

The influence of the materials used in the crystallizer on the rapid growth of monosectoral large crystals of the KDP type

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A method has been developed for determining the possibility of using materials in crystallizer designs for the method of rapid growth of KDP-type crystals (dihydrogen phosphate crystals). A potassium dihydrogen phosphate crystal with dimensions of $620 \times 560 \times 72$ mm has been obtained, grown in a crystallizer assembled on the basis of materials that had previously been tested according to the developed method.

Keywords: crystal, dihydrogen phosphate crystals, highly deuterated solutions, rapid growth, impurities, crystal growth kinetics.

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Introduction

Potassium dihydrogen phosphate crystals (KDP, KH_2PO_4) and its deuterated analogue (DKDP, KD_2PO_4) have been investigated for many years [1–5], but it is still a relevant task to produce them. Demand in large-size single-crystal optical elements used as laser radiation transmitters and electrooptical switches stimulated development a technology of rapid growth of the KDP-type crystals. In turn, a variety of materials inside a crystallizer installation can be another cause of appearance of optical inhomogeneities in the growing crystal. Pollution of solution by inorganic impurities as a result of active corrosion of crystallization equipment during growth of the KDP crystals results in increase of light absorption in the UV range in the crystal [6]. The impurities entering the solution usually degrade the quality of the crystal growing therein. Capturing the impurity by the growing crystal results in origin of a various kind of defects. Such kind of defects is exemplified by a vicinal-sectorial and zonal inhomogeneity, solution inclusions within the crystal volume. The impurities cause generation of macrosteps on the growing surface and deceleration of growth of separate crystal faces [7–9]. For example, impurities of three- and four-valence metals (Al^{3+} , Cr^{3+} , Fe^{3+} , Ti^{4+}) as well as of Si^{4+} reduces the prism face growth rate of the KDP crystal and almost do not affect the face growth rate of the bipyramid [10]. Presence of these impurities in the solutions causes increase on a supersaturation interval („dead zone“), in which the prism face growth rate is close to zero [11,12].

The technique given herein makes it possible to evaluate the effect of materials which are applied in crystallizer

structures and interact with the potassium dihydrogen phosphate solution, on the single-sector rapid growth of the large-size KDP crystal. The criterion of applicability of a structural material is a value of the „dead zone“ in the solutions with the studied sample and its change over time. Significance of the developed technique for evaluating suitability of the used materials in the crystallizer structures is added by studies with highly-deuterated DKDP solutions (a degree of deuteration of the samples is above 90%). Influence of even an insignificant impurity concentration results in defects in the grown crystal and heterogeneity of the crystal properties. Production of the DKDP crystal of the high optical quality is a relevant task in connection with development of powerful laser systems, whose peak power can be at the multipetawatt level. Presently, there are studies to create a multi-channel laser complex of an exawatt power level in Russia — the XCELS project (eXawatt Center for Extreme Light Studies) [13]. In particular, the project is based on open ultra-wideband synchrony of parametric amplification of radiation with the central wavelength of 910 nm in the DKDP crystal. The ultra-wideband synchrony exists in many crystals, but the DKDP crystal is almost the only one, which can be grown with an optical quality and aperture required to reach the multipetawatt power.

1. Measurement method

Kinetic curves of the solutions have been obtained by using an interference-polarization installation [5]. This installation is designed to measure the rates of growth of birefringent crystals in conditions of natural concentration convection. The installation consists of a laser, a cuvette, in which a seed crystal grows, a dividing plater and a

photodetector. Since during light transmission the beam is divided into a usual and an unusual one with the refractive indices n_o and n_e^* , respectively, the crystal outlet exhibits the phase difference $\varphi = 2\pi \frac{n_o - n_e^*}{\lambda} l$ therebetween, where l — an optical path in the crystal. In this case, variation of intensity of the transmitted beam will vary periodically with variation of the value of the optical path of the beams in the crystal l with the period $\frac{\lambda}{n_o - n_e^*}$. In accordance with the classic theory, with dislocation layer-by-layer face growth due to motion of a bunch of the similar elementary steps of the inclination p of the vicinal face in relation of the nearest singular surface, the normal face growth rate R is $p \cdot V_{st}$, where V_{st} — the step motion rate. In our case, the dependence of the face growth rate R on supersaturation σ can be approximate by the empirical expression $R = \beta \cdot \sigma^m$, where β — the kinetic coefficient of crystallization, while the value m is 1 or 2 [5]. The supersaturation σ is determined by the following expression: $\sigma = \ln\left(\frac{C_0(T_s)}{C_n(T)}\right) \cdot 100\%$,

