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Investigation of gamma radiation effect on chemical properties and surface morphology of some nonlinear optical (NLO) single crystals

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ABSTRACT

The effect of Co-60 gamma irradiation on L-alanine cadmium chloride (LACC), L-alanine doped potassium dihydrogen orthophosphate (KDP) and L-arginine doped KDP nonlinear optical (NLO) single crystals were studied in doses ranging from 100 krad to 6 Mrad. The crystals were grown by slow evaporation method at room temperature. The effects of gamma irradiation on the chemical, surface morphology, DC electrical conductivity, thermal and mechanical properties of the grown crystals have been studied. The functional groups of unirradiated and irradiated crystals have been identified and confirmed by Fourier transform infrared (FTIR) studies. Scanning electron microscopy (SEM) of irradiated crystals shows some morphological changes in the crystals. The dc conductivity of LACC and L-alanine doped KDP crystals were found to increase with increase in radiation dose whereas in case of L-arginine doped KDP crystals, the dc conductivity was found to decrease with increase in radiation dose. Differential scanning calorimetry (DSC) thermograms reveals that there is no significant change in the melting point of the crystals after irradiation and the crystals does not decompose as a result of irradiation. The mechanical behavior of both unirradiated and irradiated crystals is explained with the indentation effects using Vicker's microhardness tester. The Vicker's hardness number H_V and Mayer's index 'n' has been estimated and confirms that LACC belong to the hard materials.

1. Introduction

Recent research interest is focused on the search of new semi organic nonlinear optical (NLO) materials, as these materials share the advantages of both inorganic (high thermal and mechanical stability) and organic (broad optical frequency range and second harmonic conversion efficiency) materials [1–10]. After the advent of laser, the nonlinear phenomena made a big revolution in the field of optics and the frequency conversion become an important and popular for laboratory lasers [11–13]. Many of solid-state lasers are designed to work in the strong external fields of ionizing radiation. Exposure of laser materials to space radiation and to high-energy photons (>30 MeV) can result forms of damage as ionization of atoms and structural damage of the laser material [14–16]. Therefore understanding the effects of different radiations on solid state materials in particular NLO crystals is an important problem and has practical applications for use in radiation rich environments. It was shown that heavy ion and gamma irradiation on crystalline materials are changing its physical and chemical properties of the specimens [17–22]. The irradiation studies are the only way of understanding the degradation mechanisms and estimation of the lifetime of the crystals in radiation environments. Recently, considerable attention has been given on a study of the electronic excitation dynamics, radiation-induced processes and defects generation in nonlinear optical crystals, because these defects affect the optical and stimulated emission properties of non-linear optical crystals [23,24]. In this paper we made an attempt to understand the Co-60 gamma irradiation effect on some important NLO crystals. LACC is semi organic amino acid crystal possessing a monoclinic system and acquire nonlinear optical properties with a second harmonic efficiency of about 1.5 times that of KDP [2–4]. The single crystals of amino acid L-alanine and L-arginine doped KDP are found to be tetragonal systems and shows second harmonic generation properties [25–27]. In the present work, LACC, L-alanine doped KDP and L-arginine doped KDP crystals were grown from low temperature solution growth method by employing slow evaporation of the solvent. The grown crystals were exposed to Co-60 gamma radiation in the total doses ranging from 100 krad to 6 Mrad. Understanding the gamma irradiation effects on structural, chemical, optical and thermal properties is important from the viewpoint of

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their technical efficacy. For the first time, we present the results of gamma irradiation effects on crystals by different techniques such as FTIR studies, scanning electron microscopes (SEM), DC conductivity studies, differential scanning calorimetry (DSC) and Vicker's microhardness test.

2. Experimental

2.1. Synthesis and crystal growth

The single crystal of LACC was synthesized from L-alanine and cadmium chloride monohydrate taken in the equi-molar ratio. The calculated amounts of the reactants were dissolved in double distilled water and stirred well for about 2 h using a magnetic stirrer at 30 °C to form a saturated solution. The solution was then filtered twice to remove the suspended impurities and allowed to crystallize by slow evaporation of solvent at room temperature. Good transparent crystals of size around 3.4 × 3 cm were obtained in a period of about four weeks and are shown in Fig. 1. L-alanine and L-arginine doped KDP single crystals were grown by dissolving purified KDP, L-alanine and L-arginine powders in appropriate amount in double distilled water and heated at a constant temperature at 43 °C with continuous stirring using magnetic stirrer for 2 h to form a saturated solution. The solution was then filtered and allowed to crystallize by slow evaporation method at room temperature. Good transparent L-alanine doped KDP crystals of size around 2 × 1.3 cm and L-arginine doped KDP crystals of size around 1.5 × 1 cm were obtained in a period of 10–15 days as shown in Fig. 1.

