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THE MECHANISM OF FORMATION OF STRUCTURAL V-DEFECTS IN POLAR AND SEMIPOLAR EPITAXIAL GaN FILMS SYNTHESIZED ON SiC/Si(111) AND SiC/Si(100) HETEROSTRUCTURES

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Abstract. The past results related to the main features of formation of structural V-defects in polar GaN(0001) and semi-polar epitaxial GaN films are presented. The GaN films have been synthesized by hydride-chloride vapor-phase epitaxy (HVPE) on two different substrates Si(111) and Si(100) with an intermediate nano-SiC epitaxial layer. The nano-SiC layer has been formed by a method of atom substitution on the Si substrate. The experimental studies have demonstrated that V-defects on the surface of GaN(0001) films are regular hexagons of 30 μm in size, while on the surface of GaN V-defects are of the order of 1 μm. It was found that V-defects on the semipolar face are extended along the $\langle 11\bar{2}3\rangle$ direction. The size of oblique facets of V-defects on the surface of polar GaN(0001) is of about 1 μm, while on the surface of semipolar GaN$(1\bar{1}01)$ layer they are much less, measuring about 150 nm, on the average. On the basis of thermodynamics, the mechanisms of nucleation of V-defects on polar and semipolar faces of GaN epitaxial films are elucidated, and the criteria of the formation of V-defects are theoretically derived. A good qualitative agreement between the experimental results and the theoretical model was found.

1. Introduction.

Present-day needs for substrates with semi-polar GaN layers for the production of light diodes [1] and lasers [2] grow steadily. This is due to the fact that the use of polar structures in quantum-dimensional (QW) III-nitride opto-electronic devices is associated with the Stark-effect [3] which is brought about by strong piezoelectric polarization in polar structures. In the case of semi-polar structures, the piezoelectric polarization is absent, and this opens up new prospects in the production of devices of a new generation on the basis of semi-polar (Al, Ga, In)N structures.

Substrates for growing of semipolar structures are usually synthesized by the following way. Initially the thick epitaxial layers of polar GaN (0001) should be grown. Then the grown up epitaxial GaN layers are cut in such a way that semipolar planes would crop out on the crystal surface. After that, these semipolar planes are polished. The surface area of substrates so produced is not large and depends on a thickness of an initial GaN (0001) layer. The thicker the layer of an initial polar GaN film, the larger is the area occupied by GaN semipolar plane. Thick crack-free layers of GaN, as is well known, are difficult to grow. That is why the fabrication of semipolar substrates with the use of thick GaN layers is a labor consuming and highly expensive process.

The search for synthesis of GaN semipolar epitaxial films on previously prepared foreign substrates is now actively pursued. For this purpose the n-, r-Al_2O_3 sapphire substrates are coming into use [4]. However, that to use them as substrates for growing of GaN semipolar layers, both substrates of Al_2O_3 and of thick GaN must be preliminary prepared. At present, the producers of light-emitting diodes are making efforts to grow the substrates up to 12 inches in diameter [3]. Sapphire and silicon carbide substrates of such a size are not currently produced as yet. Moreover, if this were the case, the price would be unproperly high. In this regard, only sapphire substrates are most promising for the synthesis of structures on the basis of gallium nitride.

The authors of papers [5, 6], whose results are generalized in the review [3], proposed for a first time to use for the production of GaN semipolar films silicon (Si) plates coated previously with a nano-layer of epitaxial silicon carbide (SiC). It is appropriate to note that for the synthesis of GaN semipolar films, a nano-layer of SiC was grown up by a new atom substitution method, advanced in papers [7, 8]. The mechanism of growth of SiC, in this case, is fundamentally different from the classic method of growth of SiC on Si. As a result, the epitaxial films of semipolar GaN have been obtained on Si(210) [5] and Si(001) [6].

One should mention, that for the synthesis of both semipolar and polar GaN layers, the commonly accepted methods are used; these are epitaxy from organometallic compounds (MOCVD) [9], hydride-chloride vapor-phase epitaxy (HVPE) [3-6] and molecular beam epitaxy (MBE) [10].

