Chapter I

Growth of Cr-doped CdTe quantum dots

The aim of this thesis was to study the dynamic of single spin in quantum dots. The first step in this study was to grow these doped QDs. We studied to type of quantum dots: self assembled QDs and strain free QDs. The strained dots are formed by the partial relaxation of a CdTe layer on ZnTe. Strain free dots are formed by thickness variation of a CdTe quantum well between CdMgTe barriers. The growth of Cr doped samples were done in Pr. Shinji Kuroda laboratory, in the University of Tsukuba. The Mn-doped samples were grown at Grenoble, in the INAC, by Dr. Hervé Boukari.

We will focus in this chapter on the growth done at Tsukuba by Molecular Beam Epitaxy (MBE). We begin by giving some general explanation on the MBE process and the different tools that are used in it. We then go to the growth of the self-assembled quantum dots, detailing the preparation of the substrate and the actual growth, and discussing quickly the results. In the last section, we present two other kinds of sample we grew: samples with the possibility of applying an electric field on them, and the strain free dots. For each of them, we detail the growth process and discuss the results we had studying them.

I.1   Generality on Molecular Beam Expitaxy

In the MBE process, cells of pure elements are heated to control their evaporation or until they reach sublimation. Those elements will form the desired crystal on the substrate. They are kept in Knudsen cells, which consist of a crucibles of high-melting-point material with a low contaminating power (typically Pyrolytic Boron Nitride) wrapped in Tungsten filament which will act as heater. Each are closed by a shutter controlled by a computer.

When the desired cell temperature is reached, said shutter is opened to let the element travel to the substrate. The chamber containing the substrate is kept in Ultra High Vacuum (about 10^{-8} Pa), in order to avoid contamination of the sample and get a mean free path of the gas long compared to distance to the sample. This process is illustrated on Fig. I.1. Reaching the surface, the atoms diffuse before stopping, either having dissipated their kinetic energy through interaction with the surface, or (more commonly) being kept by island of previously deposited atoms. In the ideal case, the growth occur layer by layer, slowly (about 1 monolayer/s), giving a good control of the thickness of the grown material.
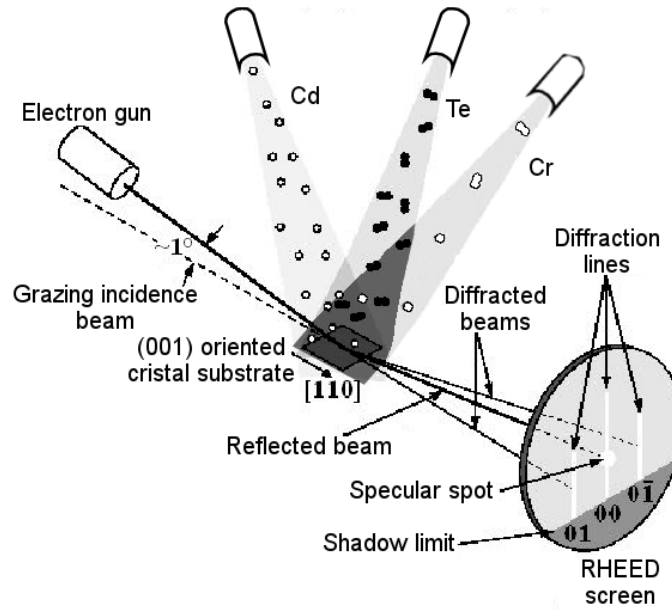

Figure I.1: Scheme of a MBE chamber and the cells. An electron gun is also fixed to the chamber in order to probe the surface of the sample.

This necessity of Ultra High Vacuum kept the MBE to be developed before the end of the 1960s [1], even though the the idea was formalized at the end of the 19th century. This method offers a good control on the growth, which make it useful for the development of nanostructures. Depositing the materials layer by layer gives the possibility to grow really thin structure, and the transition between two materials can be made abrupt, spanning over only a few monolayer (ML). Growing nano-structure is still the main use of MBE. However, this method is mainly used for research purpose, its slow growth speed and hard to fulfil growth conditions being an obstacle for the industrialization of the process.

