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Application of retrospective analysis of production control databases for quality control of GaAs single crystals

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A retrospective analysis of a production control database for growing tellurium-doped GaAs single crystals using the Czochralski method was conducted. The asymmetry and Smirnov criteria were used to evaluate the normal distribution of material and process parameters. The strength of the relationship between material parameters and process parameters was assessed using the gradation of the paired correlation coefficient ($|r_{xy}| \in 0.013\text{--}0.420$). A significant discrepancy was observed between the actual and predicted results within the regression model (the correlation coefficient R varied in the range of 0.23–0.47). Using cognitive graphics techniques, it was shown that convective mixing of the melt with a flat crystallization front is necessary to achieve high free carrier mobility at a low density of structural defects ($N_d < 1.8 \times 10^4 \text{ cm}^{-2}$).

Keywords: Czochralski method; III–V; GaAs; retrospective analysis of industrial control databases; cognitive graphics techniques; statistics.

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Introduction

Monocrystalline gallium arsenide (GaAs) is one of the most promising and demanded semiconductor materials [1]. In addition to demand for semiconducting single crystals, the development of electronics also justifies the growing requirements for their quality.

Earlier, using the example of InSb single crystals alloyed with Te, it was shown [2] that it is possible to optimize the complex of its electrophysical and structural properties by selecting an effective mode of growing single crystals. Digital instruments for monitoring single crystal growth and intermediate control of its structural and electrophysical parameters allow to obtain a representative amount of data which determines the effectiveness of the application of

post-event analysis instruments to identify critical technology factors determining the quality of products.

The post-event analysis is a passive experiment in the K -dimensional space of the production process parameters (ξ_k) or, in other words, end-to-end analysis of the technology – from source material to final product [3]. Effectiveness of such an approach competes with active experiment (search for material quality dependence γ on ξ_k within the allowed tolerance range $[\xi_k^{\min}; \xi_k^{\max}]$ based on the response $\delta\gamma_i$, however, different order of material costs determines the interest in “data mining” [4].

The purpose of this work was to evaluate the possibility of post -event analysis applied to the technology of growing GaAs single crystals

using the Czochralski method with liquid encapsulation of melt based on post-event analysis of production control databases to search for critical factors of the technology.

Object of the Study

The object of research in this work was the production control of a database of in-house technology for growing GaAs single crystals using the Czochralski method with liquid encapsulation of melt (LEC) in the crystallographic direction [100]. The database was a matrix the number of rows in which corresponded to the number of grown single crystals (168 pcs.) and the number of columns corresponded to the number of recorded process parameters (V_f , V_c – movement speed of the crystal and crucible; W_f , W_c – rotation speed of crystal and crucible with melt; T_{mh} , I_{bh} – main heater temperature and current on the background) and material (density of dislocations and free-carrier mobility – N_{dd} and μ).

Quality certification was carried out according to the adopted regulation on plates cut orthogonally to the direction of growth from the beginning and end of the single crystal [5]. The gradient value of electrophysical parameters (concentration of charge free carriers was evaluated by optical method – by reflection spectra in the far and medium infrared region [6].

The procedure of post-event analysis included the building of correlative models, regression models, histograms, which capacity (k) was determined by the minimum RMS deviation of the found distribution from the actual one which is achieved at $k \approx N^{1/3}$, where N is the volume of experimental sample [7]. The type of distribution of parameters of technology and product was estimated by the values of asymmetry coefficients A_s and excess E_s in the normal distribution type – according to the Smirnov goodness-of-fit-test, the degree of relationship between two variables γ_i and ξ_k based on the matching correlation coefficient $|r_{xy}|$ – according to Chaddock scale [8].

Results and their discussion

Correlation analysis showed that in the “technological factors – quality parameters” pair

the relationship closeness is weak ($|r_{xy}| < 0.3$) excluding the influence of V_c , T_{mh} , W_c on the dislocation density at the beginning (0.37, 0.41, 0.42, respectively) and I_{bh} (–0.32) on the mobility of free charge carriers at the end of the single crystal, corresponded to a moderate bonding strength. Such results can be a consequence of the combined influence of temperature and dynamic parameters on the quality and rejection level, which is usually described by the equation of linear regression $\gamma_i(\xi_k)$ [9]. The forecast of density dislocation (N_d) and mobility of free charge carriers (μ) relative to the temperature and dynamic parameters of GaAs single crystal growing process – W_c , V_c , W_f , V_f , T_{mh} , I_{bh} within regression models revealed a high data of actual and expected discrepancy results – correlation coefficient R varied in the range of 0.23–0.47.

