CHARACTERIZATION STUDIES OF GLYCINE AND POTASSIUM DIHYDRATE ORTHOPHOSPHATE CRYSTAL

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Abstract:

Single crystals of glycine doped potassium dihydrogen orthophosphate (KDP) were grown by Sankaranarayanan-Ramasamy (SR) method with have a size of 20 mm in diameter and 90 mm long . The grown crystals were subjected to powder X-ray diffraction (XRD), differential scanning calorimeter (DSC), thermo-gravimetric analysis (TGA), optical transmission, dielectric and Vickers microhardness studies. The TGA of the samples reveal that the grown crystals were stable up to 200 ÛC a minimum of for all samples. The important optical parameters like reflection and extinction coefficients of the grown crystal were calculated and discussed. The variation of dielectric constant, dielectric loss, a.c. resistivity and a.c. conductivity with frequency of applied field within the range from 1 kHz to 200 kHz was studied. The lower values of dielectric loss because of less of defect were observed in SR grown glycine doped KDP crystals. Vickers microhardness study shows higher mechanical stability in SR method grown crystals.

Keywords:

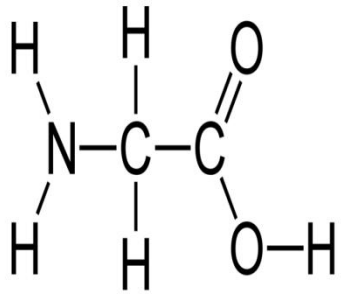
Growth from solutions; Nonlinear optical materials; Dielectric materials

Introduction

Nonlinear optical (NLO) crystals with high conversion efficiencies for second harmonic generation ar fascinating in varied applications like telecommunication, optoelectronics and device technology. There ar attention-grabbing materials each academically and industrially. KDP would possibly even be a material material documented for its nonlinear optical and electro optical (ferroelectric at coldness : Tc = 123 K)properties [1-2]. the excellent properties of KDP embody transparency throughout associate honest region of optical spectrum, resistance to wreck by device radiation and comparatively high nonlinear potency, beside consistent growth to giant size. Therefore, it is usually employed in many applications like device fusion, electro-optical modulation and frequency conversion [3]. several studies on the enlargement and properties of KDP crystals at intervals the presence of impurities ar reported [4-7]. The amino acids ar the known organic materials, play an important role at intervals the earth of nonlinear optical crystal growth. Since most of the amino acids exhibit NLO property, it's of interest to dope them in KDP [8]. With the aim of discovering new helpful materials for tutorial and industrial use an endeavor has been created to vary KDP crystals by adding some amino acids. KDP doped with amino acids like L-glutamic acid, L-histidine, L-valine were reported [9]. there ar found

modifications in optical, electrical and mechanical properties.Furthermore, natural philosophy trade desires new low material constant (Hr) materials as Associate in Nursing layer material (ILD) as a results of lowering the price of cloth constant of the ILD decreases the RC delay and lowers power consumptions [10]. Some substances once doped to KDP could yield KDP with low material constant [10-11]. Glycine was tried as a results of the dopant to scale back material constant and modify KDP crystals. There ar reported [12] that the second harmonic generation potency is found to be appreciably inflated by addition of amino acid glycine as impurity in KDP crystals grownup by typical technique. the enlargement of bulk size crystals whereas not defects would possibly even be a troublesome task for crystal farmer. The SR growth technique is appropriate to urge unifacial crystals from answer. the foremost blessings of SR answer growth technique ar easy experimental setup, unifacial growth, high solute-solid conversion, minimum thermal stress on the crystal throughout growth and hindrance of organism growth [14]. Recently several papers show that the SR technique full-grownup|grownup|mature} crystals have higher crystalline perfection than typical big crystals [15-17]. There do not appear to be any reported on the results of glycine on unifacial crystals growth by SR technique. Studies on the frequency and temperature dependence of cloth properties unveil helpful knowledge relating to structural changes, defect behavior and transport phenomena [18]. throughout this paper the enlargement by SR technique of glycine doped KDP crystals ar reported . The grownup crystals were outlined victimization XRD, TG/DSC, UV-vis NIR, material constant, material loss and Vicker microhardness to reveal the structure, thermal properties, optical transmission, dielectrics, defects and mechanical strength of the SR grownup glycine doped KDP crystals.