CdTe growth in MBE is *auto-regulated*: if only one element is open, only a given quantity of material will be deposited and then the growth will stop until the other element is also deposited. The deposited quantity of material before the growth stop depends only on the substrate temperature. This allows us to achieve a really fine control of the growth using the MBE mode called Atomic Layer Epitaxy (ALE) or Migration-Enhanced Epitaxy (MEE). In this mode, only one element is opened at a time, growing the sample layer by layer with good control on the growth. Between each opening, the sample is left under vacuum in order to relax the surface. A full cycle correspond to opening each cell once. For CdTe, a substrate temperature between 260°C and 290°C guaranty a growth of only 0.5 ML for each cycle [2]. This allow a small uncertainty on the substrate temperature while keeping a really good control on the growth of the sample.

The growth was monitored with RHEED (Reflexion High-Energy Electron Diffraction). This technique requires a high vacuum, a given since MBE asks for ultra-high vacuum condition. An electron gun sent a beam of high energy electron at a low angle, between 1° and 3°, to the surface sample. This way, the electrons will only probe the surface of the sample, entering the material only on a 3 or 4 ML. Therefore the detected pattern directly gives information on the flatness and the crystallinity of the surface.

Incident electrons have a wave vector $\mathbf{k_i} = 2\pi/\lambda_e$, with λ_e the electron wavelength, typically 6 or 7 pm for an electron gun energy between 30 and 40 kV. Since only scattered diffraction is considered, the diffracted wave vector $\mathbf{k_f}$ as the same norm as the incident one $\mathbf{k_i}$. The Ewald's Sphere has then a radius equal to the norm of $\mathbf{k_i}$. In the reciprocal space, the plane of diffraction are infinite line. So, in the case of a perfect crystal, with a perfect detector, the intersection with Ewald's sphere should be points. However, since the crystal may present some defect and neither the gun or the detector are perfect, the diffracted pattern shows lines.

Once dots are grown, though, the surface become rough at the scale of the length of coherence of the beam. The electrons can interact with more layers while passing through the dots. This can be seen on the diffraction pattern, where lines become points.

Another use of the RHEED diffraction is the monitoring of the number of layer grown. Focusing on the lowest angle reflected spot, called the specular spot, one can see small variations in the reflected intensity during the growth, such as presented on Fig. I.2. This intensity is minimal when there is half a ML grown, and maximal when the ML is fully grown. This is due to the variation of reflectivity of the surface: maximal for a flat surface, minimal for a rough one. Therefore, a period of these oscillation is exactly the growth of a single monolayer [3, 4]. We can also see the relaxation of a layer: the surface becomes then rougher, and the specular sport stays at low intensity.
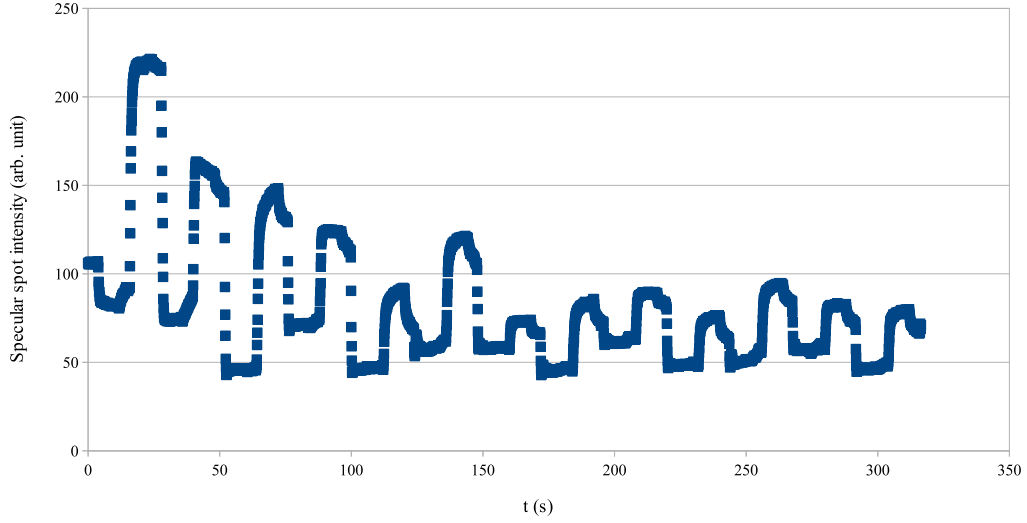
Figure I.2: RHEED oscillation for the ALE of the strained dots.

I.2 Strained dots: CdTe/ZnTE

I.2.1 Substrate preparation

The samples were grown on ZnTe(100) substrates. In order to get the best surface to grow on, we need to clean the sample. Two cleaning methods were tested: etching of the substrate in a Bromure solution, and exposition of the substrate to a hydrogen radical plasma.

