# Quantitative Scanning Capacitance Spectroscopy on GaAs and InAs Quantum Dots

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In this work, quantitative scanning capacitance spectroscopy studies on bulk GaAs and InAs quantum dots are carried out in ambient atmosphere. The experimental results are well described by a simple spherical capacitor model, and the corresponding barrier heights and sample dopings are determined from the measured data. We further find a strong dependence of the C(V) data on the applied tip force. The barrier height is decreasing significantly with increasing pressure.

## Introduction

Scanning Capacitance Microscopy/Spectroscopy (SCM/SCS) is an extension of conventional Atomic Force Microscopy (AFM) and a promising tool for two-dimensional carrier profiling in semiconductor devices. In SCM/SCS, a conductive AFM tip is used to measure the local capacitance between the tip and the sample. The current state of the art of this technique can be found in the review articles [1] – [4].

In scanning capacitance spectroscopy (SCS), the tip-sample capacitance is recorded during a DC voltage sweep to obtain a capacitance versus voltage (C(V)) curve [5]. However, quantitative SCM/SCS measurements are still a major challenge for technical and physical reasons. First, the capacitance between the AFM tip and the sample is in the aF regime only. To obtain a reasonable signal size at reasonable data collection speed for imaging, lock-in techniques are normally used. Thus, commercial SCM systems usually yield qualitative dC/dV data only. To obtain quantitative results, these data have to be calibrated. This, however, is technically complicated due to large difficulties with the reproducibility of the reference sample preparation process. In addition, the currently commercially available SCM systems do not operate at small signal conditions, which further complicates the data evaluation and makes sophisticated simulation methods almost inevitable [1] – [4].

To circumvent at least the reproducibility problems with reference samples, quantitative scanning capacitance spectroscopy can be used. In our previous work [6] we have demonstrated that this method is e.g. useful for a nanoscale analysis of high-k dielectric materials such as $ZrO_2$ and a pointwise calibration of capacitance images obtained by commercial SCM systems.

While a large amount of literature exists on SCM measurements on silicon samples, significantly fewer publications can be found on SCM on GaAs and other III-V materials. Besides very innovative approaches such as X-ray absorption measurements by SCM [7] and local capacitance measurements on InAs dot-covered GaAs surfaces by scanning capacitance microscopy [8] under high vacuum conditions, most of the SCM work on GaAs was devoted to the SCM characterization of laser structures [9], [10]. Douheret, e.g., has shown that SCM can provide a complete 2D map of the device structure, including doping variations, the location of p-n junctions, and regrown interfaces.

In this paper, bulk GaAs and InAs quantum dots are studied by scanning capacitance spectroscopy in ambient atmosphere. It is found that the experimental data agree well with capacitance spectra calculated from a simple spherical capacitor model. The donor concentrations obtained from this model are in excellent agreement with the sample parameters and the measured Schottky barrier heights are consistent with data obtained by Ballistic Electron Emission Microscopy on similar samples [12]. The influence of illumination and tip force is found to be very critical.

## Experimental Preliminaries

All samples we used for our experiments were MBE grown GaAs layers on semi insulating GaAs substrates. The layer thickness was 1 µm for all samples and the doping was $N_d = 1 \times 10^{16}$ cm$^{-3}$ and $N_d = 1 \times 10^{17}$ cm$^{-3}$ for the GaAs bulk samples, respectively. The “on surface” quantum dot sample we used had the same layer structure and was also doped at a level of $N_d = 1 \times 10^{16}$ cm$^{-3}$.

For all capacitance measurements, an ultrahigh precision, low frequency (1 kHz) capacitance bridge (*Andeen Hagerling* 2550) was used, which allows capacitance and loss measurements under well controlled, small signal conditions down to the aF regime. A (*Keithley*) source measure unit was used to provide the DC voltage component. External coax cables from the capacitance bridge were attached on both the sample and the AFM tip holder. The AFM was only used for tip positioning and for the tip-sample approach. As tips we used highly doped (p-type $1 \times 10^{20}$ cm$^{-3}$) conductive diamond tips (*Nanosensors*, Germany). The spring constant of the cantilevers was 42 N/m and the typical tip radius was 100 nm according to the data sheet. Note that this type of cantilevers is normally used for scanning spreading resistance measurements only; the reasons for using these tips are described below.