where $C_0(T_s) = a + b \cdot T_s + c \cdot T_s^2$ — concentration of the crystallizing substance in the solution, T_s — the temperature of saturation of the solution, $C_n(T) = a + b \cdot T + c \cdot T^2$ — the equilibrium concentration of the crystallizing substance at the temperature T ; a , b , c — the empirical coefficients. For the solutions $\text{KH}_2\text{PO}_4 + \text{H}_2\text{O}$: $a = 0.123$, $b = 2.719 \cdot 10^{-3}$, $c = 1.1087 \cdot 10^{-5}$; for the solutions $\text{KH}_2\text{PO}_4 + \text{H}_3\text{PO}_4 + \text{H}_2\text{O}$: $a = 0.15$, $b = 3.249 \cdot 10^{-3}$, $c = 0$ [5]. The values of the magnitudes $C_0(T_s)$ and $C_n(T)$ are presented in units of solution concentration in grams of KH_2PO_4 per one gram of the solution. The value of T_s is determined by a start of crystal dissolution. The value of $T_d = T_s - T$ determines the temperature which corresponds to certain supersaturation σ^* . The presence of the „dead zone“ ($0 - \sigma^*$) of the solutions is a typical feature of the prism face of the KDP crystals is related to adsorption of impurities out of the solution. The structural material is considered to be suitable for manufacturing crystallizer elements in contact with the solution, if T_d does not exceed 0.6°C in all the control tests. This value of T_d is selected because it is necessary to ensure single-sector growth of the crystal, during which the sector of growth of the main face (the bipyramid face for manufacturing the optical element of the frequency converter) occupies almost the entire volume of the growth cuvette with the crystal. It is possible with a certain ratio of the growth rates of an adjacent (prism) and a main (bipyramid) face, which should be such that edges between the main and the adjacent faces are shifted towards the adjacent faces during crystal growth. This ratio is close to unity when solution supersaturation is obviously exceeds the value of the „dead zone“.

Before the tests, the sample of the structural material is subjected to preparation which includes the following operations:

- 1) degreasing (wiping the sample surface in ethanol);
- 2) abundantly flushing with deionized water;

3) leaching in the orthophosphoric acid aqueous solution of the concentration 9.8%–10% at the temperature of $T = (60 \pm 2)^\circ\text{C}$ during 8–12 h;

4) extraction and abundant flushing of the sample with deionized water.

Preparation of the studied sample was followed by making a solution out of the potassium dihydrogen phosphate salt KH_2PO_4 and deionized water H_2O ($R = 21 \text{ M}\Omega$). The concentration of the impurities Al, Ba, Cr, Mg, Fe, Na, Si, Rb, Ti in the used reagents did not exceed 3 ppm. The solution has the following parameters: $T_d \leq (0.40 \pm 0.05)^\circ\text{C}$, $T_s \approx (45.0 - 53.0) \pm 0.1^\circ\text{C}$, $\text{pH} = (4.00 \pm 0.02)$. The prepared sample of the structural material was placed in a certain volume of this solution (it depends on the sizes of the studied material, $\sim 300 \text{ ml}$). In addition to a solution sample with the studied material, there is also a check sample being prepared to accompany the test of the structural material and designed to be a reference. The reference KDP solution does not contact the tested structural material and is studied in the interference-polarization installation at least once a month. In this study the following factors were taken into account: the effect temperature T — the maximum temperature of heating of the solution with the sample; the ratio of the solution volume to the area of the surface of contact of the structural material with the V/S solution.

The solution with the sample immersed therein is placed into a drying cabinet for the entire time of the experiment at the given effect temperature. The solutions are studied in the interference-polarization installation with the following periodicity: 1–3 days, 1 week, 3–5 weeks, 7–9 weeks, 11–13 weeks, 15–17 weeks. These periods are selected based on duration of crystal growth and, respectively, the time of interaction of the structural materials with the growth solution. Thus, for example, the test period of 3–5 weeks corresponds to the time of growing of the crystal with the sizes $\sim 80 \times 80 \times 80 \text{ mm}$, so does the period of 15–17 weeks to that of the crystal with the sizes $\sim 400 \times 620 \times 75 \text{ mm}$.

