



RAPID GROWTH OF POTASSIUM DIHYDROGEN PHOSPHATE (KDP) SINGLE CRYSTAL BY ROTATING CRYSTAL METHOD

MESHAM N.S.*, GAHANE N.M., KAKDE R.N., SHINGADE B.A. AND REWATKAR K.G.

Department of Physics, Dr. Ambedkar College, Deekshabhoomi, Nagpur, MS, India.

*Corresponding Author: Email-

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Abstract- Potassium Dihydrogen Phosphate [KDP] single crystals were grown by rotating method. Potassium dihydrogen phosphate is necessary nonlinear optical material used to convert the laser light to shorter wavelength for laser fusion experiment. KDP Grown crystals have been characterized using powder X-ray diffraction, (XRD), Energy Dispersive X-ray spectroscopy (EDX) and Fourier Transform Infrared spectroscopy (FTIR). The presence of Gallium into pure KDP crystal was confirmed by FTIR and EDX spectra. Crystal structure has been studied by XRD. Pure KDP and Gallium doped KDP crystals both possessed tetragonal structure. The transparency is found to increase with the increase of doping concentrations of the grown crystals as observed by UV-Vis spectra.

Keywords- KDP, Solution Growth, XRD, FT-IR, EDAX, UV-VIS, SEM

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Introduction

Single crystals produced in industrial processes are needed to be of high quality because the performance of the devices (lasers, high-speed computers, etc.) using single crystals depends highly on the crystal quality. The properties and the structure of the crystal are greatly affected by crystallization process parameters. Therefore, a better understanding of the phenomena during crystallization is essential. [1,2] This classical material has a wide range of applications such as frequency conversion, laser fusion and electro-optical modulation [3,4]. The rapid growth of good quality crystals and various studies of organic and inorganic impurities doped KDP crystals have been reported by various investigators [5]. Improvement in the quality of KDP crystal and the performance of KDP based devices can be realized with suitable dopants such as transition metals, organic and inorganic dopants added in appropriate mol%.

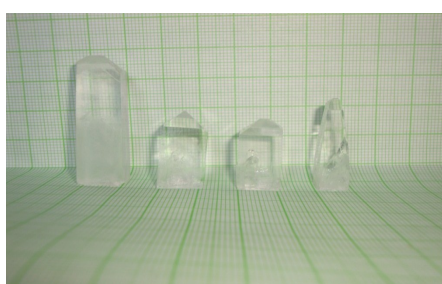
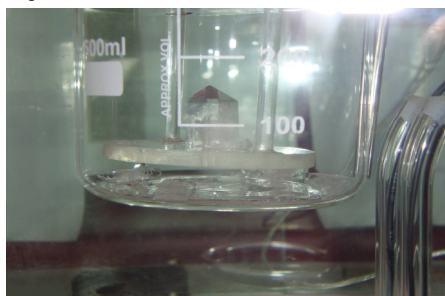
Semi organics are formed by combining organic molecules of high polarizability with mechanically strong and thermally stable inorganic molecules. These materials combine the chemical flexibility and

nonlinearity of organics and favorable physical properties of inorganics. In the present investigation, we report KDP single crystals were grown by rotating crystal technique. [6]. The crystals were characterized using powder X-ray diffraction studies, Fourier transform infrared (FT-IR) analysis, FT-Raman, UV-visible transmittance studies, Thermogravimetric Analysis and Differential Thermal Analysis (DTA). The crystal structure was revealed by single crystal X-ray diffraction studies.

