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# Doping effect of L-cystine on structural, UV–visible, SHG efficiency, third order nonlinear optical, laser damage threshold and surface properties of cadmium thiourea acetate single crystal

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ABSTRACT

The present article is focused to investigate the influence of L-cystine (LC) on linear-non-linear optical and laser damage threshold of cadmium thiourea acetate (CTA) crystal. The structural parameters of pure and LC doped CTA crystals have been determined using the single crystal X-ray diffraction technique. The functional groups of grown crystals have been identified by means of fourier transform infrared (FT-IR) analysis. The UV–visible spectral analysis has been done in the range of 200–900 nm to ascertain the uplifting influence of LC on optical properties of CTA crystal. The second harmonic generation (SHG) efficiency of LC doped CTA crystal is found to be higher than CTA and KDP crystal. The Z-scan technique has been employed to determine the third order nonlinear optical (TONLO) nature of LC doped CTA crystal at 632.8 nm. The self focusing tendency confirmed the strong kerr lensing ability of LC doped CTA crystal. The TONLO susceptibility ($\chi^3$), refraction ($n_2$) and absorption coefficient ($\beta$) has been calculated using the Z-scan data. The laser damage threshold of pure and LC doped CTA crystals has been measured using the Q-switched Nd:YAG laser and its is found to be in range of GW/cm$^2$. The surface analysis has been done by means of etching studies.

## 1. Introduction

The rapid development of nonlinear optical (NLO) thiourea metal complex (TMC) crystals has drawn huge attention due to its allied organic and inorganic features such as structural diversity, good transparency, large nonlinearity, huge laser damage threshold, high thermal stability and adequate mechanical hardness. The possession of these qualities make the TMC crystals most desirable for laser fusion systems, optical switching and data storage devices, telecommunication practices, optoelectronics and advanced NLO applications [1,2]. The growth and extensive study on TMC crystals namely zinc thiourea sulphate, bis thiourea cadmium chloride, potassium thiourea iodide, zinc thiourea chloride, bis thiourea zinc acetate, copper thiourea chloride, potassium thiourea bromide and cadmium thiourea acetate (CTA) have been reported [3,4]. As doping of external impurity plays crucial role in tuning the properties of host crystal, the most effective strategy to gain improvement in intrinsic properties of TMC crystals is to incorporate organic and inorganic impurities in a selected quantity [5]. In order to gain enhanced optical performance the TMC crystal is to be doped with organic additive offering good photo chemical stability, high charge mobility and strong polar density as such inherited by amino acids. The significant enhancement in crystal perfection, optical transparency, second harmonic generation (SHG) efficiency, mechanical strength and dielectric properties of CTA crystal has been achieved by doping amino acids (glycine and L-alanine) [6]. The L-cystine (LC) is an optically active amino acid with wide hydrogen bonding network and chiral centers which are essential qualities to enhance the optical properties of material [7]. Hitherto not a single researcher has made an attempt to investigate the influence of LC on different properties of CTA crystal. In this submission the structural, etching, UV–visible, SHG efficiency, third order nonlinear optical and laser damage threshold

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studies of LC doped CTA crystal have been concluded to explore distinguished laser assisted NLO device applications.

## 2. Experimental procedure

The CTA has been synthesized by dissolving cadmium acetate and thiourea 1:2 mol ratio in double distilled de-ionized water. The purity of CTA metal complex was gained by repetitive recrystallization process. The purified CTA compound was then dissolved in water to obtain the supersaturated solution. The measured quantity of 2 mol% of LC was added to supersaturated solution of CTA and allowed to stir at homogeneous speed for six hours. The LC doped CTA solution was filtered and kept for slow solution evaporation in a constant temperature bath at 38 °C. The as grown CTA and LC doped CTA (LC-CTA) single crystals are shown in Fig. 1.