The developed technique is also applicable to investigations of structural material interacting with the DKDP solution. A specific feature of work with the deuterated solution of potassium dihydrogen phosphate is that there is no interaction of the structural materials with protic water, as its presence results in reduction of percentage of solution deuteration that is obviously selected for the investigation. For this purpose, during preparing the sample for the investigations as per the developed technique, the sample of the structural material shall be thoroughly dried after being cleaned from mechanical impurities. This operation extends to all the technique equipment (retorts, covers, etc.) which later interacts with the deuterated solutions of potassium dihydrogen phosphate. Parameters of the DKDP solution for checking the materials: $T_d \leq (0.40 \pm 0.05)^\circ\text{C}$, $T_s < (44.0 \pm 0.1)^\circ\text{C}$, $\text{pH} = (4.00 \pm 0.02)$, the degree of deuteration of the solution is selected based on the requirements to the crystal deuteration degree.

2. Results and discussion

Using the method of rapid growth of profiled single-sector crystals oriented in a pre-defined way, the KDP crystal grows to the sizes $620 \times 620 \times 75$ mm during 6–9 months. During this time, the crystallizer (Fig. 1) shall be resistant to many physical-chemical processes inside this system, including corrosion, deformation, cracking, etc. Table 1 shows the structural materials which are potentially applicable for manufacturing the crystallizer parts in contact with the potassium dihydrogen phosphate solution in the rapid method of growth of the KDP-type crystals. Suitability of the materials has been studied as per the above-described technique for variation of the value of T_d over the time t . During checking of the materials, the effect temperature was $T = (60 \pm 2)^\circ\text{C}$, $V/S \sim (10.000 - 12.000) \pm 0.008$ cm; pH of the solution was $\sim (4.00 \pm 0.02)$.

Among the materials used in the crystallizer structure, a large area of contact with the growth solution is provided by the glass — borosilicate glass and window glass of the M1 grade, which presently has no alternative for replacement. A possible alternative to the window glass of the M1 grade was studied to be the glass of the M0 grade. But it did not

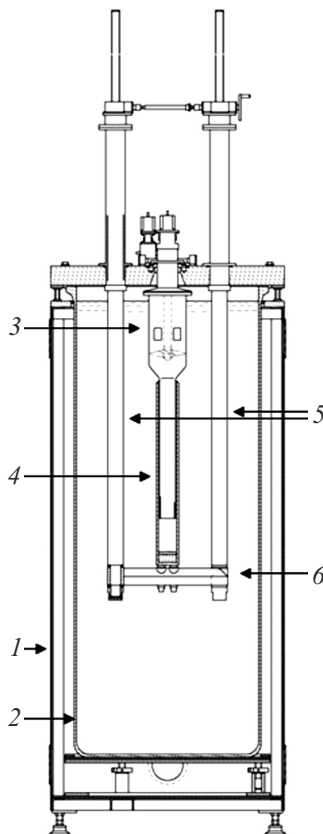


Figure 1. Crystallizer for rapid growth of the large-size single-sector crystals of the KDP type: 1 — the thermostat; 2 — the bottle; 3 — the device designed to mix the solution near the growth face; 4 — the growth cuvette; 5 — the rods; 6 — the support ring.

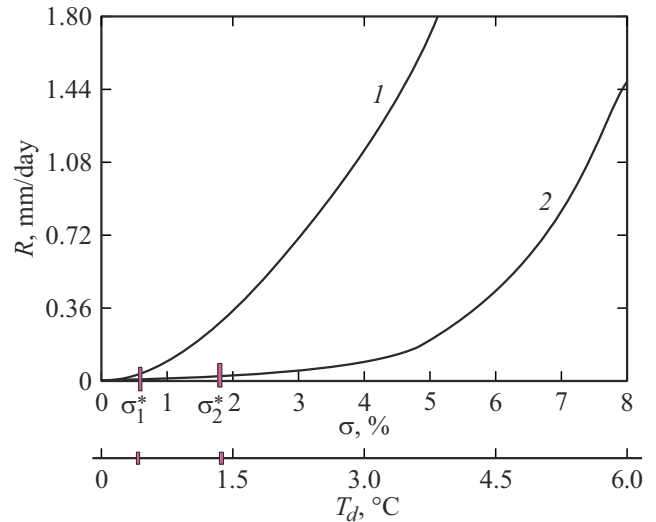


Figure 2. Dependence of the prism face growth of the KDP crystal on supersaturation in the solutions with the immersed sample made of 1 — the M1 glass and 2 — the M0 glass with $t = 28$ days.

pass the test for variation of $T_d(t)$ (Table 2). Fig. 2 shows the dependences of the prism face growth rate of the KDP crystal on supersaturation in the solutions with the samples made of the glasses of the grade M1 and M0. The values of T_d , which correspond to the supersaturations σ_1^* and σ_2^* , are marked on the additional scale of the values of $T_d(^\circ\text{C})$ to the dependences $R(\sigma)$.

