

Investigation of boron-doped delta layers in CVD diamond grown on single-sector HPHT substrates

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PACS 81.05.ug, 73.63.-b

ABSTRACT This work is devoted to experimental study of boron doped delta layers in CVD diamond. Delta layers with a thickness of 0.8 – 2 nm were grown with a concentration of boron atoms of $(1 - 1.7) \cdot 10^{21} \text{ cm}^{-3}$, and localized inside undoped defect-free diamond. The layers thickness and boron concentration were measured by secondary ion mass spectrometry (SIMS). The surface density and the Hall mobility of holes, the layer resistance at room temperature, and temperature dependences of these parameters are presented. Performed electrical measurements showed that, despite the perfect (from the point of view of the possibility of quantum effects) profile of delta layers, no significant increase was observed in the hole mobility compared to uniform doping with the same concentration of boron atoms. An explanation is proposed for the results of electrical measurements based on calculations of the delta layer profile and the concentration of delocalized holes depending on the layer thickness. It is discussed which parameters of the boron doped delta layers are needed in order to obtain a significant increase of the hole mobility in heavily doped diamond.

KEYWORDS CVD diamond, boron delta-doping, electrical measurements, hole mobility

ACKNOWLEDGEMENTS The work was carried out within the frame of the Federal research center Institute of Applied Physics of the Russian Academy of Sciences project No. 0030-2021-0003. We express our sincere thanks to Dr. J. Butler for a fruitful discussion on delta doping of diamond during his collaboration with IAP RAS in the period of 2013 – 2018.

FOR CITATION Lobaev M.A., Vikharev A.L., Gorbachev A.M., Radishev D.B., Arkhipova E.A., Drozdov M.N., Isaev V.A., Bogdanov S.A., Kukushkin V.A. Investigation of boron-doped delta layers in CVD diamond grown on single-sector HPHT substrates. *Nanosystems: Phys. Chem. Math.*, 2022, **13** (5), 578–584.

1. Introduction

Semiconductor single-crystal diamond is produced by adding dopants (boron or phosphorus) during chemical vapor deposition (CVD synthesis) of a diamond layers on a diamond substrate, usually produced by high pressure and high temperature (HPHT) method. With its unique characteristics, such epitaxial layers are considered as promising material for electronic applications [1]. But until now, diamond electronics is still under development. The main difficulty in realizing the potential of CVD diamond is the problem of obtaining a high density of charge carriers with high mobility. Boron or phosphorus atoms create deep energy levels in semiconductor diamond, which leads to low activation of impurities in the diamond. To create an acceptable conductivity level, it is necessary to increase the doping level, but in the case of boron doping, this leads to a decrease in the hole mobility in diamond [2]. In order to increase the hole mobility, the delta doping approach was proposed [3]. Inside the undoped defect-free epitaxial layer, a doped delta layer with a thickness of 1 – 2 nm and a concentration of boron atoms of more than $5 \cdot 10^{20} \text{ cm}^{-3}$ is formed. It is believed that in a heavily doped layer of nanometer thickness, all impurities are ionized, and most of the holes may be spread outside the delta layer, where they should have high mobility. The delocalization of free holes outside the delta layer occurs due to the effect of quantum penetration [4].

There were several attempts to create delta layers in CVD diamond [5–10]. In these studies, heavily doped delta layers of nanometer thickness were grown, but further electrical measurements did not show a high mobility of charge carriers [8, 9]. The results of these experiments [5–10] were summarized in Table 1 presented in the article [11]. The Table also lists the results obtained by that time on a CVD reactor [12] specially designed for delta doping. In the first

experiments on this reactor, several samples with delta layers of nanometer thickness were obtained, which demonstrated the Hall mobility of holes close to $100 \text{ cm}^2/\text{V}\cdot\text{s}$ at room temperature [11]. Along with these measurements, one of the samples from this series was used for angle resolved photoemission spectroscopy (ARPES) measurements [13]. ARPES was used to compare the electronic structure of a 1.8 nm doped delta layer with a thick 3 mm boron doped diamond film [13]. The occupied electronic structure of the boron-doped layer was found to be similar to that of a uniformly doped film, with no additional features that could be attributed to the electron-occupied quantum well states and no modification of the pre-existing bands, except for a slight modification of the effective mass [13].