The etching process was done in four steps. All of them, except the etching in Bromure-ethanol, occur in an ultrasonic cleaning device vibrating the sample at 43 kHz and last 3 minutes. We began with a cleaning in acetone, followed by one in ethanol. The third step was the actual etching: the substrate was put in a solution of Bromure-ethanol, with 3% of Bromure, during 1 minute. We finally rinsed it in methanol. Once rinsed, we keep the sample in ethanol until fixing them to the sample holder. The growth usually occur the day after the cleaning, the sample being kept in the MBE load-lock chamber, under vacuum.

Another type of cleaning of the surface was tried: using hydrogen radical (H*) to remove the impurity at the surface. This was done to get a smoother surface directly in the chamber, to avoid any contamination that might occur during the transport from the etching room to the MBE chamber. The substrate was rinsed four times, first in acetone, then ethanol and then water, for a duration of 5 min with the cleaning device vibrating at 43 kHz, and finally 5 more minutes in water with the cleaning device vibratin at 23 kHz. It was then put in the MBE main

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chamber to be clean by H* radicals. In order to form the radical gas, a hydrogen gas was ionized in a chamber by a RF power source of 300 W and with a frequency of 13.6 MHz. This gas composition is optically checked by probing the emission of the Balmer series: for a pure hydrogen gas, peaks at 656 nm and 486 nm appear clearly. During the formation of this gas, the substrate temperature is raised to 400°C and we initiate its rotation. Once the plasma is formed, the valve to the main chamber is opened and the substrate is exposed to the plasma for 15 minutes. In order to check the quality of the surface, we look at the RHEED image, that should present strikes.

I.2.2   Strained dots growth

The targeted flux chosen for the growth of the CdTe/ZnTe QDs are presented in Tab. I.1 for each cell used during the growth of strained QDs. These flux were measured via the pressure gauge inside the MBE chamber. It was shown that the best quality of ZnTe was achieved for a growth in excess of Zn [5]. Otherwise, vacancies appear in the bulk, optically visible, and the surface is more rough. Moreover, the adsorption power of the Zn is smaller than the Te. For these reason, we choose to grow the ZnTe barriers in excess of Zn.

Table I.1: Aimed flux for each cell during the growth of the strained QDs.

Elements	Targeted BEP (Torr)
Zn	6.8×10^{-7}
Te	4.5×10^{-7}
Cd	4.5×10^{-7}

As previously stated, the CdTe quantum dots was grown using ALE. In this mode, the flux of the elements has no influence on the quantity deposited at each cycle. The only consideration is whether enough material is deposited to reach the maximal thickness in a cycle. Therefore, the flux for both Te and Cd are chosen to be the same [2].

Beginning the growth, the substrate temperature was initially raised to 415°C. The Zn cell shutter was open starting at 360°C, in order to flatten the surface for the growth. While it took several minutes to raise the substrate temperature, only one Zn layer was deposited due to the auto-regulation of the growth. When the substrate temperature reach 415°C, the Te shutter was also open, in order to grow the ZnTe buffer layer. This thick ZnTe layer guaranteed us to the best possible surface for the growth of the QD layer [6]. The surface quality is checked by the RHEED picture, presenting clear lines (Fig. I.3 (b)). Once done, we position the sample is order to have the specular spot on the RHEED screen, growing

