Growth and Characterization of EDTA doped KDP single Crystal

[1]A. M. Dholariya & [2]K. G. Raval
[1]Bhagwan Mahavir College of Science and Technology, Surat-395017, Gujarat, India

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ABSTRACT: *The Potassium Dihydrogen Orthophosphate (KDP, KH_2PO_4) doped with Ethylenediaminetetraacetic acid (EDTA, $C_{10}H_{16}N_2O_8$) were successfully grown by slow evaporation method at room temperature with deionized water as a solvent with aim of improving properties of KDP crystal. The concentration of EDTA was 0.1mol% considered while synthesis of EDTA doped KDP single crystal. The grown single crystal has been analyzed with Energy Dispersive X-ray Analysis (EDAX) and X-Ray diffraction (XRD). The structural property of EDAX indicates the inclusion of EDTA in KDP crystal. XRD indicates the change in lattice parameter in EDTA doped KDP single crystal.*

Key Words: *Slow evaporation method, doping, characterizations, EDAX, XRD*

I. Introduction

Potassium dihydrogen orthophosphate (KDP, KH_2PO_4) is an interesting material because of its uses both in academic and industry [1]. As a crystal, KDP is a well-known inorganic nonlinear optical material, having piezoelectric, ferroelectric and electro optic properties [2-3]. In past, applications of the nonlinear optical (NLO) property such as second, third and fourth harmonic generators for Nd: YAG, Nd: YLF lasers have been discussed in the scientific field and KDP crystal remains interesting for its electro-optical applications such as Q-Switches for Ti: Sapphire, Alexandrite lasers [4]. Researcher have been trying to improve the properties of KDP crystal and review of literature shows that organic or inorganic impurities dopant crystals have shown improvement the material characteristics like nonlinear optical, ferroelectric properties, piezoelectric, electo optic and second harmonic generation (SHG).

In present work, the Ethylenediaminetetraacetic acid (EDTA, $C_{10}H_{16}N_2O_8$) was added as 0.1mol%, in KDP single crystal. Element analysis of Energy dispersive X – Ray analysis (EDAX) and structural analysis of X-Ray diffraction (XRD) have been carried out.

II. Experimental

2.1Growth of EDTA doped KDP crystal

Single crystals of 0.1mol% EDTA doped KDP crystals were grown by slow evaporation method in room temperature using deionized water as solvent.

For the seed crystal, super saturated solution of KDP (AR grade) was prepared in deionized water with 120 minutes of temperature controlled magnetic staring which was then filtered and kept in a Petri dish covered appropriately to get effect of slow evaporation and the seed crystal were allowed to grow for three weeks. The solubility of KDP in deionized water was 56gm/100ml at room temperature and pH of this solution is 4. After three weeks, seed KDP crystal was harvested as shown in figure 1.

Figure 1.seed crystal

To grow the crystal, a super saturated solution of KDP and 0.1 mol% solution of EDTA was prepared using deionized water as solvent. The solution was stirred using magnetic stirrer at room temperature for about

two hour for homogeneous mixture than solution was filtered using Whatmaan filter paper. The seed crystal was tied with a nylon threads and suspended into a beaker filled with super saturated solution of KDP with doped 0.1 mol% EDTA solution which was covered for slow evaporation and kept undisturbed for four weeks. After four weeks, transparent KDP crystal doped with EDTA was harvested as shown in figure 2.

Figure 2. EDTA doped KDP crystal

III. result and discussion

3.1 Energy Dispersive Analysis of X-Ray (EDAX)

Energy dispersive X-ray analysis (EDAX) is used for quantitative analysis. When a beam of electron strikes a sample, a fraction of the incident electron excites the atom of the sample which then emits X – rays on returning to their ground state. The energy of these X – ray is related to the atomic number of the element exited and therefore their detection forms the basis of elemental analysis in the electron microscope [5]. Energy dispersive X-ray analysis was accomplished using a Philips XL30 ESEM model at 0.2kv to 30kv.

The element confirmation of crystal was done by Energy dispersive X-ray analysis (EDAX), In order to confirm the presence of element O, P and K in pure KDP crystal [6] and C, O, P and K in 0.1mol% EDTA doped KDP crystal, as observed in the spectrum shown in Figure 3. It is also observed that Carbon of EDTA is present in EDTA doped KDP crystal. The stoichiometric ratio of every element in 0.1 mol% EDTA doped KDP are shown in Table 1.

