

## Studies on $\text{Ag}^{8+}$ and $\text{Li}^{4+}$ ions irradiated LAHCl single crystals

K. Sangeetha<sup>a</sup>, R. Ramesh Babu<sup>a,\*</sup>, K. Ramamurthi<sup>a</sup>, Furan Singh<sup>b</sup>, K. Asokan<sup>b</sup>

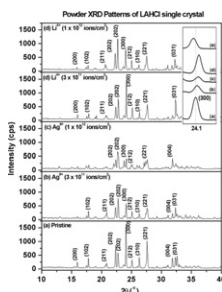
<sup>a</sup> Crystal Growth and Thin film Laboratory, Department of Physics, Bharathidasan University, Tiruchirappalli 620 024, Tamil Nadu, India

<sup>b</sup> Inter-University Accelerator Centre, P.O. Box 10502, Aruna Asaf Ali Marg, New Delhi 110 067, India

### HIGHLIGHTS

- ▶ LAHCl single crystals were irradiated with  $\text{Ag}^{8+}$  and  $\text{Li}^{4+}$  ions for the first time.
- ▶ Dielectric constant and hardness of the LAHCl increased at low ion fluence.
- ▶ Increased dielectric constant in irradiated LAHCl may enhance the electro-optic coefficient.
- ▶ The difference in effect of  $\text{Ag}^{8+}$  and  $\text{Li}^{4+}$  on physical properties LAHCl was also discussed.

### GRAPHICAL ABSTRACT



### ARTICLE INFO

#### Article history:

Received 30 January 2012

Received in revised form

19 October 2012

Accepted 21 October 2012

#### Keywords:

- B. Crystal growth
- D. Irradiation effects
- C. Powder diffraction
- D. Dielectric properties

### ABSTRACT

Semi-organic single crystals of LAHCl were grown by unidirectional solution growth method. The grown single crystals were subjected to  $\text{Ag}^{8+}$  and  $\text{Li}^{4+}$  ions irradiation of energy 100 MeV and 50 MeV, respectively. Breaking of bonds in the irradiated LAHCl molecules and the lattice deformations are analyzed by Fourier transform infrared spectroscopy and X-ray diffraction studies, respectively. The modifications induced by ion irradiation in dielectric, mechanical stability and nonlinear optical property are studied at different ion fluence.

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### 1. Introduction

When energetic ions from accelerators made to hit single crystals, transformation of energy from ion to single crystal take place mainly by two processes, elastic and inelastic collisions. The elastic collision is dominant in low energy ion irradiation and inelastic collision is dominant in high energy ion irradiation. This energy transfer induces lattice deformation and defects in the crystal that changes the properties of the crystal. In organic single crystals, ion irradiation creates ion tracks (i.e. defects with cylindrical volume) which increase the dielectric constant and refractive index of the

crystal [1]. Hence, ion irradiation is an important tool to tune the properties of the single crystals. Semi-organic single crystals are a new class of nonlinear optical crystals which are formed by the combination of organic and inorganic compounds. Semi-organic single crystals have the merits of both organic and inorganic crystals. L-Arginine monohydrochloride monohydrate (LAHCl) is a potential nonlinear optical crystal comes under the class of semi-organic and its structural [2], mechanical [3], piezoelectric [4], thermal, optical, nonlinear [5] and dielectric properties [6] were reported. In this paper, the effect of  $\text{Ag}^{8+}$  and  $\text{Li}^{4+}$  ions irradiation on LAHCl single crystals is presented. The ions interaction with the crystal is mostly in the form of electron interactions. The modifications induced by ion irradiation in dielectric, mechanical and nonlinear optical properties are studied at different ion fluence.

\* Corresponding author. Tel.: +91 431 2407057; fax: +91 431 2407045.  
E-mail address: [rampap2k@yahoo.co.in](mailto:rampap2k@yahoo.co.in) (R. Ramesh Babu).