In contrast to standard SCM/SCS measurements, where the applied frequencies are so high (≈1 GHz) that light induced charge carriers cannot follow the HF fields, our setup operates at a frequency of 1 kHz. Here, the measurement turned out to be extremely sensitive to the influence of light. Therefore, the AFM hardware was modified in order to switch off the laser and all other illumination and the feedback loop was controlled externally during the C(V) measurements [11]. To minimize vibrations and acoustic noise, the AFM system was placed on an air suspended table and put into an acoustic hood. This also allowed an operation under temperature stable conditions which turned out to be crucial. In detail, the temperature variations were kept below ±10 mK during the measurements. Due to improved shielding measures and larger signals compared to our earlier experiments [6], a reasonable signal to noise ratio was already obtained at moderate averaging times and a typical C(V) spectrum took 20 minutes.

## Results and Discussion

Figure 1 (a) shows typical C(V) curves, which were obtained in complete darkness. The force we used for these measurements was 2.9 µN. Curve (1) was recorded on bulk GaAs with a doping of $N_d = 1 \times 10^{16}$ cm$^{-3}$. Curve (2) was bulk GaAs too, with $N_d = 1 \times 10^{17}$ cm$^{-3}$, and curve (3) was obtained on a InAs quantum dot on GaAs substrate with a doping of $N_d = 1 \times 10^{16}$ cm$^{-3}$. Obviously, the onset of these C(V) spectra is shifted to lower bias for higher substrate doping and the onset of the C(V) curve obtained on a quantum dot is even lower.

As already found by Yamamoto [8], a simple parallel plate capacitor model cannot be used to describe AFM based capacitance spectra on GaAs. The reason for this lies in the nature of the Schottky contact between the tip and the GaAs underneath. In a Schottky contact, the extent of the depleted space charge region determines the capacitance of the contact. As the depletion length easily reaches several hundred nanometers on low doped samples and the tip radius is much smaller, a simple parallel plate capacitor model is not appropriate for geometric reasons. In contrast to that, the active region in Metal-$SiO_2$-Silicon (MOS) junctions is always directly at the Si-$SiO_2$ interface directly underneath the tip, which is the reason why simple parallel plate capacitor models on Si samples normally yield results which are correct at least within an order of magnitude.