Thus, as was noted, the results of [8, 9] and [11, 13] diverge from each other and do not coincide with the results of modeling [8, 14] (predicting delocalization of holes). In experiments [8, 9], the measured Hall mobility of holes at room temperature was $1 - 4.4 \text{ cm}^2/\text{V}\cdot\text{s}$, which approximately coincides with the mobility in uniformly doped diamond with the same concentration of boron atoms of $5 \cdot 10^{20} \text{ cm}^{-3}$. In the experiment [11], the mobility reached approximately $100 \text{ cm}^2/\text{V}\cdot\text{s}$, which was close to the desired mobility values. With a clear discrepancy between the results, it was natural to want to repeat the data of [11]. We assumed that the reason for the discrepancy between the results could be the imperfection of the delta layers, i.e. insufficiently sharp and narrow dopant profiles were obtained. Therefore, a series of experiments was performed to find the delta doping regime, which ensures the growth of delta layers with a boron atom concentration of 10^{21} cm^{-3} and a thickness of 1 nm. The results of growing such doped samples were published in [15, 16].

This article presents the data of electrical measurements of a new series of samples with delta layers grown in the found regime [15, 16]. Different delta layers, both surficial and buried under the surface of the epitaxial layer, were grown. The parameters of the delta layers (thickness and boron concentration) for each sample were measured by the SIMS method. It should be noted that in previous works, either surface [5] or buried [8] layers were studied. In addition, in [8], not every sample, for which electrical measurements were carried out, was controlled by the SIMS method. Such control is important for samples, since the thickness of the delta layer and the concentration of boron in it can depend on the angle of misorientation of the surface with respect to the crystallographic direction (001) and on the level of background boron in the reactor. The performed electrical measurements showed that, despite the perfect (from the point of view of the possibility of quantum effects) profile of delta layers, there was no significant increase in the hole mobility compared to uniform doping with the same concentration of boron atoms. At the same time, according to calculations [8, 14], the quantum effect should manifest itself for such structures. Therefore, questions arise whether the theoretical calculations are predictive and which profile of the delta layer should be experimentally obtained to achieve the goal. In this article, we tried to answer these questions by analyzing our latest experimental data and the results of recent numerical calculation of delta layer profiles [17], taking into account a new effect that was not taken into account earlier [8, 14].

2. Experimental conditions

CVD diamond containing boron doped delta layers was grown in home-made CVD reactor, which was described in detail in [12]. The reactor consists of a cylindrical resonator with a quartz tube located on its axis. In the tube, the plasma was maintained above the substrate holder by microwave radiation from a magnetron at a frequency of 2.45 GHz. Inside the quartz tube, a laminar vortex-free gas flow was created with a flow rate of $900 \text{ cm}^3/\text{min}$, which made it possible to quickly switch the composition of the gas mixture. A buffer layer (undoped diamond) was first deposited on an HPHT substrate (produced by the high pressure and high temperature method) in $\text{H}_2 + \text{CH}_4$ gas mixture ($\text{CH}_4/\text{H}_2 = 0.2 \%$). Then, to grow the delta layer, this mixture was rapidly (with a characteristic time of 5 seconds) switched to a gas mixture containing boron $\text{H}_2 + \text{CH}_4 + \text{B}_2\text{H}_6$ using a gas switch. Typical rates of diamond epitaxial growth were close to 60 nm/h . Next, the gas mixture was switched to $\text{H}_2 + \text{CH}_4$ mixture with the addition of hydrogen sulfide, which was used as a chemical getter to reduce the level of residual contamination of the reactor with boron during the growth of the undoped layer [18]. As a result, the top layer of undoped diamond had a low concentration of boron, usually less than 10^{17} cm^{-3} .

The main conditions for the synthesis of epitaxial diamond were chosen based on the results of our previous experiments [15, 16]. In [15], the dependence of the boron concentration in the delta layer on the substrate temperature and the ratio of boron to carbon (B/C) in the gas mixture were studied. Therefore, for the new series of experiments, the optimal ones were chosen: the deposition temperature of the delta layer and the B/C ratio equal to $850 \text{ }^\circ\text{C}$ and 25000 ppm, respectively. In [16], the effect of the surface misorientation angle relative to the (001) crystallographic direction on the boron concentration in the delta layer was studied. At small misorientation angles, a large number of defects in the form of pyramidal hillocks were found on the surface of the epitaxial layers. At large misorientation angles, an increase in the diamond growth rate occurred, at which it was not possible to obtain a delta layer of small thickness. As a result, it was found that the optimal angle of surface misorientation should be about 1° . Therefore, in the experiments, type IIa diamond substrates with (100) orientation, $3.0 \times 3.0 \times 0.5 \text{ mm}^3$ in size and with the same misorientation angles of about 1° were used.