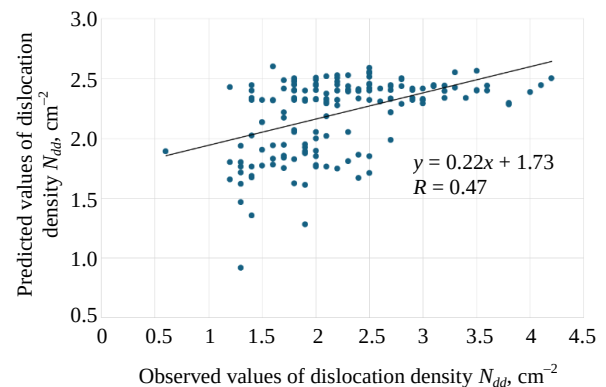


Fig. 1. Correspondence of observed and predicted values of dislocation density

One of the conditions for performing regression is the correspondence of distribution type of control parameters to the normal law. However, in our case the distribution of values W_f and T_{mh} are bimodal and have two expressed modes in the ranges of 988–1078; 1257–1347 °C and 11.6–12.6; 14.8–15.8 rpm (fig. 2). The distribution of values V_f , V_c is characterized by left-hand asymmetry A_s (–5.4 and –2.86) with its sharp spike ($E_s = 46.99$ and 15.62), while for values W_c , I_{bh} – right-hand (1.26 and 0.87) and E_s excess coefficient value (1.11 and 0.87), respectively. A similar situation is observed when analyzing the distribution of GaAs single crystal quality indicators: values of asymmetry and excess coefficients for μ and N_d were 1.31–2.06 and 0.39–0.70; 11.67–14.26 and –0.33–0.34, respectively. And if the Gaussian hypothesis not rejected for quality characteristics for N_{dk}

(at $A_s = 0.39 < A_{scr}$ with risk $\alpha < 0.01$ [9]), this primarily reflects the symmetry of the values

distribution but not always correspondence to normal law.

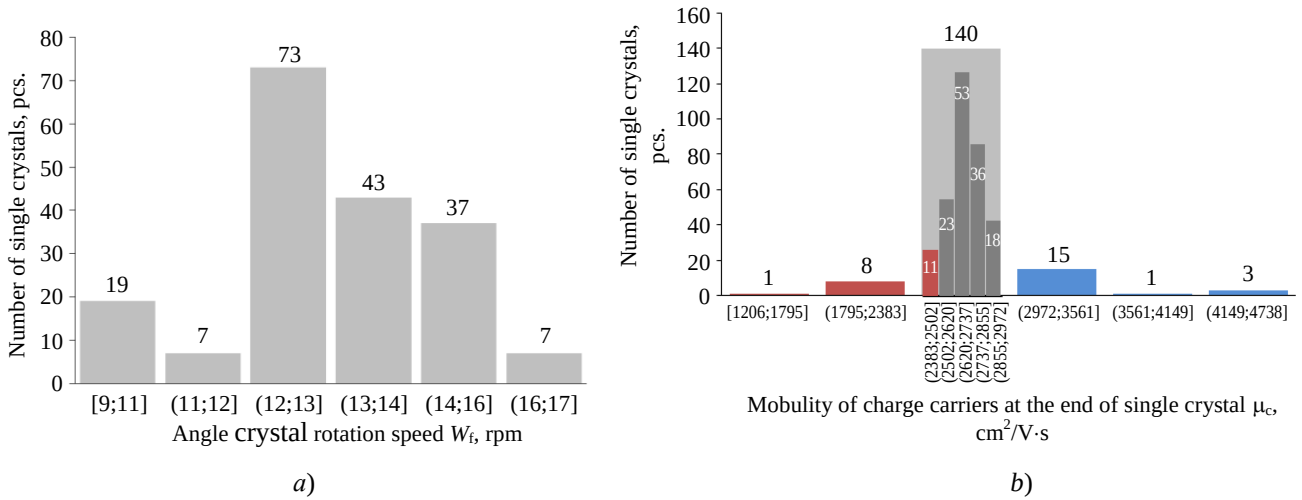


Fig. 2. Histograms of the distribution of the crucible rotation velocity (a), as well as the mobility of free charge carriers (b) at the end of GaAs single crystal

In our case the application of Smirnov non-parametric test showed that the distribution of dislocation density values at the end of GaAs single crystals is subject to normal law— $t_{exp} = 0.1$ at $t_{table} = 1.36$ $\alpha = 0.05$. In this regard, it can be useful to determine the influence of process parameters on the products quality estimated by the totality of values of its characteristics at the upper and lower limits of their distribution (tails of the corresponding distribution histograms (fig. 2b) [3].