In addition to no influence on the impurity composition, the listed materials of Table 1 shall be resistant to generation of cracks during the entire growth of the crystal. For example, the structural material — organic glass (polymethyl methacrylate) — withstands the temperatures to 100°C , but over time when interacting with the growth solution it is affected by cracking. The parameters of the experiment with acrylic glass: $T = (60 \pm 2)^\circ\text{C}$; $V/S \sim (10.000 - 12.000) \pm 0.008$ cm; $\text{pH} = (4.01 \pm 0.02)$. During the entire experiment, the value of T_d of the solution with acrylic glass did not exceed $(0.60 \pm 0.05)^\circ\text{C}$. That is why, in terms of pollution effect to the KDP solution, acrylic glass can be regarded as a material suitable for use in the crystallizer structures for rapid growth of the KDP-type crystals. However, the acrylic glass parts require control for origin of cracks which can be locations of formation of spontaneous crystals, which is unallowable during growth.

The work has been done for search of alternative up-to-date structural materials which would meet requirements of wear resistance and an allowable impurity composition. The research results are shown in Table 2. The experiment parameters are the same as in Table 1. The most suitable materials in terms of resistance to effect of the KDP solution and the temperature are polymer compounds. Thanks to high strength and elasticity, these materials make it possible to create parts of a required form and size based on the

Table 1. The value of T_d of the KDP solutions after contact during the time t with various materials of the crystallizer parts

Description of the crystallizer part	Material	$T_d, ^\circ\text{C} (t, \text{day})$					
		1	7	14	28	56	84
Rods, support ring, substrate for the seed crystal, fasteners (bolts, nuts)	Organic glass	0.4	0.42	0.42	0.4	0.42	0.42
Device designed to mix the solution near the growth face, cuvette for the sensors, separator glasses	Quartz	0.3	0.3	0.3	—	—	—
Growth cuvette	Glass of the grade M1	0.41	0.44	0.53	0.64	0.68	0.79
Crystallizer bottle	Borosilicate glass	0.3	0.4	0.7	—	—	—
Film for the aluminum bottom disc	Polyethylene	0.32	0.33	0.38	0.46	0.48	0.5
Substrate for the seed crystal, screw, shaft for the screw, fastener (washers)	Teflon	0.3	0.29	0.25	0.26	0.29	0.29

Note. The error of the value of T_d is $\pm 0.05 ^\circ\text{C}$

Table 2. The value of T_d of the KDP solutions after contact during the time t with alternative structural materials of the crystallizer parts

Description of the crystallizer part	Material	$T_d, ^\circ\text{C} (t, \text{day})$					
		1	7	28	56	84	112
Growth cuvette	Glass of the grade M0	0.3	1	1.41	—	—	—
Device designed to mix the solution near the growth face, the screw	ABS-plastic (Avaatech)	0.37	0.37	0.38	0.38	0.39	0.4
Device designed to mix the solution near the growth face, the screw	ABS-plastic (Bestfilament)	0.25	0.29	0.27	0.27	0.29	0.27
Fasteners (bolts, nuts)	Polycarbonate	0.29	0.32	0.28	0.29	0.29	0.27
Rods	Polypropylene	0.28	0.27	0.3	0.29	0.32	0.31

Note. The error of the value of T_d is $\pm 0.05 ^\circ\text{C}$

requirements to the crystallizer design. Pollution effect by ABS plastic, polypropylene, polycarbonate has been studied by the above-described technique. The parameters of the experiment: $T = (60 \pm 2) ^\circ\text{C}$ and $T = (80 \pm 2) ^\circ\text{C}$; $V/S \sim (10000 - 12.000) \pm 0.008 \text{ cm}$; $\text{pH} = (4.01 \pm 0.02)$. Fig. 3 shows the prism face growth rate of the KDP crystal on supersaturation in the solutions with samples of the materials ABS-plastic (Bestfilament) and ABS-plastic (Avaatech).

