Method to predict and optimise charge sensitivity of ungated AlGa_N/Ga_N HEMT-based ion sensor without use of reference electrode

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Abstract— In this paper we report on a methodology for theoretical prediction and optimization of charge sensitivity for ungated AlGa_N/Ga_N HEMT-based ion sensors operated in reference electrode free configuration. We have performed numerical simulations of device sensitivity, specifically the change in channel electron concentration with the change in surface potential, for different Al mole fractions and AlGa_N thicknesses. These results can be used for device optimization, signal analysis and sensor calibration purposes. To validate the model, six ungated AlGa_N/Ga_N transistor-based devices of different Al mole fraction and AlGa_N thickness were fabricated. These devices were exposed to KOH solutions with different pH values, and the voltage change at the gate area was indirectly measured as a function of ionic concentration. The gain in conductivity across the measured range of pH values was experimentally extracted for each device and closely matched the sensitivity predicted by simulation.

Index Terms—sensors, HEMT, AlGa_N/Ga_N, ISFET, sensitivity

I. INTRODUCTION

ALGAN/GAN heterostructures are showing promise for the development of chemical and biochemical sensors for a variety of applications in industrial process monitoring, pharmaceutical and medical research and product development [1], [2], [3], [4]. The potential of AlGa_N/Ga_N heterojunction-based devices for chemical sensing stems from several factors: they are stable in harsh environments and have low toxicity, their conductivity is strongly affected by surface conditions, and ungated surfaces are amenable to functionalization [5]. For AlGa_N/Ga_N grown along the [0001] axis with Ga/Al-polarity the strong spontaneous as well as piezoelectric polarization of

the strained AlGa_N top layer results in the formation of a two-dimensional electron gas (2DEG) in the narrower band gap Ga_N side of the heterojunction [6]. This 2DEG acts as a highly conductive channel for Field Effect Transistor (FET)-type devices that can be modulated by changes in the surface potential. Thus, devices without gate metallization can be used to directly sense charged particles and adsorbed molecules [6], [7], [8], [9], [10], [11]. The realisation of reference electrode free measurements permits the fabrication of small, robust and on-chip AlGa_N/Ga_N FET-based sensors, providing a clear advantage over existing glass electrode based pH and ion sensors. Although reference-electrode free pH sensing has been demonstrated [12],[13], the scope of the reported investigations has been limited in terms of reference electrode free sensor optimisation. Ion sensitivity in reference electrode free sensors has been shown to depend on AlGa_N/Ga_N composition [14], which is not surprising given that the heterostructure design determines sheet carrier density. While carrier charge density and mobility have been theoretically calculated for various AlGa_N/Ga_N FET structures in the context of transistor operation, and to a limited extent to explain the behaviour of ungated AlGa_N/Ga_N FET sensors[15], such calculations have yet to be systematically deployed to investigate the sensitivity limits of reference electrode free sensors, and/or to determine design parameters for practical sensor optimisation.

The sensitivity of AlGa_N/Ga_N pH sensors is often quantified as the change in gate voltage (V_G) in mV per pH, with the extracted value being then compared to the theoretical Nernstian response (59.9 mV/pH at room temperature). In sensor applications in which a reference electrode can be employed, the sensor can be tuned to its maximum sensitivity

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by biasing the device to its maximum transconductance (g_m). Evidently, this approach relies on the reference electrode to maintain and/or manipulate the gate potential. However, V_G cannot be measured nor manipulated without a reference electrode in ungated sensor devices. Reference-electrode free devices require a different approach, and device sensitivity in terms of induced surface potential needs to be extracted from I_{DS} or V_{DS} often employing simplifying approximations [16], [17]. In terms of heterostructure design, Brazzini et al. recently demonstrated AlInN/GaN-based sensors with high sensitivity even when employed in reference electrode free measurement configuration [13]. In their study, which included measurements with and without reference electrodes, devices fabricated employing thinner AlInN layers demonstrated better sensitivity compared to sensors with thicker AlInN layer when measured without a reference electrode. In this work, experimental results and theoretical calculations demonstrate that barrier composition and barrier thickness need to be optimised together to obtain highly sensitive AlGaN/GaN heterostructure-based sensors in a reference electrode free configuration for ion and pH sensing applications.

