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Bulk growth and analysis on luminescence, third order nonlinear optical, laser damage threshold, dielectric and thermal properties of KDP crystal doped with BTZC complex

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ABSTRACT
The 34 × 10 × 06 mm^3 bulk single crystal of bis-thiourea zinc chloride (BTZC) doped potassium dihydrogen phosphate (KDP) material has been grown from solution evaporation growth technique. The color centered photoluminescence nature of grown crystal has been investigated in visible region of interest. The He–Ne laser assisted Z-scan analysis has been carried out to explore the promising third order nonlinear optical behavior of BTZC-KDP crystal. The origin of nonlinear refraction (n_2), the absorption coefficient (β) and susceptibility (χ^3) has been discussed and their respective magnitudes have been determined using the Z-scan transmittance data. The high magnitude of laser damage threshold (GW/cm^2) of grown crystal has been determined using the 1064 nm Nd:YAG laser. The influence of increasing temperature on dielectric constant and dielectric loss of pure and BTZC doped KDP crystal has been comparatively investigated. The thermal decomposition and the melting profile of BTZC doped KDP crystal has been investigated by means of simultaneous thermogravimetric and differential thermal analysis.

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Crystal growth; Z-scan study; dielectric studies; thermal studies; LDT; photoluminescence study

1. Introduction

In the current technological era, the nonlinear optical (NLO) single crystals play the crucial role in designing, tuning, processing and fabricating the hi-tech devices that encourage the use of NLO crystals in photonics, optoelectronics, laser frequency conversion and integrated optic applications [1,2]. The potassium dihydrogen phosphate (KDP) crystal shows high technological impetus for industrial applications owing to high NLO response and excellent optical homogeneity which are prerequisite parameters for designing optical switching device. For the enhancement in desired qualities of KDP crystal various experimental approaches (doping of additives or different growth techniques) have been attempted, however, the stress has been given on doping technique. In literature majority of investigations were reported on enhancing an effect of amino acids (glycine, L-alanine, L-arginine, L-histidine, L-valine) on nonlinear optical property of KDP crystal [3–5]. The influence of formic acid, maleic acid, citric acid and oxalic acid on SHG efficiency, third order nonlinear optical (TONLO) and dielectric properties of KDP crystal have been extensively studied [6–8]. In addition, the impact of metallic impurities (Li, Ca, Ce and V) and different dyes on optical, mechanical, thermal and dielectric traits of KDP crystal has also been investigated [9–11]. In our recent investigation, a semi organic thiourea metal complex bis-thiourea zinc chloride (BTZC) has been doped in KDP crystal and interesting results have been observed. The doping of BTZC [12] and BTNN [13] has inculcated large enhancement in SHG efficiency, dielectric quality, optical transparency and crystalline quality of KDP crystal. Present communication is the extended work which has been accomplished by achieving the bulk single crystal growth and investigation of photoluminescence, dielectric, laser damage threshold, TONLO and thermal properties of BTZC doped KDP crystal to explore the vitalized technological applications.

2. Experimental procedure

The BTZC salt was synthesized using the AR grade zinc chloride and thiourea in a molar ratio of 1:2 and double distilled water as a solvent. In order to dope BTZC the supersaturated solution of KDP salt was prepared at room temperature. As SHG efficiency of 0.2 mol of BTZC doped KDP salt is found higher in our previous studies [12], the same BTZC doped KDP single crystal was further purified two times by recrystallization process. The optical quality BTZC doped KDP (BTZC-KDP) single crystal grown at constant temperature of 35 °C within the period of two weeks is shown in Figure 1(a). The dimension of BTZC-KDP crystal grown by self-nucleation process is noticeable which is found to be 34 × 10 × 06 mm^3.