## 2. Experimental details

LAHCl single crystals were grown by unidirectional solution growth method [7] along  $\langle 001 \rangle$  orientation. LAHCl single crystals grown by slow solvent evaporation method were used as seed crystals for unidirectional growth. A cylindrical glass ampoule [7] is loaded with seed crystal at the bottom with (001) plane facing the saturated solution that is filled above the seed. The ampoule is then placed inside a constant temperature bath and the bottom portion of the ampoule is cooled in order of  $0.5\text{ }^\circ\text{C/day}$  to nucleation temperature ( $23\text{ }^\circ\text{C}$ ) at which the seed crystal started its growth. The unidirectionally grown LAHCl crystals were cut and polished in  $7\text{ mm} \times 5\text{ mm} \times 1\text{ mm}$  dimension and mounted on a target ladder. The ladder was placed inside a chamber and evacuated to about  $10^{-8}$  mbar. These crystals were irradiated with  $\text{Ag}^{8+}$  and  $\text{Li}^{4+}$  ions of energy 100 MeV and 50 MeV, respectively using Pelletron accelerator at Inter-University Accelerator Centre, New Delhi. The ion fluence was varied from  $1 \times 10^{11}$  to  $1 \times 10^{12}$  ions  $\text{cm}^{-2}$ . During irradiation the ion beam current was kept constant at 1 pA for an area of about  $1\text{ cm}^2$ .

The stopping range of  $\text{Li}^{4+}$  and  $\text{Ag}^{8+}$  ions in LAHCl crystal lattice (density is  $1.446\text{ g cm}^{-3}$ ) was calculated using the software 'Stopping Range of Ions in Matter' (SRIM 2008) and is presented in Table 1.

The structural disturbance induced by irradiated ions was analyzed by powder X-ray diffraction recorded using SIEFERT X-ray diffractometer and the unit cell parameters were calculated using CHECKCELL software [8]. Fourier transform infrared spectra for pristine and irradiated crystals were recorded using Perkin–Elmer Fourier transform infrared spectrometer in the range  $400\text{--}4000\text{ cm}^{-1}$ . The relative dielectric constant ( $\epsilon'$ ) were measured for (001) LAHCl crystal plates using HIOKI 3532-50 LCR Hi-TESTER in the range 1 kHz–5 kHz and the mechanical stability was measured using microhardness tester (HMV – 2) fitted with a Vickers diamond pyramidal indenter and attached with an optical microscope. Further, second harmonic generation efficiency was measured for pristine and  $\text{Ag}^{8+}$  and  $\text{Li}^{4+}$  ions irradiated LAHCl crystalline powders using Nd:YAG laser of wavelength 1064 nm with pulse width 5 ns.

## 3. Results and discussion

### 3.1. SRIM calculations

It is observed from Table 1 that low mass and energy of  $\text{Li}^{4+}$  ions made it to penetrate a greater range of  $379.31\text{ }\mu\text{m}$  than  $\text{Ag}^{8+}$  ions ( $16.52\text{ }\mu\text{m}$ ) of high mass and energy. But the electronic and nuclear energy losses are high for  $\text{Ag}^{8+}$  ions when compared to  $\text{Li}^{4+}$  ions. This confirms that the effect induced by  $\text{Ag}^{8+}$  ions would be more than  $\text{Li}^{4+}$  ions in LAHCl single crystals.

### 3.2. Powder X-ray diffraction analysis

Fig. 1 (a–e) shows the powder XRD patterns of pristine,  $\text{Ag}^{8+}$  and  $\text{Li}^{4+}$  ions irradiated LAHCl crystals at ion fluence  $3 \times 10^{11}$  and  $1 \times 10^{12}$  ions  $\text{cm}^{-2}$ . When the ion fluence increases, the diffraction