In addition to control of the misorientation angle of the HPHT surface, the substrates were carefully selected according to another criterion. Using a scanning microwave microscope [19], the substrates were checked for the presence of growth and conduction sectors along the substrate surface. Studies have shown that a combination of dielectric and conductive regions was observed in some substrates [20]. Therefore, substrates with nonuniform surface conductivity

were replaced, and for a new series of experiments, single-sector and non-conductive HPHT substrates were selected. Note that prior to the beginning of the previous experiments [11], such a selection of substrates was not carried out. As before, all substrates were mechanically polished to a surface roughness (R_a) of 0.1 nm, measured with a Zygo NewView 7300 white light interferometer over an area of $0.22 \times 0.22 \text{ mm}^2$. A layer of 4–5 μm was uniformly etched from the substrates in ICP plasma (Oxford Instruments, Plasmalab 80) to remove defects introduced into the surface by mechanical polishing, without deterioration of the substrates roughness. As a result defect-free substrates with an atomically smooth surface were used to grow delta-doped CVD diamond.

The depth distribution of boron in the grown samples was measured by secondary ion mass spectroscopy (SIMS) on a time-of-flight SIMS setup (IONTOF TOF.SIMS-5). The depth of etch craters for calculating the depth of analysis from the etch time was determined using Talysurf CCI 2000 and Zygo NewView 7300 white light interference microscopes. Sputtering was performed with Cs^+ ions with an energy of 1 keV at an angle of 45° . Probing was carried out with Bi^+ ions with an energy of 25 keV. Quantitative calibration of the mass spectrometer was performed using a test HPHT substrate implanted with boron ions.

3. Experimental results

The performed series of experiments, in which the optimized delta doping regime was used, made it possible to obtain samples with a high concentration of boron atoms in a layer of nanometer thickness, Fig. 1. The samples had different delta layers: for sample SS4-2, the layer was located on the surface; for sample SS5-2, buried at a depth of 72 nm from the surface of the epitaxial layer; for samples SS9, SS10, and SS1-3, the layer was located at depth of 15, 12 and 13 nm, respectively. In addition, heavily doped surface layers 3 nm thick were grown on the last three samples to create ohmic contacts without additional thermal annealing for electrical measurements. The boron concentration profile in the epitaxial layer for these three samples was approximately the same, corresponding to the profile shown in Fig. 1(c) for sample SS9. Boron atoms concentration, delta layer thickness, and other characteristics are summarized for all samples in Table 1. To quantitatively describe the distribution of boron in delta-doped diamond, we considered the simplest approximation, which is a rectangular concentration distribution or “top-hat” distribution. According to SIMS measurements, the maximum concentration of boron in the delta layers was $(0.99 - 1.68) \cdot 10^{21} \text{ cm}^{-3}$. The profile width determined using the reconstruction procedure described in [15] was 0.85 – 2.01 nm. Taking into account the width of the profile and the maximum concentration of boron, the surface densities of N_{2D} boron atoms in the delta layers were calculated equal to $(14.3 - 19.9) \cdot 10^{13} \text{ cm}^{-2}$. These N_{2D} values, according to electrical measurements, were several times higher than the surface concentrations of charge carriers (holes).