## 3. Results and discussion

### 3.1. Single crystal X-ray diffraction (XRD) analysis

The grown crystals were subjected to single crystal XRD analysis using the Enraf Nonius CAD4 single crystal X-ray diffractometer. The XRD data shown in Table 1 reveals that the pure and LC-CTA crystals belong to orthorhombic crystal system. The structural parameters of CTA crystal are in good agreement with literature [5]. The cell parameters of LC-CTA crystal are slightly changed with reference to CTA which might have been occurred due to strain imposed on lattice cites of CTA crystal by dopant LC.

### 3.2. Fourier transform infrared (FT-IR) analysis

The functional groups of grown crystals have been identified by means of FT-IR spectral analysis using the Bruker α-ATR spectrophotometer. The FT-IR spectrum of single crystals was recorded in the range of 600–4000 $\text{cm}^{-1}$ and the recorded spectrum is shown in Fig. 2a. The peak observed at 687 $\text{cm}^{-1}$ is contributed due to C–C bond deformation in thiourea. The C=S stretching vibration is attributed at 1459 $\text{cm}^{-1}$. The characteristic $\text{COO}^-$ stretching vibration associated with acetate ion is expressed at 1526 $\text{cm}^{-1}$. The $\text{NH}_2$ bending vibration is evident at 1652 $\text{cm}^{-1}$. The functional peak observed at 1693 $\text{cm}^{-1}$ confirms the C=O stretching vibration. The broad absorption bands associated with C=O stretching of carboxyl group of LC are expressed at wavenumber 1740 $\text{cm}^{-1}$ and 1883 $\text{cm}^{-1}$. The absorption peak at 2360 $\text{cm}^{-1}$ is contributed by the C–C bond stretching vibration. The N–H stretching is attributed within 3610–3860 $\text{cm}^{-1}$. In Fig. 2a, the identified shift in functional frequencies confirms the incorporation of dopant LC in CTA crystal.

**Table 1**
Single crystal XRD data.

| Crystal | a (Å) | b (Å) | c (Å) | Volume $(\text{Å})^3$ | Crystal system |
|---|---|---|---|---|---|
| CTA | 7.56 | 11.87 | 15.67 | 1406.18 | Orthorhombic |
| LC-CTA | 7.54 | 11.82 | 15.89 | 1416.16 | Orthorhombic |

### 3.3. Linear optical studies

The high optical quality is a prerequisite demand for material to possess NLO properties. The optical transmittance of unpolished 2 mm thickness pure and LC-CTA crystal has been recorded within 200–900 nm using the Shimadzu UV-2450 spectrophotometer as shown in Fig. 2b. In a bulk crystal, the transmittance is majorly influenced by chemical composition and defect density. The spectrophotometer records the maximum transmittance of 55% for CTA crystal and 72% transmittance for LC-CTA crystal in visible region. The transmittance of LC-CTA crystal is found to be enhanced by 17% as compared to CTA. This ensures that LC-CTA crystal offers less scattering and absorption of light owing to least absorption tendency of amino acid [8] and minimized defects (structural and crystalline) [9] which might have been enforced by dopant LC resulting to substantial enhancement in %transmittance of LC-CTA crystal. The high transparency and low absorption tendency as observed in LC-CTA crystal may serve advantage for UV-tunable lasers and NLO devices active in blue and green spectrum [10].

The detail analysis of optical constants gives the idea of optical quality of crystal which plays decisive role in processing, tuning, calibrating and designing the technological devices. Thus, the influence of LC on optical conductivity ($\sigma_{\text{op}}$), extinction coefficient (K), refractive index (n) and reflectance (R) of CTA crystal has been investigated using the measured transmittance data. The variation of optical conductivity and extinction coefficient with reference to photon energy is shown in Fig. 3a and b respectively. It reveals that the optical conductivity increases with increase in photon energy while the reduced extinction coefficient of LC-CTA crystal facilitates less optical loss. The quality of less optical loss with increasing optical conductivity makes LC-CTA crystal highly desirable for ultrafast optical data processing, computing and signaling devices [11]. The characteristic property of material to change the path of light when passed through a medium is termed as refractive index. The change in refractive index and reflectance of grown crystals is plotted in Fig. 3c and d. It confirms that the refractive index and reflectance of CTA has been modified by LC to lower value. The materials with low refractive index are readily used in holographic data storage utilities [12] also; they find huge application as antireflection coating material for solar thermal devices [13]. The LC-CTA crystal with improved linear optical performance is suggested as potential material to be utilized in NLO device applications.

