# The electrochemical profiling of $n^+/n$ GaAs structures for field-effect transistors

© D.Yu. Protasov<sup>1,2</sup>, P.P. Kamesh<sup>2</sup>, K.A. Svit<sup>1</sup>, D.V. Dmitriev<sup>1</sup>, A.A. Makeeva<sup>1</sup>, E.M. Rzaev<sup>3</sup>, K.S. Zhuravlev<sup>1</sup>

 <sup>1</sup> Rzhanov Institute of Semiconductor Physics, Siberian Branch, Russian Academy of Sciences, 630090 Novosibirsk, Russia
<sup>2</sup> Novosibirsk State Technical University, 630073 Novosibirsk, Russia
<sup>3</sup> JSC "Zelenograd Nanotechnology Center", 124527 Moscow, Zelenograd, Russia

E-mail: protasov@isp.nsc.ru

Received September 14, 2023 Revised January 31, 2024 Accepted January 31, 2024

It is shown that when using a standard electrochemical profiling recipe that applies intensive illumination by halogen lamp with power up to 250 W of  $n^+/n$  GaAs structure to generate the holes necessary for etching, the resulting electron distribution profile differs from that set during growth for an electron concentration in the  $n^+$ -layer  $> 4 \cdot 10^{18}$  cm<sup>-3</sup> when using EDTA electrolyte. This difference is due to the appearance and development of etching pits caused by the increase in the degree of defectivity of GaAs layers with increasing concentration of the donor impurity - silicon. To obtain adequate electron distribution profiles in  $n^+/n$  GaAs structures it is necessary to limit the illumination up to 25 W.

**Keywords:** electrochemical profiling,  $n^+/n$  GaAs, etching defects, concentration profile distortions.

DOI: 10.61011/SC.2024.01.58122.5562

## 1. Introduction

Structures used at present for the fabrication of various semiconductor devices often contain several different layers with varying dopant types and doping levels. The parameters of structures need to be set and maintained precisely in order to produce devices with the needed parameters. Since the growth conditions of structures may vary with time, the parameters of grown structures need to be monitored periodically. The method of electrochemical capacitancevoltage (C-V) profiling is used to control the distribution of dopants in structure layers [1]. If an electrolyte is used both for etching and forming a Schottky contact with a semiconductor, C-V profilometry is the optimum technique for determining the profile of the carrier concentration distribution over structure layers and verifying the suitability of structures for device fabrication. This method allows one to etch a sample to a depth up to several micrometers with an accuracy no worse than 1 nm [2]. This makes it suitable for monitoring the parameters of advanced nanostructures with individual layers being as thin as several nanometers.

Heterostructures based on gallium arsenide are now used widely in the fabrication of centimeter-wave [3], millimeterwave [4], and high-frequency switching transistors [5]; photonic integrated circuits on silicon[6]; and  $n^+/n$  structures for field-effect transistors with doped layers with thicknesses ranging from several tens to several hundreds of nanometers. The methods of C-V profiling of such structures are currently being refined rapidly [7,8]. It was found that the transition from an  $n^+$ -layer to an n-layer in  $n^+/n$  GaAs structures may be strongly diffuse when the concentration profile is determined this way [9]. This makes it harder to determine the concentration and the thickness of an n-layer. However, the C-V profiling conditions inducing this alteration of concentration profiles were not analyzed in [9]. It was demonstrated in [10] that such a distortion of the concentration profile in  $n/n^+$  GaAs structures may be induced by heterogeneous electrochemical etching with an incorrectly chosen electrolyte. The C-V profiling conditions also remained unexamined.

As is known, electrochemical etching is sustained by the drift of holes in an electrical field to the semiconductor/electrolyte interface, where they "release" valence electrons of atoms. Produced positively charged ions pass into the electrolyte [1]. The formation of an  $n^+$ -layer with an electron concentration of  $10^{18}-10^{19}$  cm<sup>-3</sup> on the surface of GaAs transistor structures for enhancing the properties of ohmic contacts is a complication to the process of electrochemical etching of such structures. Since the concentration of minority holes in *n*-type wide-band layers (and especially in an  $n^+$ -layer) is low, optical radiation is commonly used to generate electron–hole pairs [11].

