Chapter 6
Complex Investigations of Sapphire Crystals Production

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The problem of optimum conditions choice for processing sapphire substrates was solved with optimization methods and with combination of analytical simulation methods, experiment and expert system technology. The experimental results and software give rather full information on features of real structure of the sapphire crystal substrates and can be effectively used for optimization of technology of the substrate preparation for electronic devices.

6.1 Introduction

The sapphire crystals have a complex of the certain physical properties so it can be widely used in microelectronics, quantum electronics, optics of high accuracy, nanotechnology. The requirements to sapphire structure quality grow with development of technologies. It is known that sapphire quality is determined by a set of criteria, and first of all it is the growth conditions. This concept also includes a set of others, such as temperature distribution in the crystallization camera, temperature gradient and its change, the processes arising on the interphase crystal-liquid boundary and their influence on total crystal structure, crystal annealing process when cooling takes place after growth and the chemical, thermal and mechanical treatment.

In recent years the certain successes have been achieved in research and development of crystal formation mechanisms, morphological stability of crystallization front and the role of impurity, the thermal and concentrated streams and other factors in crystal structure formation. However determination of functional dependences for real crystal parameters based on conditions of crystal growth remains the main task because the problem solution of processing a crystal

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material with the set of properties depends on it. The detailed analysis of processes and environment structure from which crystals grow up is necessary for the solution of this problem. It is necessary also to develop the mathematical and information support for fabrication of products from sapphire crystals.

6.2 Growth Methods of Sapphire Crystals

There are many growth methods of sapphire crystals [1, 2]. The most promising method is the growth method from the melt by horizontal directed crystallization (HDC), allowing one to obtain large-size crystals (e.g., $350 \times 150 \times 45$ mm^3) with high growth rates if we compare them with crystallization rates from solutions or gaseous media. There are growth methods with crystallization in the crucible (Verneil's method) or crystallization without it (Kiropoulos's method, Bridgmen-Stokbarger's method, Stepanov's method, Czochralsky's method, HDC method, and zone melting method).

The crystal growth methods from the melt can be divided into two groups:

(i) the methods with small volume of the melt (Stepanov's method, Verneil's method, and zone melting method),
(ii) the methods with large volume of the melt (Kiropoulos's method, Bridgmen-Stokbarger's method, Czochralsky's method, HDC method).

The melt volume influences on crystallization character and there are many methods of sapphire crystals growth [1, 2]. The most promising method from viewpoint of intensity of the crystal physical and chemical processes in melt is the HDC method [3–5], which allows one to obtain large-size crystals with high growth rates. The melt can dissociate, and dissociation products can evaporate in the atmosphere. For these substances the time of staying into melt is limited, so the crystals are grown from the small volume of the melt. The similar conditions have to be applied for substances which interact with container material and atmosphere. It is necessary to pay attention on differences of convection conditions for the both groups of the growth methods. In the large melt volume the convective streams develop early, and the convective transfer plays a noticeable role [6, 7]. In the small volume convection cannot play such role, and the weight is transferred by diffusion.

It should be noted that Kiropoulos's and Czochralsky's methods are brought to the high technological level. But they also have some features which do not promote reduction of crystal prices. First of all there are: (i) the high cost of growth equipment, (ii) the technological difficulties during large diameter sapphire growth with crystallographic orientation (0001) which is optimal for optics and optoelectronics on the nitride-gallium structures, (iii) the necessity of expensive use of the raw materials with high degree of purity (99.996 % Al_2O_3). Many of these problems are not characteristic for HDC method. This method does not demand the complex and expensive equipment, and allows one to grow the large-size crystals of any crystallographic orientation and to use the cheap raw materials with