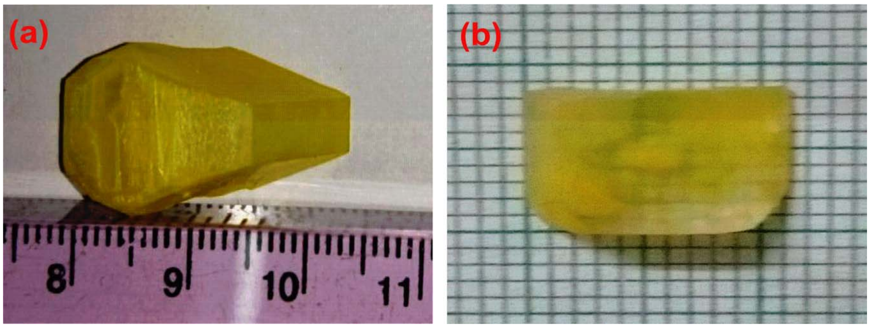

**Fig. 1.** Single crystal of (a) CTA (b) LC-CTA.

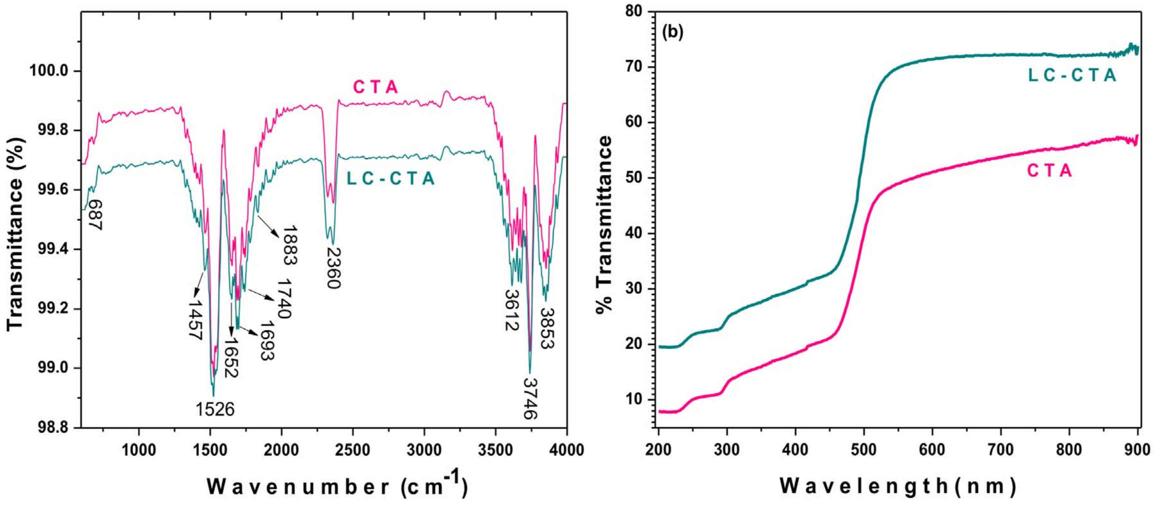

**Fig. 2.** (a) FTIR spectrum (b) Transmittance spectrum.

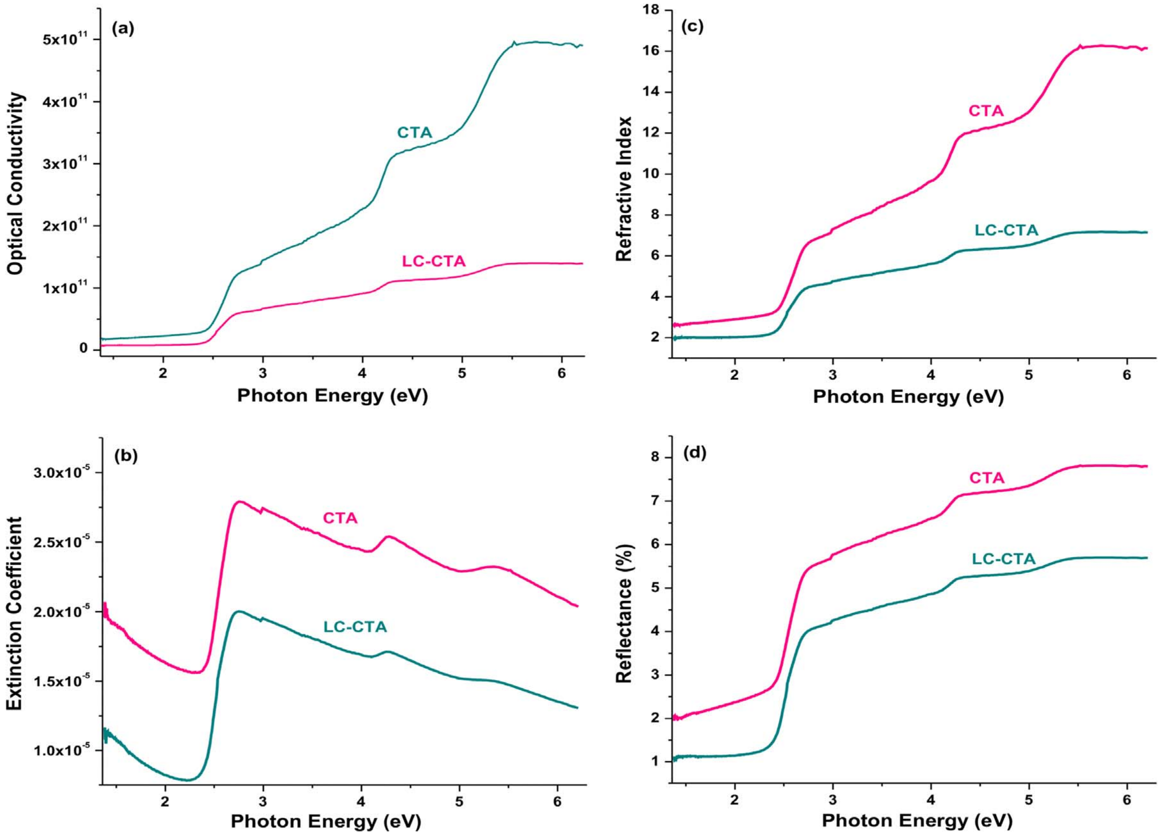

**Fig. 3.** Photonic response of (a) optical conductivity (b) extinction coefficient (c) refractive index (d) reflectance.

### 3.4. *Nonlinear optical studies*

#### 3.4.1. *SHG efficiency test*

The laser frequency doubling effect in grown crystals has been investigated using the kurtz and perry powder technique. In present analysis the Q-switched mode Nd:YAG laser operating at 1064 nm with repetition rate of 10 Hz and pulse width 8 ns has been used. The perfectly grown crystals were powdered and packed in a micro-capillary tube of uniform bore. The prepared samples were multishot normally by a gaussian laser beam and the generated output signal from the sample was collected through the array of photomultiplier tube. The emergence of sharp green light at the output confirmed the NLO behavior of materials. The optical signal of the sample was recorded in terms of voltage using the digital oscilloscope and the output voltage for KDP, CTA and LC-CTA crystal sample was 5 mV, 6.2 mV and 8.4 mV respectively. The SHG efficiency of LC-CTA crystal is found to be 1.68 times greater than KDP and 1.35 times greater than CTA material. The high SHG efficiency of LC-CTA crystal is promising quality for laser frequency conversion applications.

