

3. Yu. F. Biryulin, N. V. Ganina, et al., *Fiz. Tekh. Poluprovodn.*, 19, No. 6, 1104 (1985).
4. E. V. Solov'eva, M. G. Mil'vidskii, and N. V. Ganina, *Fiz. Tekh. Poluprovodn.*, 16, No. 10, 1810 (1982).
5. E. V. Solov'eva, N. S. Rytova, et al., *Fiz. Tekh. Poluprovodn.*, 15, No. 11, 2141 (1981).
6. V. K. Bazhenov and V. I. Fistul', *Fiz. Tekh. Poluprovodn.*, 18, 1345 (1984).
7. E. V. Solov'eva, M. G. Mil'viskii, and N. S. Rytova, *Semiconductor Crystals and Film Growth [in Russian]*, Pt. 2, Nauka, Novosibirsk (1984).
8. W. C. Mitchel and P. W. Yu, *J. Appl. Phys.*, 57, 623 (1985).
9. E. V. Solov'eva, M. G. Mil'vidskii, et al., *Fiz. Tekh. Poluprovodn.*, 16, No. 3, 566 (1982).
10. F. E. Rosztocky and F. Ermanis, *J. Appl. Phys.*, 41, 264 (1970).
11. Takatosi Nakanisi, *Jpn. J. Appl. Phys.*, 12, 1263 (1973).
12. V. I. Fistul', *Fiz. Tekh. Poluprovodn.*, 17, No. 6, 1107 (1983).
13. V. B. Ufimtsev and V. I. Fistul', *Élektron. Tekh.*, Ser. 6, No. 9 (194), 42 (1984).

ELECTRON-MICROSCOPE STUDIES OF THE SURFACE OF  
 EPITAXIAL GaAs LAYERS IN THE PROXIMITY OF THE  
 (111)A FACE

I. V. Ivonin and S. E. Toropov

UDC 621.315.592:548.522

Electron-microscope studies have been carried out on the relief of the growth surface of epitaxial gallium arsenide layers in the vicinity of the (111)A face and the quantitative characteristics of the elements of the relief have been determined: the density of growth centers on a singular face, the height of the steps, and the distances between steps in the vicinal planes. The parameters of the growth steps are shown to depend on the orientation of the face and the concentration of the growth components in the vapor phase.

The growth of epitaxial layers of gallium arsenide on substrates with a (111)A orientation or close to it is known to be very sensitive to variations in the crystallization conditions. An increase in the concentration of the growth components, affected by variation of the  $\text{AsCl}_3$  pressure upon entry into the chloride gas-transport system, causes a decrease in the depth of the singular minimum in the  $V(\varphi)$  function at a point that corresponds to the (111)A face [1]; in this case the singular face becomes kinetically unstable and can break down into nonsingular faces because of the considerable increase in the normal growth rate (frequency of formation of nuclei of new layers at the vertices of growth centers) [2]. The introduction of considerable amounts of a dopant into the vapor phase results in more pronounced changes in the anisotropy of the growth rate: the singular minimum on the (111)A face, which is observed at low pressures of arsenic trichloride, goes over into a singular maximum at high  $p_{\text{AsCl}_3}$  [1]. The changes in the nature of the anisotropy of the growth rate are accompanied by changes in the anisotropy of impurity trapping [3, 4].

In this communication we present the results of studies, which were carried out during the development of the papers [1-4] and were aimed at obtaining experimental information about the structure of the growth surfaces of epitaxial GaAs layers in the vicinity of the (111)A face. The relief of the surface was examined by means optical microscopy and electron-microscope methods of carbon replicas previously shadow-cast with platinum; we studied gallium arsenide films, highly doped with tellurium, which were grown at two values of the  $\text{AsCl}_3$  inlet pressures: 10 and 400 Pa.

---

V. D. Kuznetsov Siberian Physicotechnical Institute at Tomsk University. Translated from *Izvestiya Vysshikh Uchebnykh Zavedenii, Fizika*, No. 9, pp. 8-12, September, 1987. Original article submitted December 4, 1985.