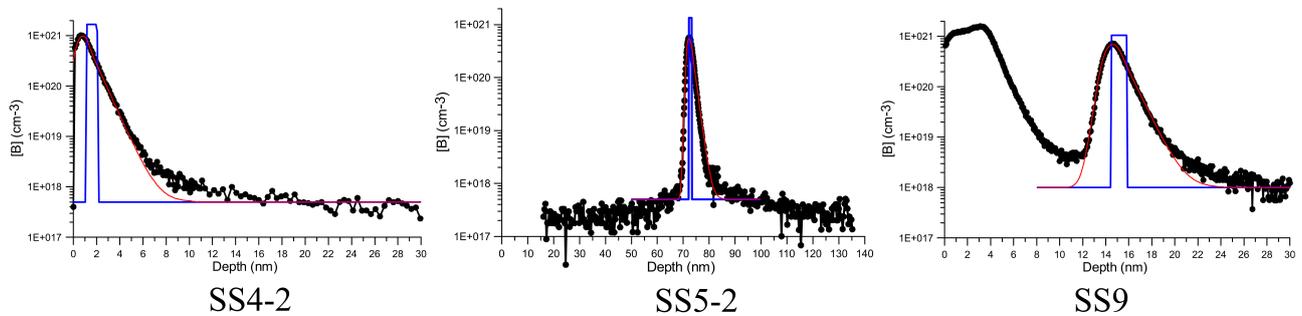


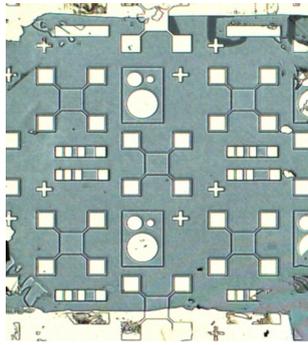
FIG. 1. Boron concentration profiles obtained from SIMS data (black dots), reconstructed boron profiles (blue line) and convolutions of the reconstructed profile and instrumental function of the SIMS instrument (red line) for samples SS4-2, SS5-2 and SS9

To carry out electrical measurements on samples with two layers (SS9, SS10 and SS1-3), measuring test cells were formed, including the process of etching mesa structures. The measurements were performed using Hall effect methods. Fig. 2 shows a photograph of a diamond surface ($0.22 \times 0.22 \text{ mm}^2$ in size) with test cells formed for Hall measurements in Van der Pauw geometry, TLM lines and CV cells, as well as a scheme for the formation of a mesa structure and an ohmic contact to the delta layer using p^+ layer on the surface. To create ohmic contacts, we chose the composition Ti/Mo/Au with a layer thickness of 20 nm for Ti, 30 nm for Mo, and 100 nm for Au. The creation of ohmic contacts to a heavily doped diamond layer was described in detail in [21]. The contact resistances were measured by the transmission line modeling (TLM) method using the Keithley SCS 4200 measuring system. The contacts turned out to be ohmic without additional thermal annealing and had a contact resistance of $10^{-4} - 10^{-5} \text{ Ohm}\cdot\text{cm}^2$ [21]. The surface p^+ layer between the contact pads was etched away; therefore, the current flow in the structure occurred only along the buried delta layer. The results of electrical measurements are also shown in Table 1.

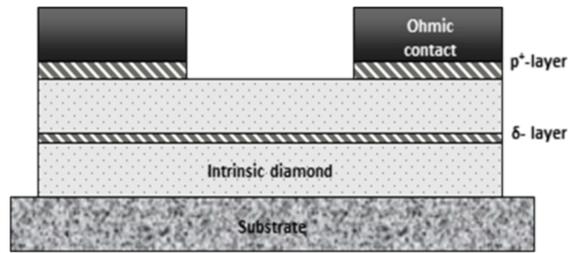
Table 1 lists the surface densities N_{2D} of boron atoms in grown delta layers. In the case of complete ionization of boron atoms in the delta layer, the value of N_{2D} in the delta layers should coincide with the measured surface concentration

TABLE 1. Boron concentration, delta layer thickness, surface concentration and Hall mobility of holes at room temperature, layer resistance

Sample label	Boron concentration (10^{21} cm^{-3})	Delta layer thickness (nm)	N_{2D} boron density (10^{13} cm^{-2})	P_s (10^{13} cm^{-2}) sheet hole concentration at $T = 300 \text{ K}$	Mobility at $T = 300 \text{ K}$ (cm^2/Vs)	Resistivity at $T = 300 \text{ K}$ ($\text{k}\Omega/\text{sq}$)
SS4-2	1.68	0.85	14.3	0.23	24.4	112
SS5-2	1.33	1.08	14.4			
SS9	1.05	1.39	14.6	2	4	70.5
SS10	0.99	2.01	19.9	11	1.4	37.75
SS1-3	1.29	1.43	18.4	7.0	1.5	58



a)



b)

FIG. 2. Photograph of a diamond surface ($0.22 \times 0.22 \text{ mm}^2$ in size) with test cells formed for Hall measurements in Van der Pauw geometry, TLM lines and CV cells (a), a scheme for the formation of a mesa structure and an ohmic contact to the delta layer using p^+ layer on the surface (b)

of holes. As can be seen from Table 1, the difference is quite significant, from 2.6 times for sample SS1-3 up to 63 times for sample SS4-2. Such a difference can be caused either by incomplete ionization of boron in the delta layer (for example, due to the formation of boron dimers) or by the presence of a sufficiently large number of traps for free holes, which can arise due to the presence of defects in the epitaxial layer [22].