#### 3.4.2. *Z-scan studies*

The study of third order nonlinear optical (TONLO) properties of single crystal at a particular wavelength enables to discover the utility of the crystal for NLO assisted ultrafast optical device applications. The TONLO characteristic features of LC-CTA crystal

**Table 2**
Optical resolution of Z-scan setup.

| Parameters and notations | Details |
|---|---|
| Laser wavelength (λ) | 632.8 nm |
| Lens focal length (f) | 30 mm |
| Optical path distance (Z) | 85 cm |
| Beam waist radius ($\omega_a$) | 3.3 mm |
| Aperture radius ($r_a$) | 2 mm |
| Incident intensity at the focus ($I_o$) | 5 MW/cm$^2$ |

(0.58 mm) have been determined using the Z-scan technique as demonstrated by Bahae et al. [14]. For present analysis the He-Ne laser operating at 632.8 nm has been used. The polarized gaussian laser beam has been focused on crystal sample through a converging lens of focal length 30 mm. Further the crystal sample placed at the focus (Z=0) was gradually translated along the beam irradiated path towards positive and negative Z-direction relative to focus and the transmitted intensity was recorded. The details of Z-scan setup are given in Table 2.

The closed aperture Z-scan technique was employed to measure the magnitude and observe the on axis change in third order nonlinear refraction of the crystal sample. The laser beam was made normally incident on the crystal sample through a converging lens and the sample was gradually translated back and forth the focus (Z=0) along the Z-direction. The transmitted intensity measured with changing Z position was recorded using the partially closed aperture of photo detector placed at far field. The change in nonlinear refraction (NLR) of LC-CTA crystal is observed in closed aperture Z-scan curve shown in Fig. 4a. It shows low transmittance in negative Z-direction i.e. pre-focus valley and high transmittance in positive Z-direction i.e. post-focus peak evidencing the presence of positive NLR in LC-CTA crystal which is the intrinsic quality of the material exhibiting self focusing tendency [15]. Similar results were observed in MMP and SSH crystals grown by solution growth technique [15,16]. The focused repetitive optical energy of laser beam is the key element which causes the localized absorption and spatial distribution of optical energy along the crystal surface causing the on axis phase shift ($\Delta\phi$) in NLR ($n_2$) about the focus for a particular crystal [17,18]. The peak to valley transmission ($\Delta T_{p\text{-}v}$ ) can be expressed in terms of phase shift as [17],

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

where $S=[1-\exp(-2r_a^2/\omega_a^2)]$ is the aperture linear transmittance, $r_a$ is the aperture radius and $\omega_a$ is the beam waist radius in front of aperture. The $n_2$ of crystals has been determined using the relation [17],

$$n_2 = \frac{\Delta\varphi}{KI_0L_{eff}} \tag{2}$$

where $K=2\pi/\lambda$, $I_0$ is the incident irradiance intensity of beam at the focus (Z=0), the effective thickness of the sample $L_{eff}=[1-\exp(-\alpha L)]/\alpha$ where, ($\alpha$) is the linear absorption coefficient and L is the thickness of the sample. The strong phase shifting ability about the focus i.e. positive NLR tendency signifies the prominent kerr lens modelocking (KLM) capability of LC-CTA crystal. The $n_2$ of LC-CTA crystal of order of $10^{-12}$ cm$^2$/W suggests its potential utility for testing the stability limits of continuous-wave mode-locked laser systems and generating the shorter laser pulses [19]. The open aperture Z-scan trace of LC-CTA crystal shown in Fig. 4b reveals the suppressed transmittance at focus evidencing the reverse saturable absorption (RSA) effect derived by the multi-photon absorption (MPA) phenomenon in excited states [20]. The MPA is a simultaneous complex event which is originated due to allied two-photon absorption (TPA) along with the absorptions associated with excited singlet and triplet states [20,21]. The nonlinear absorption coefficient ($\beta$) of LC-CTA crystal is found to be of order $10^{-5}$ cm/W which is superior to CTA, LV-CTA and other potential crystals [17,22]. The $\beta$ value has been evaluated using the equation [17].

