 5 V tip bias is given for equal donor and acceptor concentrations. In this figure it can be seen that the potential drops to zero on the sample surface in about 30 nm in lateral direction. From this value an estimated upper limit for the area where the dots are filled is $\pi(30)^2 \text{nm}^2$, which would mean that only one dot is visible per spectrum. This value appears to be far too small to be able to explain the 55 lines, which cover an energy range of some 200 meV in the spectrum. A possibility is that due to the oxide layer at the surface, the Fermi level in the sample is pinned in such a way that the dots are already filled with holes without a tip bias. The spatial resolution is then no longer determined by hole drift due to the electric field, but by electron diffusion, which acts on ten times longer length scales.

6.2 Temperature dependence and phonons

As is stated in the previous paragraph, we expect to see differences in the spectra as a function of temperature. When we compare the spectra taken at different temperatures, some general features are clearly visible. First, the spectrum is redshifted at 77 K due to the temperature dependence of the band gap. Second, at 77 K a large background is observed. A comparison of the linewidths at different temperatures shows that at 77 K, the full width at half maximum (FWHM) is about 3-4 meV, while at 4.2 K the observed width is 1 meV, which is the resolution of the monochromator. From literature it is known that the actual linewidth at this temperature is less than 50 $\mu$eV [21]. The value of 3-4 meV at 77 K seems to be reasonable [24].

A convolution of the 4.2 K spectrum with a Lorentzian profile with a width of 3.5 meV reproduces the observed background fairly well (see figure 6.5). Thus we conclude that the background is caused by the broadening of the individual lines. The magnitude of the broadening indicates that the main reason for the line broadening is the increase in phonon occupation in the sample at elevated temperatures. An increase in phonon occupation leads to higher scattering rates and the exciton lifetime is decreased. Since the linewidth of an optical transition is inversely proportional to this lifetime, the lines are
Chapter 6. Results

Figure 6.4: a) STL spectrum on wafer 2247E at 77 K compared to c) 4.2 K. The applied voltage is in both cases 4 V and the tunnel current 5 nA. In b) and d) part of the spectra are shown at enlarged scale to compare the linewidths.

broadened at higher phonon occupation and thus higher temperatures. This conclusion is supported by several measurements by other groups (e.g. Spithoven et al. [23] and Matsuda et al. [24]) and the theoretical model by Li et al. [25]. Other broadening mechanisms like dephasing due to scattering of the exciton with thermalized carriers [26] results in broadening in the order of 0.02 μeV/K [27], which can not explain the 3-4 meV found here.

Third, the number of lines that can be distinguished is about 30 at 77 K and approximately 55 at 4.2 K. When we compare this with the calculated number of holes expected per dot, we see that the ratio of the calculated hole density is 1.8, which is in very good agreement with the ratio between the observed different numbers of lines. From this result the tentative conclusion can be drawn that the number of lines is in principle related to the number of holes per dot. However, the remark should be made that lines in the 77 K spectrum are broadened. Due to this broadening it is harder to distinguish the different lines properly. For example in the convoluted 4.2 K spectrum some 40 lines can be distin-
6.3 Current dependence

Figure 6.5: a) and b) Measured spectrum at 77 K compared to c) and d) convolution of a 4.2 K spectrum with a 3.5 meV Lorentzian function.

guished, which shows that due to the broadening several of the original 55 lines merge into one broad peak.

The possibility of an enhancement of the number of visible lines by phonon replicas cannot beforehand be excluded. During the electron-hole recombination a phonon may be emitted simultaneously. When such an event occurs, the energy of the emitted photon is lowered by the phonon energy. In GaAs the typical LO phonon energy is 36 meV, so we expect the so-called phonon replicas at multiples of 36 meV to the low side of the original peak. To identify these phonon replicas, we applied an autocorrelation to the 4.2 K spectrum (figure 6.6). No significant features could be distinguished around 36 meV, so no phonon replicas are visible in the spectra.