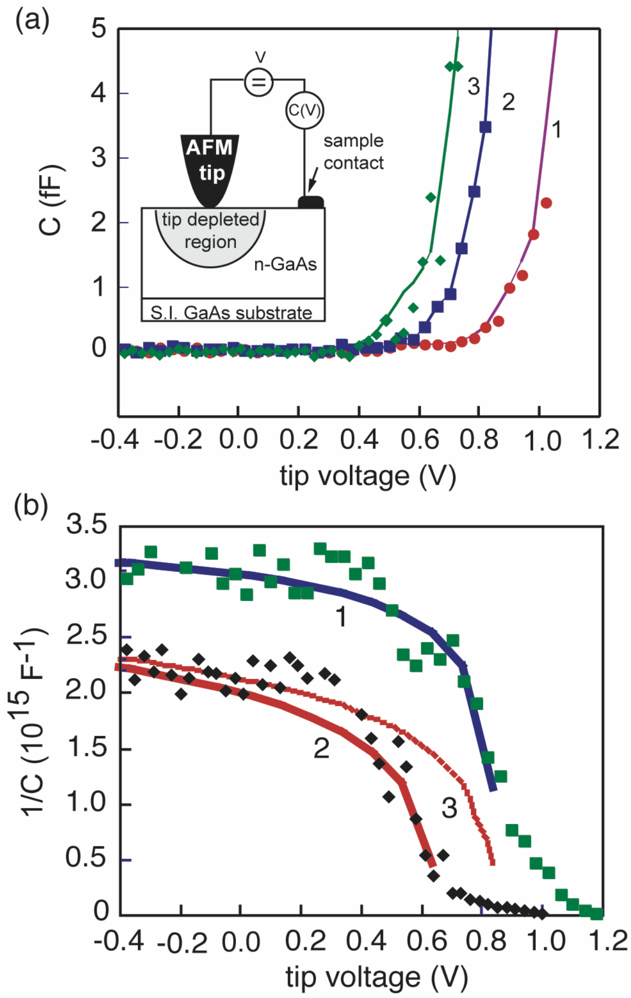

Fig. 1: (a) Capacitance – voltage (C(V)) spectra measured on three different samples (curves 1 – 3). (1): bulk GaAs with $N_d = 1x10^{16}$ $cm^{-3}$, (2): bulk GaAs with $N_d = 1x10^{17}$ $cm^{-3}$, (3): InAs dots on GaAs with $N_d = 1x10^{16}$ $cm^{-3}$. The solid lines connecting the data points are only a guide for the eye. A schematic view of the experimental setup is shown in the inset. (b) Capacitance data for the high and low doped bulk sample plotted as 1/C over bias. The solid lines were calculated using a spherical capacitor model. Details are found in the text.

To analyze our data, we therefore applied a simple model assuming a capacitor consisting of two concentric spheres. The capacitance of such a spherical capacitor is calculated as $C = 4\pi\varepsilon_r\varepsilon_0\left(\frac{r_1 r_2}{r_2 - r_1}\right)$, where $\varepsilon_0$ is the dielectric constant of vacuum, $\varepsilon_r$ is the relative dielectric constant and $r_1$ and $r_2$ are the radii of the inner and outer sphere. To estimate the capacitance of our tip-semiconductor system we simply set $r_1 = r_{tip}$, where $r_{tip}$ is the tip radius and $r_2$ is set to $r_2 = (r_{tip} + d)$, where $d$ is the depletion layer thickness in GaAs. We further assume that $\varepsilon_r = 13$ and ignore all influence of the dielectric constant of the diamond tip and the surrounding air. For a given Schottky barrier height $V_b$, the depletion layer thickness $d$ is calculated as: $d = \left(2(V - V_b)\varepsilon_r \varepsilon_0 /(eN_D)\right)^{1/2}$, where $V$ is any applied external voltage, and $N_D$ the doping concentration. Finally we take for the tip-sample capacitance $C_{TS}$ a value of $C_{TS} = C/2$ only, because we just consider the lower half sphere of the capacitor. As long as $d > r_1$, which is easily achieved on low doped substrates, this will be a reasonably good approximation.

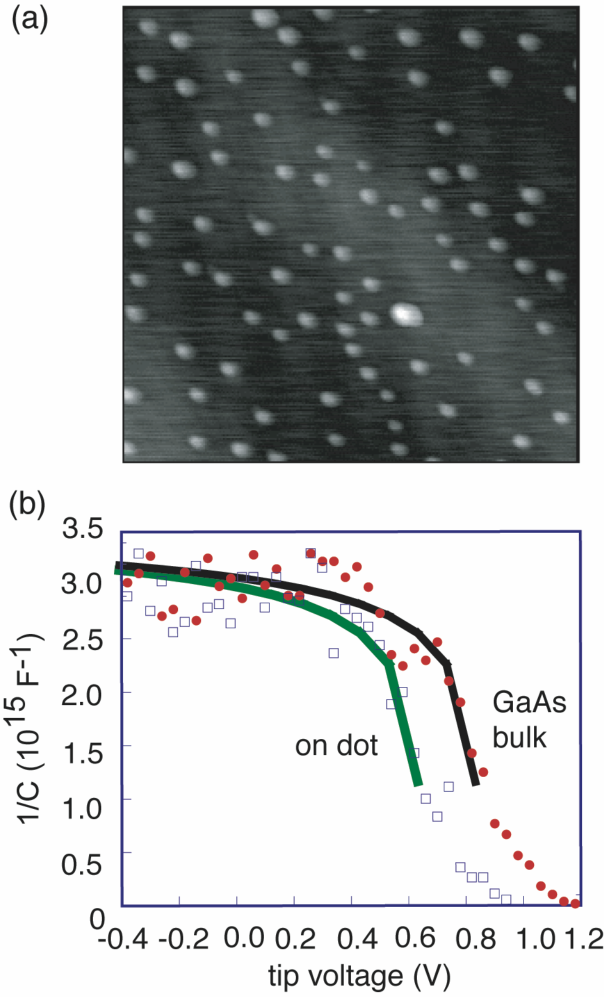

Fig. 2: (a): AFM image of the quantum dot sample. Scan size is 1µm x 1µm. The typical dot diameter is 40 nm. (b): 1/C(V) curves obtained on the low doped bulk sample and on quantum dot positions. The solid lines were calculated using a spherical capacitor model. Details are found in the text.