2.2. Irradiation methodology

The NLO single crystals of LACC, L-alanine doped KDP and L-arginine doped KDP were irradiated in Co-60 gamma chamber-5000 which is a compact, portable, self-shielded type of a Co-60 gamma irradiator located at Central Instrumentation Facility, Pondicherry University, India. The irradiation chamber has an irradiation volume of approximately 5000 cc with radial and axial dose rate uniformity within ±10%. NLO crystals were irradiated with the dose rate of 167 rad/s up to a total ionizing dose ranging from 100 krad to 6 Mrad with average gamma energy of 1.25 MeV.

3. Results and discussion

3.1. FTIR spectral analysis

The infrared spectral analysis provides useful information regarding the molecular structure and functional groups of the compound. The infrared spectrums of unirradiated and irradiated LACC, L-alanine doped KDP and L-arginine doped KDP crystals (Fig. 2) were recorded in the frequency range 400–4000 cm^{-1} using FT/IR-4100typeA, under a resolution of 16 cm^{-1} and with the scanning speed of 2 mm/s. Pellets of the mixture of each sample with KBr have been prepared and used in the experiment. The FT–IR spectrums of unirradiated and irradiated LACC crystals are shown in Fig. 2a. The NH_2 group of L-alanine is protonated by the COOH group. The presence of NH_3^+ is very easily identified in the FT-IR spectrum by the broad intense band with the absorption at 3741.2 and 3552.2 cm^{-1} corresponding to asymmetric and symmetric stretching mode of NH_3^+. The NH_3^+ symmetric stretching frequencies are overlapping with the vibrations of CH_3 group. The strong absorption at 1461.8 and 1612.2 cm^{-1} indicate the bending degenerate frequency of the carbonyl group and NH_3^+ group. The strong absorption at 1369.2 cm^{-1} indicates the bending symmetric frequency of the CH_3 group. The NH_3^+ and CH_3 rocking frequencies occur at the 1106.9 and 1014.4 cm^{-1}. The absorption at 1187.9 and 844.7 cm^{-1} corresponding to asymmetric and symmetric stretching mode of

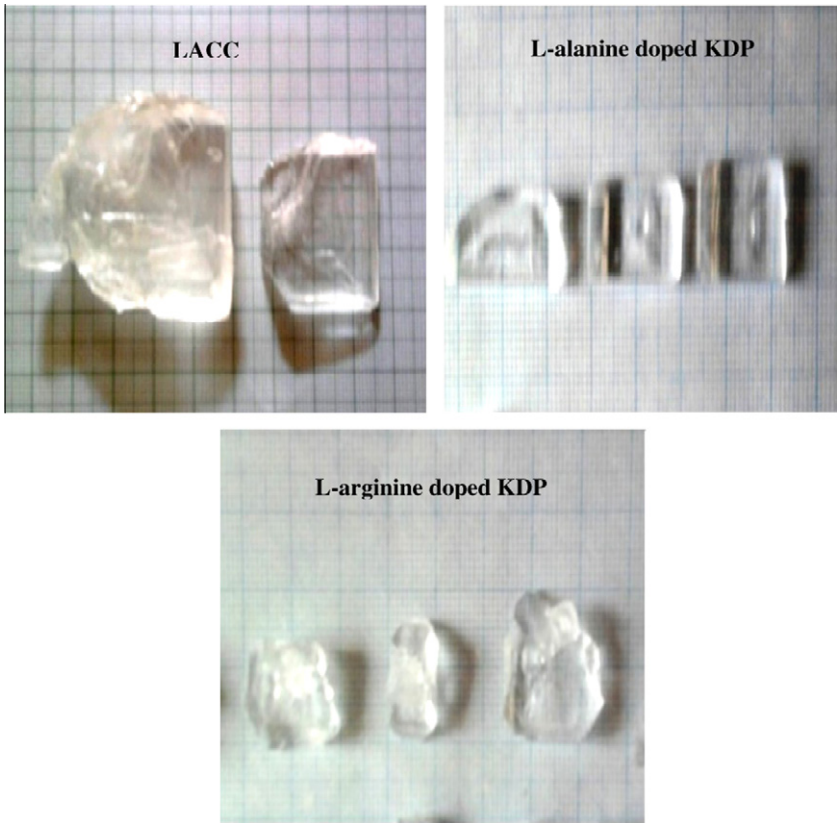

Fig. 1. The photograph of the as-grown single crystals.