$$\beta = \frac{2\sqrt{2\Delta T}}{I_0L_{eff}} \tag{3}$$

where $\Delta T$ is the one valley value obtained in open aperture Z-scan curve. The TONLO susceptibly ($\chi^3$) of the grown crystal has been analyzed by solving the following equations [17],

$$\mathrm{Re}\chi^{(3)}(esu) = 10^{-4}\left(\epsilon_0C^2n_0^2n_2\right)/\pi\left(\mathrm{cm}^2/\mathrm{W}\right) \tag{4}$$

$$\mathrm{Im}\chi^{(3)}(esu) = 10^{-2}\left(\epsilon_0C^2n_0^2\lambda\beta\right)/4\pi^2\left(\mathrm{cm}/\mathrm{W}\right) \tag{5}$$

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

where $\varepsilon_0$ is the vacuum permittivity, $n_0$ is the linear refractive index of the sample and c is the velocity of light in vacuum. The

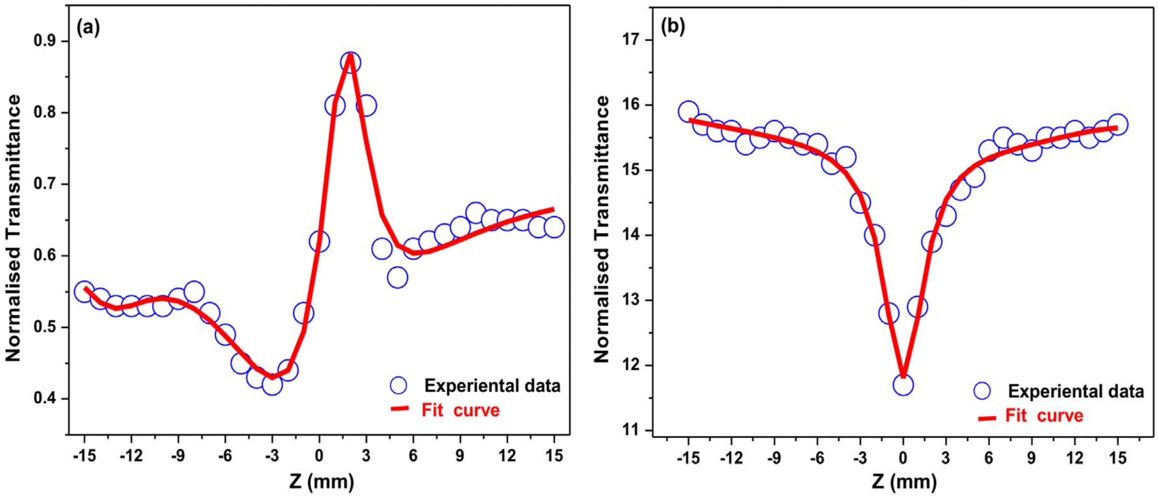

**Fig. 4.** (a) Close and (b) Open aperture Z-scan transmittance curve.

**Table 3**
TONLO parameters.

| Crystal | $n_2$ cm$^2$/W | β cm/W | $\chi^3$ esu | FOM |
|---|---|---|---|---|
| CTA [16] | $8.37 \times 10^{-11}$ | $4.70 \times 10^{-6}$ | $2.58 \times 10^{-4}$ | 35.57 |
| LC-CTA | $4.85 \times 10^{-12}$ | $1.19 \times 10^{-5}$ | $6.18 \times 10^{-5}$ | 156 |

LC-CTA crystal has a high magnitude of $\chi^3$ ($10^{-5}$ esu) which might have been facilitated by photoinduced π-electron delocalization along large bonding network also resulting to develop strong polarizing potential in crystal. The LC-CTA crystal with high $\chi^3$ is superior material than several technologically vital crystals (KDP, BBO and $LiNbO_3$) [23]. The figure of merit (FOM=$\beta\lambda/n_2$) is an essential parameter. The FOM value for LC-CTA crystal is found to be 156 which suggest its potential candidature for optical power limiting applications [24]. The LC-CTA crystal with attractive nonlinear properties (see Table 3) holds huge advantage for optical switching, optical logic gates and passive laser mode-locking systems [25]. In addition the third order nonlinear susceptibility of LC-CTA crystal is found remarkably higher than thiourea, BTZB, ZTS, BTZC and BTCF crystals [17,22,26].