To compare the model with the experimental data, it is helpful to plot $1/C_{TS}$ versus tip-bias similar like the $1/C_2$ versus bias plots, which are usually made for C(V) curves on Schottky junctions assuming parallel plate capacitors. For the low doped sample, the agreement between the experimental data and the primitive model is amazing. Figure 1 (b) shows a comparison of the measured and calculated curves. Curve (1) shows the result for a substrate doping of $N_d = 1x10^{16}$ cm$^{-3}$, a tip radius of 90 nm, and a Schottky barrier height of Vb = 0.9 eV. The background stray capacitance was also subtracted. Below the barrier height, the agreement is really excellent. For bias values above the barrier height, the model can not be applied. Curve (2) shows the result for the sample with the higher doping ($N_d = 1x10^{17}$ cm$^{-3}$). In agreement with our simulation, the reciprocal of the measured signal becomes smaller with increased doping level. To fit the shifted onset position, an “effective” barrier height of $V_b$ = 0.7eV was assumed to account for the tunneling effects through the thinner Schottky barrier at higher doping levels. However, the overall agreement between the calculation and the experiment is still not as good as for the low doped substrate. In our opinion, this is due to the smaller extension of the depleted region, where the primitive spherical capacitor approximation becomes less accurate. For comparison, the result of an equivalent simulation assuming a barrier height of 0.9 eV is also shown in curve (3).

Motivated by the good agreement between theory and experiment on bulk GaAs, the same measurements were also carried out on InAs quantum dots. An AFM topography of our sample can be seen in Figure 2 (a). The dots have a typical diameter of 40 nm on this sample. Figure 2 (b) shows the corresponding capacitance data.

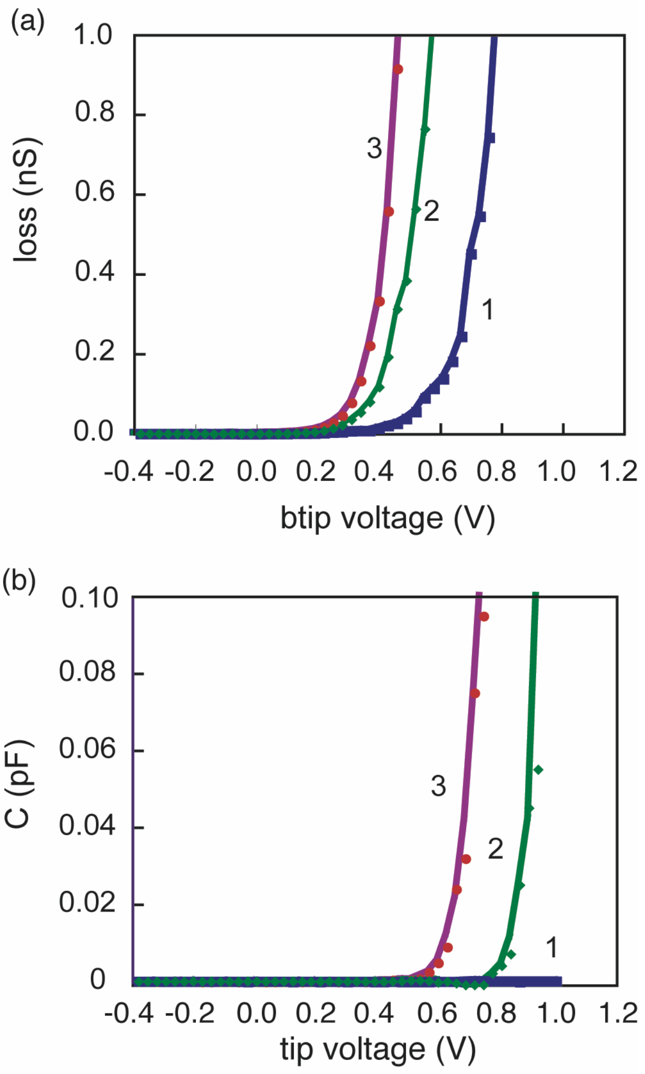

Fig. 3: (a) Loss curves recorded on bulk GaAs ($N_d$ = 1x$10^{16}$ $cm^{-3}$) using forces of 2.0 µN, 3.8 µN and 6.1 µN, for curves (1 – 3), respectively. (b) Simultaneously measured C(V)-spectra.