The thinnest of all delta layers (0.85 nm) was the layer located on the surface of sample SS4-2. The Hall mobility of holes at room temperature was the highest for this sample and was $24.4 \text{ cm}^2/\text{V}\cdot\text{s}$. It can be assumed that in this series of experiments, with a decrease of the layer thickness from 2.01 to 0.85 nm, a tendency was observed for an increase of the mobility of holes with a narrowing of the dopant profile.

For the SS10 sample, the temperature dependence of the surface concentration and the Hall mobility of holes was studied in the temperature range $100 \text{ K} < T < 500 \text{ K}$. Measurements were also performed using Hall effect methods on etched mesa structures, Fig. 3. It can be seen from the figure that the hole mobility in the delta layer of this sample varied from 1 to $4 \text{ cm}^2/\text{V}\cdot\text{s}$ over the entire temperature range. The hole density also weakly depended on temperature and varied from $6 \cdot 10^{13}$ to 10^{14} cm^{-3} . Such a weak temperature dependence of the density and the mobility of holes and the resistance of the layer indicate that in the delta layer under consideration, a transition from semiconductor to metallic conductivity (MIT transition) occurred.

The error in measuring the resistivity by the four-point probe method did not exceed 10%. With a sufficiently high mobility, above several tens of $\text{cm}^2/\text{V}\cdot\text{s}$, measurements of the mobility and concentration of charge carriers will have the same error. However, with a decrease of mobility, the measurement error of the Hall EMF increases sharply. With a mobility of $\sim 1 \text{ cm}^2/\text{V}\cdot\text{s}$, the error of determining the concentration and mobility may reach 100%. Apparently, this is the reason for the strong scatter of the experimental points for mobility and concentration in Figs. 3(b,c) as the temperature decreases below 200 K. An additional reason for such a scatter may be the occurrence of a nonlinear I-V characteristic of ohmic contacts in this temperature range. At $T = 300 \text{ K}$, the I-V characteristic is linear.

When growing epitaxial layers with doped delta layers, we studied the roughness of the diamond surface for each sample. For example, one can make an assumption about the delta layer surface by comparing the surface roughness of samples SS4 and SS5-2. Table 2 compares two values characterizing the surface roughness RMS (root mean square roughness) and Ra (mean deviation) for these samples before and after the CVD growth process.

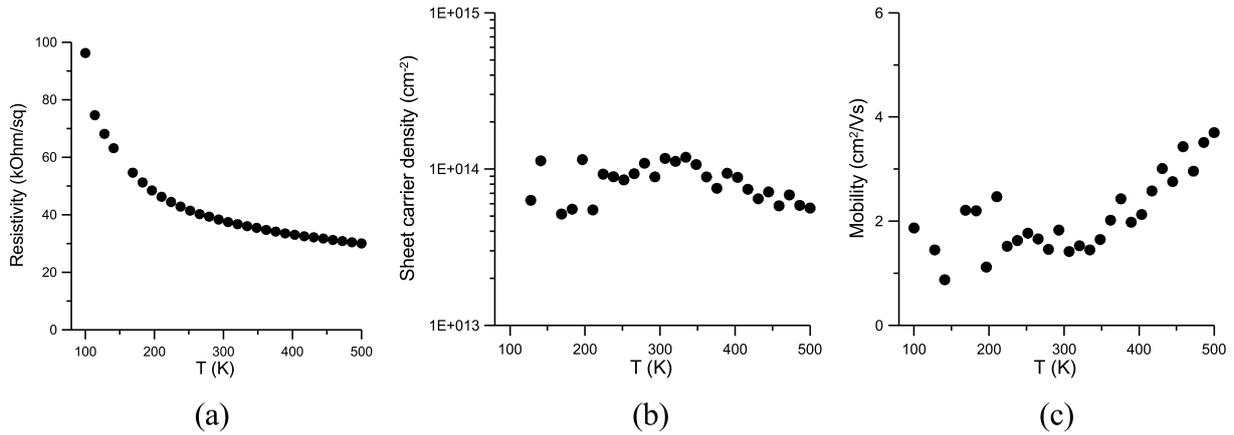


FIG. 3. Temperature dependence of surface resistance (a), Hall density of carriers (b), and Hall mobility (c) for the SS10 sample