It is known that rough [12] (or even porous [13]) layers are produced in certain regimes of electrochemical and photochemical etching of GaAs. This leads to an incorrect determination of the electron concentration profile in C-V profiling of structures with nanoscale layers with different levels of doping. In this context, the aim of the present study is to establish the relation between the etching regime and the concentration of electrons in an  $n^+$ -layer of  $n^+/n$  GaAs structures and the obtained electron concentration profile. The veracity of results of C-V profiling of GaAs layers with uniform doping was verified for this purpose. Both the optical radiation power and the concentration in a contact  $n^+$ -layer of  $n^+/n$  GaAs structures were varied. The photoluminescence (PL) method was used to examine the degree of defectivity of  $n^+$ -layers. The relation between observed electron concentration profiles and the morphology of etch pits is characterized qualitatively.

#### 2. Samples and experimental methods

GaAs structures grown by molecular-beam epitaxy on epiready semi-insulating GaAs substrates with the (100) orientation produced by Xiamen (China) were used to examine the influence of electrochemical etching regimes on the electron concentration distribution. Growth was performed in a Compact-21 T (France) system. An undoped 0.8-µm-thick buffer layer was grown first. A shortperiod Al<sub>0.2</sub>Ga<sub>0.8</sub>As/GaAs (2 nm/2 nm) superlattice with seven periods was embedded into its center. Doped structure layers were grown next. Doping with silicon was performed during growth; the electron concentration was adjusted by varying the temperature of the silicon source. The relation between the silicon source temperature and the electron concentration in a GaAs layer was established in advance by growing a series of test GaAs structures with the electron concentration in them determined via A total of eight GaAs structures Hall measurements. were examined (five uniformly doped ones and three  $n^+/n$ structures). Layer thickness values and concentrations of electrons in the studied samples obtained during growth are presented in the table.

Structures S6370, S3816, S3523, S4704, and S4172 were single-layer ones. The concentration of electrons in them was determined via van der Pauw Hall measurements. Structures S6370 and S3816 were used to verify the veracity of C-V profiling data. Structures S3523, S4704, and S4172 with a high concentration were used to establish the relation between the degree of defectivity and the doping level in PL experiments. Transistor structures (S5419, S5096, and S6375) contained two layers: an  $n^+$ -GaAs contact layer and an n-GaAs transistor channel layer with a vastly different doping level. The electron concentration in the contact layer in these structures was varied from  $4 \cdot 10^{18}$  to  $1 \cdot 10^{19}$  cm<sup>-3</sup>. The contact layer thickness fell within the range from 50 to 150 nm. The channel layer with a concentration of  $(2.5{-}4.2)\cdot10^{17}\,\text{cm}^{-3}$  and a thickness of  $150\,\text{nm}$  was positioned below the contact one.

A Wafer Profiler CVP21 setup (Germany) was used for C-V profiling. The EE electrolyte (EDTA<sub>0.1m</sub>-ED10% — solution of disodium salt of ethylenediaminetetraacetic acid in a 10% aqueous solution of ethylenediamine), which is the recommended choice for C-V profiling of  $A^{\rm III}B^{\rm V}$ 

Layer thickness and electron concentration in GaAs structures

Structure number	Thickness, nm	Concentration, cm <sup>-3</sup>
C6370	1000	$4.3\cdot 10^{17}$
C3816	1000	$6.4\cdot 10^{18}$
C3523	1000	$2.9\cdot 10^{18}$
C4704	1000	$5.1 \cdot 10^{18}$
C4172	1000	$7.1\cdot 10^{18}$
C5419	50	$1\cdot 10^{19}$
	150	$2\cdot 10^{17}$
C5096	100	$6\cdot 10^{18}$
	150	$3\cdot 10^{17}$
C6375	150	$4\cdot 10^{18}$
	150	$4.2 \cdot 10^{17}$

heterostructures [14], was used for etching. Since the examined structures had a semi-insulating substrate, the common contact was located on their surface. Prior to measurements, the sample surface at the point of contact was subjected to a high-voltage discharge spark and tinned with indium in order to enhance the ohmic contact. An etch pit was bounded by a sealing ring with an area of  $10 \,\mathrm{mm^2}$ . The software controlling the process of electrochemical etching maintained a constant current density and, consequently, a constant etching rate by adjusting the following two parameters: voltage applied to the Schottky contact and illumination intensity. Two etching regimes were used. In the "V Prefer" regime, an acceptable etching rate was set by raising the voltage at a minimum illumination intensity ( $\leq 10\%$  of the maximum); in the "Light Prefer" regime, the etching rate was adjusted by varying the emission intensity of an Osram HLX64657 halogen lamp with a color temperature of 3450 K and a maximum power of 250 W. The spectrum of radiation incident onto the studied sample was cut off in the shortwavelength region by a CalFlex X filter with a passband of 750-1100 nm and was bounded in the long-wavelength region by absorption in the aqueous electrolyte solution  $(\sim 900 \,\mathrm{nm})$ . The indicated boundary wavelengths correspond to photon energies of 1.65-1.4 eV. These energies are sufficient to produce electron-hole pairs in GaAs with a band gap of 1.42 eV at 300 K.