The assessment of the combined impact of process parameters on the limit values of material quality characteristics was carried out based on the search and separation of “dense” clouds of points when displaying the dependence $\gamma_i(\xi_k)$ on different planes $\xi_k-\xi_m$. When selecting an optimal plane $\xi_k-\xi_m$, the existing physical patterns were considered. In particular, the reducing of the non-homogeneity of the thermal filed (reduction of axial $\nabla T_a \downarrow$ and radial $\nabla T_r \downarrow$ temperature gradient) – alignment of crystallization front is required to reduce the stress level and improve the dislocation structure of GaAs. An increase of the rotation speed of the crystal W_f and decrease of the rotation speed of the crucible W_c [10] also results in a flatter crystallization front and distinct cutting of the single crystal which confirms the display of

dependence $\gamma_i(\xi_k)$ on the plane W_f-W_c (fig. 3).

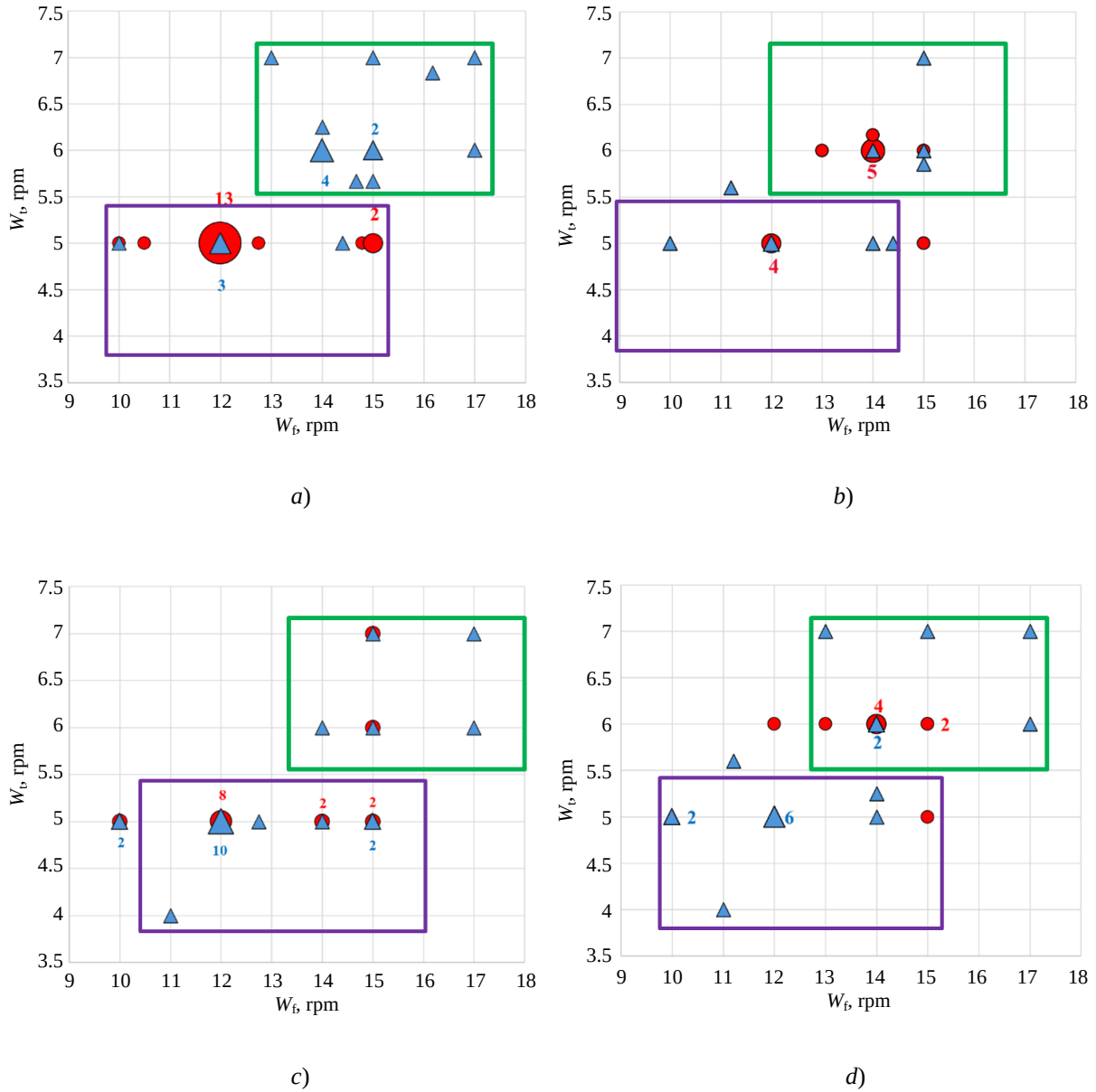
It was established that in the range of rotation speeds $W_f = 13-17$ and $W_c = 5.5-7$ rpm, it possible to obtain a low level of density dislocations at the beginning of the single crystal with almost zero probability of obtaining a high density of defects of the crystal lattice ($N_{dc} > 3.0 \text{ cm}^{-2}$). However, it was possible to obtain a low density of dislocations at the end of a single crystal ($N_{dc} \leq 1.8 \text{ cm}^{-2}$) with a risk of $\alpha = 0.29$, only at a maximum crystal rotation velocity $W_f = 17$ rpm which also provided high mobility of free charge carriers at the end of the single crystal.

