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Optics

Effect of L-arginine phosphate doping on structural, optical and strength properties of KDP single crystal

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Abstract. L-arginine-phosphate doped Potassium Dihydrogen Phosphate single crystals with 0.2…4.4 wt.% concentration in the solution was grown on a point seed by the method of temperature reduction. The grown KDP:LAP crystals were characterizied by UV-vis spectroscopy, powder XRD analysis, differential thermal and thermogravimetric analyses and second harmonic generation efficiency measurements. The mechanical and laser strength values of LAP doped KDP crystals have been evaluated. Slight variation in the unit cell parameters of KDP:LAP has been reported. It has been shown that the efficiency of second harmonic generation conversion in KDP:LAP crystals was higher by more than 3-fold as compared to the corresponding values of pure KDP. The experimental results evidence the suitability of the grown KDP:LAP crystals for optoelectronics, and the study is helpful for further searching and designing of hybrid NLO materials.

Keywords: KDP crystals, additives, L-arginine phosphate, optical and structural properties, nonlinear optic materials.

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1. Introduction

Potassium Dihydrogen Phosphate (KDP, KH_2PO_4) single crystals attract much attention due to their wide applications in different fields of nonlinear optics, optoelectronics and photonics. KDP group crystals possess high structure perfection, mechanical strength, wide range of spectral transparency, as well as relatively high values of laser damage threshold. Moreover, the growth technology makes it possible to obtain KDP crystals with well-developed growth sectors containing practically no defects. Nevertheless, relatively low value of quadratic susceptibility ($d_{36(KDP)}$ = 0.38 pm/V [1]) is one of the main functional restrictions for using the KDP crystals. One of the methods for raising the efficiency of second harmonic generation (SHG) is introduction of organic molecules, *e.g*., amino acids, which possess high nonlinear coefficients, into the matrix of KDP crystal. As shown by Xue *et al*. [2], hydrogen bonds (such as O-H and N-H/O) play a very important role in optical nonlinearities of inorganic crystals. For instance, L-arginine phosphate (LAP), a typical NLO semi-organic crystal, combines the advantages of both inorganic NLO crystals, *e.g*., high optical damage threshold, and of organic NLO crystals, such as considerable optical nonlinearity [3]. LAP belonging to KDP family crystals consists of alternating layers of the inorganic dihydrogen phosphate anionic groups, water molecules and the organic L-arginine molecules $[(H_2N)CNH(CH_2)_3CH(NH_3)COO]^+$, held together by plentiful hydrogen bonds [4]. The organic L-arginine molecule, the inorganic dihydrogen phosphate anionic group and the water molecules are all attributed to the NLO response of the crystal, but the major contribution is made by intrinsic optical nonlinearity of organic L-arginine molecule and inorganic dihydrogen phosphate group [4].

As shown in [5], the efficiency of SHG for KDP with 0.3 wt.% of L-arginine and KDP with 0.4 wt.% of L-arginine rises by the factors 1.33 and 1.74, respectively, in comparison with that of pure KDP. On the other hand, in a number of studies [6, 7] it has been revealed that doping with L-arginine and other amino acids leads to certain decrease of the values of

microhardness for KDP. It is well-known [8] that laser damage of KDP crystals essentially depends on their strength characteristics, and it is followed by the appearance and propagation of microcracks caused by mechanical or heating pulse in the process of irradiation.

In this work, we studied the influence of LAP molecules on the optical properties of KDP crystals grown from the solutions by using the temperature lowering method. The influence of LAP molecules on the structural, strength (laser-induced damage threshold and microhardness) and thermal characteristics of doped KDP crystals was investigated.

2. Experimental

Pure and doped KDP single crystals were grown from aqueous solutions (pH4.0 ± 0.1) onto a point seed measuring 5×5×10 mm by the temperature lowering method in a 6.0-L crystallizer at a saturation temperature of 50 °C as described in [9, 10]. LAP ($C_6H_{14}N_4O_2H_3PO_4 \cdot H_2O$) was used for doping KDP crystals at the concentrations 0.2, 0.5, 1.0, 2.2 and 4.4 wt.% in the mother liquor (on the weight of KH_2PO_4 salt). The LAP salt was prepared by mixing of aqueous solutions of L-arginine powder and orthophosphoric acid in a stoichiometric ratio. The temperature was lowered at the rate close to 0.3 °C/day and 0.4…0.8 °C/day for KDP and KDP:LAP crystals, respectively. The crystals were grown during 20…45 days (Fig. 1). The average crystal growth rate was 1.3 and 1.0 mm/day for pure KDP, 2.0…1.0 and 1.5…0.7 mm/day for KDP:LAP crystals in direction *z* and *xy*, respectively. The samples measuring 10×10×10 mm with the sides parallel to the planes (100) and (001) were cut out from the crystal growth sectors {100} and {101} of the grown crystals for studies of their optical transmission, laser and mechanical strength. The samples of crystals were cut out at the phase matching angle θ = 59° (Type II, *oee*) from {101} and {100} growth sectors for SHG measurements. Then all the samples were ground and polished.