A Bruker Multi Mode 8 atomic force microscope was used to measure the surface roughness. Optical images of the surface of etch pits were provided by an MMN-2 metallographic upright microscope. Etching imperfections were examined with a Hitachi SU8220 scanning electron microscope. A MicroProf200 optical profilometer was used to determine the profiles of etch pits. PL spectra were recorded by an Acton SP2500 (Princeton Instruments) spectrometer fitted with a CCD camera cooled by liquid nitrogen. A solid-state laser with an emission wavelength of 527 nm was the excitation light source. PL spectra were recorded at liquid nitrogen temperature (77 K).



**Figure 1.** Carrier concentration profiles in uniformly doped structures S6370 (*a*) and S3816 (*b*). Regimes: "Light prefer" — blue triangles; "V prefer" — red circles. Conductivity type: electron — open symbols; hole — filled symbols.

# 3. Electron distribution profiles

#### 3.1. Uniformly doped GaAs layers

The depth distributions of electron concentration for uniformly doped structures S6370 and S3816 obtained in two C-V profiling regimes are shown in Figures 1, a and b. The conductivity type was determined during profiling from the slope of C-V dependences and is also indicated in Figure 1.

It can be seen from Figure 1, a that close electron concentrations were measured for structure S6370 in two profiling regimes and the doped n-GaAs layer thickness  $(1 \mu m)$  agrees with the value set in the process of The electron concentration averaged over the growth. film thickness is  $5.7 \cdot 10^{17} \text{ cm}^{-3}$  in the "V prefer" regime and  $5.3 \cdot 10^{17} \text{ cm}^{-3}$  in the "Light prefer" regime. The electron concentration determined via Hall measurements  $(4.3 \cdot 10^{17} \text{ cm}^{-3})$  agrees with the results of C-V profiling if one takes into account the Hall factor [16] and a 15percent error of the van der Pauw method applied to the examined square samples with small contacts at the edges [15]. The parameters of electrochemical etching in two regimes are also virtually identical: current densities and etching rates fall within the ranges of  $0.5-0.48 \text{ mA/cm}^2$ and  $0.4-0.58 \,\mu$ m/h, respectively, while the applied voltage varies from -0.4 to 2.0 V. It follows from Figure 1, b that the concentration profiles measured in different etching regimes in the region of the 1-µm-thick doped layer differ more substantially in structure S3816 with a higher electron concentration. In this structure layer, the electron concentration is  $6.6 \cdot 10^{18} \text{ cm}^{-3}$  in the "V prefer" regime and  $7.9 \cdot 10^{18} \text{ cm}^{-3}$  in the "Light prefer" regime. The electron concentration determined via Hall measurements is  $6.4 \cdot 10^{18} \,\mathrm{cm}^{-3}$ , which is closer to the values obtained in the "V prefer" regime. In addition, the electron concentration does not decrease after etch removal of the conductive layer

with a thickness of  $1\,\mu\text{m}$  in the "Light prefer" regime. The measured dependence of C on V ceases to be a decreasing or increasing function typical of a Schottky diode and assumes the shape of a low-frequency capacitance-voltage characteristic with a pronounced minimum; as a result, the setup starts indicating *p*-type conductivity. However, the parameters of electrochemical etching in two regimes differ only slightly: the current density falls within the  $0.4-0.5\,\text{mA/cm}^2$  range, and the etching rates are roughly equal at  $0.57\,\mu\text{m/h}$ .

#### 3.2. $n^+/n$ GaAs structures

Figure 2 shows the profiles for three  $n^+/n$  GaAs structures with different electron concentrations in the contact layer. The electron concentration profiles obtained for these structures in different regimes differ more substantially than those corresponding to uniform samples.