II. MATERIALS AND METHODS

A. Theoretical Modelling of Sensitivity

The exposure of Ga-face AlGaN/GaN layers to ionic solutions leads to a shift of the surface potential toward a more negative value and thus to a change in the sheet carrier density and mobility (Δn_s and $\Delta \mu$) of the 2DEG. Such an influence on the 2DEG can be compared to an effective gate voltage ($V_{G,eff}$) applied to the active area of the device. It is known that the change in 2DEG parameters is maximal near the threshold voltage for any AlGaN/GaN device; this change will result in the maximum gain in the measured conductivity and therefore lead to very high sensitivity of the device. Most chemical and biological compounds should shift the surface potential of the reference-electrode-free device only by tens or hundreds of mV from initial zero volts bias (shifting by 59.9 mV/pH according to the Nernstian equation). Therefore the most sensitive reference-electrode-free devices will be obtained for near zero threshold voltage.

The experimentally measured gain in the conductivity is a product of sheet carrier density and mobility. The conductivity is the most useful and the most direct way of expressing and predicting sensitivity due to the direct relationship with measured current and inverse relationship with measured voltage. The change in measured current is directly proportional to the change in conductivity. Our aim was to predict an experimental gain in conductivity via modelling. This model can then be used for signal analysis and calibration. Although changes in mobility ($\Delta \mu$) depend on the interplay of different scattering contributions and vary with device structure and material quality, mobility data can be found in the literature and utilized in the model [18]. To demonstrate this we obtained values of mobility variation with V_G from the work of Asgari et al., one of the most sophisticated models for 2DEG density and mobility variation [18]. Four of the eight different AlGaN/GaN structures in Asgari et al.'s paper were selected (Table I) and

changes in n_s and threshold voltage were calculated for all four structures using a self-consistent 1D-Poisson solver [19]. Surface charge (modelled via $V_{G,eff}$) was varied for both mobility and carrier concentration (n_s and μ) in the range from -5V to 1V with 1V step and by specifying appropriate boundary conditions with surface pinning of -1.2 eV. There are a wide range of AlGaN/GaN surface pinning values reported in the literature. The actual value is strongly dependent on heterostructure growth conditions and device processing steps. Therefore in the model the value was chosen to best match 1D-Poisson simulations to carrier concentration obtained experimentally via Hall measurements. For a realistic figure of merit the change in n_s and μ was normalized to the overall carrier concentration and mobility including the contribution from the bulk. The gain was calculated separately for n_s , μ and conductivity (σ) according to

$$\text{Gain} = 20 \log(K) \text{ [dB]} \quad (1)$$

where K represents a relative change one of the three different 2DEG parameters $K_{ns} = \frac{\Delta n_s}{n_s}$; $K_\mu = \frac{\Delta \mu}{\mu}$; $K_\sigma = \frac{\Delta \sigma}{\sigma}$, which are related by $K_\sigma = f(K_{ns}; K_\mu)$. The conductivity was calculated from the carrier concentration and mobility according to

$$\sigma = q n_s \mu (W/L) \quad (2)$$

where $q = 1.6 \times 10^{-19}$ is electron charge, W is width and L is length of the device.

Finally the gain in sheet carrier concentration was calculated using a self-consistent 1D-Poisson solver for a much wider range of structures. Such calculations allow modelling of device sensitivity as a function of AlGaN thickness and composition in any region of transistor characteristic. However, due to the nature of reference-electrode-free measurements for pH and ionic solutions we have focused on the region of near zero bias. The effect of changes in surface charge was modelled as an effective gate potential ($V_{G,eff}$), which was varied in the range from -1.1V to 0.2V with 100mV steps and by specifying appropriate boundary conditions with surface pinning of -1.2 eV for the range of different structures. The sensitivity is presented as the gain in carrier concentration calculated for first 100mV change in $V_{G,eff}$ from zero bias to allow direct comparison between structures with different composition. Also we have demonstrated the shift in optimal structure design as $V_{G,eff}$ moves from 0.2V to -1.1V.

B. pH Experiments with Modelled Structures

In the experimental part of this work three Ga-face AlGaN/GaN heterostructures with 2nm GaN cap with high predicted sensitivity according to the modelling were compared to three uncapped samples from previous work [14] with lower predicted sensitivity according to the modelling. In addition to covering a range of predicted sensitivities, the choice of heterostructures was influenced by material quality and processing difficulties which limited the upper range of AlGaN composition and lower range of AlGaN thickness. The list of the samples can be seen in Table II. Measurements were performed using four point bar mesa-etched test structure configurations (Fig. 1), with sensing areas 1.5 mm in diameter, Al/Cr/Au ohmic contacts, at constant currents of 100 μ A using

a HP 4156A semiconductor parameter analyser. All measurements were performed under constant light conditions. Ungated devices fabricated on AlGaIn/GaN HEMT wafers were exposed to different KOH solutions of pH from 8 to 12, and the potential drop across the gate area for constant current density was measured as a function of KOH concentration (pH). Baked photo-resist and silicone gel were used for electrical isolation. Standard cleaning was applied to the AlGaIn surface (acetone, isopropyl alcohol, de-ionised water and N₂ blow-dry) after device fabrication, and ten second soak in HCl followed by rinsing in de-ionised water after exposure to each ionic solution. No other treatments were applied to the surface.