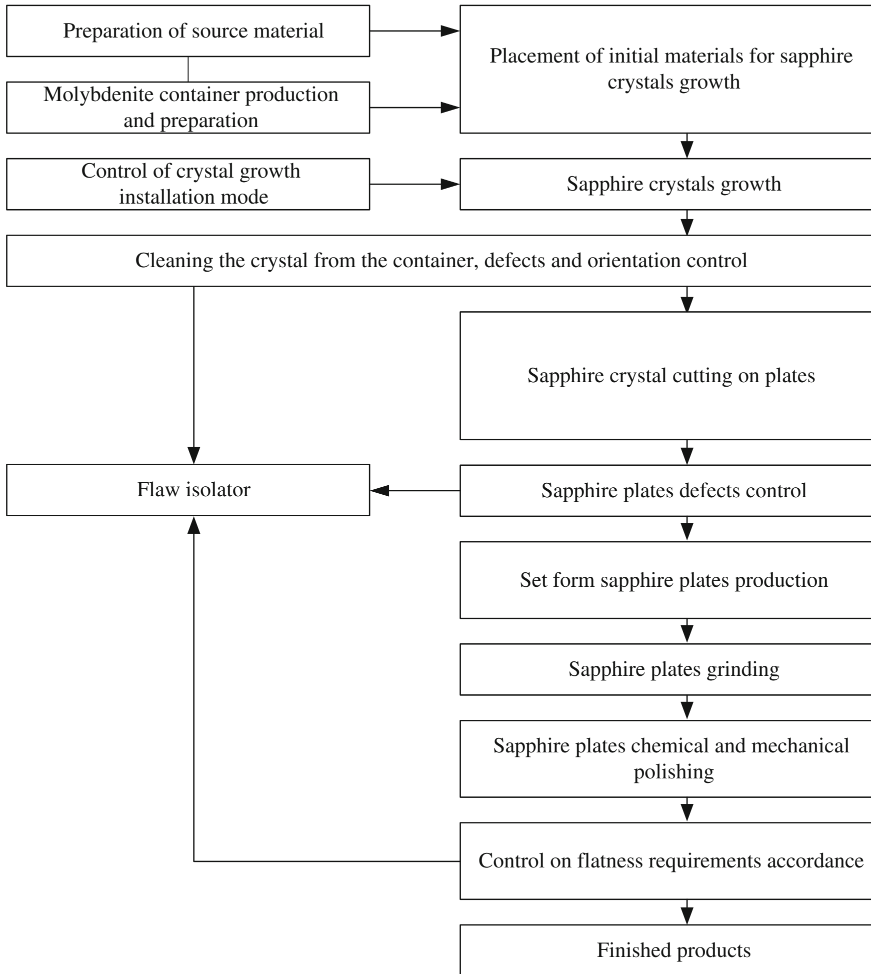

Fig. 6.1 Technological scheme of sapphire crystal production by HDC method

higher impurity concentration because of their effective evaporation in the melt crystallization process. It has allowed one to increase the crystal sizes and to expand their application. The common scheme of Al_2O_3 substrate production by HDC method [8] is presented in Fig. 6.1. It includes crystals growth and substrate production.

The HDC method has some advantages in comparison with other methods. First of all consists in that the melt height and its square are constant during a whole process (see Fig. 6.2). The large melt volume allows one to provide the effective impurities evaporation. The fuse process and boundary of phase division can be controlled visually or by means of optical devices. It creates the grounds for automation process [9, 10].

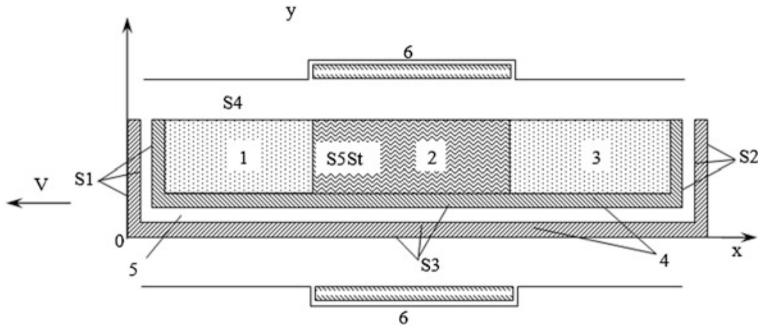

Fig. 6.2 The model for temperature distribution calculation in the system crystal-melt: *1* crystal, *2* melt, *3* powder, *4* molybdenite container and pallet, *5* gap between pallet and crucible, *6* heater, *S1*, *S2*, *S3*, *S4* emitting surfaces, *S5St*—crystallization boundary

6.3 Simulation of Heat Transfer Processes During the Sapphire Crystal Growth by Horizontal Directed Crystallization Method

In real conditions of sapphire crystal growth by HDC method the temperature of fuse decreases that defines the temperature gradient in growth crystal. The simulation of heat transfer processes in the crystal-melt system was carried out for definition of influence of the sapphire growth parameters on the crystal quality [8].

The pallet and the crucible with powder (see Fig. 6.2) move relatively to the heater with the speed of 6–10 mm/h. Therefore it is possible to consider the all process as occurring in the quasi-steady state.