It can be seen from Figure 2, a that the electron concentration in the upper  $n^+$ -layer of structure S5419 is  $1.1 \cdot 10^{19} \,\mathrm{cm}^{-3}$  in both regimes. This agrees with the data from the table. However, an n-layer and an undoped buffer layer are not observed in the "Light prefer" regime. When a layer with a thickness of 300 nm, which exceeds the overall thickness of the conductive region, is etched out, the electron concentration does not decrease and remains at a level of  $10^{19}$  cm<sup>-3</sup>. The sample illumination intensity in this regime decreased from 100 to 28% as the etching depth increased. The electron concentration profile in the "V prefer" regime matches growth data almost perfectly: the electron concentration is  $1.1 \cdot 10^{19} \text{ cm}^{-3}$  in the upper layer, and the thickness determined at the level of a twofold reduction in the electron concentration is 47 nm; the electron concentration in the channel *n*-layer is  $2.5 \cdot 10^{17}$  cm<sup>-3</sup>, and its thickness is 110 nm. The inaccuracy of determination of the channel layer thickness may be attributed to the fact that the depth resolution of C-V profiling is on the order of two



**Figure 2.** Electron concentration profiles in  $n^+/n$  GaAs structures S5419 (*a*), S5096 (*b*), and S6375 (*c*) measured in two regimes: Light prefer — blue triangles and V prefer — red circles. (A color version of the figure is provided in the online version of the paper).

Debye screening lengths [1]. The screening length for an electron concentration of  $2 \cdot 10^{17}$  cm<sup>-3</sup> in GaAs is ~ 10 nm. The illumination intensity in this regime did not exceed 10%. Note that illumination had almost no effect on the etching rate: the average rate was  $0.31 \,\mu$ m/h in the "Light prefer" regime and  $0.3 \,\mu$ m/h in the "V prefer" regime. Owing to its smallness at the center of the etching region, the etching depth could not be measured with MicroProf200 in the "Light prefer" regime. The etching depth in the "V prefer" regime was 195 nm, which is almost the same as the overall thickness of the conductive structure layer. It can be seen from Figure 2, b that the electron concentration measured in the upper  $n^+$ -layer of structure S5096 at a lower specified electron concentration of  $6 \cdot 10^{18} \, \text{cm}^{-3}$  is the same in all etching regimes:  $7.8 \cdot 10^{18} \text{ cm}^{-3}$ , which is slightly higher than the set value. As the etching depth increases further in the "V prefer" regime, a step associated with the transition to the *n*-layer of the transistor channel becomes apparent. The  $n^+$ -layer thickness determined in this regime is 93 nm, which corresponds to the  $n^+$ -layer thickness specified during growth. The obtained values of the electron concentration and the *n*-layer thickness are  $3.2 \cdot 10^{17} \text{ cm}^{-3}$  and 194 m, respectively. It is evident that the electron concentration is almost the same as the tabular value, while the thickness is  $\sim 50 \text{ nm}$  higher. However, the step in the electron distribution profile in the "Light prefer" regime is poorly resolved, and the electron concentration decreases gradually down to  $10^{14} \,\mathrm{cm}^{-3}$ . This profile allows one to determine only the  $n^+$ -layer thickness (97 nm). The illumination intensity in the "Light prefer" regime was 35% and 14% in etching of  $n^+$ - and *n*-layers, respectively. The illumination intensity in the "V prefer" regime did not exceed 11%. The etching rate did not depend on the etching regime and was equal to  $\sim 0.3 \,\mu$ m/h. The etching depth determined at MicroProf200 was 240 and 340 nm for "Light prefer" and "V prefer" regimes, respectively. Since the overall thickness of conductive layers of structures was 250 nm (see the table), conductive layers were etched out in both cases. Figure 2, c presents the profiles in structure S6375 with the lowest electron concentration  $(4 \cdot 10^{18} \text{ cm}^{-3})$ . All structure layers are seen clearly in the profiles obtained in both "Light Prefer" and "V Prefer" regimes. The electron concentration measured in the upper  $n^+$ -layer does not depend on the etching regime and is equal to  $4.8 \cdot 10^{18} \text{ cm}^{-3}$ , which agrees with the tabular value. The contact layer thickness is 144 nm in the "Light prefer" regime and < 125 nm in the "V prefer" regime. Both values are lower than the thickness of 150 nm set during growth. The second step in the concentration profiles is associated with the n-layer of the transistor channel. The electron concentration in it is  $6.7 \cdot 10^{17} \,\mathrm{cm}^{-3}$  in the "Light Prefer" regime and somewhat lower  $(5.4 \cdot 10^{17} \text{ cm}^{-3})$  in the "V prefer" regime. Both values are higher than the tabular electron concentration value  $(4.2 \cdot 10^{17} \text{ cm}^{-3})$ . The channel layer thickness is 130 nm in the "Light prefer" regime and 266 nm in the "V prefer" regime. The illumination intensity in "Light Prefer" and "V Prefer" regimes fell within the 17 - 20%range. The etching rate was also independent of the etching regime and was equal to  $\sim 0.3 \,\mu\text{m/h}$ . The etching depth determined at MicroProf200 was 230 and 290 nm for "Light prefer" and "V prefer" regimes, respectively. Only in the "V prefer" regime is it fair to say that the entire conductive part of this structure (300 nm) was etched out.