6.3 Current dependence

When we compare the spectra taken at different values of the tunnel current as is done in figure 6.7, only an increase in the total intensity of the spectrum is observed. The absence of new peaks emerging at higher injection currents confirms that the number of electrons per dot is indeed less than 1. A plot of the total intensity versus the tunnel current shows a linear behavior. This is the result we expect when there are no competing non-radiative
Figure 6.6: a) Autocorrelation of a 4.2 K STL spectrum, taken at a tip voltage of 4.5 V and a tunnel current of 5 nA. In the autocorrelation, several distinct peaks are visible, for example at 13.4 meV. b) Autocorrelations of spectra taken at the same sample with the same tip do not reproduce the peaks. In all cases no clear peak can be seen at the GaAs LO phonon energy of 36 meV.

recombination channels present which can be saturated [28]. This result also indicates that no light output saturation effects as mentioned in section 2.3.2 take place. As an increase in current from 1 to 10 nA corresponds with a change in the tip-sample distance of 1 Å at maximum, we don't expect to see a difference in the number of active dots as a function of the injection current.
6.4 Voltage dependence

In most of the measurements, the voltage dependence of the spectra shows no significant features (see figure 6.8). The total intensity remains about constant. This is not the behavior we expect to see as a result of the calculated band diagrams given in figure 4.2. In these calculated band diagrams, we see a minimum in the conduction band in the intrinsic layer above the dot layer. Electrons which are scattered before they reach the depth of the dot layer are therefore unlikely to be trapped by the dots, but will be trapped in the conduction band minimum. Here no radiant recombination is possible, because the holes are spatially separated from the electrons since they are only present in the surface accumulation layer and the dot layer. With increasing bias the conduction band minimum disappears and thus we would expect an increasing intensity of the dot luminescence as a function of the
Chapter 6. Results

Figure 6.8: a) Spectra taken at different tip voltages and constant current. For clarity, only part of the spectrum is shown. b) and c) The total intensity normalized to the value at 3.7 V tip bias seems to be independent of tip voltage on average. A fit with the ballistic electrons model evaluated between the dot layer and the p-doped layer shows no good agreement with the data, which proves that the electric field is indeed always directed towards the dots in the intrinsic layer.

applied voltage. Since we do not observe this change, the band diagrams inside the sample apparently differ from the calculated band diagrams and the calculated minimum is not present in real. This conclusion is supported by calculations with the model that describes scattering of ballistic electrons given in equation 3.4. With this model we calculated the expected voltage dependence of the dot luminescence by evaluating the number of electrons that is scattered between the dot layer and the p-doped layer. The electrons that are scattered in the layer between the top layer and the dots are assumed to be directed away from the dots towards the top layer, as we would expect from the calculated band diagrams. The model does not fit the experimental data and therefore we conclude that the field in the layer above the dot layer must be directed towards the dots. In figure 6.9 the deduced band diagram is given. From $\Delta E_V$, which is taken to be between 50 and 150 meV, we calculate a hole occupation of 9 to 27 holes per dot at a dot density of $10^{10} \text{cm}^{-2}$ under the assumption that all holes are captured by the dots and not present in the wetting layer.

Furthermore, since no extra lines appear in the spectrum at increasing tip bias, the injection area does not seem to be determined by hole drift.

However, some of the experiments at 77 K have shown intensity differences of certain peaks as a function of tip voltage. One of these results is given in figure 6.10. In this
6.4. Voltage dependence

Figure 6.9: Deduced band diagram at 2.5 V tip bias. The value of 2.5 V for $V_0$ follows from the voltage dependence of the GaAs luminescence, which is discussed in the next pages. From $\Delta E_V$ the number of holes in the dot is estimated at 9 to 27.

Figure 6.10: Spectra taken at 77 K at a tunnel current of 2 nA show a change in intensity of the peaks around 1210 meV and 1220 meV as a function of tip voltage.