The main factor retarding an intensive introducing of heterostructures based on GaN into the production is the high content of various type defects. On the surface of gallium nitride heterostructures there are originated so called V-defects [11-13], which are etching pits, usually with a crystalline faceting, located on the surface of the GaN film. The structure of V-defects formed on the GaN(0001) surface is well studied [11-13]. Thus, several models have been proposed [13, 14], in which the V-defects were suggested to be formed due to the interaction of dislocations [13] or due to elastic stresses in heterolayers [15]. According to the advanced hypothesis, elastic stresses lead to the lower velocity of the growth on the $(1\bar{1}01)$ plane compared with that on the plane (0001).

When studying the growth mechanism of GaN semipolar layers on Si and on Si coated with a layer of SiC [3, 5, 6], we have revealed that on the surface of GaN semipolar layers the V-defects are formed. As far as we know, up to now, the studies of morphology and nucleation mechanism of V-defects on the surface of GaN/Si semipolar layers have not been carried out. In this connection, our goal was to elucidate experimentally this problem for GaN semipolar layers grown up on carbide substrates of (111) and (001) orientations, coated with a nano-layer of epitaxial SiC.

2. Experiment

The nitride layers were synthesized by a combined method. At the first stage, the SiC layer was formed on Si(111) and Si(001) by using the atom substitution method [7, 8]. The thickness of SiC layer on Si(111) was of about 90 nm, and that on Si(001) of about 30 nm. After that, with the aid of HVPE method, the buffer AlN layer of thickness ranged from 300 to 1000 nm was formed. Then the basic layer of GaN has been deposited. That to synthesize the GaN film on the surface of Si(111), the technology described in [16] has been applied. In the process of the further experiment the GaN(0001) layer with a thickness of about 100 μm was grown up. On the surface Si(001) disoriented in the direction [110] by 2-7 °, the layer has been growing by the method described in [5]. The thickness of the GaN$(1\bar{1}01)$ layer was 15 μm. The structural characteristics of so produced films of GaN(0001)/AlN/SiC/Si(111) and GaN$(1\bar{1}01)$/AlN/SiC/Si(001) and their morphology have been studied by different methods: these are atomic-force microscopy (AFM), scanning electron microscopy (SEM), x-ray diffractometry and x-ray luminescence.

3. Experimental results

X-ray diffraction measurements of the structures revealed that a half-width of the x-ray diffraction curve was ω_θ = 10 arcmin for the polar GaN(0002) layers and ω_θ = 20 arcmin for semipolar GaN$(1\bar{1}01)$ layers.