III. RESULTS AND DISCUSSION

A. Theoretical Modelling of Sensitivity

The main focus of the modelling is to predict experimentally measured gain in conductivity for variable AlGaIn thickness and composition. While carrier concentration can be calculated theoretically at low computation time, mobility calculations are complex and computationally expensive. Therefore mobility data found in the literature was used for proof-of-concept purposes to show sensitivity can be predicted for a range of AlGaIn thicknesses and compositions. Variation of 2DEG density, 2DEG mobility and 2DEG conductivity with $V_{G,eff}$ are shown for four different structures (Table I) in Figs. 2a, 3a and 4a respectively. Figs. 2b, 3b and 4b demonstrate the variation in gain for the same parameters of modelled structures. As can be observed from the figures below both carrier concentration and mobility demonstrate maximum positive gain for near threshold values of $V_{G,eff}$ and so both will contribute to measured gain in conductivity.

It can be seen that gain in conductivity is maximum near threshold (structure A), but is not linear and can vary greatly as surface potential is varied on the order of volts. This is the main challenge for reference electrode free measurements. However, for careful heterostructure design and considering the very small potential influence of most chemical and biological compounds (on the order of tens of mV) the gain will not change that much and the device will remain in the sensitive near-zero bias region, when designed with $V_{th} \sim 0$. The maximum gain in conductivity is observed at near threshold voltage and can be calculated for any AlGaIn thickness and composition using just carrier concentration.

Next the gain in carrier concentration was calculated for -100mV change in $V_{G,eff}$ from zero bias to allow direct comparison of sensitivity between structures with different AlGaIn composition and thickness. Results of these calculations can be found in Fig. 5

From Fig. 5 it can be concluded that the sensitivity is increased when the distance between surface and channel is reduced until depletion of the 2DEG results in a sharp threshold edge, the position of which also depends on the Al content. For thick barriers, changes in surface charge still modulate the amount of carriers in the channel, but the relative change reflected in the sensitivity is small because of the higher carrier concentrations. The sensitivity of the most promising structures

is in the range of 20dB at near zero bias and will decrease as surface potential becomes more negative. Structures with less than optimal design will have lower sensitivity at near zero gate bias. However, sensitivity of non-optimal structures can increase as surface potential becomes more negative and the sensitivity of the optimal structure will decrease. This effect is partly demonstrated in Fig. 6 where we plotted thresholds that are close to applied surface potential and therefore the shift in optimal structures can be observed as surface potential changes from -1.1 to 0.2 V. As threshold value stays constant for a given AlGaIn thickness and composition the shift observed in Fig. 6 demonstrates what changes should be introduced to AlGaIn thickness and composition to optimise sensitivity when surface potential is likely to be shifted to more negative or more positive values (shifted away from zero bias). Also the increase and decrease in sensitivity for a given structure can be predicted through such diagrams and be used for calibration and signal analyses. In the case of more negative surface potential the shift in the optimal structure design, to keep sensitivity at around 20dB, should be towards slightly thicker structures with more negative thresholds. However, we are still looking at a rather narrow region of thicknesses and compositions. Therefore a combination approach can be used with the calculations, by designing an optimised device and then calibrating for changes in surface potential.

B. pH Experiments with Modelled Structures

All six devices with different predicted sensitivity were exposed to KOH solutions of different pH values and the data obtained were normalised to the maximum potential drop that was observed for KOH with pH12 for each device. Fig. 7 shows as-measured data for the sample 2 device which in Fig. 5 lay in the region that was predicted to be close to maximum sensitivity.

The response to different KOH solutions for all six ungated AlGaIn/GaN HEMT-based devices, normalised to the maximum response for each device (pH 12), is given in Fig. 8. The sensitivity values calculated from experimental data in terms of overall gain in conductivity from pH8 to pH12 for all devices are summarised in Table II and can be compared to Fig. 5. It can be seen that the results obtained in the experimental part of this work demonstrate significant correlation with predicted sensitivity. Moreover, from the normalised data it can be seen that the increase in measured voltage is not linear; the shape is directly related to the exponential change in KOH concentration with pH for most of the devices. However, the sample 1 device has a slightly different shape due to the fact that sensitivity itself is changing with KOH concentration, corresponding to the change in sensitivity with $V_{G,eff}$ surface potential (see Figs. 5 and 6).