TABLE 2. RMS and Ra surface roughness before and after CVD growth

	RMS for substrate, nm	RMS after CVD growth, nm	Ra for substrate, nm	Ra after CVD growth, nm	Etch pits/cm ² × 10 ⁴	Hillocks /cm ² × 10 ⁴	Σ def/cm ² × 10 ⁴
SS4-2	0.179	0.584	0.102	0.171	3.4	2.5	5.9
SS5-2	0.181	0.817	0.096	0.220	3.15	2.8	5.95

The surface analysis of the samples was carried out using a Zygo NewView 7300 white light interferometer. As can be seen from Table 2, the roughness of the epitaxial layer increased compared to the roughness of the substrate. The deterioration of the measured roughness after the CVD growth process is largely associated not with the deterioration of the roughness of the entire surface, but with the appearance of a small number of point relatively deep (high) defects: etch pits and pyramidal hillocks. This can be seen from the surface profile image shown in Fig. 4 (blue/red spots), and by the ratio of RMS/Ra values. Ra after growth, which is less sensitive to point defects, deteriorated by only 0.07 – 0.12 nm (1.7 – 2.3 times), while RMS deteriorated by 0.40 – 0.60 nm (3.3 – 4.5 times). The defect density for sample SS4-2 was $5.9 \cdot 10^4$ def/cm², and for sample SS5-2 it was $5.95 \cdot 10^4$ def/cm². Despite such a low density, apparently, surface defects of the SS4-2 sample influenced the results of electrical measurements (reduced the hole mobility).

4. Discussion

In [11], higher values of the hole mobility in samples with delta layers were obtained than in the present work. However, according to our latest studies, the results of [11] can not be unambiguously interpreted as the mobility of delta layers. In [11], multisectoral diamond substrates were used, which could contain both dielectric and conductive regions, which was not analyzed in [11] when choosing substrates. Such an analysis became possible only due to the use of local microwave conductivity microscopy [20]. All the substrates used in this work underwent such an analysis and did not contain conductive inclusions.

In our experiments single-sector and non-conductive substrates were used; layers with a boron atom concentration of 10^{21} cm⁻³ and a thickness of 0.85 – 2.01 nm were grown on them. However, the hole mobility did not increase significantly compared to uniform doping, which would be a consequence of a noticeable delocalization of holes. An increase in the mobility of holes is achieved due to the quantum penetration of the wave functions of holes from the potential well formed by the doped layer into the surrounding undoped diamond. As a result, their scattering by ionized boron atoms located in the doped layer decreases, while the mobility increases. When choosing the doping regime (to obtain the required concentration of boron atoms in diamond and the thickness of the delta layers), we were guided by the results of earlier calculations [8, 14], which predict a noticeable delocalization of holes for the above layer parameters.

The results obtained in the new series of experiments can be explained using the results of recent calculations of the delta layer profiles [17]. These calculations took into account the increase in the energy of the upper boundary of the valence band, which was not previously taken into account in [8, 14], which arises in such layers due to the high concentration of ionized boron atoms and leads to a significant deepening of the potential wells for holes created by them. As a result, the penetration of the wave functions of the holes into the undoped material surrounding the delta layer turns out to be much weaker than was previously thought. Thus, calculations show that at a concentration of boron atoms in the delta layer of the order of 10^{21} cm⁻³ and its thickness of 2 nm, only 7 % (and not 50 % as stated in [14]) of holes