### 3.5. Laser damage threshold (LDT) analysis

In addition to high linear-non-linear optical properties, the single crystal must deliver large tolerance to laser power intensity [27], to subject it to high-tech laser assisted NLO applications. Therefore, the LDT of pure and LC-CTA crystal has been determined using the Q-switched mode Nd:YAG laser (1064 nm, 10 ns, 10 Hz). Practically LDT is determined by single shot and multi shot method. In present study the LDT of grown crystal has been measured by multi laser shot method. The laser beam of 1 mm diameter was attenuated by variable attenuator and focused on respective crystal sample via converging lens of focal length 30 cm for 30 s/shot. The output energy causing the damage to crystal surface was measured using the coherent energy/power meter (Model No. EPM 200). The laser induced damage in material is governed by the allied intrinsic complex optical effects and several external optical parameters. In practical, if the pulse width of laser belongs to regime of nano seconds, the laser induced damage in a material is dominantly contributed by the localized thermal effects. The irradiation of beam causes a thermal conduction in the atomic lattice leading to photo-ionization of crystal surface in the form of melting or decomposition or crack or fusion of material, eventually resulting to damage [28]. It is found that the laser beam of energy of 253 mJ imposed a damage of 1 mm on CTA crystal while the energy of 193 mJ caused a damage of 1.8 mm on surface of LC-CTA crystal. The LDT value for pure and LC-CTA crystal is found to be 32.22 GW/cm$^2$ and 7.58 GW/cm$^2$. It is observed that the LDT of pure and LC-CTA crystal is higher by large margin as compared to KDP (0.2 MW/cm$^2$), BTZA (12.44 MW/cm$^2$) and benzimidazole (1.9 MW/cm$^2$) crystals [29].

### 3.6. Etching studies

The etching study gives the clue of reciprocity, growth habit and structural defects associated with the crystal surface. In present analysis the crystal surfaces were etched by water and patterns were recorded after definite interval of time. The etch pattern of pure and LC-CTA crystal recorded after 10 s are shown in Fig. 5a and b respectively. The etch pattern of CTA shows large number of micro-pits and randomly arranged intermixed layer pattern while the step growth pattern indicates the symmetrical growth in LC-CTA crystal along the studied plane. The similar patterns were observed after 20 s (Fig. 5c and d) for pure and LC-CTA crystal. The layered growth habit, absence of pits, less