Glycine Structure:



Properties:

Molecular formula : C2H5NO2

Molecular weight : 75.07g\mol

Appearance : White solid

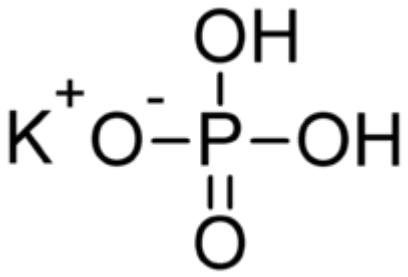
Odor : odorless

Melting point : 233 °C

Uses:

In the US, glycine is usually sold in 2 grades: u. s. book ("USP"), and technical grade. USP grade sales account for roughly eighty to eighty five % of the U.S. marketplace for glycine. wherever the customer's purity necessities exceed the minimum needed underneath the USP normal, as an example for a few pharmaceutical applications like blood vessel injections, pharmaceutical grade glycine, typically made to proprietary specifications and generally sold at a premium over USP grade glycine, is also used. Technical grade glycine, which can or might not meet USP grade standards, is sold at a cheaper price to be used in industrial applications; e.g., as Associate in Nursing agent in metal complexing and finishing.

Potassium phosphate Structure:



Molecular formula : KH2PO4

Molecular weight : 174.4g/mol

Odor : Odorless

Melting Point : 252.60C

Uses:

Potassium phosphate may be a extremely water soluble salt that is commonly used as a fertilizer,food additive and buffering agent.It is common supply of element and potassium.it is employed as a food additive;dipotassium is used in imitation dairy farm creams,dry powder beverages mineral supplements.

Experimental technique:

Crystals can grow from resolution if the answer is concentrated.Crystals could also be grown from a saturated resolution by slow evaporation, slow cooling,and diffusion strategies of all the strategies of crystal growth,slow evaporation methodology is simplest one for growing single crystals.In this methodology the saturated resolution is allowed to evaporate slowly at temperature with none disturbance.Single crystals of fine quality square measure commonly created by this methodology.In the gift work,water is employed as a solvent to grow the crystals of glycine mix metal phosphate.

Glycine metal phosphate: Saturated solutions of glycine and metal phosphate were ready from 20ml of distilled water.Then the answer were stirred well employing a magnetic stirrer for one hour.The saturated resolution was filtered and unbroken duringa beaker coated with perforated paper.After two days glycine metal phosphate crystal seeds were harvested.

CHARACTERISTIC TECHNIQUE UV - STUDIES

Ultraviolet–visible chemical analysis or ultraviolet-visible spectrophotometry (UV - Vis or UV / Vis) refers to absorption chemical analysis or coefficient of reflection chemical analysis within the ultraviolet- visible spectral region. this suggests it uses lightweight within the visibleand adjacent (near-UV and near-infrared (NIR)) ranges. The absorption or coefficient of reflection within the visible vary directly affects the perceived color of the chemicals concerned.during this region of the spectrum, molecules bear electronic transitions. this method is complementary to visible radiation chemical analysis, in this visible radiation deals with transitions from the excited state to the bottom state, whereas absorption measures transitions from the bottom state to the excited state.ULTRAVIOLET- VISIBLE SPECTROMETER: The instrument employed in ultraviolet-visible chemical analysis is termed a UV/Vis spectrophotometer. It measures the intensity of sunshine passing through a sample (I), and compares it to the intensity of sunshine before it passes through the sample (Io). The quantitative relation I / Io square measure referred to as the transmission, and is sometimes expressed as a share (%T). The absorbance, A, is predicated on the transmittance:

$$A = -\log (\%T / 100\%)$$

The UV-visible photometer also can be designed to live coefficient of reflection. In this case, the photometer measures the intensity of sunshine mirrored from a sample (I), and compares it to the intensity of sunshine mirrored from a reference material (Io)(such as a white tile). The quantitative relation I / Io is termed the coefficient of reflection, and is sometimes expressed as a share (%R).