...
state of the dot. As our dots are filled with approximately 10 holes, the charged excitons probably consist of exited hole states. These states may very well be less localized than the electron. If this is the case, the argument used in chapter two can be reversed and the binding energy is increased instead of decreased with an increasing number of holes. From this argumentation we can explain the change in the spectrum by additional holes in the exciton complex. The reason why we do not observe such features at 4.2 K is probably that at that low temperature, all available hole levels in the dot are already occupied. Since the hole occupation is calculated to be higher at 4.2 K than at 77 K, it seems reasonable that extra holes can be added at 77 K whilst this is impossible at 4.2 K.

Behavior of the GaAs peak

![Graph](image)

Figure 6.11: a) Voltage dependence of GaAs peak intensity. The peak intensity is normalized to the intensity of the total spectrum, which is shown in b). The spectrum includes both the GaAs peak and the dot luminescence. A 150 lines grating is used to obtain this spectrum.

We measured the intensity of the GaAs peak as a function of voltage (figure 6.11). When we fit equation 3.1 we find a scattering rate $\Gamma_s$ of $3.5 \cdot 10^{13} / s$. As this value is in good agreement with the LO phonon scattering time documented in literature (see chapter 3), we conclude that the dominant relaxation mechanism is indeed due to LO phonon scattering. From the scattering time, we calculate the ballistic mean free path to be 21 nm at 0.1 V excess energy. The fitted values for $V_0$ vary from 2.4 V at 5 nA to 2.7 V at 10 nA. Self-consistent calculations with HETMOD yield a value of 2.0 ± 0.2 V for $V_0$. Comparison of these results gives rise to the idea that instead of tunneling through the thin Schottky barrier, the electrons are only injected into the dots when their initial energy is higher than the height of the barrier. This observation confirms the idea that the surface is pinned by the oxide layer.
6.5. Spatially resolved luminescence

6.5 Spatially resolved luminescence

Figure 6.12: Position dependent luminescence maps at a tip bias of 4.5 V and tunnel current of 5 nA. Spectra are taken in a grid of 16 x 16 points while the tip is constantly scanning. The integration time is 2 s and the slit width of the monochromator is 20 μm. The peak intensity is normalized to the total intensity in the entire area. Roughly 7 different patterns can be distinguished, which are depicted here.

Luminescence maps are made by following the intensity of a particular peak as a function of the lateral tip position. To exclude background features, the peak intensity is normalized to the total intensity of all the spectra in the total area. The disturbing influence of cosmic rays is filtered out by clipping the intensity of the peaks above a certain value, which is chosen well above the maximum intensity of the dot peaks. The dimensions of the mapped area have been estimated. As the STM x-y piezo controller is calibrated at room temperature, reference measurements on a pure gold surface have been done to calculate the response of the piezo element at 4.2 K. Features at the surface appear 5 times larger at 4.2 K than at room temperature, and therefore the mapped area is about 860x860 nm in real when the controller is set to 4300x4300 nm.

In the maps given in figure 6.12, about 7 different patterns can be observed for the 55 lines. This means that on average 8 lines are emitted per dot, which is not unexpected at the estimated value of 10 to 20 for the number of holes. The shape of the spectra suggests
groups of lines to emerge from one dot. In figure 6.13 it is shown that these lines give similar patterns in the luminescence maps. The separation of 3 meV between the lines could be caused by exchange interactions due to the hole distribution in the dots. However, no estimate for the number of lines per dot can be made by just counting the number of lines per group, as for example a line at 50 meV higher energy shows the same position dependence.

Figure 6.13: In the spectra, different groups of lines can be distinguished. Lines in some of these groups show the same position dependence, although lines from other groups can also result in similar patterns in the luminescence maps.

A comparison of maps made at different tip voltages shows that the spatial dependence of the intensity of the different peaks is reproduced (e.g. the maximum intensity of the peaks appears in the same region of the probed area). No structural differences in injection length scales could be determined from these measurements. We think that the 7 different patterns correspond to 7 different active dots in the spectrum. From the dot density, we would expect to see about 64 dots in this large region. Different reasons can be given for this large discrepancy.