3. Results and discussion

3.1. Optical studies

The study of relaxation of excited electrons from higher to lower energy states under certain conditions gives a specific color centered emission spectrum which is an ideal characteristic of a given material known as photoluminescence (PL) study. Following this study information of material can be scrutinized

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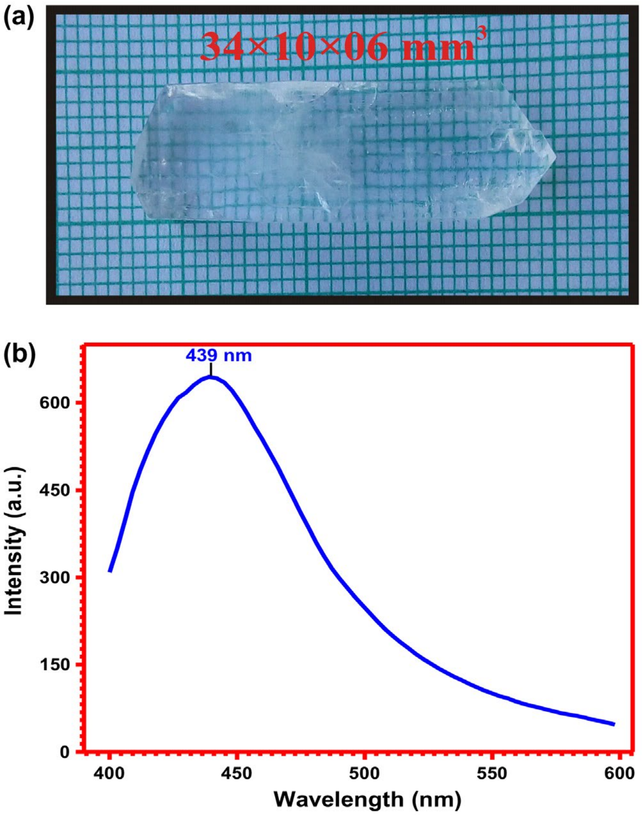

Figure 1. (a) BTZC-KDP crystal and (b) PL emission spectrum.

Table 1. Details of Z-scan setup.

Parameters	Magnitude
Laser wavelength (λ)	632.8 nm
Laser power (P)	10 mW
Lens focal length (f)	20 cm
Optical path distance (Z)	113 cm
Beam waist radius (ω_a)	1 mm
Aperture radius (r_a)	1.5 mm
Incident intensity at the focus (I_0)	2.3375 KW/m^2