The studied solutions with the samples made of polypropylene, polycarbonate, ABS-plastic (Bestfilament) and ABS-plastic (Avaatech) have shown weak variation of the magnitude T_d within the measurement error $\pm 0.05 ^\circ\text{C}$. The solution with the sample made of ABS-plastic (Avaatech) has exhibited a higher growth of the value of „dead zone“ (Fig. 3). Nevertheless, the sample of the studied material is assumed to be applicable for manufacturing the crystallizer elements interacting with the growth solution,

since the value of T_d was at most $0.6 ^\circ\text{C}$ during each of the control test periods. ABS-plastic (Avaatech) is produced as a result of copolymerization styrene, butadiene and acrylonitrile. In order to increase a transparency degree of ABS-plastic, methyl methacrylate is added into it. This product is exemplified by ABS-plastic (Bestfilament). Obvious difference in the chemical composition of these ABS-plastics is a possible cause in change of behavior of the value of the „dead zone“ of the solutions. Based on the dependences $T_d(t)$ obtained here, some crystallizer parts made of organic glass have been replaced with alternative ones made of polymer materials. Besides, applicability of additive technologies made it possible to replace one of the main crystallizer units (the device designed to mix the solution near the growth face), which was previously made of quartz glass. Despite resistance to large temperature gradients and no interaction of the quartz material with the potassium dihydrogen phosphate solution, the crystallizer

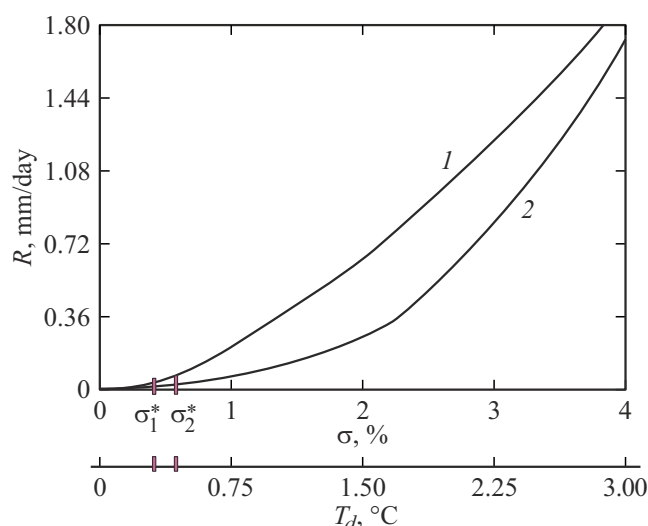


Figure 3. Dependence of the prism face growth of the KDP crystal on supersaturation in the solutions with the immersed sample: 1 — ABS-plastic (Bestfilament); 2 — ABS-plastic (Avaatech) with $t = 112$ days.

parts made of quartz glass are a fragile and less flexible structure as compared to the parts made of the polymer materials.

In the crystallizer, whose components are made of the materials tested for suitability as per the above-described technique, the KDP crystal of the sizes $80 \times 80 \times 80$ mm was grown in the method of rapid growth of the profiled single-sector crystals oriented in a pre-defined way for further manufacturing of a nonlinear-optical element of the frequency converter of the I type. The average crystal growth rate was ~ 3 mm/day. The solution of the 10 l-volume for growth of the KDP crystal was made of the potassium dihydrogen phosphate salt KH_2PO_4 and deionized water H_2O . The concentration of the impurities Al, Ba, Cr, Mg, Fe, Na, Si, Rb, Ti in the used reagents did not exceed 3 ppm. The solution saturation temperature was $T_s = (49.9 \pm 0.2)^\circ\text{C}$, the magnitude $T_d = (0.29 \pm 0.05)^\circ\text{C}$, $\text{pH} = (4.02 \pm 0.02)$. After completion of the crystal growth, the solution saturation temperature was $T_s = (33.1 \pm 0.2)^\circ\text{C}$, the magnitude $T_d = (0.53 \pm 0.05)^\circ\text{C}$, $\text{pH} = (4.06 \pm 0.02)$. The identical crystal was produced in the crystallizer before investigating a number of structural materials. The solution of the 10 l-volume had the following parameters: the solution saturation temperature was $T_s = (50.1 \pm 0.2)^\circ\text{C}$, the magnitude $T_d = (0.30 \pm 0.05)^\circ\text{C}$, $\text{pH} = (4.06 \pm 0.02)$. After completion of the crystal growth, the solution saturation temperature was $T_s = (33.3 \pm 0.2)^\circ\text{C}$, the magnitude $T_d = (0.76 \pm 0.05)^\circ\text{C}$, $\text{pH} = (4.07 \pm 0.02)$. The parameters of the solution before and after crystal growth have been compared to show a higher value of the dead zone of the solution after crystal growth in the crystallizer assembled before replacement of the number of the materials (acrylic glass, quartz, the glass of the M0 grade).