a few more ZnTe level to see the intensity variation of the reflected spot. The substrate temperature was then lowered to 295°C, the Zn cell being open until the temperature reach 360°C, preparing the growth of the dots doped with Cr atoms.
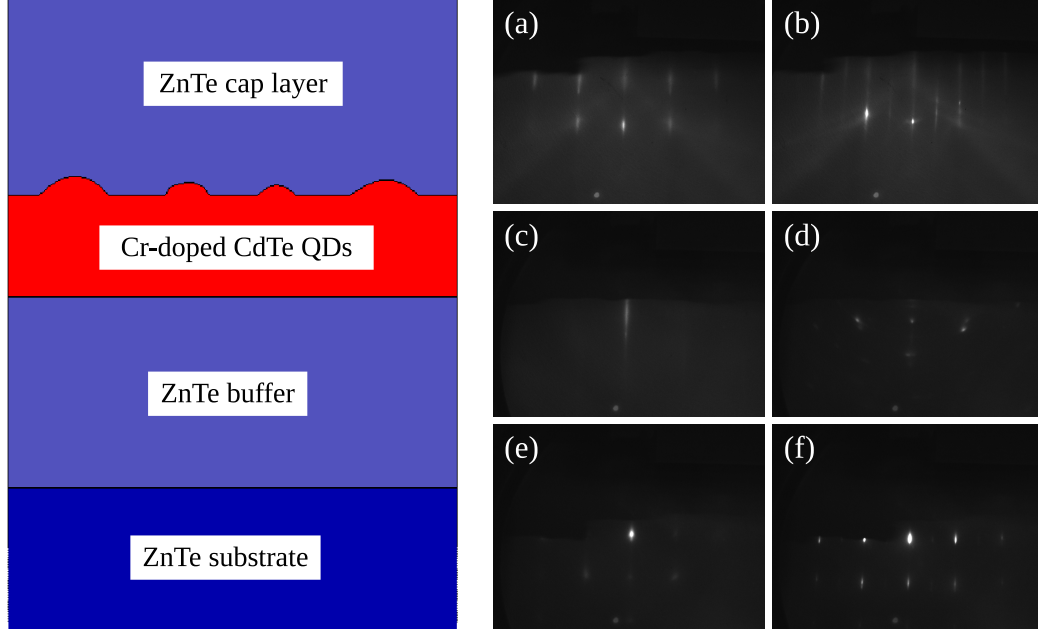

Figure I.3: Left: Layer structure of the strain Cr-doped CdTe QDs samples. Right: RHEED pattern taken at different key moment of the growth: (a) before the growth of the ZnTe buffer, (b) after the growth of the ZnTe buffer, (c) after the (Cd,Cr)Te ALE, (d) after the Te deposition, (e) during the Te evaporation (the picture was taken at $T_{substrate} = 177$°C) and (f) after the growth of the ZnTe cap.

One of the main goal of this work was to calibrate the Cr flux in order to embed only a single Cr atom in most of the QDs of the sample. To achieve this, the Cr density must be of the same order as the QDs density at the surface of the sample. This means a really small flux, with a BEP of the magnitude of 10^{-10} Torr, which is about one order lower than the main chamber pressure and therefore not measurable with our technique. The optimisation was done starting with the know how acquired in Grenoble on the Mn and trying to optimise it for the Tsukuba machine, through a feedback loop with the micro-PL characterization in Grenoble.

This really small flux was achieved by heating the Cr cell around 1000K, low compared to its sublimation temperature, and opening the cell only once during the ALE, for only 5s. In order to have big enough QDs, emitting at right wavelengths, 6.5 ML of CdTe is the optimal thickness. However, the critical thickness of CdTe on ZnTe is 6.5 ML. Dislocations and defect will form in the layer for a higher

thickness. Therefore, some samples were also grown with a 5.5 ML thickness in order to not get too close to the limit. At the chosen temperature, it correspond to either 13 cycles of ALE (for 6.5 ML) or 11 cycles (for 5.5 ML). The Cr cells was opened during the 7th cycle, halfway through the growth of the QD layer, in order to allow the Cr atoms to diffuse without going out the QD layers. The whole ALE recipe to grow the QDs layer is given in the Fig.I.4.
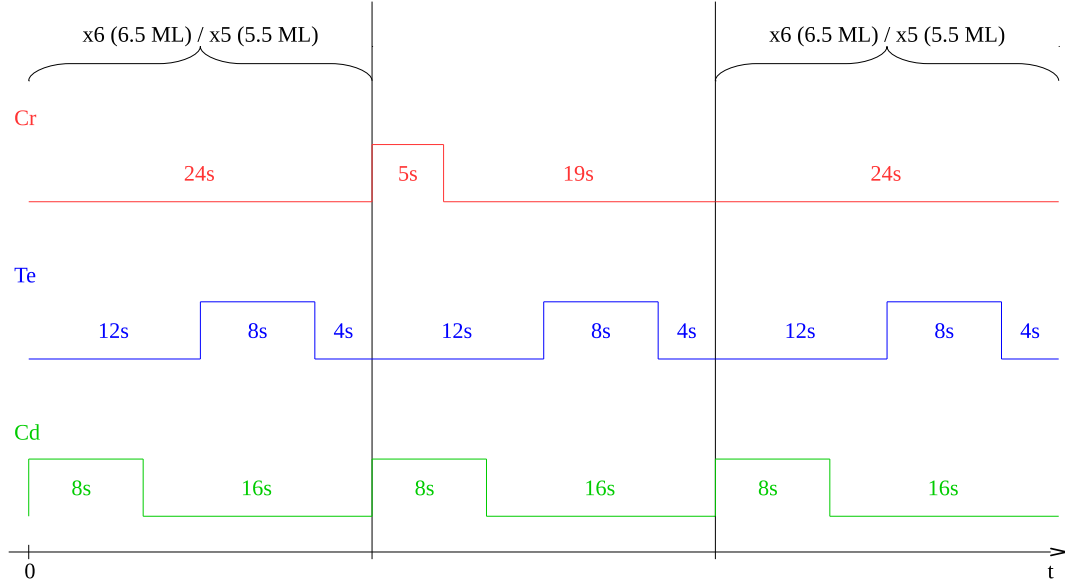