Two stationary positions of crucible with material relatively to the heater were considered: (i) the crystal, melt and powder be in crucible, (ii) the finish of the technological process took place, when the crystal and melt were in the crucible (see Fig. 6.2). The process occurred in the vacuum, so the heat transfer between free surfaces is caused by radiation [8].

The problem of temperature distribution determination in the system "crystal-melt-powder" consists of equations set solution [11] including the thermal conductivity equation:

$$-\nabla k \cdot \nabla T = q, \tag{6.1}$$

with the boundary condition for S5St [12]:

$$-\lambda_{\kappa p} \frac{\partial T_{kp}}{\partial x}\bigg|_{x=0} = \rho_{\kappa p} \cdot L \cdot V - \lambda_{p} \frac{\partial T_{p}}{\partial x}\bigg|_{x=0}, \tag{6.2}$$

and the heat balance equation, written in accordance with the Stefan-Boltzmann law for radiation surfaces S_1,S_2, S_3, S_4 [11]:

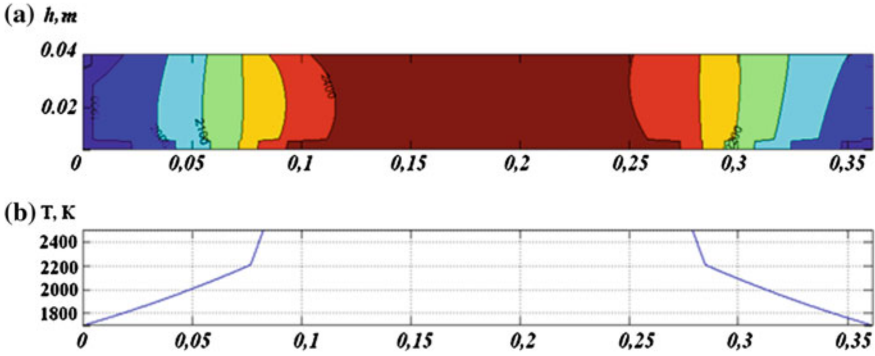

Fig. 6.3 The crystal, melt and powder disposed in the container: **a** The temperature distribution in the system "crystal-melt-powder", **b** The heater temperature

$$-k\frac{\partial T}{\partial n} = \sigma \cdot \beta \cdot (T^4 - T_0^4), \tag{6.3}$$

where k is the material heat conductivity, T is the temperature in the system "crystal-melt-crystal powder", q is the heat release on the boundary "melt-crystal", ρ is the material density, L is the hide crystallization heat, σ is the Stephan-Boltzmann constant, β is the coefficient of reflection.

The heat balance equation at the boundary S5St, written in accordance with the Stefan-Boltzmann law for radiation surfaces and Lambert law for an absorbing medium has the form [11]:

$$q_\psi = E_0 \int \cos\psi \cdot \exp(-\alpha \cdot r(\psi)) \cdot d\omega, \tag{6.4}$$

where q_ψ is the heat stream density due to radiation, $E_0 = \beta \cdot \sigma \cdot (T^4 - T_0^4)$ is the stream density emitted by the melt in crystal, $d\omega$ is the angle under which from the point of emitting it can be visible the elementary crystal surface area, ψ is the angle between the normal to the surface and the emission direction, α is the medium absorbance, r is the distance between the radiation point and the crystal surface.

The system of differential equations (6.1)–(6.4) is solved by the numerical method based on the finite differences method [13]. The two-dimensional numerical model was developed. It includes the translucent crystal area and the opaque melt area, separated by a diffuse boundary [8].

This model allowed us to conduct the calculation for estimation of effects of the thermo-physical material properties and external temperature conditions on the crystallization process in rectangular system "crystal-melt-powder" in the quasi-stationary approximation.

The results of temperature field calculation in the system "crystal-liquid–powder" are presented in Fig. 6.3. The results indicate that the temperature in the container

has the non-linear distribution. The heat streams in the upper part of the crystal are substantially different from the thermal streams in the bottom part. The pallet-vacuum gap-container play a role of screen and change the form of crystallization front making it inclined to the growth direction (see Fig. 6.3).

The crystallization front changes the form with the crystal length increase. It becomes convex in the melt. This fact embarrasses the bubbles emersion on the melt surface so the bubbles accumulate at the bottom part of container. The simulation results showed that the small sapphire absorbance in the solid phase, α, was the explanation for this solidification front distortion.