The impurity composition of the potassium dihydrogen phosphate solution was investigated by means of the atom emission method on the inductively coupled plasma spectrometer iCAP 6300 Duo (Thermo Electron Corporation, Great Britain). Random error of the analysis results is characterized by a value of relative standard deviation ≤ 0.10 . The obtained results did not reveal deterioration of the impurity composition of the solution during growth of the crystal and, respectively, interaction of the parts of the crystallizer structures with the solution in the crystallizer after replacement with the alternative materials (Table 3). And, respectively, the obtained high values of the „dead zone“ with growth of the crystal from the crystallizer assembled before replacement of some materials are confirmed by a high content of the impurities in the solution.

The performed studies shown applicability of some alternative materials in the crystallizer structures. However, one of the unsolved problems related to leaching of some impurities from the material of the crystallizer structures, which interacts with the solution, is still use of glass parts of the crystallizer. The glass provides good visibility of the crystallization process and it has good thermal stability and resistance to mechanical effects. But despite these advantages, the glass actively interacts with the potassium dihydrogen phosphate solution (Tables 1 and 2). The effect of leaching of the impurities from the glass is particularly pronounced in stoichiometric solutions. In nonstoichiometric solutions, due to presence of an active reagent (acid or alkali), the part of the impurities that arrive from the glass to the potassium dihydrogen phosphate solution transfers into inactive state in terms of deceleration effect on motion of the growth steps on the crystal faces. But change of the hydrogen index of the solution does not completely exclude a problem of negative effect of the impurities leached by the solution from the glass. This problem is especially pronounced during growth of large-size crystals in the crystallizer with the growth tank volume up to 700 l. Search for possible solutions for replacement of or methods of protection of the structural material — glass — against impurity leaching when interacting with the potassium dihydrogen phosphate solution is the subject of our further research.

In the crystallizer, whose components are made of the materials tested for suitability as per the above-described technique, the KDP crystal of the sizes $620 \times 560 \times 72$ mm (Fig. 4, a) was grown in the method of rapid growth of the profiled single-sector crystals oriented in a pre-defined way for further manufacturing of a nonlinear-optical element of the frequency converter of the I type.

The average crystal growth rate was ~ 3 mm/day. The solution of the 700 l-volume for growth of the KDP crystal was made of the potassium dihydrogen phosphate salt KH_2PO_4 , orthophosphoric acid H_3PO_4 and deionized H_2O . The concentration of the impurities Al, Ba, Cr, Mg, Fe, Na, Si, Rb, Ti in the used reagents did not exceed 5 ppm. The studies of pH effect on the kinetics of growth of the

Table 3. Concentrations of the basic impurities in the solutions before and after KDP crystal growth in the crystallizer before and after replacement of the structural materials with the alternative ones

Description of the solution sampling stage		Mass fraction of the impurity, ppm								
		Al	Ba	Cr	Mg	Fe	Na	Si	Rb	Ti
Crystallizer before replacement of structural materials with the alternative ones	Before KDP growth	0.02	0.005	0.007	0.001	0.01	0.06	1.1	0.26	0.003
	After KDP growth	0.06	0.048	0.007	0.016	0.01	0.17	3.5	0.27	0.003
Crystallizer after replacement of structural materials with the alternative ones	Before KDP growth	0.02	0.007	0.007	0.001	0.01	0.06	1	0.14	0.003
	After KDP growth	0.02	0.005	0.007	0.001	0.01	0.08	2.9	0.05	0.003

Table 4. Concentrations of the basic impurities in the solutions before and after growth of the KDP crystal

Description of the solution sampling stage	Mass fraction of the impurity, ppm								
	Al	Ba	Cr	Mg	Fe	Na	Si	Rb	Ti
Before KDP growth	0.02	0.009	0.007	0.002	0.01	0.12	0.6	0.09	0.003
After KDP growth	0.02	0.005	0.007	0.017	0.01	0.073	1.9	0.07	0.003

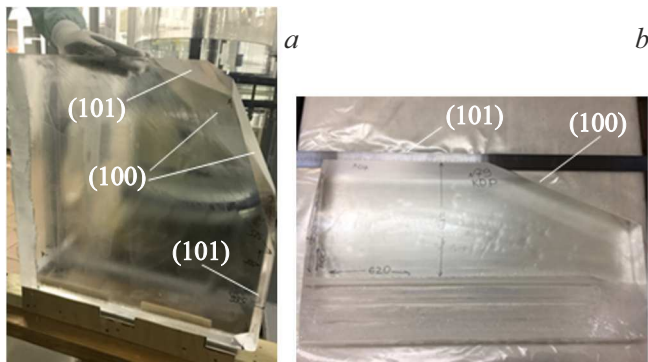


Figure 4. *a* — the KDP crystal of the sizes 620 × 560 × 72 mm; *b* — the KDP crystal of the sizes 620 × 219 × 72 mm.