#### 3.3. Surface morphology of etch pits

It was determined by atomic force microscopy that the surface roughness at the center of etch pits in all samples depends only weakly on the etching regime. The rms roughness value was 1.5-3 nm. The surface morphology at the edge of pits depends on the electron concentration in a sample and the etching regime.

Semicircles in Figures 3, *a* and *b* denote the position of the sealing ring that bounds the etching region. The etched region is within this semicircle. It can be seen that no marked surface defects are present in an etch pit formed in the "V prefer" regime. In contrast, numerous etching imperfections are seen at the edge of an etch pit in the "Light prefer" regime. It was determined by scanning electron microscopy that the average diameter of these etching imperfections is  $20 \,\mu\text{m}$  and their depth is  $7 - 9 \,\mu\text{m}$ . Etching imperfections produced in the "Light prefer" regime for structure S5096 with an electron concentration in the contact layer of  $6 \cdot 10^{18} \,\text{cm}^{-3}$  were an order of magnitude smaller, and no imperfections were observed for structure S6375 with an electron concentration of  $4 \cdot 10^{18} \,\text{cm}^{-3}$ .

#### 4. Discussion

The charge propagating along the circuit in the process of electrochemical etching is equal to the overall charge of ions passing from a semiconductor to an electrolyte. Having measured time dependence I(t) of current in the circuit and knowing the semiconductor parameters (molar mass M, density  $\rho$ , and valence  $N_V$ ), one may calculate the volume of the etched layer using the Faraday law for electrolysis [1]:

$$V_{etch} = \frac{\int\limits_{0}^{t} I(t)dt}{q \cdot N_V} \cdot \frac{M}{N_A \cdot \rho},$$
 (1)

where q is the elementary charge and  $N_A$  is the Avogadro number. Since the etching area is set by the size of the sealing ring, the etching depth is calculated from the etched volume. Let us estimate the volume of material removed from the  $n^+$ -layer and from etching imperfections at the edge of an etch pit. The area of the sealing ring in our experiments was 10 mm<sup>2</sup>. The volume of the  $n^+$ -layer for structure S5419 is estimated as  $V_{n^+} = S_k \cdot d_{n^+} \approx 5 \cdot 10^{-4} \text{ mm}^3$ . It can be seen from Figure 3, b that  $\sim$  50 etching imperfections are found within 1/4 of the etch pit perimeter. The overall number of etching imperfections in an etch pit is then  $N_d = 200$ . The volume of a single etching imperfection is calculated as  $V_d = \pi \cdot a^2 \cdot h/4$ , where a is the average size of an etching imperfection  $(20 \,\mu\text{m})$  and h is its average depth  $(8 \,\mu\text{m})$ . The overall volume of etching imperfections is then

$$V_{\sum d} = N_d \cdot V_d \approx 6 \cdot 10^{-4} \,\mathrm{mm}^3.$$

Thus, it follows from our evaluation that the volumes of the  $n^+$ -layer and etching imperfections are roughly equal. Therefore, an overestimated value is obtained if expression (1) is used to calculate the thickness of the etched layer. This conclusion is verified by the results of measurement of the etch pit profile with MicroProf200. Owing to the smallness of etching depth, this setup could not determine it in the "Light prefer" regime for structure S5419. Let us examine the diagram in Figure 4 that helps evaluate the measured capacitance.