A possibility we must consider is that the tip is not actually scanning over the surface as we meant it to be, but sticks at a certain point. This problem has been discussed in chapter 5 and a new method has been developed to prevent this effect from happening. Some preliminary results obtained with the new method are presented in figure 6.15.
6.5. Spatially resolved luminescence

Figure 6.14: Reproducibility of the luminescence maps at different tip voltages. The spectra are taken in a 16x16 points grid with an integration time of 2 s per spectrum. The tip is not stopped at the measurement positions. In between the two different measurements, the tip is not retracted from the sample surface.

Furthermore, although the size distribution of the dots is not homogeneous, we cannot exclude the possibility that different dots emit photons with the same wavelength within the resolution window of the monochromator. This idea is supported by for example the map made of the 1324.6 meV peak, where we can distinguish two different regions of high intensity (in the upper left and the lower middle of the map). These regions seem to be too far apart to be assigned to the same dot, as we do not expect the injection region to be over 500 nm in length. Another argument originates from the width of the PL dot spectrum. We conclude that the size distribution of the dots causes about 10% of the lines from different dots to overlap.

Our results are supported by similar experiments done by Hess et al. with a SNOM technique on dot-like centers in a GaAs/AlGaAs quantum well [29]. They present the same kind of luminescence maps with a few reproducing patterns. Their interpretation of the data is that the dimensions of the bright spots reflect the diffusion lengths of the carriers. Different regions of high intensity correspond to different luminescence centers and thus in our case different dots. When we interpret our data this way, the typical diffusion lengths appear to vary from 100 to 400 nm. This value corresponds well with the diffusion lengths observed in similar structures [22]. From this observation we conclude that indeed the diffusion is the limiting factor in the spatial resolution of the technique and apparently the remark made in section 6.1 that the dots are already filled with holes before a bias is applied is true.

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Chapter 6. Results

One remark has to be made here though: the maps shown here are interpolations of data measured in a grid consisting of 16x16 points. The interpolation can make the areas seem larger than they are in real. Another point of consideration with the grid is that 16x16 points in an area of 860x860 nm results in a distance of approximately 54 nm between the spectra. This distance is in the same order of magnitude as the expected interdot distance. To be able to study the injection trajectory in more detail, a finer grid should be used. In figure 6.15 some results of measurements with a finer grid are presented. Here we see the same kind of regions with higher intensity, but there appears to be a sub-structure within these regions.

From the maps it is clear that the injection mechanism is not radially symmetric at all, so the diffusion cannot be described by equation 3.8. The diffusion coefficient obviously changes as a function of the position in the sample. The observed non-uniformity in the carrier transport is probably caused by the potential landscape in the sample. First, different dots are occupied with different numbers of holes. Electrons are attracted to the dots with higher hole densities due to the larger positive charge. Second, the presence of ionized impurities alters the field locally, which can focus the carriers to certain dots and block them from entering others. Third, variations in the thickness or indium concentration of the wetting layer around the dots can cause barriers that prevent the carriers from entering the dot [30].
6.5. Spatially resolved luminescence

Figure 6.15: The left column labelled a) shows two typical results of the use of a grid of 32x32 points whilst scanning constantly during the measurements. The integration time is 2 s. In the maps, the areas with high intensity are not as pronounced as in the 16x16 grid, but still the 1310 meV shows a distinct area of higher intensity at the left of the map. In the middle, the two maps labelled b) depict the result of an experiment where the tip is retracted to a tunnel current of 0.01 nA in between measurements. The used grid is 20x20 points and the integration time is 10 s per point. In the maps, the areas with high intensities seem elongated in the scanning direction. To find out if this is due to the tip sticking to the surface during the experiment, the scanning procedure was changed. Instead of scanning every line from left to right, an S-shaped path is chosen. This means that the first line is scanned from left to right, the second line from right to left, the third from left to right again and so on. The results of this experiment are depicted in the right column labelled c). In these maps it is indeed clear that the tip is being dragged over the surface in the scanning direction. From the alternating high and low intensity in the scan lines we conclude that the tunnel current originates from a different point of the tip apex while going from left to right compared to the reverse direction.