faces of the KDP crystals have shown that the maximum rate of face (100) growth is reached at the stoichiometric pH 4.2 [14]. However, it is known that over time such solutions are subjected to degradation [15,16], which is indicated by an increased value of the „dead zone“ for a time based on the results of the present study. The authors of the paper [17] have shown that it is possible to increase the prism face growth rate of the KDP crystal with adding orthophosphoric acid. Despite the proved efficiency, adding a new component into the solution often reduces the quality of the crystal [18]. However, growth from the nonstoichiometric solution with the reduced pH can reduce, as compared to the stoichiometric solution, an arresting effect of impurities of ions of metals entering the solution, including from the crystallizer materials which have not alternative replacement and contact with the

potassium dihydrogen phosphate solution. The solution saturation temperature was $T_s = (54.5 \pm 0.2)^\circ\text{C}$, the magnitude $T_d = (0.41 \pm 0.05)^\circ\text{C}$, $\text{pH} = (2.41 \pm 0.02)$. After completion of the crystal growth, the solution saturation temperature was $T_s = (37.9 \pm 0.2)^\circ\text{C}$, the magnitude $T_d = (0.43 \pm 0.05)^\circ\text{C}$, $\text{pH} = (2.47 \pm 0.02)$. Fig. 5 shows the dependence of the prism face growth rate of the KDP

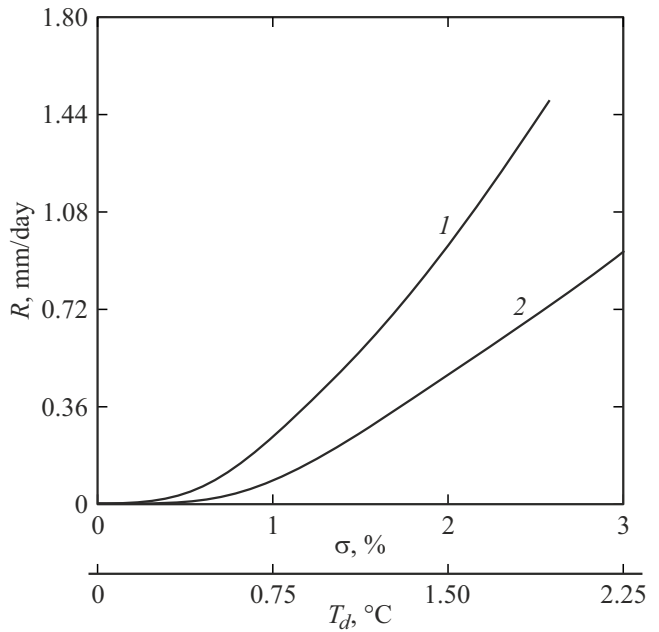


Figure 5. Dependence of the prism face growth rate of the KDP crystal on supersaturation in the solutions: *1* — before growth of the KDP crystal and *2* — after it.

crystal on supersaturation in the solutions before and after growth of the KDP crystal.

During the period of crystal growth, taking into account reduction of the saturation temperature from 54.5 °C to 37.9 °C, the value of T_d varied from 0.41 °C to 0.43 °C, which is consistent due to reduction of the saturation temperature. The study of the impurity composition of the solution has not detected deterioration of its impurity composition for the quite long period of crystal growth and, respectively, interaction of the parts of the crystallizer structures with the solution (Fig. 4).

The mass fraction of impurity of the basic elements Al, Ba, Cr, Mg, Fe, Na, Si, Rb, Ti in the prepared solution and in the solution after growth of the KDP crystal does not exceed 2 ppm. Fig. 4, b shows the KDP crystal of the sizes 620 × 219 × 72 mm, which was grown in the crystallizer, before studies of some structural materials. The crystal growth was stopped due to violation of a single-sector condition of the rapid method of growth owing to increase of the prism face sector in the crystal, thereby indicating high values of the „dead zone“ and, respectively, higher content of the impurities in the used solution.

Conclusion

The study has developed the technique of checking the materials used for manufacturing the crystallizer parts in contact with the solution during KDP-type crystal growth by the method of fast growth of the profiled single-sector crystals oriented in a pre-defined way. The criterion of applicability of the material as per the present technique is the value of the „dead zone“ of T_d of at most 0.6 °C during each of the control test periods. Based on the obtained dependences $T_d(t)$, it was shown that it was possible to use some polymer structural materials, in particular, ABS plastic, polypropylene, polycarbonate when manufacturing the crystallizer parts interacting with the KDP solution, which had been previously made of acrylic glass and quartz.

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Conflict of interest

The authors declare that they have no conflict of interest.