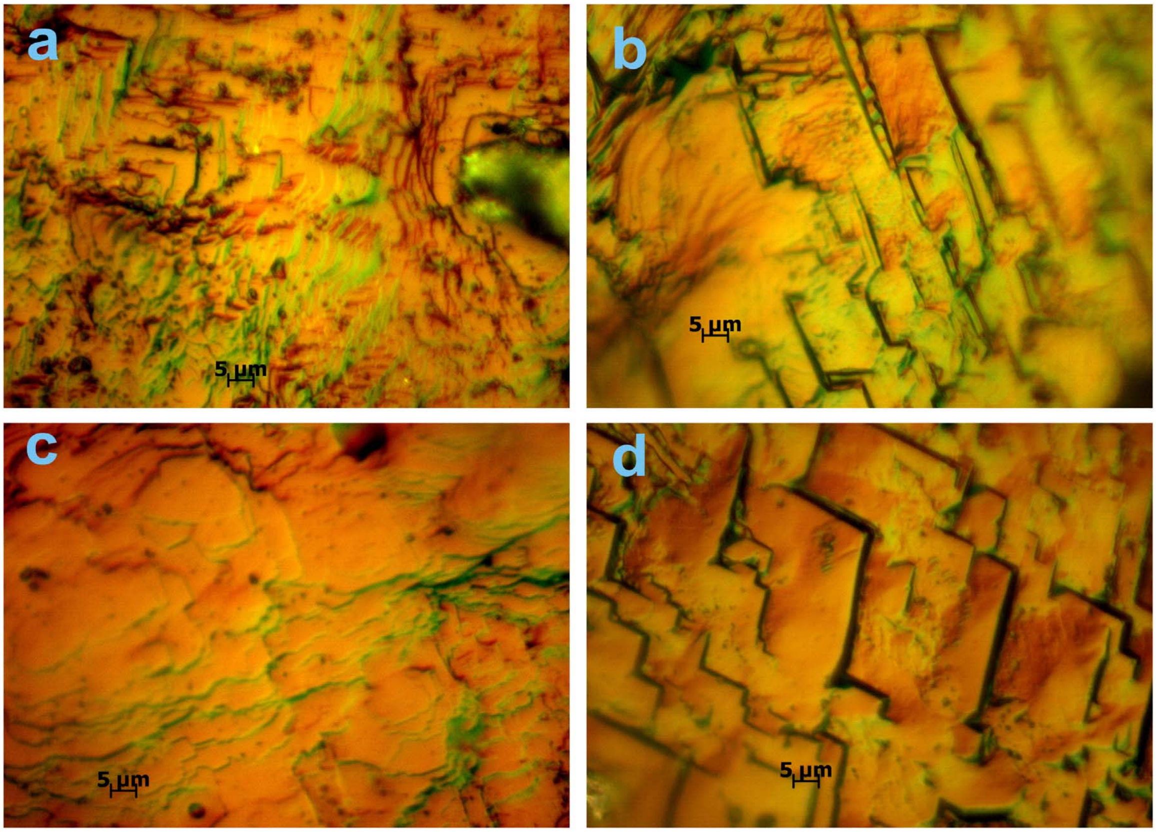

**Fig. 5.** (a) CTA and (b) LC-CTA etch pattern after 10 s, (c) CTA and (d) LC-CTA etch pattern after 20 s.

dislocations determines the two dimensional symmetrical nucleation mechanisms along the plane [30], asserting the enhancement in crystalline quality of LC-CTA crystal.

## 4. Conclusion

In present analysis good quality pure and LC-CTA crystals have been grown by slow evaporation solution technique. The XRD analysis confirmed the orthorhombic structural symmetry of grown crystals and the unit cell parameters have been determined. The FT-IR analysis confirmed the functional groups of grown crystal. UV–visible studies revealed that LC-CTA crystal has higher optical transparency, increasing optical conductivity, lower extinction coefficient, less refractive index and reflectance with reference to CTA crystal which are vital for distinct NLO applications. The promising NLO behavior of LC-CTA crystal has been confirmed by means of SHG efficiency test and Z-scan analysis. The SHG efficiency of LC-CTA crystal is found to be 1.68 times that of KDP and 1.35 times higher than CTA crystal. The positive NLR or self focusing tendency of LC-CTA crystal revealed the strong kerr lens modelocking ability of the crystal confirming its selective utility for laser stabilization and shorter pulse generation systems. The origin of photoinduced multiphoton absorption confirmed the existence of reverse saturable absorption in LC-CTA crystal with a magnitude of $1.19 \times 10^{-5}$ cm/W. The enhanced charge mobility and strong polarizing nature resulted to high FOM and excellent TONLO susceptibility ($6.18 \times 10^{-5}$ esu) in LC-CTA crystal. The localized photo-ionization due to thermal effects originated the laser induced surface damage in pure and LC-CTA crystals. The LDT of CTA and LC-CTA crystal is found to be 32.22 GW/cm$^2$ and 7.58 GW/cm$^2$. The surface analysis through etching studies disclosed the symmetrical layered growth habit, less pits and minimum dislocations in LC-CTA crystal confirming its enhanced quality and crystalline perfection. All above studies concur that LC-CTA crystal is a promising optical material with adequate LDT which can be used for laser assisted NLO applications such as mode-locking, photonics, optical limiting, SHG, data processing, holographic data storage devices vital for high-tech technological systems.

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