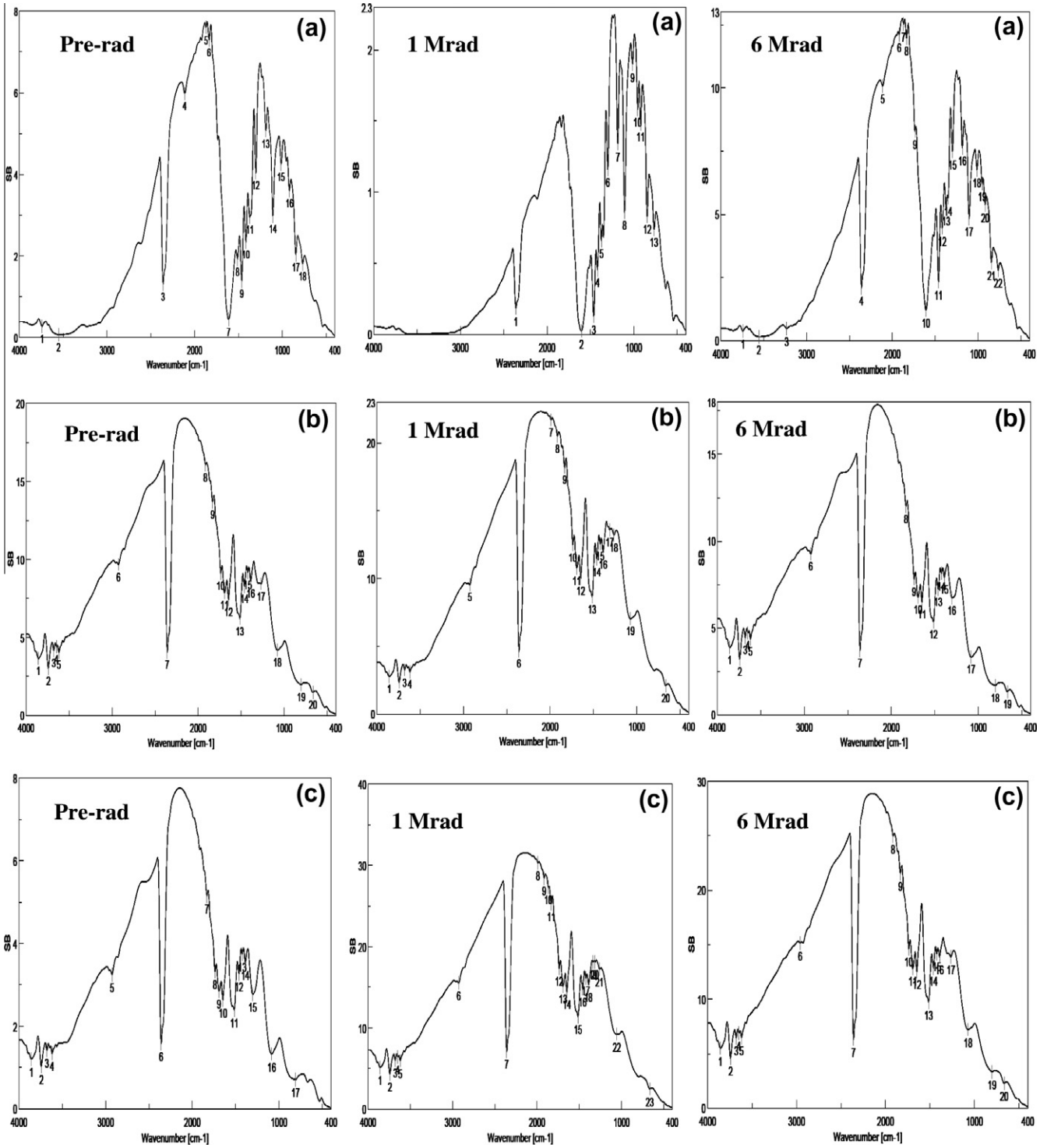

Fig. 2. FT-IR spectra of unirradiated and irradiated crystals (a) LACC, (b) L-alanine doped KDP and (c) L-arginine doped KDP crystals.