3. Optical transmission studies

The optical transmission of the crystals was studied within the spectral range of 200…1100-nm wavelengths by using a Lambda 35 PerkinElmer spectrophotometer. KDP and KDP:LAP (0.2, 0.5, 1.0, 2.2 wt.%) crystals were transparent and did not contain visible inclusions (Fig. 1). The introduction of LAP additives influenced the change of crystal morphology. Slight blocking the {100} faces of the growing crystal as compared to pure KDP crystal was observed at 4.4 wt.% LAP. The increase in the concentration of LAP up to 4.4 wt.% also caused blocking of the {101} faces.

As seen, the transmission of the samples of the pure and doped crystals is close to ~80…90% within the region 300…800 nm. On the other hand, the spectra of the samples cut out from the sectors {100} of pure KDP and from the sectors {100} and {101} of KDP:LAP contain absorption bands in the UV region (Fig. 2). The appearance of the absorption band in the prismatic growth sector {100} of the nominally pure KDP crystal

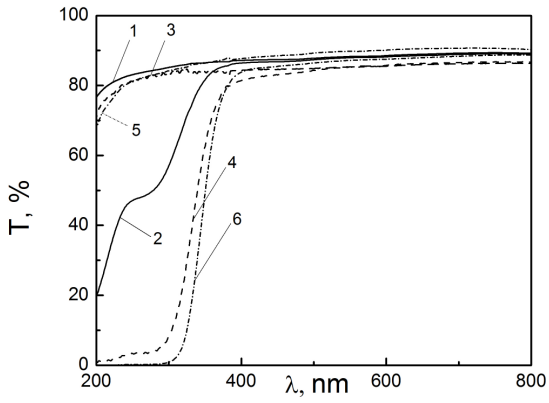
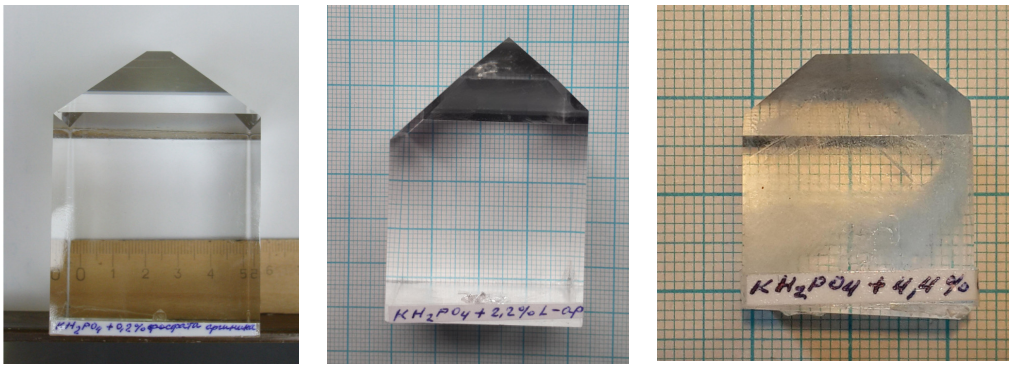


Fig. 2. UV-vis-NIR absorption spectra of KDP:LAP crystals for the sector {101} with LAP concentrations in the mother liquor, wt. %: 0 (*1*), 0.2 (*3*), 1.0 (*5*), and for the sector {100}: 0 (*2*), 0.2 (*4*), 1.0 (*6*).



a) b) c)

Fig. 1. Photo of KDP:LAP crystals for various concentrations in the mother liquor: 0.2 wt.% (a), 2.2 wt.% (b) and 4.4 wt.% (c).

discussed in a number of papers was attributed to the impurities of polyvalent metals [11, 12]. Accordingly, in our case, the absorption band at 270 nm in pure KDP crystal is related with the background cation impurities (Fe, Cr, Al, Pb, Mg, Mn *etc*.) in the initial KH_2PO_4. The strong absorption peak is defined by the impurity concentration, and it does not depend on the type of cation impurity. As seen, the spectrum of KDP:LAP crystal contains absorption bands with the maxima at 219 and 270 nm. In our opinion, the presence of intense absorption bands in the UV region of the spectrum of KDP:LAP crystal observed for both growth sectors {101} and {100} is related with the entering of the amino acid molecules into the matrix. It is confirmed by the fact that the observed position of the absorption maxima coincides with that of the absorption bands in the UV region of the spectrum related with the electron transitions in L-arginine molecule in the aqueous solution from the carboxyl group to the carbon chain [4].