Figure I.4: Opening and closing cycles of each cell for the ALE of strained (Cd,Cr)Te samples.

After the growth of the CdTe layer, we lowered the substrate temperature to 210°C to deposit the Te layer. It was deposited during 5 minutes. This step allows the CdTe layer to relax and form the quantum dots [7]. We then heated up the substrate again until 320°C, were we stayed for 20s in order to evaporate all the deposited Te [8]. If the dots were formed, we saw a spotty pattern like the one presented on Fig. I.3 (f). The Zn and Te cells were then opened, while the substrate temperature was raised to 350°C in order to grow a protective layer above the QDs.

I.2.3 Results

The samples were studied in Grenoble, at the Neel Institute. A high refractive index hemi-spherical Solid Immersion Lens (SIL) was mounted on the sample before their study, to improve the spatial resolution and enhance the collection efficiency

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of a single dot photoluminescence (PL) in a low temperature (T = 5K) optical microscope.

The characterization of the samples came in two times. First, we took macro-photoluminescence spectra, on a large energy range, typically between 1.8 and 2.3 eV, with a laser exciting at 2.9 eV. This allow us to test the luminescence of the sample: if the Cr concentration is too high, it may kill the PL of the dot layer, and thus it will not be seen in the macro-PL. If this luminescence is seen, the sample is then studied by micro-photoluminescence (μ-PL), on a much narrower energy band (about 10 meV), in order to be able to study the dots individually. We scan randomly the sample, searching for dots. We judge the quality of the sample by the proportion of thin emission peaks versus broad ones, large quantity of broad peaks hinting at a high Cr concentration, and by the number of actual dots we found with single Cr embedded inside.

Table I.2: List of samples where Cr-doped dots growth was successfully achieves.

Sample	Cleaning process	CdTe MLs	Cr aimed concentration (%)
dot358	Br etching	6.5	0.06
dot359	Br etching	6.5	0.11
dot363	Br etching	6.5	0.21
dot383	Br etching	5.5	0.19
dot385	Br etching	5.5	0.17

The samples where the Cr concentration was found to be good are listed in Tab. I.2. The Cr concentration was estimated using the Cr flux and the known Mn concentration in CdTe in function of the flux of Mn during the growth. Since the Cr flux can't be measured, it was estimated using Arrhenius law. We see that, for most of the sample, the aimed Cr concentration is above 0.15%. dot358 and dot359 had a lower aimed Cr concentration, but, even though some good dots were found in them, the probability to find one were still low. Tests were done with an aimed Cr concentration at 0.25%, but no luminescence of the dot layer was found. Therefore, in order to maximize the probability of finding a dot with a single Cr while keeping a good PL, we settled for an aimed Cr concentration of 0.17%.

I.3 Other kind of samples

I.3.1 Charge control samples

Charge control samples are pretty straightforward. These samples are grown in order to be able to apply an electric field on the dot layer: a Schottky gate is formed by two conductive layers, deposited on the back and the surface of the sample. Gluing electrode to these conductors, we are able to apply an electric field on the dot layer without inducing a current in. In order to have a conductive back, we did the growths on p-doped ZnTe substrate. Same steps were followed, but with thinner buffer and cap layers, in order to be able to apply a stronger electric field on the dot layer. We chose to do both about 150 nm thick.

Table I.3: Measured conductivity of the Au layer on GaAs for different deposition time.

Gold deposition time (s)	Resistance (Ω)
15	300
20	80
25	25
30	20
35	0
60	0

The conductive surface was formed by a thin, semi-transparent gold layer, deposited by sputtering. The samples were kept in nitrogen atmosphere during the transport. The exposition time of the sample in the sputtering machine was calibrated using gold deposited on GaAs substrate. In order to keep a collection of the light emitted by the quantum, a thin layer was necessary. However, for the applied electric field to be unformed, we also needed a gold layer thick enough to be uniformly applied on the entire surface. Such a uniform layer means the resistance of the sample surface fall to 0 Ω. Results of the resistance measurement are presented in Tab. I.3. A deposition time of 35 s was chosen.