When backward voltage is applied to the Schottky contact, space charge regions (SCRs) emerge both in the  $n^+$ -layer and in the *n*-layer and the undoped structure region (*n*-layer). The SCR thickness is governed by the Debye screening length, which is calculated as

$$L_{D_i} = \sqrt{\frac{kT\varepsilon_s\varepsilon}{e^2N_i}},\tag{2}$$

where k is the Boltzmann constant; T is the measurement temperature (300 K); static permittivity of GaAs  $\varepsilon_s = 12.9$ ;  $\varepsilon$  is the absolute permittivity; e is the elementary electrical charge; and  $N_i$  is the electron concentration in  $n^+$ -, n-, and n-layers (a concentration of  $1 \cdot 10^{15}$  cm<sup>-3</sup> was set for the last listed layer). The calculated SCR width values in these layers are 1.4, 9.6, and 135.7 nm, respectively. Let us estimate layer capacitances  $C_{n^+}$ ,  $C_n$ , and  $C_{n^-}$  in the plane capacitor approximation. The plate area is equal to the area bounded by the sealing ring  $(10 \text{ mm}^2)$  for  $C_{n^+}$ , to the side surface area of a cylinder with a height of 150 nm ( $\approx 1 \cdot 10^{-5} \text{ mm}^2$ ) for  $C_n$ , and to the sum of areas of the side surface of a cylinder  $8 \mu \text{m}$  in height and the base ( $\approx 2 \cdot 10^{-3} \text{ mm}^2$ ) for  $C_{n-}$ .



Figure 3. Images of the surface of an etch pit in structure S5419: a - V prefer regime (optical microscope), b - Light prefer regime (optical microscope), <math>c - etching imperfections at the edge of a pit imaged with a scanning electron microscope.

SU8200 5.0 kV 13.1 mm ×700 SE(UL) 02/07/202



**Figure 4.** Diagram clarifying the influence of etching imperfections on capacitance values measured in the course of profiling of  $n^+/n$  GaAs structures.

The values of these capacitances are calculated with the overall number of etching imperfections taken into account:  $C_{n^+} = 8 \cdot 10^{-7} \text{ F}, C_n = 2 \cdot 10^{-11} \text{ F}, \text{ and } C_n = 3 \cdot 10^{-10} \text{ F}.$ Since these capacitances are connected in parallel, their total value exceeds the greatest one. Therefore, the total capacitance is specified by the capacitance of the  $n^+$ -layer, while the contributions of other structure layers are much smaller. Specific features of current flow between the base electrode and the Schottky contact in the studied  $n^+-n$ structures are the reason why most etching imperfections are positioned near the sealing ring. Possible current paths are indicated by red arrows in Figure 4. Owing to its high conductivity, the current should spread uniformly in the  $n^+$ -layer. Since etching is generally directed down, the current enters the Schottky contact from the substrate side. Naturally, its density is maximized near the edge of an etch pit (the region where etching imperfections are localized). Similar edge effects were observed in electrochemical etching of silicon [17] and were attributed to the nonuniformity of the current density distribution under illumination [18].

Therefore, the measured dependence of electron concentration on depth for structure S5419 with the most heavily doped  $n^+$ -layer may be explained in the following way. A large flux of holes to the electrolyte-semiconductor interface is produced under illumination. Since they pass through the  $n^+$ -layer, a considerable number of holes recombine and do not reach the interface. The probability of passing through the  $n^+$ -layer and setting free an atom on the surface is higher in an electrical field, since holes then propagate faster in the  $n^+$ -layer due to drift. In local etching of the  $n^+$ -layer, the etching rate increases sharply due to an increase in probability of holes reaching the electrolyte-semiconductor interface, and the etching imperfection depth grows. Notably, etching proceeds mostly in etching imperfections, maintaining the needed current density. This current density is converted into etching depth, which exceeds considerably the etched thickness of the  $n^+$ -layer, in accordance with expression (1). Since the measured capacitance is at all times specified by the  $n^+$ -layer capacitance, the concentration calculated based on these measurements also remains unchanged. When the concentration in the upper layer drops to  $6 \cdot 10^{18} \text{ cm}^{-3}$ , etching imperfections become smaller; the  $n^+$ -layer is etched away completely in both regimes. Apparently, it is etched nonuniformly, which is manifested in tailing of the concentration profile in the *n*-layer and a smooth transition to the *n*-layer concentration.