References

- [1] N.I. Zaitseva, L.J. Carman. Conf. Progress in Crystal Growth and Characterization of Materials, **43** (1), 118 (2001). DOI: 10.1016/S0960-8974(01)00004-3
- [2] H. Robey, R. Floyd, R. Torres, A. Burnham. *LLNL Rep. UCRL-ID-133365* (Lawrence Livermore National Laboratory, Livermore, Calif., 1999)
- [3] L.N. Rashkovich. *KDP — Family Single Crystals* (1st ed.) (CRC Press, 1991), DOI: 10.1201/9781003062844
- [4] I.A. Batyreva, V.I. Bespalov, V.I. Bredikhin, G.L. Galushkina, V.P. Ershov, V.I. Katsman, S.P. Kuznetsov, L.A. Lavrov, M.A. Novikov, N.R. Shvetsova. *J. Crystal Growth*, **52**, 832 (1981). DOI: 10.1016/0022-0248(81)90385-7
- [5] V.P. Ershov. *Skorostnoi rost monosektorial'nykh profilirovannykh kristallov gruppy KDP* (Kand. diss. N. Novgorod, 146, 2007) (in Russian).
- [6] E.P. Efremova, N.P. Zaitseva, A.Yu. Klimova, T.M. Okhrimenko, M.L. Barsukova, V.D. Spitsyna, V.A. Kuznetsov. *Izv. Akad. Nauk SSSR, Neorg. Mater.*, **27** (12), 2600 (1991).
- [7] M. Rak, N.N. Eremin, T.A. Eremina, V.A. Kuznetsov, T.M. Okhrimenko, N.G. Furmanova, E.P. Efremova. *J. Crystal Growth*, **273**, 577 (2005). DOI: 10.1016/j.jcrysgro.2004.09.067
- [8] T.A. Eremina, V.A. Kuznetsov, N.N. Eremin, T.M. Okhrimenko, N.G. Furmanova, E.P. Efremova, M. Rak. *J. Crystal Growth*, **273**, 586 (2005). DOI: 10.1016/j.jcrysgro.2004.09.068
- [9] D.A. Vorontsov, V.P. Ershov. *Vestnik NNGU. Ser. Fizika tverdogo tela*, (in Russian). **1** (9), 132 (2006).
- [10] L.N. Rashkovich, O.A. Shustin, T.G. Chervich. *FTT*, **42** (10), 1869 (2000) (in Russian).
- [11] T.N. Thomas, T.A. Land, J.J. De Yoreo, W.H. Casey. *Langmuir*, **20** (18), 7643 (2004).
- [12] L.N. Rashkovich, N.V. Kronskey. *J. Crystal Growth*, **182**, 434 (1997).
- [13] E.A. Khazanov, A.A. Shaikin, I.Yu. Kostyukov, V.N. Ginzburg, I.B. Mukhin, I.V. Yakovlev, A.A. Solov'ev, I.I. Kuznetsov, S.Yu. Mironov, A.V. Korzhimanov, D.N. Bulanov, I.A. Shaikin, A.A. Kochetkov, A.A. Kuz'min, M.A. Mart'yanov, V.V. Lozhkarev, M.V. Starodubtsev, A.G. Litvak, A.M. Sergeev. *Kvantovaya elektronika*, **53** (2), 95 (2023) (in Russian).
- [14] S.K. Sharma, S. Verma, V.K. Wadhawan, B.B. Shrivastava. *J. Crystal Growth*, **244** (3–4), 342 (2002). DOI: 10.1016/S0022-0248(02)01658-5
- [15] H. Robey, R. Floyd, R. Torres, A. Burnham. *Impurity leaching rates of 1000 liter growth tanks* (Lawrence Livermore National Laboratory, 1999), DOI: 10.2172/8048
- [16] H. Robey, M. DeHaven, C. Steffani, I. Fine, W. Olund, D. Schumann. *A controlled study of KDP solution contamination from Halar-coated aluminum growth platforms* (LLNL internal document, in preparation, Jan., 1999)
- [17] D.A. Vorontsov, E.L. Kin, V.N. Portnov, V.N. Trushin. *Vestnik Nizhegorodskogo un-ta im. N.I. Lobachevskogo*, **5** (1), 50 (2011) (in Russian).
- [18] E.P. Efremova, V.A. Kuznetsov, A.Yu. Klimova, O.V. Kachalov, I.L. Smol'skii, V.S. Naumov, M.I. Kolybaeva, V.I. Salo. *Kristallografiya*, (in Russian). **38** (5), 171 (1993).

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