Estimation of doping density in low doped n-InAs by electrolyte-based capacitance-voltage measurements in the deep depletion mode

D Frolov and V Zubkov
Department of micro- and nanoelectronics, St. Petersburg Electrotechnical University “LETI”, Prof. Popov str. 5, St. Petersburg 197376, Russia
E-mail: dsfrolov@etu.ru

Abstract. Doping density profiling in InAs is difficult due to lack of good quality Schottky contacts and the narrow bandgap nature of the material. The electrolyte can be used to form a Schottky-like contact but the inversion layer formation does not allow obtaining a dopant concentration in low doped InAs. To overcome this issue, a pulse CV technique has been implemented to drive a sample into the deep depletion mode which makes it possible to use the depletion approximation to calculate a carrier concentration. The measured capacitance-voltage characteristics were compared with the simulation and showed good agreement with it.

1. Introduction

The capacitance-voltage profiling technique requires a junction in which a space-charge region can be created [1]. Schottky contacts are traditionally used for this purpose, but due to formation of an accumulation layer at the surface, the junction between metal and n-InAs has a negative barrier height [2,3]. The Schottky contact in InAs can only be formed in undoped or p-type structures [4]. As an alternative to the Schottky contact some other structures, like a MOS capacitor [5], a p-n barrier junction [6] or an electrolyte-semiconductor interface [7], can be used to make a space-charge region in InAs.

Choosing the electrolyte to form a Schottky-like junction has some advantages over other methods since it does not require complicated technological processes to create contacts and can be used together with the electrochemical etching of InAs. These advantages of electrolyte are implemented in the electrochemical capacitance-voltage technique (ECV) [8].

Although ECV is used for characterization of vast number of semiconductors, its application to InAs faced some difficulties [7,9]. InAs is a narrow band gap material and its conduction band edge is located below the electrochemical potential of the electrolyte. Because of that, the accumulation layer is formed at InAs surface in thermal equilibrium, whereas in most of other materials the depletion occurs. That makes the CV behavior in InAs similar to one in metal oxide semiconductor (MOS) structures, with a Helmholtz layer at the electrolyte-semiconductor interface acting as an isolator. Additionally, in the standard CV method, the capacitance is measured with a small voltage step and a long integration time comparing to minority carriers response time. This slow variation of bias voltage leads to the formation of an inversion layer in InAs that makes the doping concentration extraction very difficult.
Similarly to MOS structures, when a voltage pulse of a duration shorter than the thermal generation time constant is applied to the electrolyte-semiconductor interface, no inversion layer can be formed. This fact is used in certain CV techniques to drive a sample into the deep depletion mode. The deep depletion can be accomplished by a linear-sweep technique [10], a pulse CV technique [11] or by applying the fast ramp voltage [12]. In present work, a pulse CV technique has been used since it allows more precise control of the timing between measurement cycles.

2. Measurement technique
To implement pulse CV measurements the following instrumentation was assembled (figure 1(a)). The differential capacitance was measured using a standard technique by superposing a small ac voltage (of small amplitude) on the bias voltage and measuring the ac current through the sample [13]. An arbitrary waveform generator (PXI-5412) was used to generate both dc bias and ac test signal. The generator was connected to the electrochemical cell through an operational amplifier in the potentiostat configuration to control the voltage difference between the sample and the reference electrode (RE).

Because of the small energy bandgap of InAs, there is only a very small bias range to make capacitance-voltage measurements. Leaving the electrochemical cell connected to the circuit one can get uncontrolled formation of an anodic layer. To prevent the surface of the sample from oxidation during delay time between voltage steps, the output of an amplifier was connected to the counter electrode (CE) through the reed relay. The relay was controlled by PXI-4132 SMU (a source-measure unit) which was also used to generate the trigger signal to synchronize the start of the waveform generator with relay actuation.

The second operational amplifier in transimpedance configuration was used to make the direct conversion of the current into a voltage signal. The reference voltage and the voltage signal at the output of transimpedance amplifier were captured by a digital oscilloscope. The schematic representation of applied voltages and the measured current shown in figure 1(b).