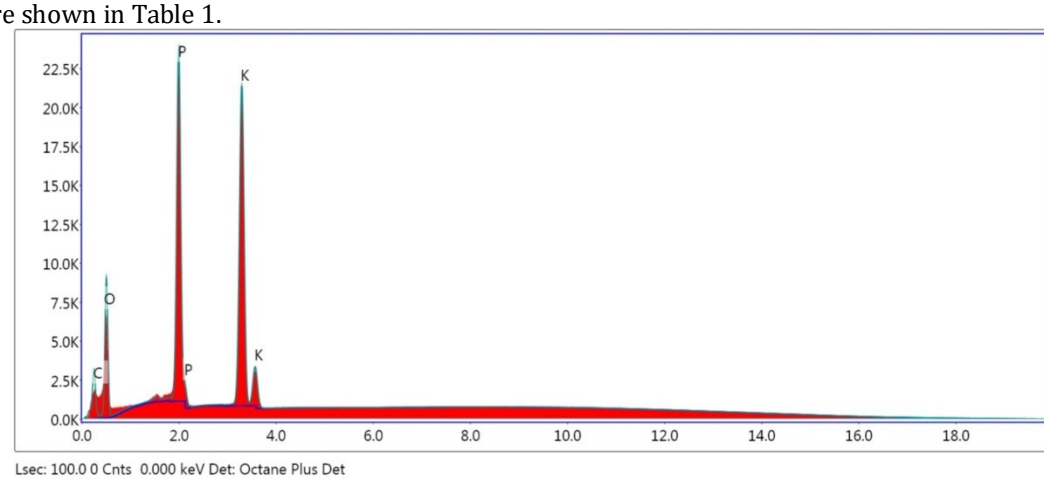

Figure 3. EDAX spectra for 0.1mol% EDTA doped KDP crystal

Table 1. Stoichiometric ratio of element of 0.1 mol% EDTA doped KDP crystal

Element	Weight%	Atomic%
C K	6.55	11.66
O K	43.41	58.01
P K	20.72	14.30
K K	29.31	16.03

3.2 Powder X-Ray Diffraction (XRD)

X – Ray diffractometer (XRD) is an analytical technique is used for phase identification of a crystalline material and unit cell dimensions. The X – Ray diffraction patterns of the crystal using a RigakuMiniflex X-

Ray powder diffractometer with monochromatic CuKα radiation λ = 1.54 A° and 2θ range is 0° to 80°. First, crystal was crushed to powder form. Lattice parameters were determine from the data using the xpowder software and equation 1 for d-space determination in a tetragonal lattice.

$$\frac{1}{d^2} = \frac{h^2 + k^2}{a^2} + \frac{l^2}{c^2} \qquad (1)$$

Where, h, k and l are miller indices and a, c are lattice parameter [7].

The experimental values of lattice parameters of 0.1mol% EDTA doped KDP crystal is good agreement with JCPDS card no. 350807 as shown in Table 2. XRD spectra of 0.1mol% EDTA doped KDP crystal is shown in Figure 4. XRD experimental data and hkl value of 0.1mol% EDTA doped KDP crystal is shown in Table 3.

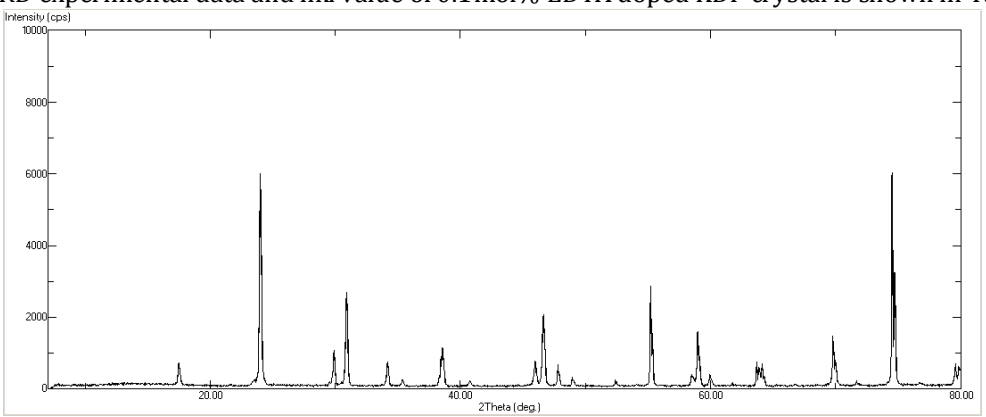

Figure 4. XRD spectra 0.1mol% EDTA doped KDP crystal

Table 2. Comparison of lattice parameters of 0.1mol% EDTA doped KDP crystal

Parameters	0.1 mol% EDTA doped KDP Crystal	ZCPDS Card data of KDP crystal NO.350807
a (A°)	7.4185	7.453
b (A°)	7.4185	7.453
c (A°)	6.9858	6.974
v $(A°)^3$	384.45	385.54
α = β = γ	90°	90°
Structure	tetragonal	tetragonal

Table 3. XRD data of 0.1mol% EDTA doped KDP crystal

d (obs)	d (cal)	h	k	l
2.8861	2.9073	1	1	2
1.5648	1.5636	3	3	2
1.4508	1.4537	2	2	4
1.3153	1.3114	4	4	0

IV. CONCLUSION

0.1mol% EDTA doped KDP single crystals grown by slow evaporation method at room temperature with deionized water as solvent. The effect of EDTA in KDP crystals were shown in Energy dispersive X – Ray analysis (EDAX) and X – Ray diffraction (XRD). The presence of all elements and the doping of EDTA were confirmed by EDAX test. The carbon in EDTA was present in doped KDP crystal. The lattice parameters are found to be fairly matched with JCPDS card No. 38507 and a tetragonal structure was confirmed by powder XRD analysis.

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