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KDP crystals as an optical element in high-power laser system

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Abstract. This research presents a study of the dependence of the addition of ethylenediaminetetraacetic acid to the KDP crystal on the crystal growth kinetics and its physical-optical properties: width of the dead zone, growth rate of crystal faces, and transmission spectra. Obtained data on the change of dead zone width of growth solutions with concentrations of 0, 0.001, 0.005, 0.015, 0.02 mol% EDTA. KDP crystal was grown by the method of high-speed growth of oriented crystals, its transmission spectra in the range from 200 to 1100 nm were obtained. The possibility of adding EDTA when growing KDP crystals by the high-speed method is discussed.

Keywords: KDP crystal, potassium dihydrophosphate, EDTA, high-power lasers

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Кристаллы KDP как оптические элементы в мощных лазерных системах

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Аннотация. В работе исследуется влияние этилендиаминтетрауксусной кислоты (EDTA) на кинетику роста кристалла KDP и его физико-оптические свойства. Получены данные изменения ширины мертвой зоны ростовых растворов с концентрациями 0, 0,001, 0,005, 0,015, 0,02 mol% EDTA. Методом скоростного роста профилированных, заданным образом ориентированных кристаллов выращен кристалл KDP, получены его спектры пропускания в диапазоне от 200 до 1100 нм. Обсуждается возможность добавления EDTA при выращивании скоростным методом кристаллов KDP.

Ключевые слова: кристаллы KDP, дигидрофосфат калия, EDTA, мощные лазеры

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Introduction

Potassium dihydrogen phosphate (KDP) crystals are a non-alternative material for optical elements in high power laser sources. As a rule, such lasers generate radiation in the infrared range. Optical elements made from KDP crystals are used for conversion to the visible and ultraviolet ranges. Also, optical elements made of KDP crystals play the role of a laser pulse shaper.

The main advantages of optical elements based on KDP crystals are their transparency in a wide frequency range, high threshold laser-induced damage and non-linearity coefficients. Another important factor is the nature of KDP crystals. They are grown from aqueous solutions based on the KH_2PO_4 salt at temperatures near to room temperature. The peculiarities of KDP growth made it possible to develop methods of growing large crystals, up to 1 m in cross-section [1]. Rapid growth techniques are used for growing KDP crystals up to 1 m in cross-section [1, 2]. Studies have shown that rapid growth techniques are more sensitive to the presence of trivalent metal impurities (e.g., Al^{3+}, Fe^{3+}) in the growth solutions. It is known that the addition of chelating agents to the growth solution leads to reduce the influence of such impurities on the growth process. One of such chemical compounds are ethylenediaminetetetraacetic acid (EDTA). Also experiments show that addition of EDTA to the solution leads to leads to higher growth rate of the boundary prism face that is why there is interest in detailed study of EDTA influence on morphology and properties of crystals [3].

Results and Discussion

The following concentrations of impurity in the solution were chosen for the study: 0, 0.001, 0.005, 0.015, 0.02 mol%, the low concentration of EDTA is a consequence of the poor degree of solubility in water. Elemental analyses of growth solutions are in the table below (Table 1). The dependences of crystal growth rate on the value of solution supersaturation were determined. It follows from the data obtained that the addition of EDTA to decrease in the dead zone and an increase in the growth rate of the prism faces depending on supersaturation. The dead zone is the inert interval of the face growth rate at which the relative supersaturation of the solution grows, but there is no crystal face growth.

At low supersaturations (up to 0.3 °C) all investigated samples show the increase of prism face growth rate. At increasing supersaturations, the greatest increase in the growth rate of the prism face shows solutions with concentrations of 0.001, 0.005, and 0.015 mol%. Such supersaturation is corresponding to the supersaturation region used in the rapid methods of growth.

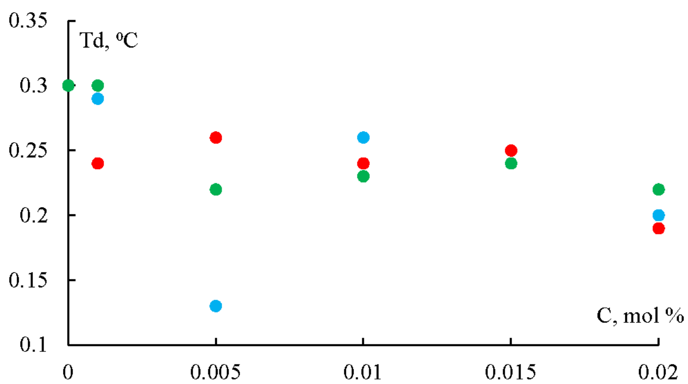

Fig. 1. Dependence of dead zone width (T_d) on solution concentration (C).
(Green is the solution without holding, blue holding for 2 weeks, red holding for 4 weeks))

To study the degradation of the solution over time after two and four months from the first study, the samples were retested. The results of the three studies are summarized in Fig. 1.

It can be seen that the dead zone width and saturation temperature change insignificantly with time in the solution. It can be noticed that the solution with EDTA concentration of 0.005 mol% in the second run has an anomalous value of the dead zone width. This is due to experimental errors: formation of stray crystals in the cuvette, laser beam moving away from the crystal or passing through the interface, hardware errors in data processing. It can be seen from the plot that the solution over time has retained the same values of the width of the dead zone.