CCN. The COO^- rocking and bending frequencies occur at the 1415.5 and 767.5 cm^{-1}. The absorption peaks at 917.9 and 844.7 cm^{-1} are assigned to C–C–N symmetric stretching vibrations. These vibrations prove the presence of expected functional groups in the compound [2,3]. The FT–IR spectrums of unirradiated and irradiated L-alanine doped KDP crystals are shown in Fig. 2b. The broad envelopes observed between 2360.4 and 3856.9 cm^{-1} are due to NH_3^+ stretching vibration, O–H stretching. C–H stretching of CH_2 is observed at 2927.4 cm^{-1} and C═O stretching is revealed by minor absorption peak at1646.9 cm^{-1}. The C–O bending attributes to the absorption at 1388.5 cm^{-1}. The absorptions occur at 1079.9 and 802.24 cm^{-1} are due to CH_2 twisting and O–H bending, respectively [25,26]. The FT–IR spectrums of unirradiated and irradiated L-arginine doped KDP crystals are shown in Fig. 2c. The broad envelopes observed between 2360.4 and 3856.9 cm^{-1} are mainly due to P–OH stretching of H_2PO_4, O–H stretching of COOH, N–H stretching of NH_3^+, C–H stretching of CH_2 and CH. The broadness is generally considered to be due to hydrogen bonding interaction of $H_2PO_4^-$, $COOH^-$ and NH_3^+ with adjacent molecules. The C═O stretching and –C═NH_4 stretching are revealed by absorption peak within

1693.2 cm^{-1}. The CH_2 bending attributes to the absorption at 1303.64 cm^{-1}. The absorption occurring at 1083.8 and 809.9 cm^{-1} are due to P–OH stretching [25,27]. It can be seen from Fig. 2 that the FTIR spectrum of irradiated samples are closely similar to that of unirradiated samples. Thus, as a result of irradiation there may not be any change in the chemical nature of samples. There are some of the extra absorption bands in irradiated samples and at the same time the other absorption bands completely destructed after irradiation. The destruction of these bands with irradiation may further enhance the amorphous in nature of the sample [17,28,29].

3.2. Scanning electron microscopy studies (SEM)

The SEM studies provide the information about topographical features, morphology, phase distribution, compositional differences, crystal orientation etc. The SEM images of the unirradiated and irradiated LACC (Fig. 3), L-alanine doped KDP (Fig. 4) and L-arginine doped KDP (Fig. 5) crystals have been carried out using S-3400 N Scanning Electron Microscope. The SEM images show some darker and brighter areas and this is due to solvent inclusions, which is most commonly observed in solution grown crystals [22,28,30]. The SEM images of irradiated crystals suggest that there are some morphological changes in the crystals after irradiation and show the formation of pores and cracks on the irradiated surface. It has been suggested that in the irradiated samples, oxygen moves into an interstitial site and creates oxygen-vacancy complex and modified stoichiometry may result in the near surface region.

3.3. DC conductivity study

The dc electrical conductivity study helps to understand the behavior of charge carriers under a dc field, their mobility and activation energy. Conductivity in ionic crystalline solids is mainly due to the presence of point defects in the lattice and due to different types of mobile charges as given in this relation:

$$\sigma = \sum_k n_k q_k e \mu_k \quad (1)$$

Where summation is taken over all the charged species k, n indicates the number of mobile charges of the type k having net charge $q_k e$ and μ_k represents electrical mobility. The dc conductivity (σ_{dc}) of unirradiated and irradiated crystals was calculated using the relation;

$$\sigma_{dc} = \frac{t}{RA} \quad (2)$$

where, R is the measured resistance, t the thickness of the crystal and A the area of the face of the crystal in contact with the electrode [31–34]. The surface of irradiated and unirradiated crystals were polished and coated with silver paste, which acts as electrodes. The conductivity measurements were carried out for both irradiated and unirradiated crystals using Keithley dual channel source meter model 2636A. The variation in dc conductivity of LACC, L-alanine doped KDP and L-arginine doped KDP crystals with Co-60 gamma dose is shown in Fig. 6 up to 6 Mrad of total dose. The dc conductivity values of unirradiated and irradiated crystals are given in Table 1. From the Table 1 it can be seen that, the dc conductivity of LACC and L-alanine doped KDP crystals were found to increase with increase in irradiation dose due the radiation induced defects. The conductivity results from the interplay between the drift mobility and the carrier concentration. If the conduction occurs by hopping of carriers, with a mobility reflecting the hopping rate, modifications in the drift mobility and carrier concentration as a result of defects and disorder brought about by irradiation and the conduction in the irradiated sample is expected to get modified [17,35,36]. In contrast, the dc conductivity of L-arginine doped KDP crystals was found to decrease with increase in radiation dose and it is unexpected.