It was established that at $W_f = 13$ rpm and $W_c = 5$ rpm at the end of single crystal with risk of $\alpha = 0.25$ channel inhomogeneity is formed (facet effect), expressed as separate segments at its periphery. Area enriched with tellurium due to the active capture of its atoms by planes $\{111\}$ was not significant on the scale of the plate and is $\sim 4 \text{ mm}^2$ with a range of FCC values $\sim 0.7 \times 10^{18} \text{ cm}^{-3}$ which however can reduce the material's resistance to brittle structure – reduction of fracture toughness (K_c value) [11].

For those processes where the identified patterns were not observed, a consistent species-by-species analysis of the technology trajectories

which in some cases allowed us to identify the reasons for this. On this basis, a flow chart of scenarios for the passing of the technological

process of growing GaAs was formed with a risk assessment of obtaining products with an acceptable quality (fig. 4).



▲ – $N_{dd} < 1.4 \times 10^4 \text{ cm}^{-2}$; $N_{dc} < 1.8 \times 10^4 \text{ cm}^{-2}$; ● – $N_{dd} > 3.0 \times 10^4 \text{ cm}^{-2}$; $N_{dc} > 6.2 \times 10^4 \text{ cm}^{-2}$; ▲ – $\mu_n > 3437 \text{ cm}^2/\text{V}\cdot\text{s}$; $\mu_c > 2972 \text{ cm}^2/\text{V}\cdot\text{s}$; ● – $\mu_n < 2992 \text{ cm}^2/\text{V}\cdot\text{s}$; $\mu_c < 2502 \text{ cm}^2/\text{V}\cdot\text{s}$

Fig. 3. The combined influence of the crucible rotation speed W_c with melt and crystal W_f on the dislocation density (N_d) and mobility of free charge carriers (μ) in the beginning (a, b) and end (c, d) of the GaAs single crystal, respectively