The chemical analysis of KDP crystals grown in the presence of LAP with the concentrations 0.2…4.4 wt.% showed that the coefficient of incorporation of LAP molecules into the crystal in both sectors of growth is 0.01…0.017.

4. XRD analysis

The lattice parameters of KDP samples were measured on a general-purpose X-ray diffractometer using $CuK\alpha_1$ radiation and graphite monochromator in a primary beam. Possible adjustment errors were minimized by means of the Bond method [13]. The rocking curves (RC) were obtained with double crystal spectrometer at $CuK\alpha_1$ radiation with silicon monochromator adjusted to the (400) reflection.

The rocking curves and parameters of the crystal cell *a* and *c* of KDP and KDP:LAP crystals were measured. The study of KDP and KDP:LAP crystals perfection (2.2 wt.% of LAP, growth sectors {101} and {101}) showed that the structural perfection of the doped crystal corresponded to that of pure KDP crystal (Table 1).

The change in the parameter *c* of the doped crystal by $5\cdot10^{-4}$ Å in the sector {100} and by $9\cdot10^{-4}$ Å in the sector {101}, and also the change in the parameter *a* by $4\cdot10^{-4}$ Å in the sector {101}, in comparison with that of pure KDP, perhaps, is caused by the entry of LAP molecules into the crystal lattice of KDP (Table 2). Thus, KDP:LAP (2.2 wt.% of LAP) crystal is characterized by a "contraction" of the unit cell along the directions [001] and [100] in the sector {101}, and also along the direction [001] in the sector {001} in comparison with pure KDP crystal.

5. Laser-induced damage threshold and microhardness

The laser-induced damage threshold (LIDT) and Vicker's microhardness (H_V) of the samples of pure KDP and KDP:LAP crystals were studied as described in [9, 10]. LIDT was investigated at the fundamental wavelength of YAG:Nd^{3+} laser operating at $\lambda = 1.06$ μm. The laser damage criterion was a spark of high-temperature glow visually observed at the crystal breakdown. Vicker's microhardeness is a good parameter to determine the firmness degree of matters. This test was performed using the crystal KDP of size 10×10×10 mm. Measurements were carried out with the PMT-3 tester fixed to a Vicker's diamond pyramidal indenter attached to a microscope. Test was made on the (100) and (001) faces of crystal in the load range 10…200 g for 10 s.

The results of the effect of LAP on the mechanical and laser strength (Fig. 3, Table 3) of the doped crystals showed that the effect of dopant on the properties of KDP in the {101} and {100} sectors is different. Doping the crystal with LAP molecules (concentration of 0.2…1 wt.%) led to an increase in the microhardness in the {101} sector by 8% and a slight decrease in the microhardness at the additive concentration 0.2 wt.% in the {100} sector. The increase in the concentration of the additive to 1 mass.% contributed to the hardening of the crystal in the {100} sector.

KDP:LAP crystals, as well as KDP:L-arginine crystals [14], are characterized by somewhat larger microhardness values measured on (001) faces as compared to those for (100) faces. A similar change in the hardening of the faces was observed in [15] for a KDP crystal doped with urea.

Addition of LAP slightly reduced the laser strength of the KDP crystal in both growth sectors (Table 3),

Table 1. Full width at half maximum (FWHM) values measured in pure KDP and KDP:LAP crystals, *hkl* (008).

Samples	FWHM, arc. sec.
pure KDP, {101}, *z*	57.0
pure KDP, {100}, *z*	48.0
KDP:LAP (2.2 wt.% LAP), {101}, *z*	60.7
KDP:LAP (2.2 wt.% LAP), {100}, *z*	56.2

Table 2. Crystal lattice parameters of pure KDP and KDP:LAP (2.2 wt.% LAP) crystals.