Results

We successfully grew these samples and studied them with μ-PL. We were able to look at the luminescence of a dot and see the evolution of its PL under the application of an bias voltage. Fig. I.5 (a) presents the results of such an experiment.

The main result of the experiment is the apparition of different species depending on the applied electric field. We see for example that, for a bias voltage of +3

V (Fig. I.5 (b)), the neutral species are almost extinguish and the charged species X^+, X^{++} and X_2^+ have the more intense PL. Similarly, at negative electric field, only the charged species X^-, X^{--} and X_2^- remain, X^{--} and X_2^- disappearing for an applied bias voltage higher than -2 V. This shows that we can select efficiently the charge of a studied quantum dot applying a bias voltage on it.
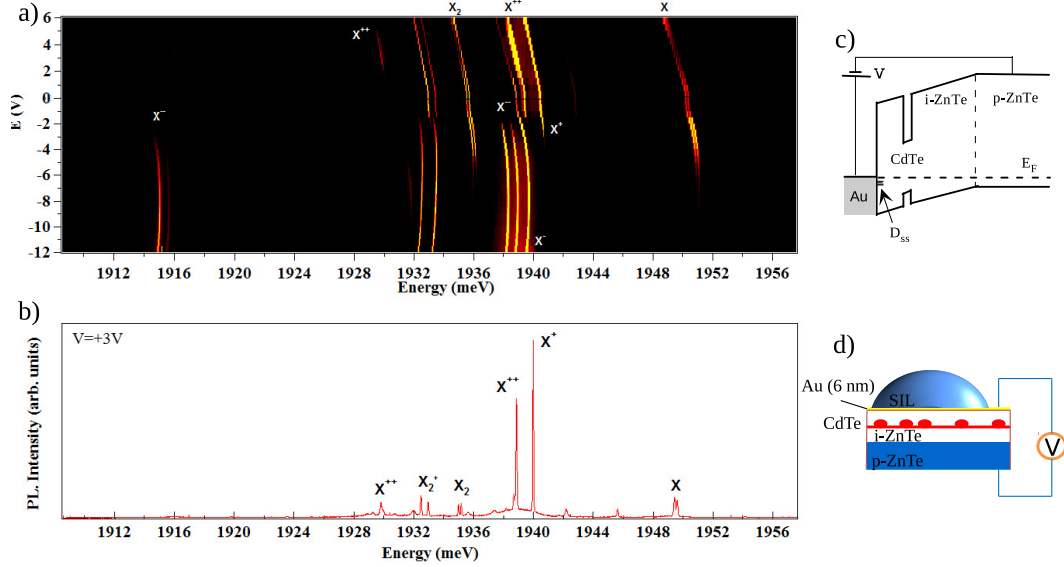

Figure I.5: (a) Evolution of the PL of a single dot under application of a bias voltage. (b) Spectra of the dot under application of a bias voltage V = +3 V. (c) Evolution of the energy structure of a CdTe dot and the i-ZnTe barrier under application of an electric field via the Schottky gate. (d) Schema of a charge control sample used for the μ-PL experiments.

Fig. I.5 (c) gives a schematic view of the Schottky gate. In this compound, the Fermi levels of the metal, semiconductor and p-doped semiconductor align via Fermi level pinning. Applying a bias voltage to the back and front of the sample change the position of the Fermi level, and therefore the probability of injection of hole or an electron in the dot, depending on the direction of the applied voltage. Moreover, the energy barriers at the interfaces keep the current to flow in the non-doped part of the semiconductor. We can then apply an electric field on the sample, and thus control the charge of the sample, without having a current going through it.

Only one charged sample containing Cr atoms was studied: dot390. It was cleaned by H* plasma. The QD layer was 5.5 ML thick and the Cr concentration was aimed to be 0.16%. However, no Cr doped quantum dots were found. Some dots presenting a emission close to the one expected for Cr atoms were found, but

they were revealed to not have any magnetic atom inside. These are discussed in more in Sec. IV.4.