The model allows us to calculate the temperature distribution in the system "melt-crystal" for sapphire crystallization by the method of horizontal directed crystallization. It takes into the account the radiation from crystal surface and diffusive front boundary.

Thus the results showed that the sapphire crystal transparency in the solid phase for thermal radiation made the radiating component prevailing in the thermal stream from front along the crystal. For this reason the crystal growth rate enlarges with crystal length increase and the crystallization front form changes also. These factors are the principal causes of defects formation in crystals.

It is necessary to calculate the temperature distribution in the growth equipment of sapphire crystals by the HDC method in three-dimensional case for more exact defect diagnostics. The temperature distribution determination in the system "crystal-melt-crystal powder" includes solution of the heat conductivity equations [14, 15]:

$$\begin{gathered}\frac{\partial T_i(x,y,z,\tau)}{\partial\tau}=\left(\frac{\partial}{\partial x}a_i\frac{\partial T_i(x,y,z,\tau)}{\partial x}+\frac{\partial}{\partial y}a_i\frac{\partial T_i(x,y,z,\tau)}{\partial y}+\frac{\partial}{\partial z}a_i\frac{\partial T_i(x,y,z,\tau)}{\partial z}\right)\\ -W\frac{\partial T_i(x,y,z,\tau)}{\partial x},\\ 0<x<x_L,\ 0<y<y_L,\ 0<z<z_L,\end{gathered}\tag{6.5}$$

here $i=1,2,3$ is the crystal, melt and crystal powder, respectively, a_i is the heat diffusivity coefficients ($a_i=\frac{\lambda_i}{\rho_i\cdot C_i}$, where λ_i is the thermal conductivity coefficient; ρ_i is the material density; C_i is the specific heat); W is the container movement speed.

The container movement speed is small enough, so we can conclude that the growth process is quasi-steady and the temperature distribution can be found by the following formula [11]:

$$\operatorname{div}(\lambda_i \operatorname{grad} T_i(x,y,z))=0. \tag{6.6}$$

The boundary conditions for system of (6.6), reflecting continuity of thermal fields and streams on bounds of media section, can be found by using the following expressions:

Fig. 6.4 Temperature fields in the system "crystal-liquid-powder"

$$\lambda_1 \frac{\partial T_1(x_T, y, z)}{\partial x} = \lambda_2 \frac{\partial T_2(x_T, y, z)}{\partial x}, \quad (6.7)$$

$$\lambda_2 \frac{\partial T_2(x_T + \Delta x, y, z)}{\partial x} = \lambda_3 \frac{\partial T_3(x_T + \Delta x, y, z)}{\partial x}, \quad (6.8)$$

$$q_{s_1} = q_{s_2} = q_{s_3} = \sigma\beta(T^4 - T_{hot}^4), \quad (6.9)$$

where σ is the Stephan-Boltzmann constant, β is the radiation coefficient, T_{hot} is the function with the heat temperature distribution.

The final volume method on the unstructured grid was used for simulation. The discrete analog of heat conductivity equation for the final volume (tetrahedron) is:

$$\sum_j \frac{\lambda(T_j - T_i)A_{ij}}{(x_j - x_i)n_{jx} + (y_j - y_i)n_{jy} + (z_j - z_i)n_{jz}} = 0, \quad (6.10)$$

where volume i is the volume for which the heat conductivity equation is solved, volume j is the volume next for volume i, A_{ij} is the area of common face for i and j volumes, $\overrightarrow{n_j}(n_{jx}, n_{jy}, n_{jz})$ is the normal to the next i and j tetrahedrons, $\overrightarrow{l_j}(x_j - x_i, y_j - y_i, z_j - z_i)$ is the direction along which the thermal stream is defined.

The results of temperature calculation in the system "crystal-liquid-powder" are presented in Fig. 6.4. The temperature fields in the system "crystal-liquid-powder" are showed in Fig. 6.4a. The temperature fields in vertical cross-section of the crystal are presented in Fig. 6.4b.

The calculation results showed that sapphire crystals transparency in the crystal phase and the opacity in the melt influenced directly on the radiation streams in the system. Considering their large influence on the common heat exchange, this factor affects also on the temperature field and the crystallization front location. The temperature fields influence on the radiation heat transfer intensity. For this influence estimation, the calculations with temperature increase on 200–300 K in the system "crystal-liquid-powder" were carried out. The results showed that the

melt width increased and as result the crystallization front location changed. So we can conclude that we have possibility to control the crystallization front location owing to heat temperature increase.