Experimental

Saturated solution of KDP(KH_2PO_4) with specific concentration of Ga^{3+} was prepared which is been subjected to grow point seed by mehod of temperature rduction in $50^\circ - 30^\circ \text{ c}$. the seed made in the from of 5mm X 5 mmX 2mm cubes were fixed on a crystal sample holder located at the crystallizer bottom . the seed planes were oriented with respect to the crystallographic planes(100) (010) and (001)is an accuracy not worse than 30° arc min . the solution was stirred by a mixture at 60 rpm .the solution temperature was kept constant . The crystal were grown from stoichiometric solution with the Ph 4.0. the crystal growt rate 3mm per day

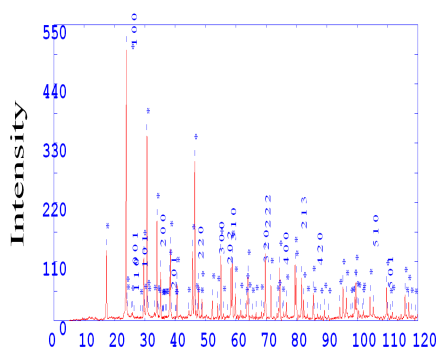
was established as an average value equal to the ratio of crystal dimension to the growth time. The physical and characteristics of the crystal were investigated using 10mm X 10mm X 10mm safe site sample obtained from the growth sector (100) with working surface subjected to grinding and optical properties. The crystal is shown in figure



Characterization

Powder crystal XRD analysis

KDP crystal belongs to the tetragonal scalerothedral symmetry with space group I42d having dimensions $a = b = 7.5243\text{\AA}$ and $c = 3.698\text{\AA}$. The sample was scanned in the range $10\text{--}90^\circ$ at a scan rate 2° per min. The finely powdered materials of the grown crystal were used for the analysis. The powdered diffraction pattern with indices are shown in figure. The prominent well resolved Bragg's picks of specific 2θ angle reveals the high crystalline nature of the crystal [7].



Fourier Transform Infrared spectroscopy

The Fourier transform infrared analysis of powdered sample was carried out between 4000 and 400 cm^{-1} by recording the spectrum using Varian FTIR spectrometer by ATR technique in order to reveal the metal complex coordination. The obtained result testify that all the IR spectra are practically identical and agree with the available literature data [8]. The assignments confirm the presence

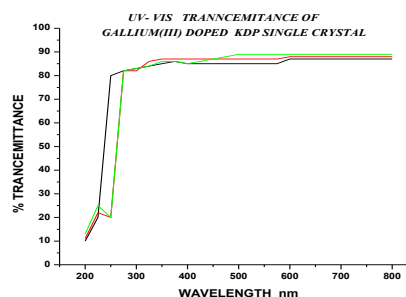
of various functional groups present in the material, tabulated in Table

Table 1-

	Wave number	functional group
1	3605	O-H
2	3333	O-H
3	2919	P-O-H
4	2839	P-O-H
5	2461	P-O-H
6	2358	P-O-H
7	1650	O=P-OH
8	1295	P=O
9	1100	P=O
10	904	P-OH
11	535	HO-P-OH

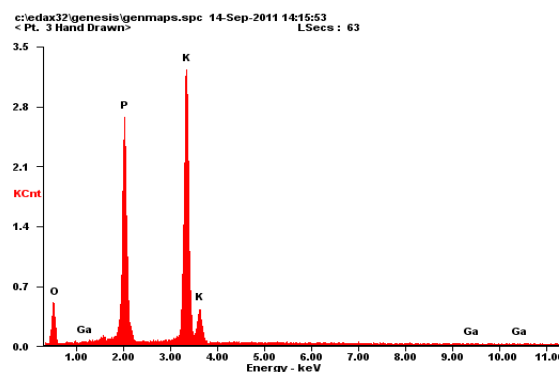
UV-VIS spectral analysis

The UV-Vis-NIR spectral transmittance was studied using a Shimadzu UV-1061 UV-Vis spectrophotometer with a single crystal of 6 mm thickness in the range of $200\text{--}1200\text{ nm}$. The recorded spectrum is shown in figure. The crystal has sufficient transmission in the entire visible and IR region [9]. The lower cut off wavelength is around 250 nm ; the transmission percentage of 2% Gallium added KDP crystal is around 93%, as compared to pure KDP, which is around 87%.