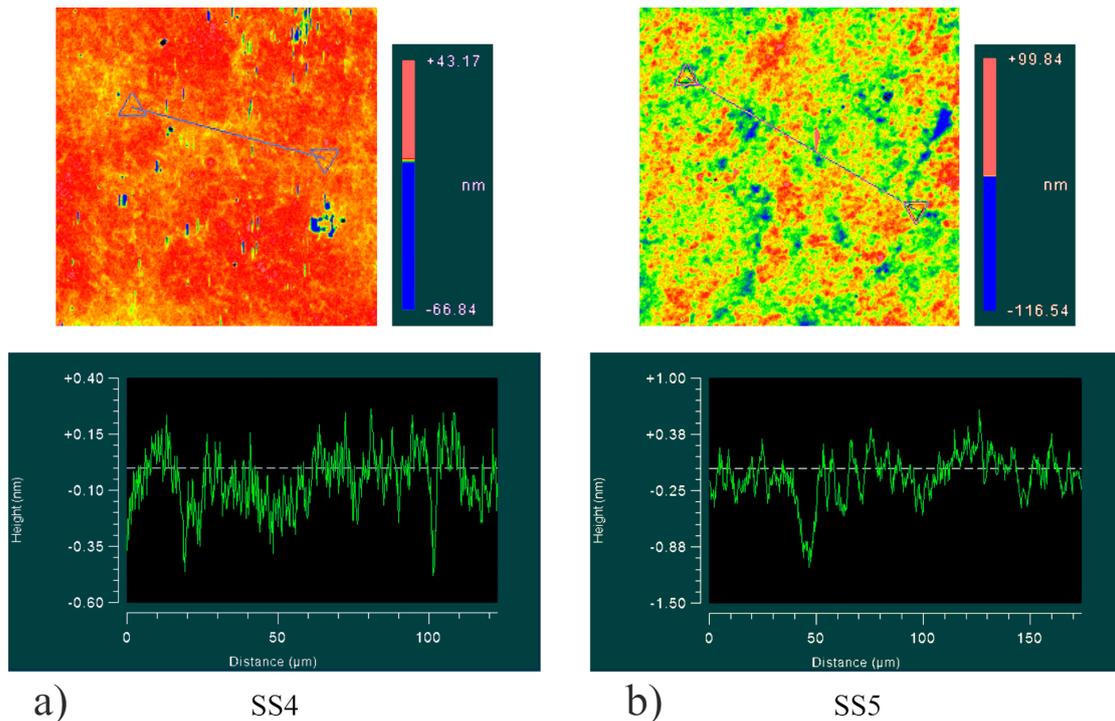


FIG. 4. Surface defects of the epitaxial layer of samples SS4-2 (a) and SS5-2 (b), investigated area $0.22 \times 0.22 \text{ mm}^2$

are outside it, at 1 nm, 19 % (and not 70 % as in [14]) and at 0.5 nm – 47 % (and not 75 % as in [14]). Thus, an increase in the hole mobility in delta layers, as compared with homogeneous doping with the same concentration of boron atoms, can only be expected at delta layer thicknesses of the order of 0.5 nm or less.

In the presented results (Tables 1 and 2), the obtained hole mobility values, firstly, can be explained by the insufficiently small thickness of the delta layers and a small fraction of delocalized holes. Second, effects other than quantum penetration can manifest themselves, additionally affecting the resulting mobility and density of free holes in the structure. Such effects may include the scattering of holes on the surface roughness of the layer and the presence of defects (traps) in the delta layer. Based on the temperature dependence of the surface resistance, Hall density, and hole mobility, it can be assumed that an insulator–metal phase transition occurred and all boron atoms were ionized, but the density of free holes was significantly lower (Fig. 3). In our experiment, the measured density of defects was quite high (Table 2) and such a high density of defects (traps) can explain the experimental difference between the density of ionized boron atoms and the measured density of charge carriers.

We also note the results of the recent work of the authors [23], which presents data on the mobility of holes in delta-doped boron layers grown on multisectoral HPHT substrates. In deep delta layers, about 1.5 nm thick, a hole mobility of $200 \text{ cm}^2/\text{V}\cdot\text{s}$ was observed, and in a layer on the surface, up to $600 \text{ cm}^2/\text{V}\cdot\text{s}$. However, the two-dimensional concentration of free carriers was only 0.1 % of the two-dimensional concentration of boron atoms, which indicated an extremely low concentration of delocalized holes. These results also confirm the main conclusion of this work about the need for thinner delta layers to create a conductive 2D channel.

Thus, to achieve high hole mobility in deep delta layers in CVD diamond, it is necessary to obtain both a thin (about 0.5 nm thick) delta layer with a high surface hole concentration of $5 \cdot 10^{13} \text{ cm}^{-2}$, and a sufficiently smooth layer surface with a minimum number of defects, providing a slight increase in roughness compared to the substrate surface, for which the RMS value is less than 0.2 nm. In such a structure, in our opinion, one can expect a significant increase of the mobility of charge carriers in semiconducting diamond. It seems to us that such layers can be obtained by improving of our doping regime.

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Submitted 25 July 2022; revised 8 September 2022; accepted 15 September 2022

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Conflict of interest: the authors declare that they have no conflict of interest.