In parallel with consideration of temperature fields, the analysis of mechanical stresses in the crystal was carried out. The calculations of the heater construction parameters should be fulfilment on the base of the strain, displacement and stress fields in the crystal. It means that mathematical simulation and calculations should be carried out for all equations which are included in system, namely: the equations of heat conductivity and thermal elasticity. So these equations [15–17] can be written as

$$\begin{cases} \mu\Delta u + (\lambda+\mu)\dfrac{\partial\delta}{\partial x} = -\dfrac{\partial(\alpha T)}{\partial x} - F_x, \\ \mu\Delta v + (\lambda+\mu)\dfrac{\partial\delta}{\partial y} = -\dfrac{\partial(\alpha T)}{\partial y} - F_y, \\ \mu\Delta w + (\lambda+\mu)\dfrac{\partial\delta}{\partial z} = \dfrac{\partial(\alpha T)}{\partial z} - F_z, \\ \dfrac{\partial}{\partial x}(a_i\dfrac{\partial T}{\partial x}) + \dfrac{\partial}{\partial y}(a_i\dfrac{\partial T}{\partial y}) + \dfrac{\partial}{\partial z}(a_i\dfrac{\partial T}{\partial z}) = 0, \end{cases} \tag{6.11}$$

where F_x, F_y, F_z are the components of volume sources; u, v, w are the components of displacements; $\phi = \{u, v, w\}$; α is the coefficient of volume heat expansion; λ and μ are the Lamé coefficients; Δ is the Laplace operator; $i = 1, 2, 3$ is the crystal, melt and crystal powder, respectively; a_i is the thermal diffusivity coefficients.

The boundary conditions for the system (6.11) are written in the form of relations [15].

The model allows us to make the numerical experiments and to study the influence of input parameters on the crystals quality. This approach allows one to improve the crystal growth process and to receive the crystals with reduced defects level.

6.4 Research of Stages of the Sapphire Crystal Treatment

Reviews [14, 18–20] showed that for the sapphire surface, corresponding to the substrate requirements for heteroepitaxy, it is necessary to carry out processing in several stages with the abrasive size decrease. In this case the fixed abrasive tool is applied on the initial treatment stages, and then the free abrasive with different granularity is used. The transitions number and the abrasive size are defined experimentally. The research of different tools and influence of treatment methods on sapphire surface was carried out for development of treatment method of the sapphire surface. After cutting the sapphire crystals, the plates were treated sequentially by three diamond grinding tools which allowed making the roughing

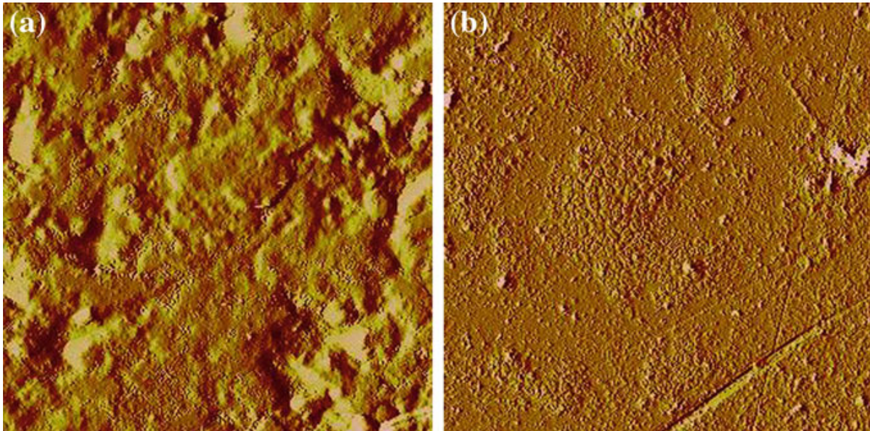

Fig. 6.5 Photomicrographs of sapphire surface after treatment with diamond powder ACM 28/20 and ACM 1/0

and finishing surface treatment. The problems of sapphire substrates processing such as oriented crystal cutting on the plates, substrates grinding, and finishing chemical and mechanical wafers polishing are considered. The two-sided grinding technology was investigated for the stage of mechanical treatment. After study of plates grinding by the free abrasive it was found [8] that these plates had the smooth matted surface without processing line traces. The free abrasive grinding provides the better surface quality. The roughness of surfaces is reduced, and their texture and purity are improved with decreasing diamond powder grain size in the tool or abrasive suspension (deionized water—66.7 %, glycerol—11.11 %) after the transition from hard grinding tools to the elastic and soft polishing tools in the final stages of processing (see Fig. 6.5).