All measurements are performed with different tips on different samples.
Chapter 7

Conclusions and Recommendations

• From calculations we estimate the dots to be filled with less than 1 electron and approximately 10 to 20 holes. Current dependent measurements confirm that indeed only 1 electron is present per dot. From the position dependent measurements we conclude that the 55 lines in the spectrum emerge from 7 different dots, which means that some 8 lines are emitted per dot. This amount of lines per dot is not unexpected at a hole occupation in the order of 10 to 20 per dot. Further calculations of the energy levels and possible transitions in the dots could be a good start to estimate the number of lines that is expected to emerge from a single dot and thus the number of dots that is visible in a single spectrum. Cross-sectional STM could give definite answers to the questions what the shape, size distribution and areal density of the dots is. With this knowledge, more accurate predictions can be done for the positions of the lines. A next step could be to apply a magnetic field to the sample and study the field dependence of the different peaks.

• We expected the limiting factor in the spatial resolution to be the region where the holes are injected into the dots. Calculations of the area of this region with simple models give values which appear to be too low when compared to the experiments. In contrast to our expectations, we conclude that in our experiments the injection area is determined by the diffusion lengths of the electrons. The impression is given that the dots are always filled with holes and an external electric field from the tip is not necessary to fill the dots. This could be due to pinning of the Fermi level by the oxide layer surface states. Improvement of the model by incorporating these surface states can lead to a better understanding of hole occupation in the dots and the influence of tip bias on the number of holes per dot.

• In principle, the hole injection area and therefore the spatial resolution of STL is determined by the voltage applied to the surface of the sample. Decreasing this voltage would mean decreasing the area and thus improving the spatial resolution. But in the current setup, we need a minimum voltage of approximately 3 V to be
Chapter 7. Conclusions and Recommendations

able to inject the electrons through a tunnel process into the sample. A possible solution could be the addition of a metal gate with holes etched in it. The voltage needed to get the electrons to tunnel to the sample can then be applied between the mask and the tip, instead of between the sample surface and the tip. This way, the voltage at the sample surface can be much smaller and the injection area is decreased. However, a problem with this method is, that the tip of the STM has to be positioned straight above such a hole to enable the electrons to reach the semiconductor instead of tunneling into the metal top gate.

- Comparison of measurements at temperatures of 4.2 K and 77 K shows an increase in linewidth from < 1 meV to 3-4 meV. We assign this increase to enhanced phonon scattering at higher temperatures. Studies of the temperature region in between these two values could give more insight in the physical concept that causes the broadening. Also, the origin of the background can be studied in more detail.

- The scattering rate in the sample is determined at $3.5 \cdot 10^{13}/s$. This value is in good agreement with literature [14] and confirms the idea that the main scattering mechanism for relaxation is indeed due to LO phonons.

- From the linear dependence of electroluminescence intensity to the current, we conclude that no additional recombination channels exist which can or have to be saturated before the dots are filled.

- From the lack of voltage dependent change in the spectra at 4.2 K, we conclude that the minimum in the conduction band in the calculated band diagrams does not exist in the actual sample. This is caused by the simplifications in the calculations, in particular the unknown properties of the surface layer.