The threshold nature of production of etching imperfections (i.e., their lack at a concentration of  $4 \cdot 10^{18} \text{ cm}^{-3}$ in the  $n^+$ -layer, emergence at  $6 \cdot 10^{18} \text{ cm}^{-3}$ , and growth at  $1 \cdot 10^{19} \text{ cm}^{-3}$ ) is associated with an increase in the degree of defectivity of the  $n^+$ -layer with increasing doping level. As the dopant concentration grows, the concentration of both gallium vacancies ( $V_{\text{Ga}}$ ) and ( $V_{\text{Ga}}$ -Si<sub>Ga</sub>) complexes increases in GaAs structures [19]. In order to estimate the degree of defectivity of our samples, PL spectra were measured at 77 K for a series of uniformly doped GaAs layers grown under the same conditions as those corresponding to  $n^+$ -layers of  $n^+/n$  structures from the table.

The concentration of electrons in these samples is indicated in the table. Three bands with maxima energies around 1.5, 1.47, and 1.25 eV are seen clearly in the PL spectra in Figure 5. The high-energy band with an energy of  $\sim 1.5 \,\mathrm{eV}$  corresponds to band-to-band recombination. This band shifts as the electron concentration increases and levels in the conduction band get filled (Burstein-Moss shift). The relatively narrow band with an energy of  $\sim 1.47\,eV$  corresponds to band-to-neutral acceptor recombination  $(e, A_0)$ . Both a background carbon impurity and Si<sub>As</sub>- may act as a neutral acceptor, since, according to calculated data, this form of incorporation of silicon atoms is the most energetically favorable after  $Si_{Ga^+}$  [20]. The third band with an energy of  $\sim 1.2 \,\mathrm{eV}$  is often found in heavily  $n^+$ -doped GaAs and corresponds to recombination via  $V_{\text{Ga}}$ -shallow donor complexes [21]. In the present case, it is evident that



**Figure 5.** PL spectra for uniformly doped  $n^+$ -GaAs structures at 77 K.

the intensity of this peak rises sharply as the doping level increases, indicating an increase in the degree of defectivity of samples. It is known that defects are etched in the process of electrochemical etching of GaAs layers [22,23]. As a result, the etching rate in the course of local etching of the  $n^+$ -layer in the region of a defect increases manyfold, since the probability that photogenerated holes reach the surface of the *n*-layer (and especially the *n*-layer) is higher than the corresponding probability for the  $n^+$ -layer. This results in the emergence of etching imperfections that distort C-V profiling data.

# 5. Conclusion

The electron distribution profile obtained in electrochemical profiling of  $n^+ - n$  GaAs structures depends both on the profiling conditions and the concentration in the  $n^+$ -layer. At an electron concentration in the  $n^+$ -layer of  $4 \cdot 10^{18} \text{ cm}^{-3}$ , an electron distribution profile close to the one set during growth may be obtained in layers with a thickness of  $\sim 100 \,\mathrm{nm}$  both under illumination ("Light prefer" regime) and without it ("V prefer" regime). The profile of etch pits is near-rectangular in both regimes. When the electron concentration in the  $n^+$ -layer increases to  $6 \cdot 10^{18} \, \text{cm}^{-3}$ , local etching imperfections grouped at the edge of a sealing ring start to form due to nonuniformity of the current density through an electrolyte-semiconductor contact under illumination and an increased degree of defectivity. The electrolyte then comes into contact not only with the  $n^+$ -layer, but also with lower-lying n- and n-layers of the structure. The measured capacitance of the structure changes as a result, and the obtained electron distribution profile gets distorted in the region of the *n*-layer. The electron distribution profile obtained in the "V prefer" regime is close to the one set during growth. At an electron concentration of  $1 \cdot 10^{19}$  cm<sup>-3</sup> in the  $n^+$ -layer, electrochemical etching in the "Light prefer" regime proceeds exclusively in local etching imperfections with a size of  $20 \times 20 \,\mu$ m and a depth of  $7 - 9 \,\mu$ m. No etching is observed in the center of a pit. As a result, the structure capacitance does not change during etching, remaining unaffected by the presence of *n*and *n*-layers. The "V prefer" regime provides an opportunity to determine correctly the parameters of layers of the  $n^+/n$ structure.

In order to obtain a correct electron concentration profile in  $n^+/n$  GaAs structures, one should either perform profiling without illumination or limit the illumination intensity. The halogen lamp power in the present study was limited to 25 W to prevent distortion of the concentration distribution profile.

Equipment provided by the "Nanostructures" common use center was used in measurements with an atomic force microscope and a scanning electron microscope.