The possibility to use the dependences of crystal microfracture on the singular abrasive grain for estimation of processing mass abrasive grains was investigated in [14]. In the real conditions, when the large amount of abrasive grains influence on the crystal, the final surface dislocation structure is defined by the dynamic balance between the crystal grinding rates and speed of new dislocations formation. On the basis of relation between the plasticity zone and depth of grain implantation into the crystal, it should be noticed that hardness increase of the abrasive and grinding tools will enlarge the layer with high dislocation density.

The dependences of material grinding tool and the abrasive radius on the depth of the surface damaged layer and the depth of formed lateral cracks in sapphire are presented in Fig. 6.6.

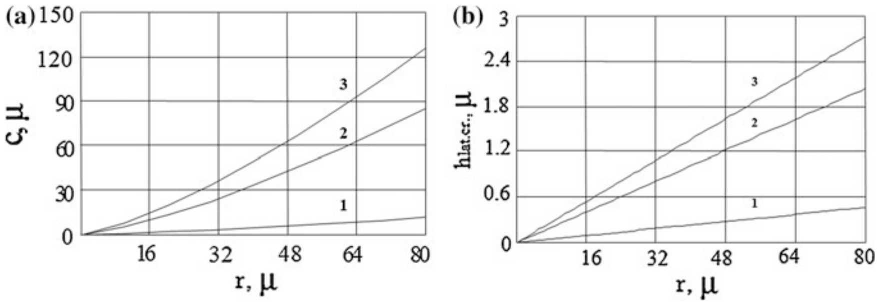

Fig. 6.6 The dependences of material grinding tool (*1*—brass, *2*—cast iron, *3*—glass) and the abrasive radius on the depth of the surface damaged layer (**a**), and the depth of formed lateral cracks in sapphire (**b**)

6.5 Method of Development of the Mathematical and Information Support for Sapphire Crystals Processing

Now, there is the intensive development of information technologies. It gives the unlimited possibilities to use computer possibilities in the crystal production. The complexity of use these methods is defined by incompleteness of mathematical description of the technological models for crystallization processes and absence of information support, which allow optimization of the technology [21].

For successful use of the information technology in the crystal production, it is necessary to develop new approaches and to improve the existing methods.

The main differences of technique applied in the work are: (i) the use of the expert estimation and experiment planning methods with the purpose of model creation. It allows one to receive dependences of the output parameters (levels of defects, etc.) on basic data (growth rate, heater power, etc.); (ii) the use of specific crystal growth and treatment database allows us to consider more adequately technological features; (iii) the complex consideration of three main stages of production (crystal growth, grinding and polishing) of the sapphire substrates.

As a work result, the method of mathematical and information support for designing growth of the sapphire crystals was developed. The algorithm is presented in Fig. 6.7.

The optimization can be divided into three main parts: (i) input data for the calculation (this process is the most time-consuming), (ii) calculations of parameters and defects, (iii) technological process optimization. After receiving the data, the models of dependence of the defect level on growth parameters of the sapphire crystals are built. The final part is the construction of model for growth optimization of the sapphire crystals. The objective function (time, defect level, etc.) is determined and restrictions (temperature, defect levels, speed, etc.) are entered.