- We have developed a method to perform spatially resolved measurements. The first results from these measurements indicate a strong non-uniform transport of carriers through the sample. Since it is not clear if this non-uniformity is caused by for example the amount of charge present in the dots or the potential landscape around the dots, further investigations are necessary.
Bibliography


[4] D.M. Bruls et al. (to be published)


Bibliography


Appendix A. Derivations

In thermal equilibrium, the drift and diffusion currents exactly cancel:

\[ 0 = en_{\text{equ}}(z)\mu_n E_{\text{int}}(z) + eD_e \frac{\partial n_{\text{equ}}(z)}{\partial z} \]  

(7.1)

The internal electric field gives rise to an internal potential which is defined as

\[ E_{\text{int}} = -\frac{dV(z)}{dz} \]  

(7.2)

From these two equations it follows that

\[ n_{\text{equ}}\mu_n \frac{dV(z)}{dz} = D_e \frac{\partial n_{\text{equ}}(z)}{\partial z} \]  

(7.3)

The potential energy of an electron in a potential \( V(z) \) is \( W_{\text{pot}}(z) = -eV(z) \) in thermal equilibrium. Furthermore, if the Fermi energy is at least a few \( k_B T \) below the conduction band energy, then \( n_{\text{equ}} \) is given by the Boltmann distribution

\[ n_{\text{equ}} = N_C(T)\exp\left( -\frac{W_{\text{pot}}(z)}{k_B T} \right) = N_C(T)\exp\left( \frac{eV(z)}{k_B T} \right) \]  

(7.4)

with \( N_C \) the effective density of states in the conduction band. Combining equations 7.4 and 7.3 gives

\[ N_C(T)\exp\left( \frac{eV(z)}{k_B T} \right) \mu_n \frac{dV(z)}{dz} = N_C(T)D_e \frac{e}{k_B T} \exp\left( \frac{eV(z)}{k_B T} \right) \frac{dV(z)}{dz} \]  

(7.5)

From this equality, the Einstein relation 3.10 follows directly.
Appendix B. SRM program

path20x20.pat

5, 5, 1; 10, 5, 1; ... 100, 5, 1; 5, 10, 1; ... 100, 10, 1; ... 100, 100, 1;

Position definition, first two numbers stand for $\%x$, $\%y$ of total scan area. Last number stands for event number to be carried out at this position.

event1.mac

reset_v;
reset_i0;
set_signal, 12;
reset_signal, 12;
loop_times, 20;
delay, 500000;
loop_end;
set_v, 4.0;
set_i0, 0.1;

Voltage restored to value experiment.
Current restored to value experiment, tip pushed closer to sample.
Trigger to open shutter of monochromator on pin 12 of status port.
Reset signal on pin 12.
Tip stopped at same position for 10 seconds.
Delay loop of 20 x 5 $\mu$s.
Voltage set to scanning value.
Current set to scanning value, tip retracted from sample.
Dankwoord

Het zal niet onverwacht zijn dat niet de fysica, maar de schrijfster zelf hier het laatste woord neemt. Ik zal mij hier echter beperken tot een bedankje. Verdere lappen tekst van mijn hand zullen hopelijk vanaf binnenkort in andere boekjes te bewonderen zijn.

Mijn afstudeerwerk zou zonder een aantal mensen een stuk minder zijn geweest. Op de eerste plaats hoort hier een grote spotlight op Martijn Kemerink. Niet alleen vanwege zijn erg aanstekelijke enthousiasme voor natuurkunde en wetenschappelijk onderzoek en zijn niet aflatende energie om overal een theorie voor te bedenken, maar ook omdat hij gewoon een heel sympathiek mens is om mee samen te werken. Martijn, hopelijk tot ziens op jouw Nobellezing! Jos van Ruyven en Peter Nouwens, enorm bedankt voor alle reddingsoperaties aan mijn opstelling! Verder wil ik Jos en Wil van het heliumbedrijf bedanken voor alle honderden liters helium, Paul voor mijn afstudeerplaats, Tom voor de PL metingen, Peter Offermans voor het programma om 3D banden te berekenen en Huub voor zijn ontelbare pogingen tot missiewerk. Ook de rest van de groep HGF bedankt voor het geklets tijdens de koffiepauze, ik zal 'De avonturen van Struuffke' niet snel vergeten!