I.3.2 Strain-free quantum dots

Strain-free quantum dots are formed by the thickness fluctuations of a CdTe quantum well in $Cd_xMg_{1-x}Te$ barriers. These fluctuations form steps localizing the carriers, acting as QDs. The higher the Cd concentration will be, the closer the lattice parameter will be, but it will also reduce the gap energy difference, reducing the confinement. The best strain to confinement ratio was found for CdTe QW in $Cd_{0.7}Mg_{0.3}Te$. The needed flux for this growth are shown in Tab. I.4.

Table I.4: Aimed flux for each cell during the growth of the strained samples.

Elements	Targeted BEP (Torr)
Cd	4.5×10^{-7}
Mg	1.6×10^{-8}
Te	5.26×10^{-7}

The strain-free sample were grown on an hybrid substrate. It was made from a GaAs substrate, cleaned using H* plasma. We grew on it a CdTe layer of about 3 μm, in order to recover the lattice parameter and not have defect close to the surface. With this thickness, the remaining strains in the layer should be of about 0.1% [9, 10]. A thin layer (about 7 nm) of ZnTe was grown between the GaAs and the CdTe in order to help the relaxation of strains [11]. The RHEED taken after the growth of the CdTe layer (Fig. I.6 (d)) shows sharp straight line, hinting at the recovery of a flat surface. A protective layer of amorphous Te was grown on the surface to keep it from being damaged outside the MBE chamber.

We began to heat the substrate temperature to 300°C, in order to remove the protective amorphous Te layer. We waited a few seconds at this temperature to remove all the deposited Te, and then resumed the heating to go to 360°C. Starting at 320°C, we opened the Te cells in order to stabilize the surface. When the substrate temperature was stabilized at 360°C, we opened the Cd cells and grew a 2.35 μm layer of CdTe, in order to be as close as possible of a total lattice parameter recovery [9, 10].

We grew the first $Cd_{0.7}Mg_{0.3}Te$ barrier on this buffer layer. In order to be sure that there will be no relaxation in the quantum well, we choose to stick to the maximum cumulated thickness of the CdTe on a CdZnTe lattice, which has been shown to be lower than the one of CdTe on a CdMgTe lattice. This correspond to a maximum cumulated thickness of 130 nm [12]. We chose to grow 40 nm below the QW, and 90 nm above it, in order to have a thicker protective layer.
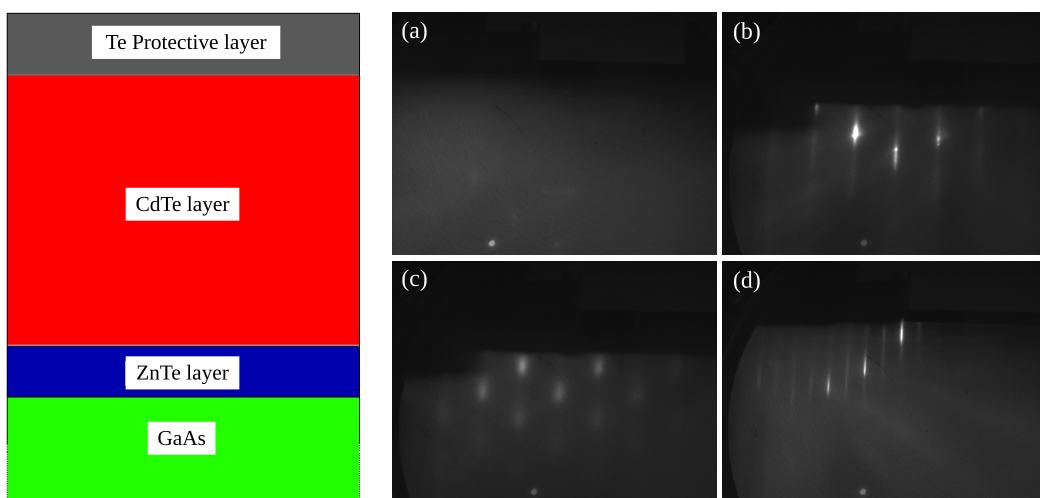
Figure I.6: Left: Layer structure of the hybrid substrate with its protective Te cap. Right: RHEED pattern taken at different key moment of the growth: (a) before H* cleaning of GaAs, (b) after H* cleaning of GaAs, (c) after the growth of the ZnTe layer, (d) after the growth of the CdTe layer.

Once the 40 nm barrier layer was grown, we lowered the substrate temperature under Te flux. Growing the QW layer in a Te environment smooth the surface layer of the sample and help having a flat surface to grow the well. Once the substrate temperature reach respectively 295°C, we began the ALE of the QW. Two QW thickness was tested: either 4 ML or 2 ML. In both case, the growth was done growing CdTe layer as done for the strained sample, opening the Cr cell for 3 s for one cycle every two cycles. The whole recipe is described in Fig.I.7.