IV. CONCLUSION

In this paper we report on a methodology for quick and simple prediction and optimization of AlGaIn/GaN device sensitivity operated in reference electrode free configuration desirable for miniaturised sensors. We have demonstrated that by optimisation of the AlGaIn layer (composition and

thickness), the sensitivity of reference-electrode-free AlGaIn/GaN heterostructure-based ion sensors can be dramatically improved. A model was applied to estimate the sensitivity of different structures and showed sensitivity can be maximised by designing a structure to have near-zero threshold voltage. One example of an ISFET device with maximal sensitivity is demonstrated. However there are many other heterostructure designs that would produce similar sensitivity. In particular, due to practical limitations high- x_{Al} or $x_{\text{Al}}=1$ (AlN/GaN) structures were not experimentally investigated in this work but are certainly indicated to be highly sensitive for very thin Al(Ga)N layers. Future improvements in this technology could include investigation of thin AlN layer devices with low threshold voltage. The model allows selection of appropriate design taking into account other factors such as complementary devices, material quality, or processing issues that may influence selection of specific AlGaIn composition or thickness. Experimental results confirmed the predictions of the model, with high signal to noise ratio and gain in conductivity with KOH exposure for the devices grown on the structures with near zero thresholds. These results can be used to improve performance for future chemical and biochemical sensors

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TABLE I
ALGAN/GAN STRUCTURES USED FOR SIMULATION

Structure	Al mole fraction (%)	AlGaN thickness (nm)	Threshold Voltage (V)
A	22	20	-3.5
B	22	24	-5
C	25	35	-9
D	30	35	-10.5

TABLE II
SUMMARY OF DIFFERENT SAMPLE STRUCTURES WITH MEASURED HALL
DATA AND EXPERIMENTALLY OBTAINED SENSITIVITY.

Sample ID	Growth method	Structure	n_s [cm^{-2}]	μ [$\text{cm}^2\text{V}^{-1}\text{s}^{-1}$]	Sensitivity [dB] (Gain from pH8 to pH12)
1	MOCVD	12nm $\text{Al}_{0.23}\text{Ga}_{0.77}\text{N}$ with 2nm GaN cap	5.09×10^{12}	1785	-4.9
2	MBE	10nm $\text{Al}_{0.3}\text{Ga}_{0.7}\text{N}$ with 2nm GaN cap	6.34×10^{12}	856	-7.8
3	MBE	20nm $\text{Al}_{0.2}\text{Ga}_{0.8}\text{N}$ with 2nm GaN cap	5.15×10^{12}	1101	-17.7
4	MOCVD	29nm $\text{Al}_{0.15}\text{Ga}_{0.85}\text{N}$ (with AlN interlayer)	3.78×10^{12}	2093	-26.5
5	MOCVD	23nm $\text{Al}_{0.23}\text{Ga}_{0.77}\text{N}$ (with AlN interlayer)	6.53×10^{12}	2254	-34.8
6	MOCVD	22nm $\text{Al}_{0.29}\text{Ga}_{0.71}\text{N}$ (with AlN interlayer)	7.34×10^{12}	2230	-44.7

Fig. 1. Measurement configuration for Kelvin probe measurement with four ohmic contact pads on mesa-etch structure.

(a)

(b)

Fig. 2. (a) Variation of carrier concentration with gate bias and (b) variation of gain in carrier concentration with gate bias (for step of 1V) calculated according to equation 1, for four different structures described in Table I.

(a)

(a)

(b)

(b)

Fig. 3. (a) Variation of carrier mobility with gate bias and (b) variation of gain in carrier mobility with gate bias (for step of 1V) according to equation 1, for four different structures described in Table I.

Fig. 4. (a) Variation of conductivity for variable gate bias and (b) variation of gain in conductivity for variable gate bias (for step of 1V) calculated according to equation 1 and 2, for four different structures described in Table I.

Fig. 5. Contour plot of the calculated sensitivity in terms of $\text{Gain}=20 \log (\Delta \text{ns/ns})$ [dB] for a change in zero surface potential of -100 mV for ungated AlGaIn/GaN devices with a 2 nm GaN cap layer. Numbers from 1 to 6 correspond to sample IDs in Table II.

Fig. 6. Contour plot of thresholds near different surface potentials varied from -1.1 to 0.2V with -100mV step for the range of $\text{Al}_{1-x}\text{Ga}_x\text{N}/\text{GaN}$ structures.

Fig. 7. Response to KOH solutions of sample 2 measured as change in voltage for fixed current. For each voltage curve, the point at which the voltage sharply increases corresponds to the point at which the KOH solution was applied.

Fig. 8. Maximum potential drop for six ungated AlGaIn/GaN HEMT-based devices exposed to different KOH solutions (normalised to the value obtained for pH12 for each device).