Tot zover bedankjes voor mijn afstudeerperiode. Maar omdat hiermee ook het einde is aangebroken van mijn in alle opzichten fantastische studententijd, horen hier ook wat andere namen. Zonder Van der Waals en iedereen die daar bij hoort, zouden de afgelopen 6 jaren een stuk minder snel zijn omgevlogen. In het bijzonder Frans, dankjewel voor al je vriendschap en dat je er altijd voor me was! Peter, Jelle, Yolanda, Manon, Ingrid, Patrick en Jimi ontzettend bedankt voor al het lachen, en het luisterend oor als het nodig was. Ook aan het Natuurkundecircus heb ik mooie herinneringen en het heeft me laten inzien wat ik echt leuk vind om te doen. Jan, bij deze sta je dus weer in een door mij geschreven dankwoord, sorry van de vorige keer!

En tot slot, omdat het dan extra opvalt, hoort zeker mijn familie te delen in de roem. Op de eerste plaats mijn ouders, die mij altijd in mijn keuzes gesteund hebben, hoe vreemd die soms ook mochten lijken. Pa en ma, na al die tijd ben ik dan eindelijk klaar hoor! Karin en Jeroen, voor de lachtherapieen in de Heremiet, John en Tiny, voor de interesse in mijn bezigheden en Ed, voor oneindig veel meer dan ik hier op 1 bladzijde kan (of wil) vertellen. Zonder jou waren de afgelopen jaren sowieso een stuk minder gelukkig geweest.

Ontzettend bedankt dus allemaal! Sonja
Samenvatting voor leken

Voor de lezer zonder natuurkundige achtergrond zal het voorgaande verhaal waarschijnlijk nogal overkomen als abacadabra. Omdat ik het erg zonde vind als iemand die dit verslag wil lezen alleen het dankwoord kan volgen, volgt hier een samenvatting van de belangrijkste inhoudelijke punten in het Nederlands. Het verhaal is zoveel mogelijk verteld in voor iedereen bekende analogieën, waardoor eenieder zich hopelijk tenminste een beeld kan vormen van waar ik me het afgelopen jaar mee bezig gehouden heb.

Licht uit halfgeleiderbolletjes

Dit verslag beschrijft onderzoek gedaan aan extreem kleine halfgeleiderstructuurtjes, quantum dots genaamd. Halfgeleiderstructuren zijn belangrijke bouwstenen van onze hedendaagse technologische omgeving. Denk bijvoorbeeld maar aan de chips in je computer of aan de laser in je cd speler. Als je nu van zo’n halfgeleider een bolletje maakt dat in alle richtingen duizend keer kleiner is dan de dikte van een menselijke haar (namelijk zo’n 25x15x5 nm, met 1 nm = 1 miljardste meter), gebeuren er heel interessante dingen.

In een halfgeleider kunnen zowel positief als negatief geladen deeltjes aanwezig zijn. Deze deeltjes kunnen worden ingevangen in het bolletje. Ze kunnen echter niet overal in de bol gaan zitten: er is alleen een soort tribune waar de deeltjes zich op kunnen bevinden. In natuurkundige termen is er een “quantisatie van de energieniveaus” opgetreden. Om je er iets bij voor te kunnen stellen, kun je het bolletje -of in vaktermen de quantum dot- beschouwen als een soort knikkerputje, met een aantal plateaus (de tribune) waar de knikkers op kunnen liggen.

Als een deeltje eenmaal in zo’n putje is gevallen, komt hij er in principe niet zomaar meer uit. Dat zou op zich heel oninteressant zijn, ware het niet dat er zoals gezegd verschillende soorten deeltjes bestaan: de positief en negatief geladen exemplaren. In de natuurkunde spreekt men over elektronen die licht, heel mobiel en negatief geladen zijn (in knikkertermen een soort eentjes) en veel zwaardere gaten, die positief geladen zijn (je voor te stellen als van die reuzebonken van vroeger). Als zo’n elektron en een gat elkaar tegenkomen, kunnen ze samensmelten (recombineren) en licht uitzenden. Afhankelijk van het hoogteverschil tussen de plateau’s waar de deeltjes op liggen voordat ze recombineren (het energieverschil van de deeltjes) wordt een andere kleur licht uitgezonden. Door de deeltjes gecontroleerd op bepaalde plateau’s neer te leggen en zo te bepalen hoe de recombinatie gaat plaatsvinden, kun je bijvoorbeeld een laser maken die een bepaalde kleur licht uitzendt. Omdat in zo’n quantum dot die plateaus veel netter gebouwd zijn dan in de gangbare lasers, is