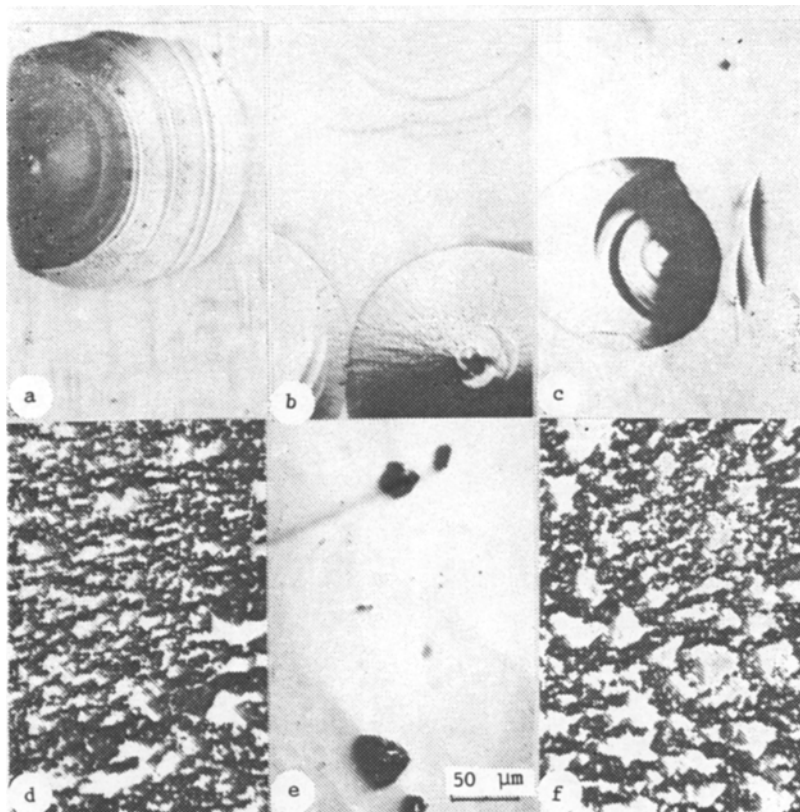


Fig. 1. Optical photograph of the surface of epitaxial GaAs layers in the vicinity of the  $\langle 111 \rangle_A$  band for two values of  $p_{\text{AsCl}_3}$  (in Pa): a-c) 10, d-f) 400. Substrate orientation: a, d)  $2^\circ$   $(111)_A(110)$ ; b, e)  $(111)_A$ ; c, f)  $2^\circ$   $(111)_A(001)$ .

#### EXPERIMENTAL RESULTS AND DISCUSSION

An examination of the morphology of layers grown at an arsenic trichloride pressure of 10 Pa shows that they have a mirror surface on the  $(111)_A$  face and its vicinals, which deviate by  $2^\circ$  and  $4^\circ$  in the directions toward  $(001)$  and  $(110)$ . The growth figures on them are round, are beveled at the vicinals, and have a large number of growth steps (Fig. 1a-c). Steps without clear faceting appear on the smooth surface between figures. At deviations of  $6^\circ$  and  $8^\circ$  from the  $(111)_A$  face the surface of the layers becomes dull. The roughness of the relief, which is revealed in an optical microscope, becomes finer and more uniform as the angle of deviation from  $(111)_A$  increases.

The morphology of the grown layers changes when the arsenic trichloride pressure is increased to 400 Pa. The  $(111)_A$  face grows as a mirror face and rare growth pits are visible in an optical microscope (Fig. 1e). Under oblique illumination, the  $(111)_A$  surface can be seen to consist of vicinals with a very small tilt ( $<1^\circ$ ). Growth on substrates that deviate  $2^\circ$  or more from  $(111)_A$  cause the formation of numerous growth pits, which almost entirely cover the surface (Fig. 1d, f).

Electron-microscope examination of the relief of the growth surface of GaAs layers showed that the very distinct step-layer structure of the vicinal  $\varphi^\circ(111)_A$  planes is observed only at a very low (10 Pa) initial pressure of arsenic trichloride (Fig. 2a-c). Numerous  $[(4-5) \cdot 10^7 \text{ cm}^{-2}]$  growth islands and steps exist on the  $(111)_A$  face (Fig. 2b); these features are due to uncontrolled deviations of the substrate surface from the  $(111)_A$  crystallographic plane, which arise during the preepitaxial preparation ( $\pm 30'$ ). Statistical processing of the photograph indicates that the polymolecular growth steps (height 1-3 nm) on the vertical planes are distributed unevenly and that the nature of the distribution (Fig. 3a) and the average distance  $\lambda$  (Fig. 3b) between steps depend on the angle of deviation. Growth centers are observed between steps when the distances between the steps are considerable; this is typical for small  $\varphi \leq 2^\circ$  (Fig. 2c).