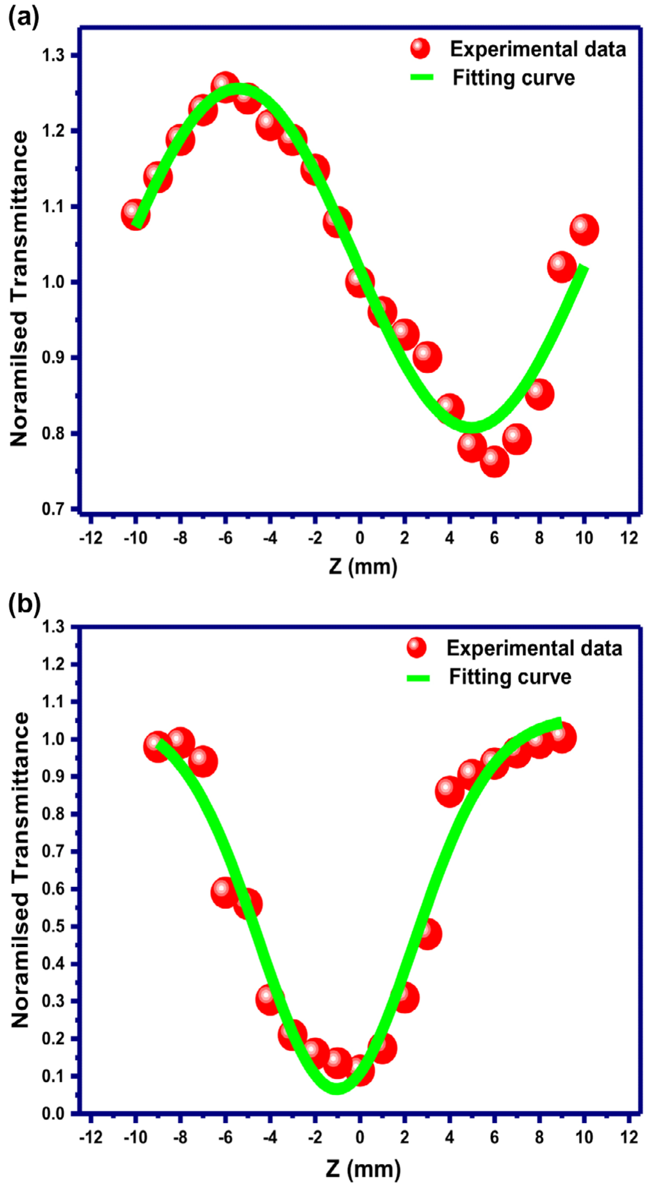

Figure 2. Z-scan transmittance curve (a) close and (b) open.

such as intrinsic defects, surface quality, electron transition profile and the associated energy states. Therefore, PL has extended realm of applications in biochemical, medical and chemical domain of research [14,15]. In present analysis, the PL studies have been carried out using the Hitachi FL-7000 spectrophotometer (emission and excitation slit width = 2.5 nm, response time = 0.1 S) and the PL emission spectrum (Figure 1(b)) was recorded in the range of 400–600 nm. The PL emission spectrum shows a single colored emission with peak maxima centered at 439 nm corresponding to the energy of 2.82 eV. This confirms the prominence of blue colored emission in the grown crystal material. The smooth spectrum and the single peak intensity confirm the good crystalline nature of BTZC-KDP single crystal.

The Z-scan is the most sensitive technique developed by Bahae et al. [16] to examine the TONLO nature of materials. In current investigation, the CW mode operated He–Ne laser (λ = 632.8 nm) based Z-scan setup (Table 1) was used to evaluate the nonlinear refraction (n_2) and absorption (β) of grown crystal employing the close and open aperture Z-scan configuration respectively. The polished and transparent crystal complying the Raleigh criteria of thickness was placed on the sample holder positioned at the focus ($Z = 0$) of the beam irradiated path i.e. Z-direction. The incident laser beam was filtered through the gaussian lens and focused on the sample. The position dependent profile of transmitted intensity from the sample was respectively recorded using the close aperture detector placed at far placed and graphically represented in Figure 2(a). It reveals that the grown crystal inherits the transmitted intensity from pre-focus peak to post-focus valley. This confirms the origin of negative nonlinear refraction (n_2) effect in the crystal which is the characteristic property of material attributing self-defocusing nature [17,18]. The phase change in propagation of light with change in position in crystal medium is governed by the fact that the irradiated laser beam intensity with high repetition rate causes the localized absorption of optical energy along the crystal surface which leads to thermal lensing effect [19,20]. The materials such BTZC-KDP crystals with negative phase change in refraction seek huge demand for night vision optical sensor devices [21,22]. The peak to valley transmittance (ΔT_{p-v}) and on-axis phase shift ($\Delta\Phi$) is related as [16],

$$\Delta T_{p-v} = 0.406(1 - S)^{0.25}|\Delta\phi| \tag{1}$$

where, $S = \left[1 - \exp\left(-2r_a^2/\omega_a^2\right)\right]$ is the aperture linear transmittance, r_a is the aperture radius and ω_a is the beam radius at the aperture. The magnitude of n_2 has been calculated using the relation [15],