To obtain the capacitance, the real and imaginary parts of the impedance have been calculated first, see equation (1).

\[
Z = \frac{v_m}{i_m} e^{j\Delta\phi}
\] (1)

The impedance magnitude was calculated from the amplitude ratio of the voltage applied to the cell \(v_m\) and the displacement current \(i_m\) through it. The phase angle of complex impedance
A parallel equivalent circuit was used for capacitance calculation due to the fact that InAs has a large surface conductivity which acts as a competitive path of current. The admittance of the parallel equivalent circuit may be written as

\[ Y = \frac{1}{Z} = G + j\omega C_p \]  \hspace{1cm} (2)

where \( G \) is conductance and \( C_p \) is capacitance.

The described experimental setup was successfully tested with various combinations of resistors and capacitors connected in place of electrochemical cell at a range of frequencies up to 1 MHz.

3. Results and discussion

The epitaxial InAs structures were grown by HVPE on \( n^+ \)-InAs substrates. Doping densities were studied in epi layer and in substrates. The pulse capacitance-voltage measurements were performed with the experimental setup described above. For standard measurements a LCR meter Agilent E4980A have been connected directly to the electrochemical cell. The aqueous solution of 0.1 M NaOH was used as an electrolyte. Contact area between the electrolyte and the sample was 0.1 cm\(^2\). All CV measurements were performed at room temperature.

The model used for capacitance-voltage simulation was described in details in our previous work [9]. CV characteristics were calculated from the numerical solution of the nonlinear Poisson equation with modified Tomas-Fermi approximation. Deep depletion capacitance was calculated by setting the minority carrier concentration to zero. CV plots were calculated for doping concentration \( N_D = 1 \times 10^{15} \text{ cm}^{-3} \).

The capacitance-voltage characteristics for InAs measured in standard and pulse modes are compared in figure 2(a). Both characteristics have been measured with the frequency of 300 Hz and amplitude (\( v_m \)) of 30 mV. The frequency was selected to minimize the dissipation factor, but to be high enough to have at least 10 cycles during the measurement time. In pulse CV at every measurement point the dc voltage was pulsed from an accumulation voltage (\( v_{\text{acc}} \)) to the voltage at which the data are to be taken (\( v_{\text{bias}} \)), as shown in figure 1(b). The settling
time before the start of main measurements was about 10 msec. The delay time between each measuring cycle was ranged from 100 msec to 1 sec.

In the standard CV mode the inversion starts at positive biases, which results in an increase of capacitance and a drop on $C^{-2} - V$ curve. In contrast, the good linearity of Mott-Schottky plot can be seen in pulse CV at voltages above +0.05 V. Using the Mott-Schottky equation in this region we calculated the corresponding free carrier concentration to be of $1 \times 10^{15}$ cm$^{-3}$ that is in good agreement with the expected doping concentration. It should be noted that depending on the delay time between each measurement, the bias range, in which $C^{-2} - V$ follows the linear behavior is changing. It can be related to the growth and dissolution of anodic layer at the surface of InAs.

Figure 2(b) shows the transient capacitance measured with the bias step from accumulation to depletion. The experimental data were fitted by an exponential function. The time constant was calculated to be 1.5 sec which is much longer than the selected settling time.

4. Conclusions
At lower doping levels in n-InAs the standard (equilibrium) CV does not provide the correct value of dopant concentration. This happens for the reason that the inversion starts too early due to a small band gap of indium arsenide. To prevent the inversion layer formation, a pulse CV technique has been used. Here the fast bias pulse drives a sample into the deep depletion mode. The concentration obtained from the linear approximation of $C^{-2} - V$ in the deep depletion mode is in good agreement with the known doping concentration in low doped InAs. The proposed technique can be used for characterization of InAs with doping levels from $1 \times 10^{15}$ cm$^{-3}$ to $1 \times 10^{20}$ cm$^{-3}$. At doping levels below $1 \times 10^{15}$ cm$^{-3}$ the intrinsic carrier concentration will dominate at room temperature. At very high doping levels the parasitic conductivity through the surface will decrease the accuracy of capacitance measurement results.

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