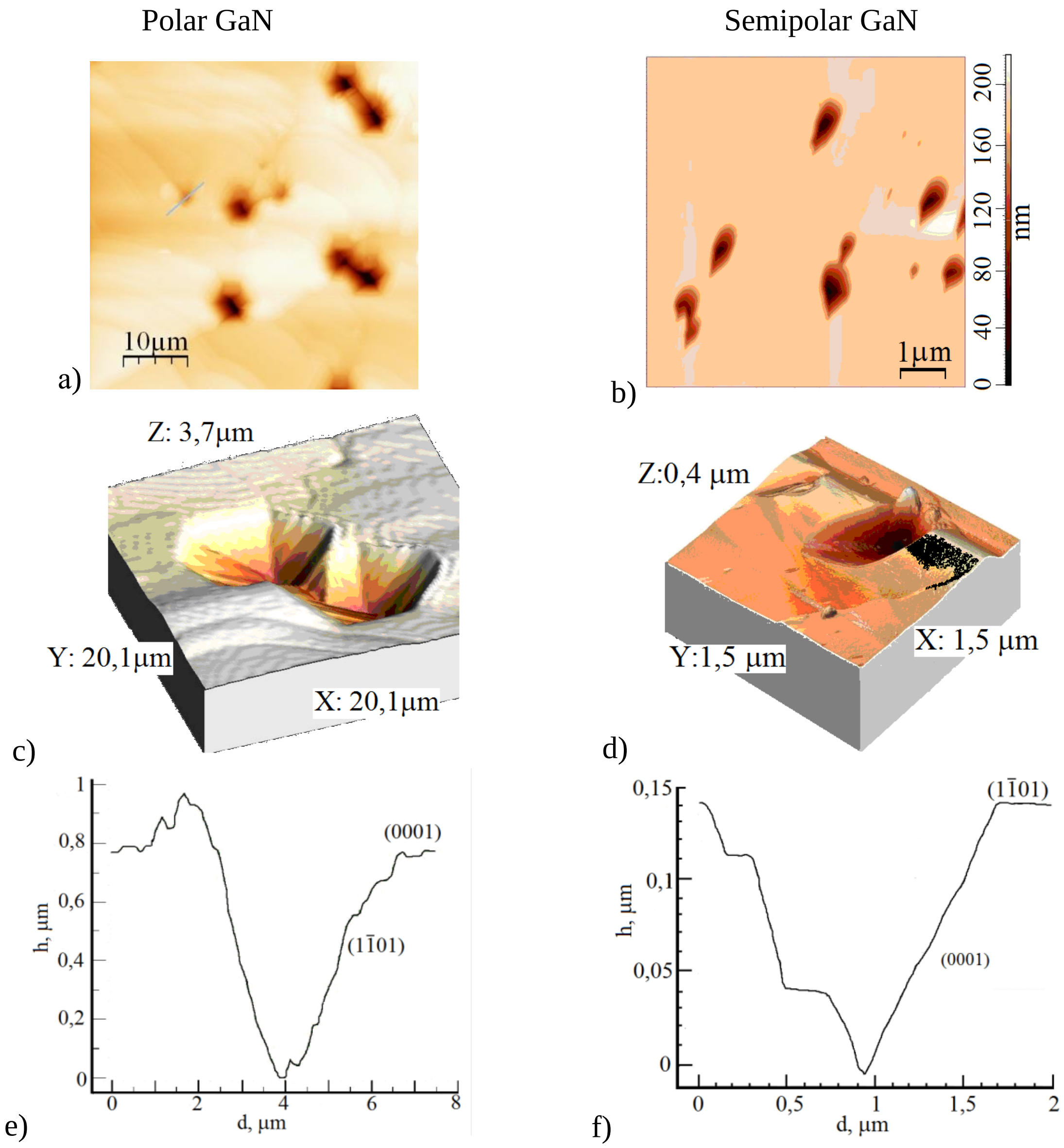

Fig. 1. AFM image of the surface of GaN layer and the profiles of the surface inhomogeneity distribution in directions $\langle 11\bar{2}3\rangle$. Figures c and d have scale X, Y, Z = 1 cm.

The morphology of V-defects on the surface of polar and semipolar GaN turned out to be essentially different. On the surface of GaN(0001) film the V-defects were regularly shaped hexagons with a transverse size 30 μm (Fig. 1a,c). On the surface of GaN$(1\bar{1}01)$ layer the V-дефектs were of the order of 1μm. The V-defects on the surface of the semipolar GaN $(1\bar{1}01)$ layer were observed to be extended along directions $\langle 11\bar{2}3\rangle$ (Fig. 1b,d). The sizes of the inclined facets of the V-defects on the surface of semipolar GaN(0001) were found to be of the order of 1 μm. The facets of the V-defects on the surface of the semipolar GaN$(1\bar{1}01)$ layer were much less and averaged 150nm. The profiles of the V-defects in the direction of growth of polar GaN(0001) and semipolar GaN$(1\bar{1}01)$ were also essentially different (Fig. 1e,f). The profile of the V-defect on the surface of polar GaN exhibited the symmetric monotonic decrease and rise (Fig. 1e). The profile of the V-defect in the direction $\langle 11\bar{2}3\rangle$ on the surface of semipolar GaN$(1\bar{1}01)$ was quite different: it had a stepwise decrease and a monotonic rise free of steps and fractures (Fig. 1f).

4. Qualitative analysis of the results

Thus, the experiment has shown that the morphology and sizes of V-defects formed on the surface of the polar GaN layers are unlike those formed on the surface of semipolar GaN layers. There is nothing strange in this fact. It is clearly apparent that the V-defects, which are nothing else than the etching pits on the crystalline surface, should be originated at places of the emergence of dislocations as it takes place in the case of other crystals [17]. It was unambiguously shown [18] that the dislocations, which were formed at the boundary of the silicon substrate and AlN layer, grow up in the direction of the C-axis and become the sources of V-defects on the surface of the GaN film. On the densely packed GaN-surface the etching pits will have the symmetric hexagonal faceting corresponding to the symmetry of the crystal [17]. The similar etching pits are also observed in other hexagonal crystals, e.g. in CdS crystals [17]. The V-defects on the GaN(0001) surface also exhibit the densely packed faces $(1\bar{1}01)$. At the same time, the surface energy of the $(11\bar{2}0)$ face is less than that of the [19]. Therefore, the surfaces $(1\bar{1}01)$ in conditions of undersaturation in components, needed for the growth of the film, i.e. in atoms of Ga and N, will be broken up into parallel steps [17]. This is precisely what we observed in the experiment (see Fig. 1e). Figure 2 is a schematic drawing of the V-defect on the surfaces (0001) and $(1\bar{1}01)$ of GaN.