$$n_2 = \frac{\Delta\phi}{KI_0L_{eff}} \quad (2)$$

where $K = 2\pi/\lambda$ (λ is the laser wavelength), I_0 is the beam irradiance intensity at the focus, $L_{eff} = [1-\exp(-\alpha L)]/\alpha$, is the effective thickness of the sample depending on linear absorption coefficient (α) and L is the thickness of the sample. The order of magnitude of n_2 is found to be 10^{-12} esu. The open aperture Z-scan analysis is crucial technique to identify the nature of nonlinear absorption in the material. In present analysis, the open aperture Z-scan curve has been recorded as shown in Figure 2(b). It is observed that as the sample has been translated towards the focus the transmittance has been reduced to the lower value which indicates the existence of photon absorption effect by different energy states associated with the crystal material. The phenomenon of minimized transmitted intensity at the focus is termed as reverse saturable absorption (RSA) [23]. The RSA effect is contributed by the dominance of two-photon absorption assisted by multiphoton absorption effect [24,25]. The TONLO absorption coefficient (β) of grown crystal has been evaluated using the equation [16],

$$\beta = \frac{2\sqrt{2}\Delta T}{I_0L_{eff}} \quad (3)$$

where ΔT is the one valley value at the open aperture Z-scan curve. The β of BTZC-KDP crystal is found to be of 9.47×10^{-6} cm/W. The TONLO susceptibility (χ^3) helps to analyze the polarizing ability of the material. The higher the susceptibility higher is the polarizing nature of the material [26]. The χ^3 has been evaluated using the relation [16],

$$\mathrm{Re}\chi^{(3)}(\mathrm{esu}) = 10^{-4}(\varepsilon_0C^2n_0^2n_2)/\pi(\mathrm{cm}^2/\mathrm{W}) \quad (4)$$

$$\mathrm{Im}\chi^{(3)}(\mathrm{esu}) = 10^{-2}(\varepsilon_0C^2n_0^2\lambda\beta)/4\pi^2(\mathrm{cm}/\mathrm{W}) \quad (5)$$

$$\chi^{(3)} = \sqrt{(\mathrm{Re}\chi^{(3)})^2 + (Im\chi^{(3)})^2} \quad (6)$$

where ε_0 is the vacuum permittivity, n_0 is the linear refractive index of the sample and c is the velocity of light in vacuum. The higher magnitude of χ^3 of BTZC-KDP crystal is found to be of order 10^{-5} esu. The TONLO parameters of BTZC-KDP crystal are excellent which are highlighted in Table 2.

The continuous exposure to the high intensity of optical irradiation causes the surface damage of the material hence the laser damage threshold analysis has become the key factor for scrutinizing a material so as to be utilized in laser assisted device applications [27]. The laser damage threshold analyses of grown crystal have been determined using the Q-switched mode operated Nd:YAG laser (1064 nm, 10 Hz, 10 ns) employing the multi-shot method. The laser beam was attenuated and the crystal sample was shot. The energy of the sample was increased till the sample showed prominent cracks/damage. In current analysis, crystal showed prominent damage at a beam energy of 104.4 mJ. As the crystal is bombarded by the high-intensity laser beam the major factors contributing to the LDT are electron avalanche effect, localized photo-ionization due to thermal gradient and multiphoton absorption [28,29]. The LDT of BTZC-KDP crystal is found to be 1.64 GW/cm^2 which is significantly higher than KDP and Urea [30], as discussed in Table 3.

Table 2. TONLO parameters.

Crystal	n_2 (esu)	χ^3 (esu)	Reference
BTZC-KDP	5.47×10^{-12}	2.33×10^{-5}	Present case
KDP	2.34×10^{-13}	3.72×10^{-14}	[6]
FA-KDP	-1.14×10^{-5}	3.81×10^{-7}	[6]
CA-KDP	-6.1×10^{-16}	7.39×10^{-4}	[7]
OA-KDP	2.25×10^{-5}	1.90×10^{-7}	[8]
MA-KDP	7.92×10^{-5}	2.13×10^{-7}	[8]

Table 3. LDT data.