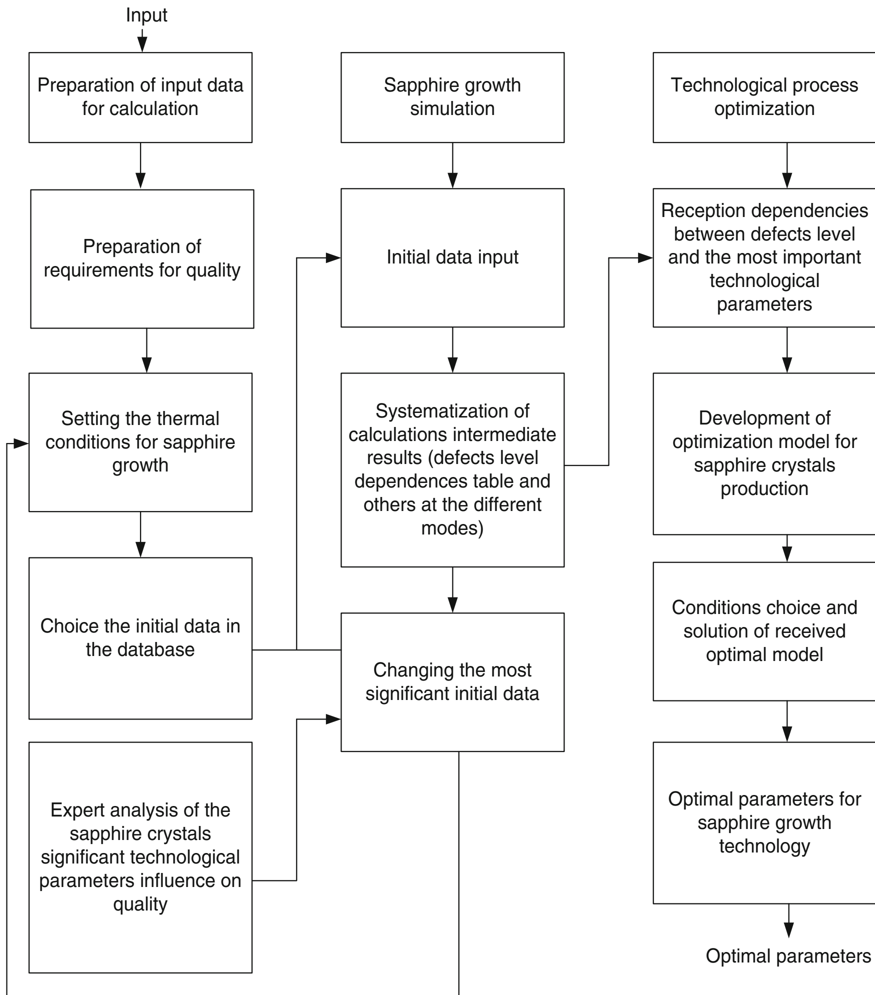

Fig. 6.7 The algorithm of mathematical and information support for the sapphire production process

The purposes of the sapphire production process and optimization criterions are based on the key process indicators, determining the production efficiency and competitiveness. These indicators are grouped in two categories: (i) quality indicators, (ii) process cost indicators.

In the modern market the sapphire production should be directed to the realization of the following objective function:

$$F(KK, P) \quad \rightarrow \quad \text{opt}, \tag{6.12}$$

where KK is the quality criteria for sapphire products, P is the product price.

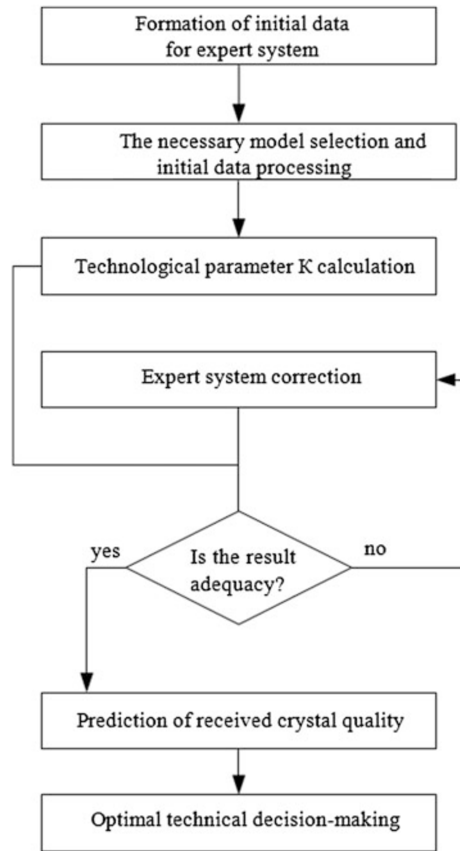

Fig. 6.8 The algorithm of the expert system for selection of decision for optimal technical solutions

The quality indicator is the multifaceted product property. The total estimation of number of quality indicators is defined by the formula:

$$y = \sum_{i=1}^{n} k_n a_n, \tag{6.13}$$

where k is the factor taking into account the relative importance of process parameters on the crystal quality ($\sum_{i=1}^{n} k_n = 1$), a is the factor taking into account importance of the processing parameters, n is the number of the considered processing parameters.