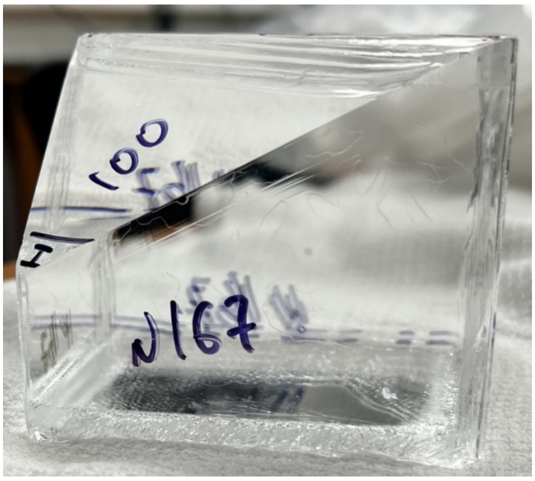

Fig. 2. Obtained KDP crystal (size of 80×80×56 mm^3)

A crystal KDP with 0.005 mol% EDTA in the growth solution was grown by the method of high-speed growth of oriented crystals profiled in a certain way. A dimension of the obtained crystal was 80×80×56 mm^3 (Fig. 2). The growth took place in a 10-liter crystallizer. The whole growth process took about a month.

Transmission spectrums for KDP crystal with EDTA addition in the growth solution were obtained (Fig. 3) with the UV-3600i Plus high-sensitivity spectrophotometer. The values correspond to the bipyramid sector with the orientation of the optical element of the type 1 frequency converter.

The impurity increased the transmittance threshold in the range of 5% in the 300–1100 nm. A characteristic ‘pit’ was formed at the beginning of the graph. Comparing the values we obtained with [3], we can see that the graphs have a general character of dependence in the studied area.

Table 1

Impurity composition of the prepared solution

Impurity	Mass fraction of impurity, ppm(wt)	Impurity	Mass fraction of impurity, ppm(wt)
Al	≤0.02	Pb	<0.01
As	<0.04	Pr	<0.03
Au	<0.05	Rb	0.14
B	<0.1	S	<0.5
Bi	<0.05	Sb	<0.06
Ca	0.08	Se	<0.04
Ce	<0.04	Si	1.0
Cs	<0.01	Sm	<0.03
Fe	≤0.01	Sn	<0.01
Ga	<0.05	Ta	<0.05
Gd	<0.01	Tb	<0.03
Ge	<0.06	Te	<0.09
Na	0.06	Th	<0.04
Nb	<0.01	Tl	<0.05

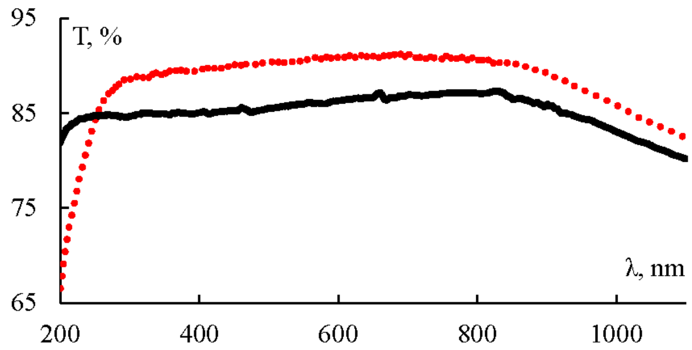

Fig. 3. Transmission spectra of the obtained crystal (red is the KDP crystal with EDTA addition, black is the clean KDP crystal).

Conclusion

A study of the effect of EDTA on KDP crystal morphology and properties showed that the addition of EDTA to the growth solution leads to an increase in the growth rate of faces (both prism and pyramid) while not disturbing the crystal structure. Within four months, the impurity solution retained the original width of the dead zone, which may indicate a long degradation time of its properties.

Analyzing the obtained transmittance spectra, we can conclude about the increase of the transmittance threshold in the wavelength region of interest. In the future it is also planned to study the threshold voltage value.

The addition of EDTA to the KDP growth solution leads to the improvement of its optical characteristics and positively affects the growth morphology and kinetics. The increase of crystal growth rate, in turn, makes the process cheaper by reducing the technological costs, which also has a positive effect on the growth technology at all. Without the influence of EDTA on the structure of the KDP crystal, EDTA can be used for fast growth of large KDP crystals. In this way, the effect of chelating agents on the morphology and properties of KDP crystals remains a promising topic for more detailed research in the future.

REFERENCES

1. **Zaitseva N., Carman L.,** Rapid Growth of KDP-type Crystals.

2. **Bespalov V.I., Bredikhin V.I., Ershov V.P., Katsman V.I., Kiseleva N.V., Kuznetsov S.P.,** Optical properties of KDP and DKDP crystals grown with high speed. Quantum Electronics, (9) (11) (1982).

3. **Vinh Trung Phan, Khanh Duy Nguyen, Hung Vu Tuan Le,** Effect of some hydroxyl additives on the growth and optical properties of KDP crystals, Journal of Crystal Growth, Vol. 587 (2022) 126643.

4. **Ershov E.V.,** High-speed growth of monosectoral profiled single crystals of the KDP group. RAS, 2007.

5. **Chuprunov V.N., Portnov V.N.,** The appearance and growth of crystals. M.: fizmatlit, 2006.

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