### Funding

This study was carried out under the state assignment of the Institute of Semiconductor Physics of the Siberian Branch of the Russian Academy of Sciences (project No. FWGW-2022-0005).

#### **Conflict of interest**

The authors declare that they have no conflict of interest.

## References

- [1] P. Blood. Semicond. Sci. Technol., 1, 7 (1986).
- [2] http://www.wepcontrol.com/cv-profiler/
- [3] K.S. Zhuravlev, D.Yu. Protasov, A.K. Bakarov, A.I. Toropov, D.V. Gulyaev, V.G. Lapin, V.M. Lukashin, A.B. Pashkovskii. Optoelectron., Instrum. Data Process., 56 (5), 478 (2020).
- [4] A.B. Pashkovskii, S.A. Bogdanov, A.K. Bakarov, A.B. Grigorenko, K.S. Zhuravlev, V.G. Lapin, V.M. Lukashin, I.A. Rogachev, E.V. Tereshkin, S.V. Shcherbakov. IEEE Trans. Electron Dev., 68 (1), 53 (2021).
- [5] D. Yu Protasov, D.V. Dmitriev, K.S. Zhuravlev, G.I. Ayzenshtat, A.Y. Yushchenko, A.B. Pashkovsky. Mater. Sci. Semicond. Process., 153, 107148 (2023).
- [6] M.O. Petrushkov, D.S. Abramkin, E.A. Emelyanov, M.A. Putyato, O.S. Komkov, D.D. Firsov, A.V. Vasev, M.Yu. Yesin, A.K. Bakarov, I.D. Loshkarev, A.K. Gutakovskii, V.V. Atuchin, V.V. Preobrazhenskii. Nanomaterials, 12, 4449 (2022).
- [7] G.E. Yakovlev, M.V. Dorokhin, V.I. Zubkov, A.L. Dudin, A.V. Zdoroveishchev, E.I. Malysheva, Yu.A. Danilov, B.N. Zvonkov, A.V. Kudrin. Semiconductors 52 (8), 1004 (2018).
- [8] George Yakovlev, Vasily Zubkov. J. Solid State Electrochem., 25, 797 (2021).
- [9] T. Clarysse, G. Brammertz, D. Vanhaeren, P. Eyben, J. Goossens, F. Clemente, M. Meuris, W. Vandervorst, R. Srnanek, R. Kinder, B. Sciana, D. Radziewicz, Zhiqiang Li. Mater. Sci. Semicond. Process., 11, 259 (2008).

- [10] M. Kaniewska, I. Slomka. Cryst. Res. Technol., 36 (8–10), 1113 (2001).
- [11] P.A. Kohl, F.W. Ostermayer. Ann. Rev. Mater. Sci., 19, 379 (1989).
- [12] Z. Mazouz, L. Beji, J. Meddeb, H. Ben Ouada. Arabian J. Chemistry, 4, 473 (2011).
- [13] Emna Ben Amara, Amira Lebib, Lotfi Beji. J. Electron. Mater., 49 (9), 5281 (2020).
- [14] A.R. Clawson. Mater. Sci. Engin., 31, 1 (2001).
- [15] E.V. Kuchis. Gal'vanomagnitnye effekty i metody ikh issledovaniya (M., Radio i Svyaz', 1990). (in Russian).
- [16] D.L. Rode. Semiconductors and Semimetals, Transport Phenomena (Academic Press, N.Y., 1975) v. 10, p. 1.
- [17] A.A. Nechitailov, E.V. Astrova. Tech. Phys. Lett., 33 (8), 682 (2007).
- [18] E.V. Astrova, A.A. Nechitailov. Semiconductors, 42 (4), 470 (2008).
- [19] J. Gebauer, R. Krause-Rehberg, C. Domke, Ph. Ebert, K. Urban. Phys. Rev. Lett., 78, 3334 (1997).
- [20] John E. Northrup, S.B. Zhang. Phys. Rev. B, 47 (11), 6791(R) (1993).
- [21] V. Swaminathan. Bull. Mater. Sci., 4 (4), 403 (1982).
- [22] Ákos Nemcsics, Janos P. Makai. FTP, **37** (6), 657 (2003).
- [23] M. Kayambaki, K. Tsagaraki, M. Lagadas, P. Panayotatos. Mater. Sci. Engin. B, 80, 164 (2001).

Translated by D.Safin