The formula for translation of the technological parameters [16]:

$$K = \frac{y - y_{\min}}{y_{\max} - y_{\min}}, \tag{6.14}$$

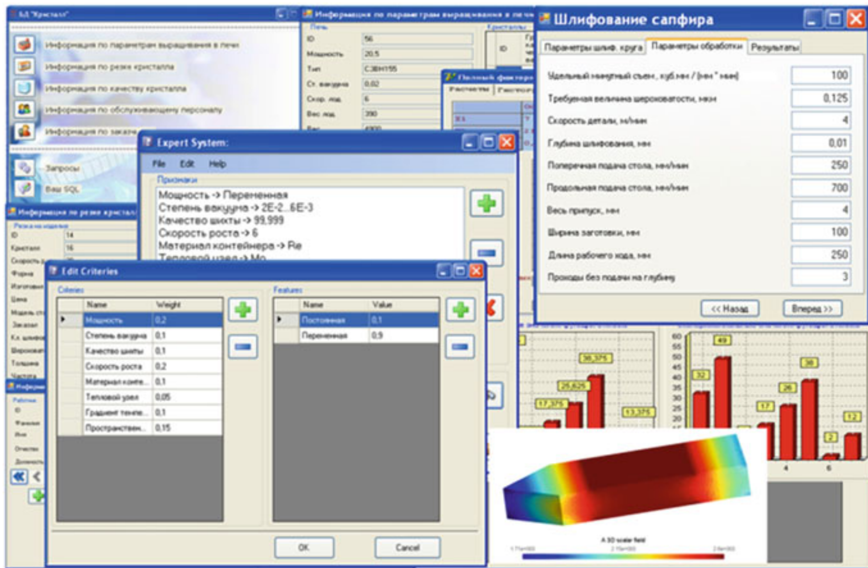

Fig. 6.9 Program interface for intellectual support during sapphire growth and treatment

where y, y_{min}, y_{max} are the current, minimum and maximum values of the processing parameters influence characteristic of the crystal quality, K changes from 0 to 1.

The range of values of the parameters and their importance were identified on the basis of experimental research [15] carried out in the equipment for the sapphire growth by HDC method. The correspondence between the real processing parameters and the parameters received by us was obtained.

These principles are used in the expert system for sapphire crystal production. The algorithm of the expert system for selection of decision on optimal solutions is presented in Fig. 6.8.

The expert system allows us to estimate the obtained crystal quality. The expert system includes database and knowledge base. It allows one to systematize the large information volumes. On the basis of the suggested models and algorithms the computer programs were developed. The interface of developed programs is presented in Fig. 6.9.

In total, the system consists of three calculating modules: (i) database of crystals and their parameters, (ii) information system allowing one to define optimum parameters of the technological process for receiving the best crystal quality, (iii) expert system allows one to make the expert analysis of an initial technological situation, to estimate the current technological situation concerning the database, to present the prediction of category of the processed crystal quality.

The system has the following purposes: (i) to give the exhausting information on crystals, (ii) to order the scattered information on crystallization processes,

(iii) to help the engineer-technologist in choice of growth parameters, (iv) to receive mathematical model for estimation of influence of the parameters on crystal quality, (v) to receive a prediction of crystal quality.

6.6 Conclusion

The complex investigations of sapphire crystals production for electronic equipment were carried out.

The favourable opportunity to control the crystallization parameters is one of the HDC method advantages. So the HDC method has the high reproducibility of growth conditions. It gives a possibility to grow the same crystals according to qualitative characteristics of serial crystallization experiments.

It was developed the model allowing one to investigate the temperature gradient influence on the position and crystallization front shape. On the basis of this model the temperature distribution in the system "melt-crystal" at different growth stages of sapphire production by the HDC method was studied. The three-dimensional numerical model for solution of the heat transfer problem in sapphire crystals allows one to develop computer programs for calculation of the temperature fields. The developed numerical model allows one to study the influence of thermal and physical material properties on sapphire crystallization.

In this work, the treatment processes of sapphire surface and investigation of influence of the different tools and treatment methods on the sapphire surface were studied.

On the basis of the suggested models and algorithms the computer programs were developed. The computer programs were based on intellectual support at decision-making for sapphire growth and treatment. It allowed us to increase the efficiency of substrate production. The experimental results and software give rather full information on features of real sapphire crystal substrate structure and can be effectively used for optimization of the substrate preparation technology for electronic devices.

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