EDAX analysis

Energy dispersive X-ray analysis (EDAX) used in conjunction with all types of electron microscope has become an important tool for characterizing the elements present in the crystals. In the present study, the crystal was analyzed by INCA 200 energy dispersive X-ray micro analyzer equipped with LEO – Steroscan 440 Scanning electron Microscope. The recorded EDAX spectrum is shown in figure. Presence of Gallium is confirmed from the EDAX spectrum [10].

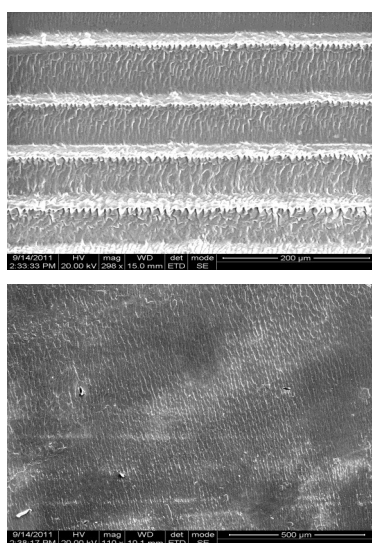


SEM Photograph

Effect of growth kinetics and surface morphology of KDP crystal by Cr³⁺ and Fe³⁺ ions were well known examples of concept of impurities absorption on the crystal faces.

Trivalent impurities like Cr³⁺, Al³⁺ and Fe³⁺ form chain like structure and disturb the crystal lattice dislocation mechanism. The Ga³⁺ ions are incorporated into superficial crystal growth layer and affect slightly growth process because they generate weak lattice stresses [10].

Scanning Electron Microscope (SEM) investigations of the as grown samples of Ga added KDP on the (100) plane reveal the isolated centers of Ga³⁺ in the form of small growth hillocks (hundredth parts of millimeter in diameter) whereas they are absent in pure KDP [11-12]. The recorded SEM images are shown in figure



Conclusions

Pure and Trivalent Ga³⁺ ion added KDP crystals were grown by slow evaporation method. The XRD spectrum shows the excellent crystalline nature of Gallium added KDP crystal. The transmission spectrum reveals that the crystal has sufficient transmission in the entire visible and IR region. All functional groups were present in crystals and are confirmed by FTIR spectrum.

Ga³⁺ ions are adsorbed on the crystal faces and create isolated centers. The Scanning electron microscope (SEM) pictures show the small growth hillocks in Ga³⁺ added KDP crystals. The presence of Gallium was confirmed by EDAX analysis.

References

- [1] Santhanu Bhattacharya, Parthasarathi Dastidar T.N. Guru Row (1994) *Chem. Mater.* 61, 531.
- [2] Renuka Kadirvelraj, Santhanu Bhattacharya, Tayur N. Guru Row (1993) *J. Inclusion Phenom. Mol. Recogn. Chem.* 30, 321.
- [3] Christer B. Aakeroy, Peter B. Hitchcock (1993) *J. Mater. Chem.* 3(11), 1129.
- [4] Chernov A.A., Rashkovich L.N., Mkrтчan A.A. (1987) *Sov. Phys.-Cryst.* 32, 432.
- [5] Rashkovich L.N., Yu B., Shekunov, (1992) *Growth of Crystals*, 18, 107.
- [6] Rashkovich L.N., Moldazhanova G.T., *Crystallogr. Rep.* 39

(1994) 135.

- [7] Chernov A.A. (1962) *Growth of Crystals*, 3, 35.
- [8] Cabrera N., Vermilyea D.A. (1958) *Growth and Perfection of Crystals*, Wiley, 393.
- [9] Bringly J.F., Rajeshwaran M. (2006) *Acta Cryst.*, E62.
- [10] Vijayan N., Balamurugan N., Rameshbabu R., Gopalakrishnan R., Ramasamy P., Harrison W.T.A. (2004) *J. Crystal Growth* 267, 218.
- [11] Prasad N.V., Prasad G., Bhimasankaran T., Suryanarayana S.V., Kumar G.S. (1969) *Indian J. Pure Appl. Phys.* 14(5), 639.