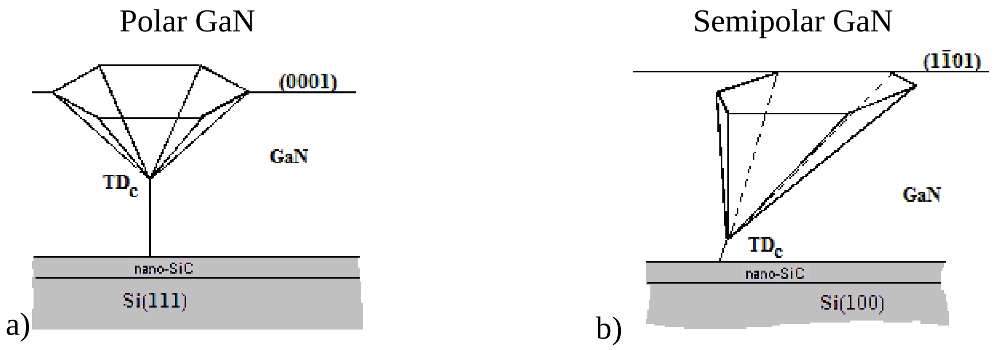

Fig. 2. Schematic drawing of the V-defect: a) GaN(0001), b) GaN$(1\bar{1}01)$.

In the case of semipolar GaN, the oval configuration of the V-defect (Fig. 1 b,d) and its profile (Fig. 1f) point out to the explicitly asymmetric behavior of the V-defect in the direction $\langle 11\bar{2}3\rangle$. This face is featured by large Miller indexes, and the walls of its "crater" form a series of parallel steps with the terraces parallel to the plane $(1\bar{1}01)$ and the side-on walls parallel to the plane (0001). It is precisely this effect that we experimentally observed.

The presence of terraces and steps on the given surface indicates convincingly that this surface is in conditions of undersaturation in components. In the opposite case, as well known, the facets with the large Miller indexes will grow fast and "wedge-out" [17]. In the present studies, as was mentioned above, we used the HVPE method to synthesize GaN. With the use of this method, the growth of the film was realized according to the chemical reaction:

$$GaCl+NH_3=GaN+HCl+H_2 . \quad (1)$$

Evidently, on faces (0001) and $(1\bar{1}01)$, in conditions of undersaturation in components, the relation between the gaseous flows which is V/III, may be violated. It, as is well known, will produce a significant change in the growth parameters of the GaN film, which result in difference in the morphology and sizes of V-defects on the faces (0001) and $(1\bar{1}01)$. One can explain this fact by invoking the simple qualitative model described in [17]. According to this model, the average slope of an etching pit, formed on the place of emergence of dislocations, is $\tan\theta = v_n/v_t$, where v_n is the velocity of etching of the pits, normal to the surface; v_t is the tangential (lateral) velocity of etching of the pits, describing the propagation velocity of the elementary steps along the surface. If the pits are formed with the constant velocity, then the average slope of the etch pit with a diameter D and the growth time t are linked by the simple functional relation [17]:

$$\tan\theta = \frac{1}{v_t}\frac{D}{t} + \frac{v_p}{v_t}, \quad (2)$$

where v_p is the velocity of dissolution of the crystal surface; $D/t= v_n - v_p$. It follows from the above equation that, when $v_n \gg v_p$, the straight line, describing the dependence $\tan\theta = f(D/t)$, will pass in the vicinity of the origin of the coordinates, and all pits, regardless of the defect type, on which they have originated, will be of the same width. If the velocity of etching, v_p, is sufficiently high, then deep pits will be wider than shallow ones. When the V-defects are growing on the surface GaN(0001), the pits, as it follows from Fig. 1e, have a significantly larger diameter and depth, compared to those on the surface $(1\bar{1}01)$. According to equation (2), this means that the velocity of etching of the surface (0001), υ_p exceeds that of the surface $(1\bar{1}01)$, on which the pits are of significantly less diameter and not so deep.