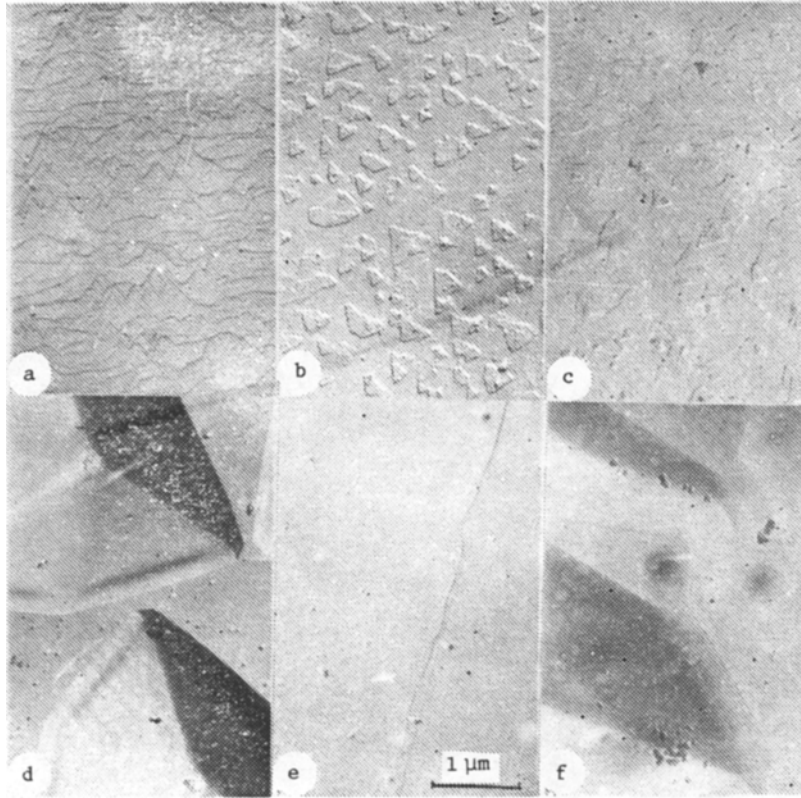


Fig. 2. Electron photomicrograph of the surface of epitaxial GaAs layers in the vicinity of the  $\langle 111 \rangle_A$  band for two values of  $p\text{AsCl}_3$  (in Pa): a-c) 10, d-f) 400. Substrate orientation: a, d)  $2^\circ (111)_A(110)$ ; b, e)  $(111)_A$ ; c, f)  $2^\circ (111)_A(001)$ .

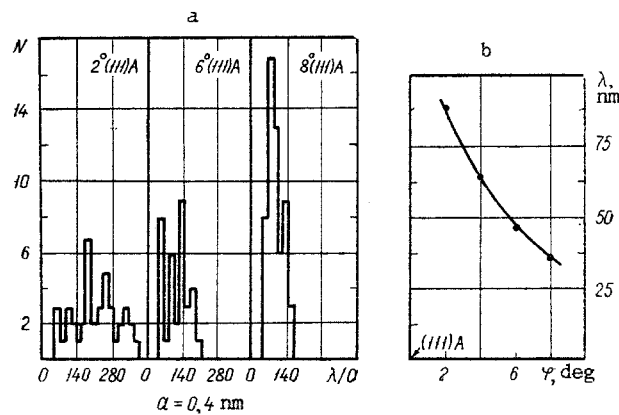


Fig. 3. Histograms of the distribution of experimentally observed steps with respect to distance between them for GaAs vicinal faces (a) and the dependence of the average distances between steps on the angle of deviation (b) for  $p\text{AsCl}_3 = 10$  Pa.

We should note that such centers arise only when the deviations are toward the (001) plane. Deviations toward (110) are characterized by the formation of echelons of polymolecular steps with a large number of macrokinks (Fig. 2a). The average size of the terraces coincides, to within the measuring error, with the average size for deviations toward the (001) plane.

The microrelief of the surface of epitaxial GaAs layers, grown at high  $p\text{AsCl}_3$ , depends weakly on the initial crystallographic orientation of the substrate (Fig. 2d-f). The most perfect relief is observed on the singular  $(111)_A$  face, such that with deviation from it the

morphological defect structure becomes more pronounced because of the formation of micron-sized growth pits, whose density is different in different parts of the sample. The variation of the angle of deviation from the close-packed face alters the geometry of the pits. Growth steps are observed only near surface irregularities, which allows us to assume that their height is close to the monomolecular height, 0.326 nm (the limiting resolution of the height of steps in the method we used is 1 nm [5]). The direction of misorientation does not affect the height of the steps, but the shape of the pits changes.

The structure of the surface formed during the growth of epitaxial GaAs layers in the vicinity of the <111>A band, therefore, depends essentially on the AsCl<sub>3</sub> pressure. While in the case of a high content of growth material in the vapor phase the acting steps have a height of no more than three monolayers, at low AsCl<sub>3</sub> pressures the echelon of steps is highly nonuniform with respect to both heights and distances between them. This circumstance must necessarily be taken into account in any analysis of the kinetic data.