Crystals	*c*, Å	*a*, Å	Δ*c*, Å	Δ*a*, Å
Pure KDP,{101}	6.9734	7.4528	–	–
Pure KDP,{001}	6.9732	7.4526	–	–
KDP:LAP,{101}	6.9725	7.4524	$-9\cdot10^{-4}$	$-4\cdot10^{-4}$
KDP:LAP,{001}	6.9727	7.4526	$-5\cdot10^{-4}$	0

whereas in [9, 14] we showed that addition of L-arginine promoted an increase in the laser strength of KDP in the {101} sector and a decrease in the {100} one. A certain discrepancy between the mechanical and laser strength of all the doped crystals (KDP:L-arginine, KDP:NN'DU, KDP:LAP) is caused by differences in the conditions of formation of mechanical and laser damage in the crystal.
The microhardness values of doped crystals, as compared to those in pure KDP, increase, and the laser strength slightly decreases. It may be related with the fact that the entry of LAP into the crystal leads to appearance of stresses in the lattice, which relax with appearance of additional defects in the crystal (dislocations). The movement of dislocations in the process of deformation with the indenter is difficult: the values of microhardness increase. At the same time, additional defects introduced into the lattice of the doped crystal enable to reduce its laser strength.

6. SHG measurements

The NLO properties of the crystals were studied using YAG:Nd^{3+} laser at $\lambda = 1.064$ μm as described in [10] (with 1 Hz pulse frequency, 7 ns pulse duration, 1 mm laser beam diameter). The samples of pure and doped KDP crystals were cut off at the phase matching angle $\theta = 59°$ (Type II, *oee*) from {101} and {100} growth sectors. The experiments were repeated for different input powers, and the corresponding output power was measured. The values of SHG efficiency were obtained from the ratio of the output to the input power.

The study of the NLO properties of KDP:LAP and pure KDP crystals has revealed the rise of SHG efficiency in the doped crystal by the factor close to 1.7 for the sectors {101} and 3.2 for the sectors {100} as compared to that in pure KDP (Table 4).

From this study, it is clear that doping with LAP leads to an enhancement in SHG efficiency, and the doped KDP single crystals are more useful than the pure ones for laser related device applications. The increase of SHG efficiency in the {100} sector relatively to that for the {101} sector in KDP:LAP crystal is probable due to both the entry of the molecules and formation of additional hydrogen bonds between the molecules and the growing face of the crystal.

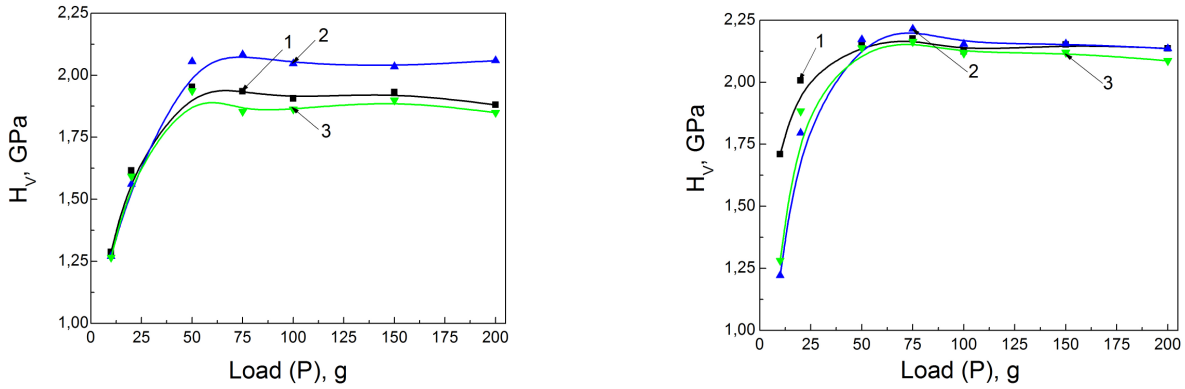


Fig. 3. Microhardness as a function of loading for the sectors {101} (a) and {100} (b) of the plane (001): pure KDP (*1*), KDP:LAP (1.0 wt.% LAP) (*2*), KDP:LAP (2.2 wt.% LAP) (*3*) crystals.