It is appropriate to note, that formula (2) describes the growth of etch pits under conditions when the crystal is purposely placed in chemical etching solutions. In our case, it is the process of growing of the crystal which is studied, but not its etching. However, the formation of the V-defects, i.e. etch pits, indicates clearly that at later stages of the growth, the crystal is under conditions close to undersaturation in components forming the film (below we prove this statement). A small deviation from stoichiometry, in accordance with reaction (1), will lead to the dissolution of the crystalline surfaces. The face (0001) is densely packed. From the crystal growth theory [20] it follows, that the growth of the densely packed crystal surfaces occurs through the formation of two-dimensional seeds. To do this would require to maintain the elevated oversaturation in gallium and nitrogen in the system. The calculations [21] have shown that when a temperature of the growth is higher than 850 °C, the GaN seeds do not practically originate. In this region the layer-spiral mechanism is responsible for growth of GaN layers and the formation of steps is probably initiated by screw dislocations. If there are no dislocations, then in conditions approaching the equilibrium, the etching of the surface, even with the substance supply, may commence. For the growth of the face $(1\bar{1}01)$ the two-dimensional seeds are not necessary. The steps, already formed on this face, are the seeds themselves. When the faces (0001) and $(1\bar{1}01)$ are in equal growth conditions, the face $(1\bar{1}01)$ will grow faster than the face (0001). This means that the velocity of etching v_p, entering into formula (2), will be higher for the face (0001). Hence, and according to formula (2), the etch pits, formed on the dislocations, will be wider and deeper.

In this connection we mention, that in the vicinity of the emergence of dislocations and on the faces (0001) and $(1\bar{1}01)$, the relation between gaseous flows of the components, V/III, may be violated, see Eq. (1).

5. Thermodynamics and kinetics of the nucleation of V-defects in polar and semipolar epitaxial GaN films

A large body of models describing the nucleation of etch pits on dislocations, are fully considered in the monograph [17]. Among them the Cabrera [22] and Schaarwehter [23] models hold the lead. According to the Cabrera model, the formation of a disk-shaped (or coin-shaped) etch pit results in a decrease in free energy by $hA\kappa \ln r/r_0$, where $A = G^{GaN}b^2/4\pi$, G^{GaN} is a shear modulus of GaN; b is the Burgers vector of dislocations in GaN; $\kappa = 1/(1-\nu)$; ν is the Poisson coefficient of GaN; and r_0 is a radius of the dislocation nuclei, beyond which the theory of elastic continuum is valid, r and h are a radius and a depth of an etch pit, respectively. Schaarwehter [23] has shown that the Cabrera model holds only beyond the dislocation nucleus, which is not, however, proved experimentally. He proposed to take for calculations another form of the potential, namely, the potential of the form $hA\kappa(r/r_0)$, i.e. the deformation energy, along Schaarwehter, increases linearly with radius, not logarithmically as it does in the Cabrera model. As a result, Schaarwehter has derived the expressions for the critical radius r'_{cr} and the free energy $\Delta \mathrm{F}'_d\,(r'_{cr})$ of an etch pit nucleated on dislocations. These dependences are of the form:

$$r'_{cr} = \frac{\gamma\Omega}{\Delta\mu}\left(1 - \frac{A\kappa}{2\pi\gamma r_0}\right) = \sqrt{p}r_{cr}, \tag{3}$$

$$\Delta \mathrm{F}^*_d(r'_{cr}) = p\Delta F^*(r_{cr}). \tag{4}$$

Here $p = \left(1 - \frac{A\kappa}{2\pi\gamma r_0}\right)^2 = \left(1 - \frac{r_F}{r_0}\right)^2$; $r_F = \kappa G^{GaN}b^2/8\pi^2\gamma$ is the Frank radius; γ, surface energy; Ω, a volume per atom, in our case, per a molecule of GaN; $\Delta\mu = \mathrm{kT}\ln C/C_o$; C_o, the concentration of saturation in the vapor phase; C, the concentration of the matter at the place of emergence of dislocations; $r_{cr} = \gamma\Omega/\Delta\mu$, the critical size of an etch pit, formed on the smooth surface, without regard for the dislocation energy; $\Delta F^*(r_{cr}) = \pi h\gamma^2\Omega/\Delta\mu$, the free energy of formation of an etch pit of depth h on the crystal surface, without considering the dislocation energy.