Crystal	LDT value(GW/cm^2)	Reference
BTZC-KDP	1.64	Present case
KDP	0.2	[30]
Urea	1.5	[30]

3.2. Thermal analysis

The thermogravimetric and differential thermal analysis (TG/DTA) of BTZC-KDP crystal has been recorded in a homogeneous nitrogen atmosphere in the range of 30–500 °C by employing the temperature increase at a rate of 10 °C/min. The thermogram recorded using the Shimadzu DTG-60H thermal analyzer is shown in Figure 3(a). The presence of solvent inclusion is a more prone phenomenon at lower temperatures, however; the analysis of TG curve reveals no decomposition of material up to 221 °C confirming the absence of water or solvent inclusions in the grown BTZC-KDP crystal material. Further the DTA curve shows an absence of phase transition peak of KDP crystal which is at 193 °C [3] while the major endothermic peaks were consequently observed at 228 and 266 °C. The simultaneous weight loss has been observed in minor steps in the temperature range 221–338 °C. The major and sharp endothermic peak in DTA curve at 228 °C confirms the preliminary melting point of grown crystal which is vital parameter to subject the crystal for laser application.

3.3. Dielectric studies

The dielectric constant and dielectric loss of pure and BTZC doped KDP crystal have been examined at a constant frequency of 100 kHz in the temperature range of 40–120 °C using the HIOKI 3532 LCR tester. In order to gain accuracy in measurements the surface of respective crystal samples were finely polished and applied by the silver paste. The response of dielectric constant with applied temperature is shown in Figure 3(b). The dielectric constant is facilitated by the relaxation time of molecular dipoles which are controlled by the polarization mechanisms associated with the material. Eventually, the dielectric constant of bulk materials is attributed to the combined effect of electronic, ionic, dipolar and space charge polarization activity [31]. It is observed that at higher temperature the magnitude of dielectric constant increases which is attributed by dominance of space charge polarization mechanism [32,33]. At the same time, it is noteworthy that the BTZC-KDP crystal contributes significantly lower dielectric constant relative to KDP crystal. The exhibition of the lower dielectric constant by BTZC-KDP crystal outstand it as a potential candidate for applications in designing broad band electro-optic modulators, optoelectronics, and field detector devices [34,35]. The dielectric loss in a bulk crystal is majorly associated with the intrinsic and extrinsic defects [36] which lead to the dissipation of electromagnetic energy. The variation of dielectric loss