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een dot een lichtbron die veel preciezer één kleur licht uitzendt. Dat kan bijvoorbeeld een toepassing vinden in nieuwe devices voor optische communicatie, iets wat getuige alle activiteit op dit gebied zeker belangrijk zal zijn in de toekomst.

De knikkervinger en de putjes

In dit verslag worden experimenten beschreven waarbij quantum dots gevuld worden met elektronen en *gaten* met behulp van een STM (scanning tunneling microscoop). In principe is het hart van een STM een hele dunne naald, die een klein stukje boven een oppervlak zweeft. Als je nu een elektrische spanning aanlegt tussen die naald en het oppervlak van de halfgeleider, steken in ons geval de negatief geladen elektronen van de naald over naar het oppervlak.

De dots zijn bolletjes van het halfgeleider materiaal indium-gallium-arsenide en zitten middenin een lagenstructuur van de halfgeleider gallium-arsenide. De toplaag van de structuur bevat een overschot aan elektronen, de middenlaag met de dots is elektrisch neutraal en de onderste laag bevat een overschot aan *gaten*, de positieve deeltjes dus. Als de naald nu vlakbij het oppervlak gebracht wordt en een elektrische spanning wordt aangelegd, schieten elektronen van de naald door de bovenste laag naar de dots. Tegelijkertijd worden de positieve *gaten* uit de onderlaag naar de dots getrokken door de aangelegde spanning. Je duwt dus eigenlijk met één vinger twee soorten knikkers tegelijk van twee verschillende kanten richting dezelfde put. Het licht dat na samensmelting van de deeltjes wordt uitgezonden, wordt opgevangen en in de verschillende kleuren gescheiden met een soort prisma. Analyse van de intensiteit en kleur van dit licht zegt nu iets over wat er in die dots gebeurd moet zijn. Bij het hier besproken onderzoek, worden er gemiddeld maar hooguit één elektron en een stuk of tien tot twintig *gaten* tegelijk in een dot gebracht.

Hoe hardere hoe beter?

Uit de experimenten blijkt dat het varieren van de elektrische spanning - dus in principe hoe hard je je elektronen duwt en hoe sterk de *gaten* worden aangetrokken- meestal niks uitmaakt voor de kleur en hoeveelheid licht die er uitgezonden wordt. Dat is vreemd. Je zou verwachten dat als je harder duwt, de deeltjes verder kunnen komen. Er wordt namelijk in de experimenten op een heel veld met dots tegelijk geschoten. Elke dot bevat een andere tribune en zendt dus een eigen kleur licht uit. Bij hogere spanningen verwacht je dat ook de dots die iets verder van de naald af liggen, gevuld worden. Verder verwacht je dat er bij hogere spanningen meer *gaten* in een dot terechtkomen en dat een electron op een ander plateau in het putje terecht komt, omdat hij er harder in geschoten wordt. Als we de hoeveelheid *gaten* in het systeem kleiner maken (in de experimenten door de temperatuur te verhogen), blijken er inderdaad ineens meer *gaten* in de dots te komen met toenemende spanning. Een nadeel van het verhogen van de temperatuur is wel dat de plateau’s minder netjes gemaakt zijn. Het licht dat de deeltjes uitzendende heeft dan niet meer zo precies één kleur, maar is een beetje uitgesmeerd over meerdere kleuren. Een verhoging van de stroom - het aantal elektronen dat per seconde naar de dot geduwd wordt- levert ook bij de laagste