The expression for the chemical potential $\Delta\mu$, entering into formulas (2) and (3), is applicable to one -component systems only. It is can, however, be generalized, by invoking the recently developed theory of nucleation of a new phase in chemical reactions [24], in which the chemical affinity ξ plays a role of the quantity $\mathrm{kT}\ln C/C_o$. In our case, with regard for the reaction (1) of formation of GaN, for the chemical affinity ξ we have the expression

$$\xi = kT\ln\frac{P_{H_2}P_{GaCl}}{P_{NH_3}K_p^{GaN}}, \tag{5}$$

where $\mathrm{K}_\mathrm{p}^\mathrm{GaN} = P^0_{H_2}P^0_{HCl}/P^0_{NH_3}$ is the equilibrium constant of the chemical reaction (1); $P^0_{H_2}, P^0_{GaCl}, P^0_{NH_3}$ are the pressures of hydrogen, gallium chloride and ammonia, respectively, which are equilibrium at temperature of the synthesis; $P_{H_2}, P_{GaCl}, P_{NH_3}$ are the operating pressures [at which the synthesis of GaN occurs along the reaction (1)]. One should keep in mind, that on dissolving of the surface, the operating pressures in the gaseous phase may be comparable (or even lower) to the equilibrium pressures in the vicinity of the dislocation nucleus.

Let us estimate the Frank radius for the faces (0001) and $(1\bar{1}01)$ of GaN. In epitaxial layers with wurtzite structure of a lattice, the dislocations of three types are observed: the edge dislocations (mixed dislocations (b_m) and screw nano-tubes (b_{nano}). The corresponding Burgers vectors are:

$$b_e = \frac{1}{3}(11\bar{2}0),\ \left(b_e = a, b_e^2 = a^2\right);\ b_m = \frac{1}{3}(11\bar{2}3),\ (b_{rmm} = \sqrt{c^2 + a^2},\ b_m^2 = 3.66a^2);$$

$$b_s = [0001],\ (b_s = c, b_s^2 = 2{,}66a^2);\ b_{nano} = n \times b_s,$$

parameter n may take the following values n=1,2,3… and a=0,312 nm; c=0,519 nm. The values of the shear modulus G^{GaN}, of the Poisson coefficients ν of GaN, and of surface energies γ for planes (0001) and $(1\bar{1}01)$ are given in the paper [19]: $G^{GaN}_{(0001)} \approx 113$GaP, $G^{GaN}_{(1\bar{1}01)} \approx 90$GaP, $\nu_{(0001)} \approx 0.336$; $\nu_{(1\bar{1}01)} \approx 0.187$, $\gamma_{(0001)} \approx 4.41\,Jm^{-2} \gamma_{(1\bar{1}01)} \approx 3.23\,Jm^{-2}$. We note, that for $\gamma_{(1\bar{1}01)}$ we used the value of the surface energy calculated in [19] for the face $(11\bar{2}0)$ since from the crystallographic view point, the faces $(1\bar{1}01)$ and $(11\bar{2}0)$ differ insignificantly. As a result, for the Frank radius on the surface (0001), in the case $n = 1$, we have $r_F^{(0001)} \approx 5.5 \cdot 10^{-11} m$. When $n = 2$, $r_F^{(0001)} \approx 3 \cdot 10^{-10} m$. The Frank radius on the plane $(11\bar{2}0)$ is $r_F^{(0001)} \approx 2.8 \cdot 10^{-11} m$. One can assume for estimates that r_0 is of the order of the Burgers vector for the given face.

For the face $(1\bar{1}01)$ the factor $\sqrt{p}$ in formula (3) takes the following values: for the plane (0001) at n=1, $\sqrt{p}_{(0001)} \approx 0.88$. For the plane $(11\bar{2}0)$ - $\sqrt{p}_{(11\bar{2}0)} \approx 0.91$. By Schaarwehter theory [23], when the ratio $r_F/r_0 < 0.5$ holds, the etch pits are nucleated in accord with the classic theory. In this case, the value of the surface energy is changed only and should be renormalized, according to (3), as $\gamma' = \gamma \cdot \sqrt{p}$. Since $\sqrt{p} < 1$, the critical size of the pit, at the same value of the chemical affinity, will be less, than that calculated without regard for effects of dislocations on the nucleation. The nucleation barrier will also be decreased (4), resulting in higher velocity of the nucleation compared with the case of a smooth surface. In our case, as follows from the above-given estimates, the ratio $r_F/r_0 < 0.5$ takes place. Thus, in order to estimate the critical size and the nucleation velocity of etch pits, one should use the renormalized value of γ. If the condition $r_F/r_0 < 0.5$ holds both on the plane $(1\bar{1}01)$ and on the plane (0001), then, at the same value of the chemical affinity, the etch pits will be nucleated more intensively on the plane $(1\bar{1}01)$ where their critical sizes will be less. The reason is that the surface energy of an etch pit on the plane $(1\bar{1}01)$ is less than on the plane (0001). It is appropriate to note, that we ignored the lateral faceting of the etch pits. In a general case, taking it into account, we could come to the opposite result. In our model we assumed that the etch pits have a shape of plane disks.