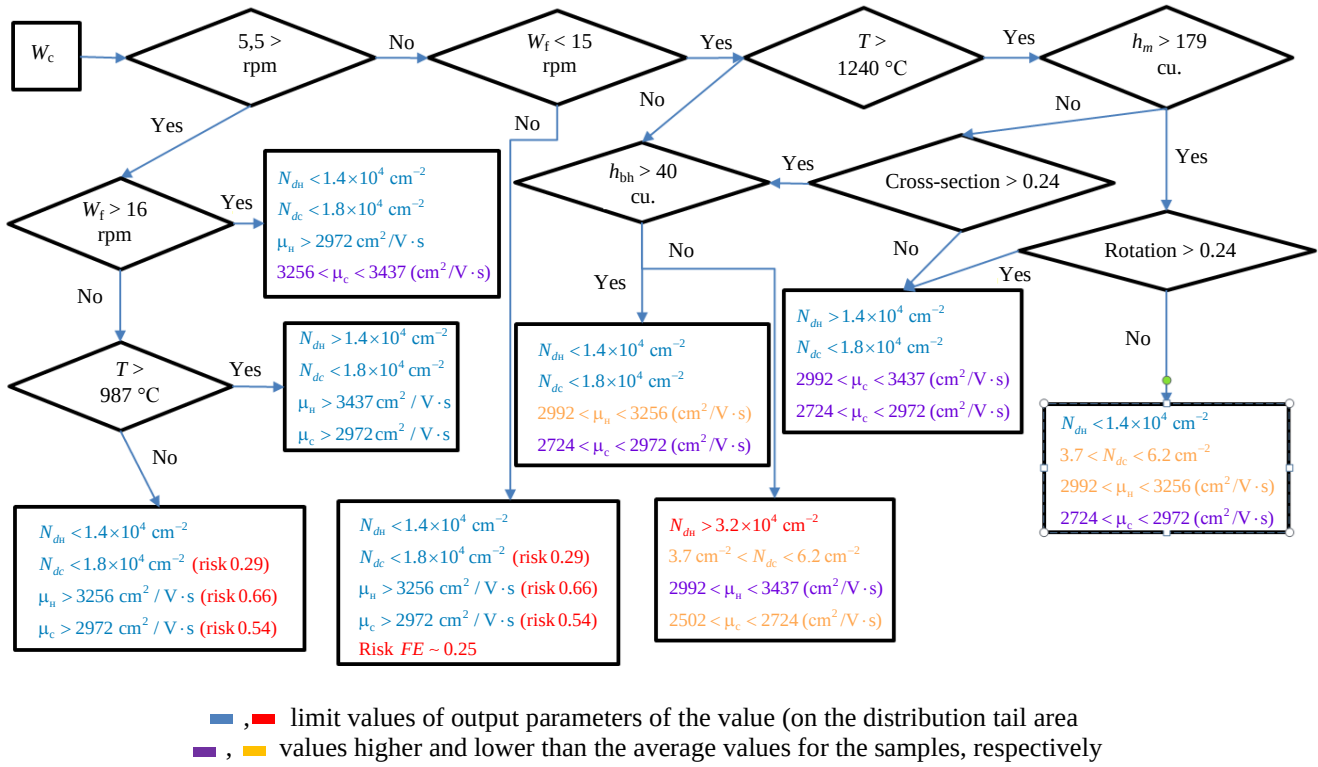


Fig. 4. A fragment of flow chart for the GaAs growing process

The presence of such flow chart allows to optimize the passage of technological process – limiting, in particular, the ranges of changes of certain technological parameters. Thus, for example, a decrease in the rotation speed of crucible with melt lower than 5 rpm naturally (due to a decrease in its convective mixing) resulted in the increased number of structural imperfections and decreased mobility of free charge carriers. It turned out to be possible to compensate for such effect of such disturbance on free carriers μ_h by setting the following temperatures and dynamic conditions – $T_{mg} > 1000$ °C, $I_{bh} > 90$ A, $W_f > 14$ rpm, and on μ_c N_{dc} at crystal rotation velocity < 15 rpm. In turn, the effect associated with an increase of dislocation density N_{dd} at decreased crystal rotation velocity ($W_f < 17$ rpm) was neutralized by a change in the value of the T_{mh}/I_{bh} ratio from 10.69 to 10.58 °S/A, for example due to a current increase at the background heater, which is likely due to a change in the axial radial gradient at the crystallization front [12].

It was also established that it is possible to ensure the limit quality level of GaAs single crystals ($N_{dh} < 1.4 \times 10^4$ cm⁻²; $N_{dc} < 1.8 \times 10^4$ cm⁻²; $\mu_h > 3437$ cm²/V·s; $\mu_c > 2972$ cm²/V·s) with the following combination of growing process

parameters: $W_c > 6$ rpm, $W_f < 16$ rpm, $T_{mh} > 987$, $I_{bh} > 90$ A.

Conclusions

1. It was established that the distribution of the values of the parameters of GaAs single crystals growing technology using the Czochralski method and its product is not subject to normal law except for the distribution of dislocation density values at the end of GaAs single crystal – experimental values of the Smirnov criterion $t_{exp} = 0.1$ at $t_{table} = 1.36$ and risk level $\alpha = 0.05$. Asymmetry coefficient and excess of experimental samples varied in the ranges – 0.95–46.9 and –5.44–0.87, respectively.

2. Correlation analysis showed that in the “input-output parameters” system a low correlation ratio according to the Chaddock scale is observed $|r_{xy}| = 0.0125$ –0.298, except for the effect of movement V_c and rotation W_c of the crucible, as well as the temperature of main heater T_{mh} on the density of dislocation and current at the background heater I_{bh} on the mobility of free charge carriers that are characterized by a moderate correlation ratio – $|r_{xy}| = 0.37, 0.41, 0.42$ and 0.32 respectively.

3. There was a significant discrepancy between actual and expected results within the

regression model (correlation coefficient R varied within the range of 0.23–0.47). This is due to the deviation of the type of distribution of material parameter values and process from the normal law, as well as the presence of unaccounted data on the passage of technological process.

4. The effectiveness of cognitive graphics techniques was confirmed for identifying critical

technology parameters (and their risk zones within tolerance extent) which was used during drawing up a flow chart of scenarios of GaAs single crystals growing processes. Its presence is the basis for the adjustment of growing technology in order to improve the quality of GaAs single crystals.

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