Indeed, according to [6] the experimental dependences of the anisotropy of the growth rate and impurity trapping are usually analyzed on the assumption that steps on the growth surface are monomolecular, are distributed uniformly, and their density increases linearly with deviation from a singular face. Comparison of the calculated and experimental  $V(\varphi)$  and  $N(\varphi)$  curves enables us to estimate such characteristic parameters of the surface processes as the effective length of surface diffusion of atoms of the growth material ( $\lambda_s$ ) and the impurity ( $\lambda_{sj}$ ). When the real situation (formation of a nonequidistant echelon of poly-molecular growth steps on vicinal planes at low pAsCl<sub>3</sub>) is taken into account, the number of numerical values of  $\lambda_s$  and  $\lambda_{sj}$  estimated from the kinetic relations should increase.

For atoms (molecules) of the main material the value of  $\lambda_s$  can be estimated directly from morphological observations, with the assumption that diffusion interaction exists between steps. Then

$$\lambda_s = \lambda/2, \quad (1)$$

where the average ( $\lambda$ , Fig. 3b) or most probable ( $\lambda_{mp}$ , Fig. 3a) distance between the observed steps can be used as  $\lambda$ . The legitimacy of this approach to the evaluation of  $\lambda_s$  is confirmed indirectly by the formation of growth centers on terraces in the case of large distances between steps, when the diffusion fields of the steps do not overlap.

When the asymmetrical capture of atoms by a step, which apparently occurs during the growth of GaAs layers in the vicinity of the <111>A and [5] is taken into account, we obtain

$$\lambda_s \simeq \lambda. \quad (2)$$

In this case we obtain  $19 \leq \lambda_s \leq 37$  nm for small values of pAsCl<sub>3</sub> on the assumption that by the time of observation a steady-state surface relief has formed and that the diffusion interaction between steps occurs most completely for a deviation angle of 8°.

The decrease in the height of the acting growth steps and the distance between them\* for high values of pAsCl<sub>3</sub> is evidently due to a decrease in  $\lambda_s$ . For pAsCl<sub>3</sub> = 400 Pa we obtain  $\lambda_s \leq 8$  nm. If we take into account the known data on the composition of the adsorbed layer on the (111)A face and its variations as pAsCl<sub>3</sub> varies [7], we can assume that the decrease in  $\lambda_s$  is due to a reduction in the vacancy density in the adsorbed layer with rising inlet pressure of the arsenic trichloride. Evaluation of the variations in  $\lambda_s$ , using the expression

$$\lambda_s = a\theta_{vac}^{1/2} \exp\left(\frac{U_{ads} - U_{sd}}{2kT}\right) \quad (3)$$

and the dependence  $\theta_{vac}(pAsCl_3)$  [7], shows that the measured quantity varies more rapidly than follows from (3). We can assume, therefore, that the energy characteristics of the diffusion process also change ( $\Delta E = U_{ads} - U_{sd}$  decreases) when the density of the adsorbed layer increases.

In conclusion, we point out two consequences that flow from the joint consideration of the micromorphology of the growth surface and data on impurity trapping and the formation of optically active complexes [3, 4].

\*The initial orientation of the surface persists during the growth of the epitaxial layer.

First, a correlation exists between their formation rate and the height of an acting step: for a low step this process occurs more rapidly, although it may well be that only an increase in the steps plays a role here. This follows from a comparison of the data for two values of  $\text{pAsCl}_3$ .

Second, the rate of formation of complexes depends on the structure of the end of the step and is higher when a large number of macrokinks exists at the front of the step. This follows from consideration of the data for  $\text{pAsCl}_3 = 10 \text{ Pa}$ .

In our experiment we were unable to isolate and make a detailed analysis of the effect of an impurity on the structure of the crystallization front of epitaxial GaAs layers since in order to do this it is necessary to carry out the process with an independent impurity source, which would allow the impurity vapor pressure to be varied while the inlet pressure of the arsenic trichloride remained constant.

#### LITERATURE CITED

1. S. E. Toropov, L. P. Porokhovnichenko, and L. G. Lavrent'eva, *Izv. Vyssh. Uchebn. Zaved., Fiz.*, No. 6, 46 (1983).
2. L. G. Lavrent'eva, I. V. Ivonin, and L. P. Porokhovnichenko, *Izv. Vyssh. Uchebn. Zaved., Fiz.*, No. 12, 24 (1977).
3. S. E. Toropov, L. G. Lavrent'eva, et al., *Izv. Vyssh. Uchebn. Zaved., Fiz.*, No. 2, 92 (1985).
4. I. A. Bobovnikova, L. G. Lavrent'eva, and S. E. Toropov, *Izv. Vyssh. Uchebn. Zaved., Fiz.*, No. 2, 96 (1985).
5. I. V. Ivonin, Author's Abstract of Candidate's Dissertation, Kharkov Polytechnic Institute, Kharkov (1978).
6. L. G. Lavrent'eva, *Izv. Vyssh. Uchebn. Zaved. Fiz.*, No. 1, 23 (1980); No. 10, 31 (1983).
7. A. A. Chernov and M. P. Ruzaikin, *J. Cryst. Growth*, 45, 73 (1978).