We then raised the substrate temperature up to 360°C, under a Te flux, in order to proceed to the growth of the upper barrier, acting also as a protective layer. The opening time was there calculated to grow 90 nm of $Cd_{0.7}Mg_{0.3}Te$.

Results

Four sample of strain-free dots doped with Cr were produced, listed in Tab. I.5.

The sample presented thin and intense peaks. It hints at a better confinement of the carriers in the QDs than what have been expected from dots formed by the thickness variation of a quantum well. This may be caused by higher steps than expected at the $CdTe/Cd_{0.7}Mg_{0.3}Te$ interface.

As discussed in Sec. I.4, the presence of a magnetic atom splits the emission of the exciton into several peaks, the number depending on the spin of the magnetic atom. However, such complex wasn't found in the strain-free samples. It may be
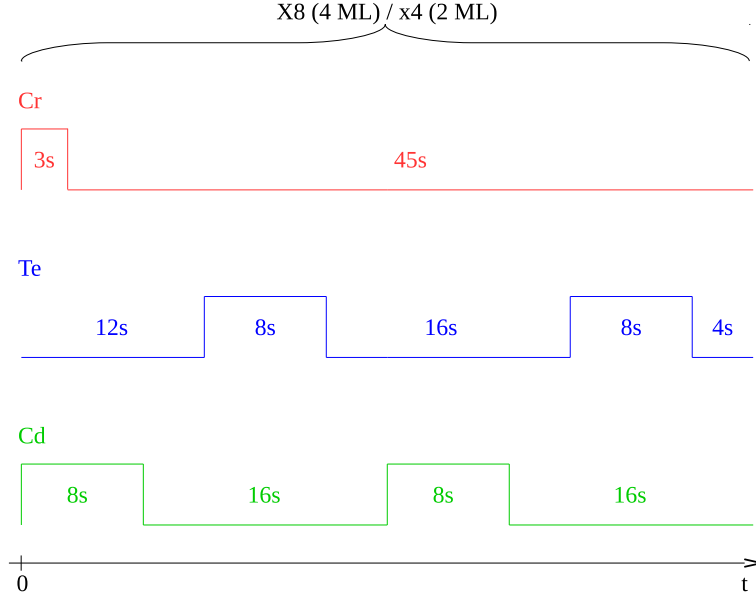
Figure I.7: Opening and closing cycles of each cell for the ALE of strain free (Cd,Cr)Te samples.

Table I.5: List of sample grown trying to incorporate Cr in SFD dots.

Sample	CdTe MLs	Cr aimed concentration (%)	Probability of Cr-doped QD
SFD4	4	0.35	None found
SFD5	2	0.15	None found
SFD6	2	0.54	None found
SFD7	2	0.35	None found
SFD8	2	0.75	None found

caused by the the absence of static Jahn-Teller effect. In strain dots, this effect increase the probability of the Chromium to have a quantization axis along the growth axis z, making the splitting visible in our experimental setup. Without strain, the Jahn-Teller effect doesn't discriminate anymore between the three different axis, and thus the Chromium spin might have a quantization axis along x or y. In this case, there is no visible splitting for the quantum dot in our experimental setup, and the magnetic atom goes undetected.

In order to counter that, it was proposed to slightly strain the quantum well. This might be done by incorporating a low density of Zn in the $Cd_{0.7}Mg_{0.3}Te$

barrier. The strains created this way should be enough to increase the probability of quantization along the z axis, while staying negligible in regard of the Cr spin energy structure and dynamics. Such samples should be grown in the near future.

Conclusion

We saw in this chapter how we grew the samples that will be studied in the next chapters. We used the characterization of the samples done in Tsukuba to narrow down on a Cr flux for the growth of Cr-doped QDs, and we decided to aim for a Cr estimated concentration in the sample of 0.17%. We then went to two other kind of sample: one enabling the application of an electric field on the dots, and one with strain free dots. The charge control samples growth was a partial success: we were able to apply an electric field on the sample, but no dot containing a single Cr was found. Strain-free dot presented promising fine peaks in their emission, but we were not able to find Cr in them either. More experiments will be done in order to successfully grow sample with the possibility of charge control, and strain-free dots.

In the next chapters, we will study with μ-PL dots found in the samples grown in Grenoble (Chap. III) and Tsukuba (Chap. IV and Chap. V).

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