When the nucleation occurs on nano-tubes with, we have $r_F/r_0 \approx 0.6$ and, according to the paper [23], different scenarios of the pit growth can develop, depending on the chemical affinity value. Thus, when $r_F/r_0 \approx 0.6$, there exists some critical potential difference $\Delta\mu^*$ which has the same dimension as the chemical affinity does. This potential difference is of the form:

$$\Delta\mu^* = \frac{2\pi^2\gamma^2\Omega}{\kappa G^{GaN} b^2}. \tag{6}$$

When $\xi > \Delta\mu^*$, the barrier for dissolution does not need to exist. In this case the pits will be formed as a result of the spontaneous nonstationary dissolution. When $\xi = \Delta\mu^*$, there may occur either the stationary spiral dissolution around the screw dislocation, or the repeated process of the two-dimensional formation of nuclei, initiated by edge dislocations. Let us estimate the quantity $\Delta\mu^*$. By using the above-calculated data on the shear modules, surface energies, Burgers vectors and other parameters, we find that for the face (0001) with nano-tubes, at $n = 2$, $\Delta\mu^*_{(0001)} = 1 \cdot 10^{-19} J$. We have grown the GaN film at 1080 °C. At this temperature the affinity is $\xi \sim 0.14 \cdot 10^{-19} J$, and, hence, the condition $\xi \ll \Delta\mu^*$ turned out to hold. This is to say that the stationary process of layer-spiral etching of the plane (0001) around the screw dislocations takes place. This conclusion is fully sustained by the image of the V-defect with a characteristic layer-spiral relief, shown in Fig. 1. Such a result is evidence in favor of the decisive role of nano-tubes in the course of etching of the GaN(0001) plane. In the presence of nano-tubes, the etching on the plane is governed by the mechanism of the layer-spiral dissolution of the film.

On such dissolving, the morphology of the pit will be symmetric. The pit will dissolve fast, and its diameter and depth exceed significantly the sizes of the pit nuclei on the $(1\bar{1}01)$ surface, since on this surface etch pits are nucleated in accord with common classic mechanism, though with renormalized surface energy.

As a result, the growth velocity and the sizes of V-defects on the surface of polar GaN(0001) will be large, compared with that on the surface of semipolar GaN$(1\bar{1}01)$. It is precisely these phenomena that we observed in the experiment. In essence, the pit on the GaN(0001) surface dissolves (in an opposite sense) similarly to the growth of crystal: the role of the growth center is played, in this case, by a screw dislocation.

As is well known, in such conditions, the crystal will grow at arbitrary small oversaturations. For its growth there is no need for the two-dimensional nucleation of steps. The steps are formed by screw dislocations.

6. Conclusions

The studies have revealed the following:

1. V-defects, formed on the surface of polar GaN(0001), synthesized on AlN/SiC/Si(111) heterostructure, are regularly hexagons.
2. V-defects on the surface of semipolar GaN$(1\bar{1}01)$, synthesized on AlN/SiC/Si(100) heterostructure, exhibit the asymmetric morphology and are extended along the $\langle 11\bar{2}3 \rangle$ direction.
3. The dislocation nano-tubes, formed by screw dislocations, play a decisive role in the formation of V-defects on the GaN(0001) surface. In the presence of nano-tubes, the etching on the plane (0001) is governed by the mechanism of the layer-spiral dissolving of the film.
4. V-defects on the surface of semipolar GaN$(1\bar{1}01)$ are originated according to the common classic mechanism of nucleation of a new phase, though with the renormalized value of the surface energy γ. The renormalized value of γ depends on elastic properties of a medium (a film material) and on the value of Burgers vector of dislocations present on a given surface.

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