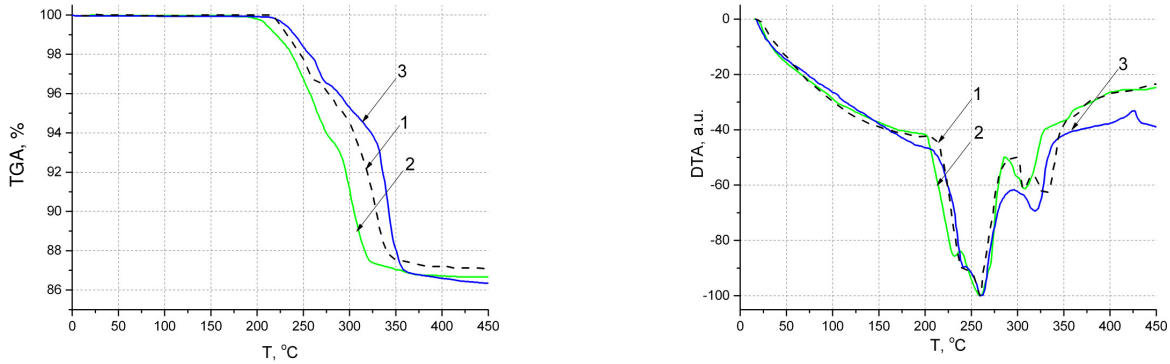


Fig. 4. TGA (a) and DTA (b) curves of KDP crystal growth, sector {101}: nominally pure KDP crystal (dashed line *1*), KDP:LAP (2.2 wt.% LAP) crystal (green line *2*) and KDP: LAP (4.4 wt.% LAP) crystal (blue line *3*).

Table 3. LIDT in KDP and KDP:LAP crystals for various LAP concentrations.

Concentration LAP in the mother liquor, wt.%	LIDT, J/cm^2	
	Sector {101}, face (001)	Sector {100}, face (001)
0	46.8	46.8
0.2	40.3	40.3
1.0	38.7	37.7
2.2	31.6	24.6

Table 4. SHG efficiency in KDP and KDP:LAP crystals for various LAP concentrations in the mother liquor.

Concentration LAP in the mother liquor, wt.%	Sector {101}	Sector {101}
Pure	1	1
0.2 wt.% LAP	0.8	1.7
0.5 wt.% LAP	1.3	2.8
2.2 wt.% LAP	1.7	3.2

7. Thermal properties

Differential thermal (DTA) and thermogravimetric (TGA) analyses of all crystals were investigated using the MOM Q-1500D derivatograph (Hungary) in 20…550 °C temperature range with the rate of heating 2.5 deg/min. Aluminum alpha-oxide was used as a standard; the samples of crystals (~1 g) for these measurements were crushed in a corundum mortar.

The TGA and DTA curves of the samples of pure KDP and KDP: LAP crystals (growth sector {101}) are shown in Figs 4a, 4b. Continuous weight loss of the studied samples is observed up to the temperature 450 °C and is approximately 12-14%. The thermogravimetric curve has three sections corresponding to the temperature ranges of 20…200 (220) °C, 200 (220)…320 (360) °C and 320 (360)…450 °C. For the first and third sections, the mass loss is insignificant and is caused by the removal of adsorbed water (the first section) and removal of residual dehydration products (the third section). The most intense mass loss is observed in the second section, where it is 12 (13)%. Within the region 200…380 °C, KDP crystals are dehydrated. The DTA curve contains several exothermic peaks within the temperature range 210…380 °C that corresponds to the dehydration of KDP crystals [16, 17]. It should be noted that for the growth sector {100}, the effect of the dopant on the course of the TG curves was similar.

For the TGA curves inherent to the doped crystals (2.2 wt.% LAP) in the growth sector {101}, the shift to the low-temperature region of the section of intense mass loss is observed: for nominally pure KDP crystals, this section is within the temperature range 215…350 °C, grown in the presence of LAP with the concentration 2.2 wt.% – 200…320 °C (Fig. 4b). The decrease in the thermal stability of KDP crystals is due to the possible loosening their structure upon doping. With the further increase in the concentration of LAP up to 4.4 wt.% of the beginning of the site of intense mass loss coincides with that for nominally pure KDP, and the end of intensive mass loss shifts by 10 °C towards the higher temperatures.

At the same time, the peaks in the DTA curves almost coincide. An increase in the thermal stability of the crystals in the {101} growth sector at the LAP concentration 4.4 wt.% relatively to the crystal grown at the LAP concentration of 2.2 wt.% may be caused by a more perfect lattice structure due to slow crystal growth related to blocking its faces by additive molecules.

8. Conclusion

Large-size optically transparent KDP:LAP (0.2…4.4 wt.% of LAP in the solution) have been grown using the method of temperature reduction. The grown crystals have been subjected to the NLO study to measure the SHG efficiency in comparison with that of pure KDP. The study of the NLO properties of KDP:LAP and pure KDP crystals has revealed the rise of SHG efficiency in the doped crystal by the factor close to 1.7 for the sectors {101} and 3.2 for the sectors {100} as compared to those for the pure KDP. The choice of L-arginine amino acid as a modifying dopant is related with the fact that it contains the amino group with a strong electron acceptor property, which may essentially influence the electron density distribution in the molecule and, consequently, the value of NLO response of the hybrid system.

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