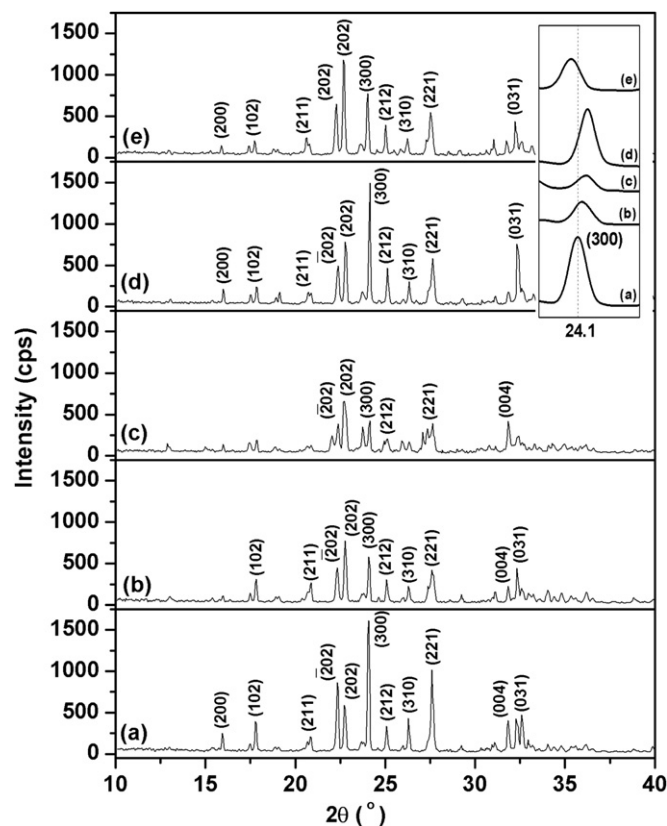


Fig. 1. XRD patterns of LAHCl (a) pristine,  $\text{Ag}^{8+}$  irradiated at (b)  $3 \times 10^{11}$  ions  $\text{cm}^{-2}$ , (c)  $1 \times 10^{12}$  ions  $\text{cm}^{-2}$ ,  $\text{Li}^{4+}$  irradiated at (d)  $3 \times 10^{11}$  ions  $\text{cm}^{-2}$ , (e)  $1 \times 10^{12}$  ions  $\text{cm}^{-2}$ .

peaks are (i) decreased in intensity, (ii) broadened and (iii) shifted from its position. These observations clearly confirm the localized lattice deformations and decrease in crystallinity [9] of the irradiated LAHCl. The lattice deformation is localized because the diffraction peaks are observed even at high fluence which indicates the unaffected planes in the irradiated crystals. The shift in diffraction peak position of (300) plane is clearly shown in the inset of Fig. 1. Further, it is observed that the decrease in diffraction intensities are high for  $\text{Ag}^{8+}$  ions irradiated LAHCl compared to  $\text{Li}^{4+}$  ions irradiated LAHCl. This shows the greater effect of  $\text{Ag}^{8+}$  ions in LAHCl crystal because  $\text{Ag}^{8+}$  ions have higher mass and energy than  $\text{Li}^{4+}$  ions.

The calculated unit cell parameters (Table 2) were disturbed slightly due to irradiation because ions traveling into the crystal lattice create volume defects through the displacement of atoms around its track.

### 3.3. Fourier transform infrared (FTIR) spectroscopy

The FTIR spectra of pristine and  $\text{Ag}^{8+}$  and  $\text{Li}^{4+}$  ions irradiated LAHCl are shown in Fig. 2(a). Fig. 2(b) represents absorption intensities of  $\text{COO}^-$  rocking (1),  $\text{COO}^-$  wagging (2), N–H wagging (3), C–CH in-plane deformation (4) and  $\text{H}_2\text{O}$  asymmetric stretching (5) vibrations observed for pristine and  $\text{Ag}^{8+}$  and  $\text{Li}^{4+}$  irradiated LAHCl. The intensity and sharpness of absorption of the above vibrations decrease with increasing ion fluence for irradiated crystals when compared to the absorption intensities of the same vibrations in pristine LAHCl. This indicates that the bonds were broken during ion irradiation as the traveling of ions initiates bond breaking and side chain scission processes in the LAHCl molecule by transferring its energy [10,11]. The bond breakings, side scission

Table 1  
SRIM calculations for  $\text{Li}^{4+}$  and  $\text{Ag}^{8+}$  ions irradiated LAHCl single crystals.

Ions	Energy (MeV)	Electronic energy loss ( $\text{eV } \text{Å}^{-1}$ )	Nuclear energy loss ( $\text{eV } \text{Å}^{-1}$ )	Projected range ( $\mu\text{m}$ )
$\text{Li}^{4+}$	50	7.532	0.0043	379.31
$\text{Ag}^{8+}$	100	1071.92	4.567	16.52

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