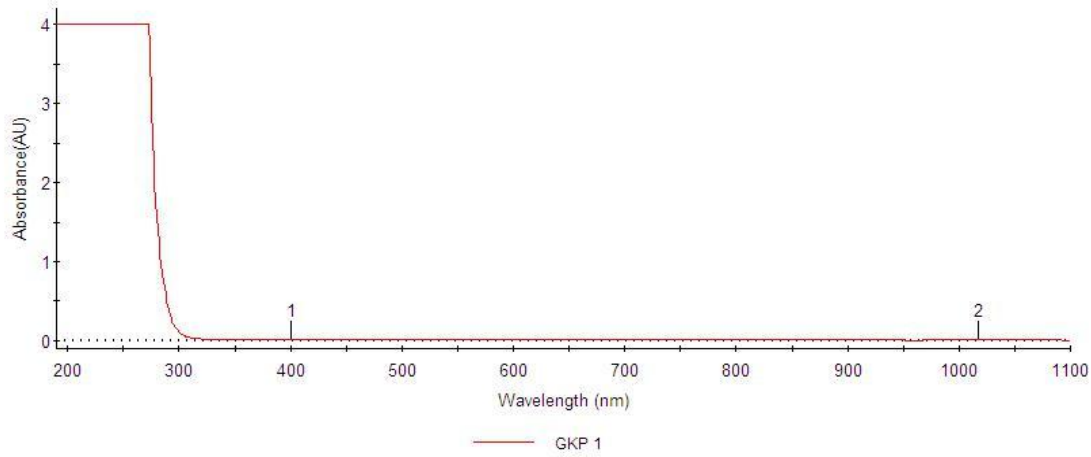
MICROHARDNESS STUDIES ON CRYSTALSHARDNESS TEST:

This hardness technique of subjecting a crystal to comparatively high at intervals a localized space with the assistance of appropriate alternative of indenter material and easy instrumentality construction will be simply applied to any or all crystalline materials beneath completely different condition. Also, specimen preparation is least. however the sole condition to be maintained is that the specimens ought to have flat and swish surfaces.Hardness of a cloth will be measured by scratch tests, processing tests, rebound tests,cutting tests, abrasion tests, and erotion tests and even by static indentation tests within which a ball, or a cone or a pyramid is forced into a surface. The load per unit space of impression provides directly the worth, as worn out brinell, Vickers, and illustrator and Knoops tests. VICKERS HARDNESS TEST: Among the various tests of hardness measurements, the foremost straightforward and reliable methodology is vickers hardness check methodology within which a pyramid indenter is employed for indentation almost like that in knoop methodology. The Vickers hardness check was developed in 1921 by parliamentarian L.Smith and martyr E. Sand land at Vickers Ltd as another to the Brunel methodology to live the hardness of

fabric. furthermore during this methodology indentation is formed on the crystal surface by suggests that of a diamond pyramid whose opposite faces contain associate degree angle. Hardness is generallydefined because the quantitative relation of the applied load to the area of the indentation. In Vickers hardness check a sq. based mostly diamond pyramid is employed rather than a parallelogram base pyramid as employed in knops methodology.

STUDIES AND ANALYSIS OF UV HARDNESS UV ANALYSIS:

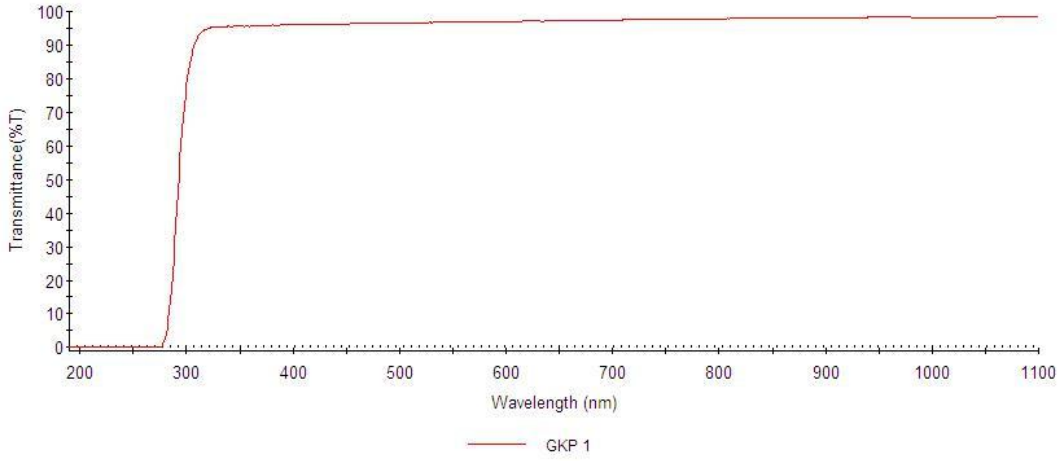
ABSORBANCE:



Name	No.	Peak(nm)	Peak(AU)
GKP	1	401.30	0.0175
	2	1,016.90	0.0092

UV ABSORBTION

The optical absorption spectrum of grow crystal.The wavelength range of 200 to 1100 nm.From the spectrum it is no absorbtion of light in the entire visible region.The widened transparency is observed in the region of 401.3 to 1016.9nm.



UV TRANSMITTANCE

The UV - Vis transmittance spectrum was recorded.It is observed that UT crystal have high transmittance in the entire visible region.The UT cut off range is found to be at 270nm.The wide range of transference 100%.

The second gap of energy of insulators its higher>4evand<3ev but lowest for semiconductor.

BAND GAP=1240/
=1240/270
=4.5925ev.

MICRO HARDNESS ANALYSIS:

Micro hardness measurement is commonly used to determine the mechanical strength of the material which is related to bond strength and defect structure . Optically clear and defect free crystal plate taken perpendicular to the growth direction was subjected to indentation tests at room temperature. The diagonal length of the indentation (d) in m for various applied load (P) in g was measured for a constant indentation period of 15 s. The Vickers's hardness number (Hv) was calculated using the relation:

$$Hv = 1854.4\ p/d2\ kg/mm2 \quad \text{---------- (1)}$$

According to the indentation size effect (ISE), micro hardness of crystals decreases with increasing load and in reverse indentation size effect (RISE) hardness increases with increasing load. In our case, Hv increases with load up to 100 g and becomes load independent for P≥100 g. The traditional Meyer's law gives the relationship between load P and size d :

$$P=Adn \quad \text{---------- (2)}$$

where the exponent n is the Meyer's number and A is a constant.

According to Hays–Kendall's approach, load dependent hardness may be expressed by :

$$P = W + A1dn \text{ ---------- (3)}$$

Where W is the minimum load initiate plastic deformation, A1 is the load independent
constant and the exponent n = 3. The value of W and A1 can be calculated by plotting the experimental P against d2. The relationship between indentation test load and indentation size is

$$P - w = k1d2 \text{ ---------- (4)}$$

Where k1 is a new constant and (P−W) is the effective indentation test load. The plot between dn and d2 is drawn and intercept W/k1, from fig.4 the value of W is calculated.

Test Mode : Vickers Hardness Test
Sample Name : GKP

Load: 25grm

S.No	L1	L2	HV
1	39.20	42.77	28.4
2	37.27	40.70	30

Load: 50grm

S.No	L1	L2	HV
1	44.14	48.05	42.7
2	44.76	48.41	45.4

Load: 100grm

S.No	L1	L2	HV
1	54.52	58.63	55.9
2	55.98	60.17	53.8

Creak Length:

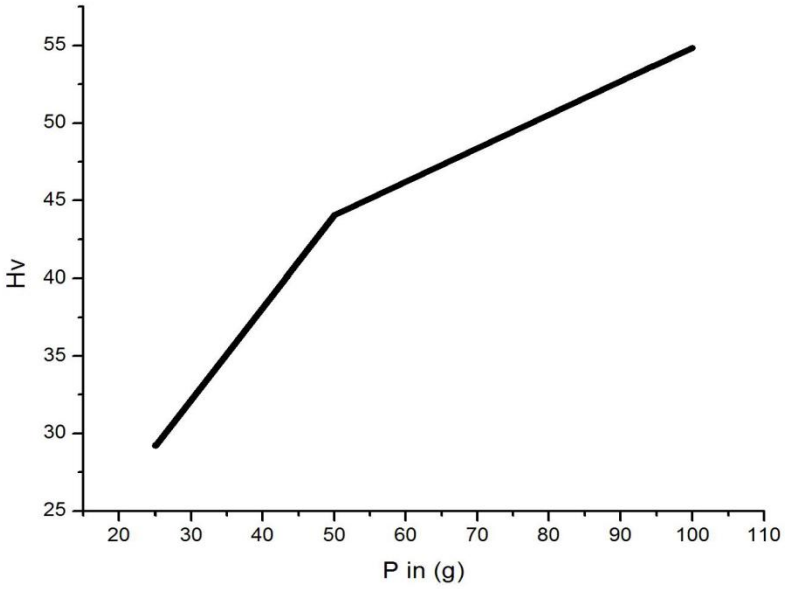
Left :34.98

Right: 26.86

UP: 22.02

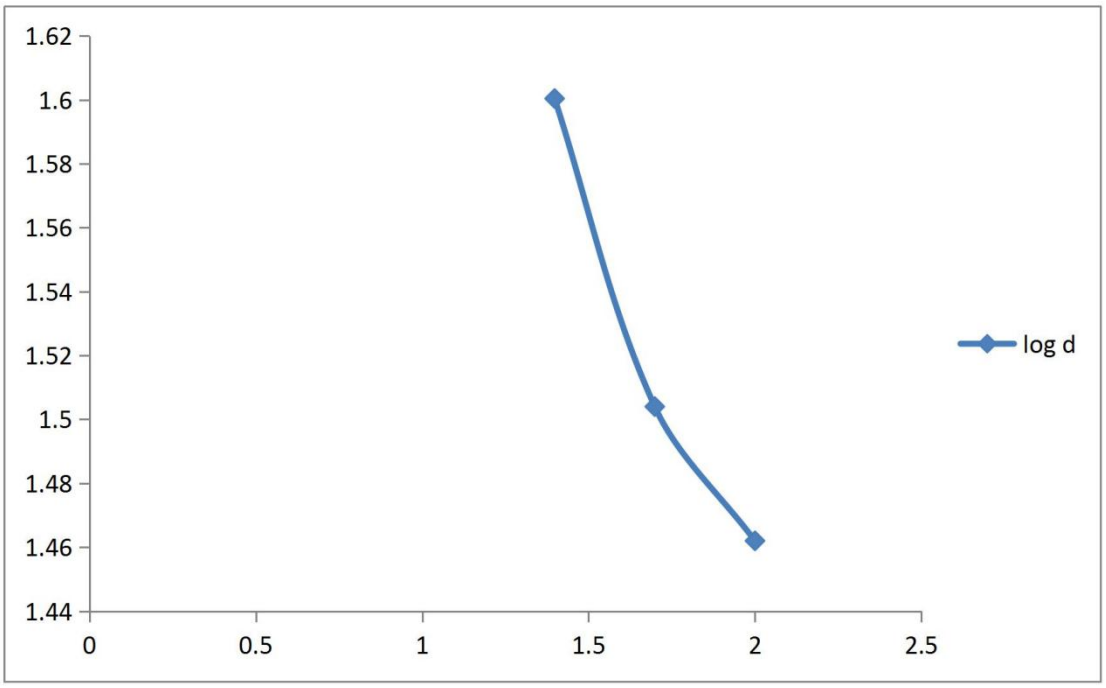
Down: 27.98

P	Hv
25	29.2
50	44.05
100	54.85



log P	log d

1.3979	1.6004
1.6989	1.504
2	1.462



Conclusion:

Glycine potassium phosphate crystal is grown by slow evaporation method.Grown
crystals are characterized by UV and HARDNESS study.This is a optical material, Glycine Potassium Phosphate crystals have been grown by slow evaporation method at room temperature. The band gap energy of uv spectrum is 4.5925ev.

The hardness studies are carried out using vicker's method.The hardness number was
calculated for different .

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