Compared to the GaAs bulk data, the “on dot” curve looks qualitatively the same and is just shifted to the left. The calculated “on-dot” curve was obtained using a barrier height of $V_b$ = 0.65 eV, which is in excellent agreement with the band offset between InAs dots and GaAs determined earlier [12] by Ballistic Electron Emission Microscopy (BEEM).

This indicates, that the InAs dot acts as intermediate contact to the tip and that the capacitance is dominated by the behavior of the InAs-GaAs system and not the tip. A similar behavior was already found by Yamamoto et al. [8].

As final point we want to discuss the influence of the tip-sample force, which turned out to have a significant influence on the results. Figure 3 (a) shows typical loss (differential conductance) spectra on the low doped GaAs bulk sample as a function of tip-force. Curves (1) – (3) were measured using tip forces of 2.0 µN, 3.8 µN and 6.1 µN, respectively. As in conventional spreading resistance measurements, the loss increases with increasing force between tip and sample. It must be pointed out, however, that the minimal force ($F \approx 1$ µN) to obtain reproducible loss data is surprisingly high. For all our measurements we had to use diamond coated “scanning spreading resistance cantilevers” with a high spring constant ($C = 42$ N/m). With conductive diamond coated “contact mode cantilevers” ($C = 1$ N/m) the forces turned out to be too low to achieve reliable conductance data. The physical origin for this behavior is probably found in the ambient conditions where the measurements were carried out. Under ambient conditions, all samples are usually covered with a thin film of water. Especially on semiconductors, one can expect an additional native oxide, too. Thus, the existence of a force threshold to penetrate these films with an AFM tip is not that surprising, the rather high amount of force, however, is.

For local capacitance measurements, the situation is even worse. Even at tip forces where a clear local conductance via the tip is already observed (Figure 3 (a), curve (1)), no capacitance signal can be detected (Figure 3 (b), curve (1)). At a tip force of 3.8 µN, which turned out to be the minimum value for reproducible capacitance measurements on our sample, we obtain typical data as they are shown in curve (2). Under these conditions, we obtain a barrier height of $V_b = 0.9$ eV as discussed above. At a tip force of 6.1 µN (curve (3)), the capacitance curve is shifted to the left and the barrier height is reduced and our model yields a significantly reduced value of $V_b = 0.55$ eV. Going to higher pressures was impossible, since this normally led to tip destruction before further significant changes could be detected.

On the reasons why the threshold force for reproducible capacitance measurements is even higher than for conductance measurements, we can only speculate. Most probably, however, any intermediate layer (water, oxide) had to be penetrated completely by the tip before a reasonable Schottky contact is established. In contrast to that, thin oxide layers would show a finite conductance already much earlier due to tunneling effects.

On the quantum dot sample, systematic pressure studies were unfortunately impossible, since the dots were considerably damaged at higher tip forces. In addition, we noticed that tip forces greater than 4.7 µN had to be used before any capacitance signal could be recorded on off-dot positions on the wetting layer. Since the samples were not grown freshly, the wetting layer was probably oxidized completely. However, we have no explanation why an oxidized InAs wetting layer is significantly thicker or less conducting than native GaAs oxide and we also found no information in the literature on this topic.

## Summary

In summary, quantitative AFM based capacitance studies were carried out in air on bulk GaAs and InAs quantum dots. All C(V) data were well described by a simple spherical capacitor model on all samples, and substrate doping as well as the corresponding barrier heights were determined and in agreement with reference data. We also found a force threshold for reproducible capacitance measurements and a significant influence of the tip sample force on the obtained results.

## Acknowledgements

This work was sponsored by "Fonds zur Förderung der wissenschaftlichen Forschung (FWF)" project No P16337-N08. The authors are grateful to J. Prinzinger for technical assistance.

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