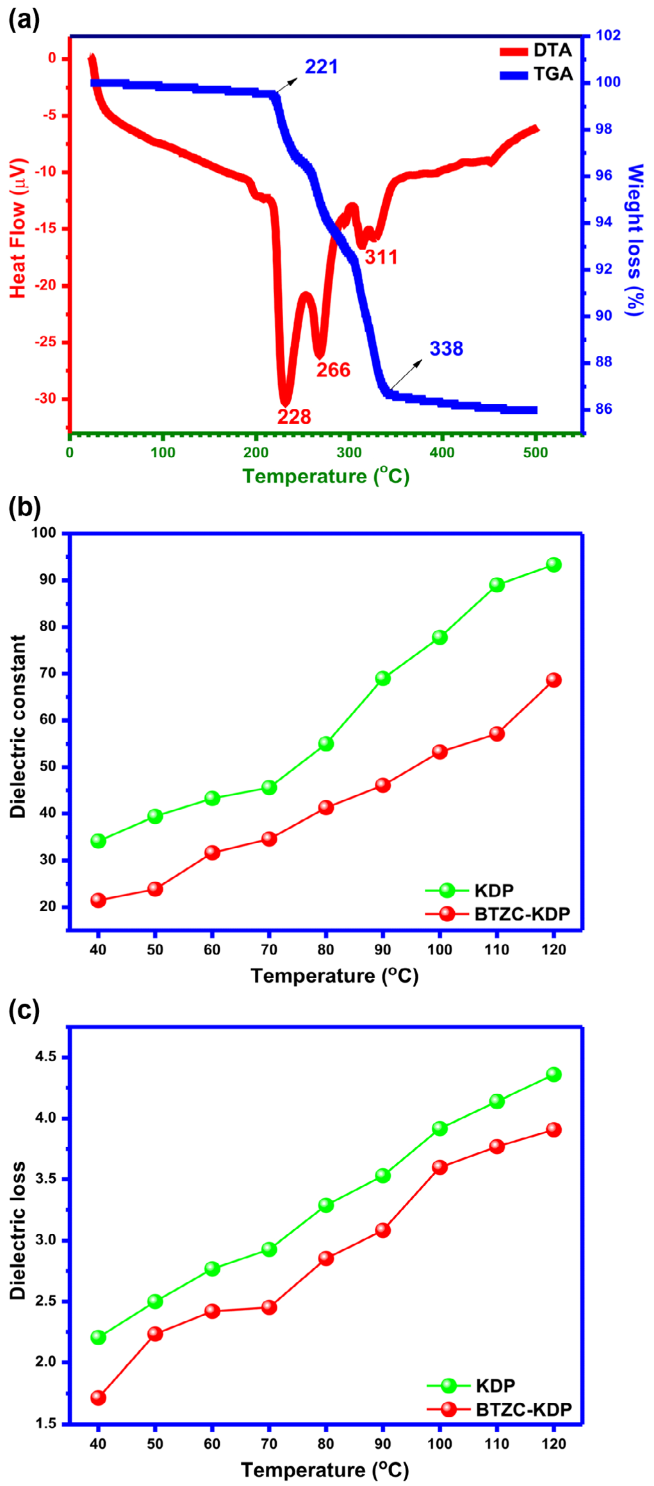

Figure 3. (a) TG-DTA thermogram, (b) temperature dependent dielectric constant and (c) temperature dependent dielectric loss.

is depicted in Figure 3(c). The dielectric loss of BTZC-KDP crystal is found to be lower than KDP which confirms that the doped KDP crystal possesses minimum electrically active defects which are vital to sustain dissipation of energy through the crystal medium [37]. The lower dielectric constant and dielectric loss of BTZC-KDP crystal suggest its potential candidature for microelectronics and photonics devices [38,39].

4. Conclusions

The optical quality BTZC-KDP bulk single crystal of dimension 34 × 10 × 06 mm^3 has been grown by slow evaporation solution technique. The PL analysis revealed the prominent blue colored emission with the maximum intensity of emission centered at 439 nm. The absence of intermediate electronic states confirmed the predominant electronic purity of grown crystal. The Z-scan analysis revealed the promising TONLO nature of BTZC-KDP crystal at 632.8 nm. The close aperture Z-scan analysis revealed the origin of negative nonlinear refraction. The BTZC-KDP crystal with n_2 of magnitude 5.47×10^{-12} esu could be a suitable candidate for optical night vision sensor devices. The dominance of multiphoton absorption effect confirmed the presence of RSA phenomenon in BTZC-KDP crystal. The magnitude of third order nonlinear absorption coefficient β is found to be 9.47×10^{-6} cm/W. The cubic susceptibility (χ^3) of BTZC-KDP is found to be 2.33×10^{-12} esu. The LDT of BTZC-KDP crystal determined using the Nd:YAG laser is found to be 1.64 GW/cm^2 which is higher than KDP crystal under same experimental conditions. The BTZC-KDP crystal is found thermally stable up to 221 °C and the major decomposition is found to be at 338 °C. The DTA curve evidences the gradual melting of compound throughout the temperature range. The dielectric constant and dielectric loss of KDP crystal have been successfully tuned to lower magnitude due to an addition of BTZC. The overall impression of above studies concludes that the BTZC-KDP crystal exhibits potential features which are highly desirable for designing optoelectronics and photonics devices.

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Disclosure statement

No potential conflict of interest was reported by the authors.

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