3.4. Differential Scanning Calorimetric studies

Differential scanning calorimetry (DSC) thermograms of unirradiated and irradiated LACC crystal was performed using Universal

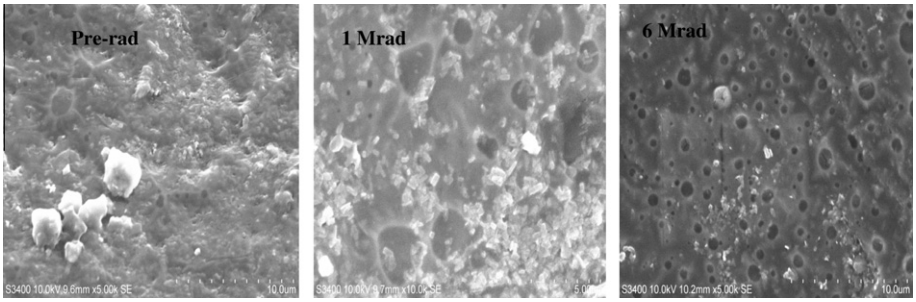

Fig. 3. SEM micrographs of unirradiated and irradiated LACC crystals.

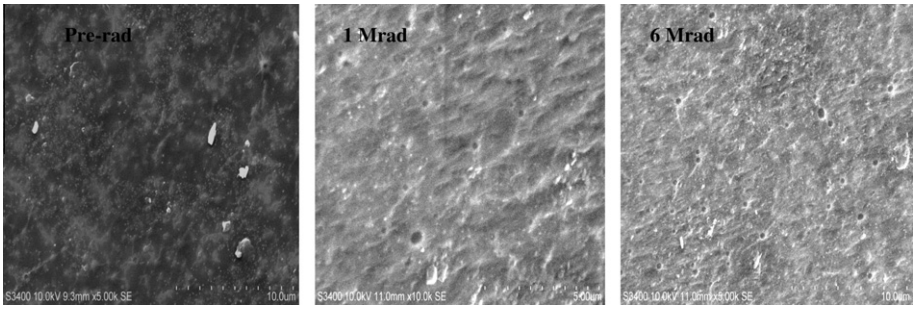

Fig. 4. SEM micrographs of unirradiated and irradiated L-alanine doped KDP crystals.

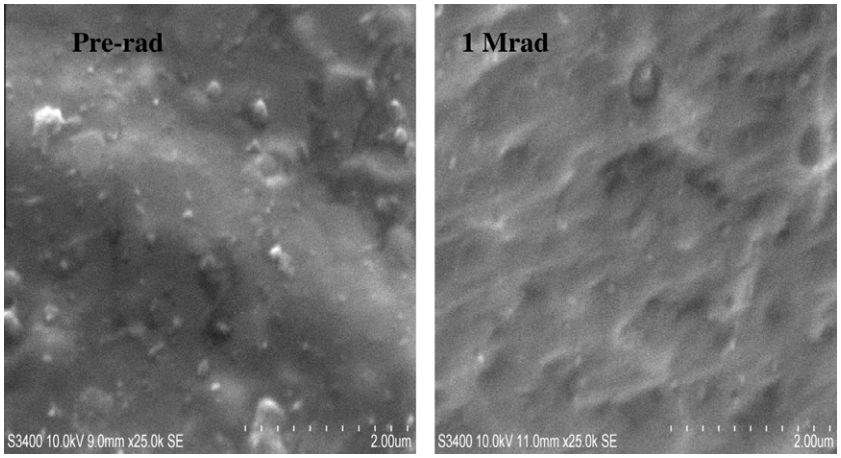

Fig. 5. SEM micrographs of unirradiated and irradiated L-arginine doped KDP crystals.

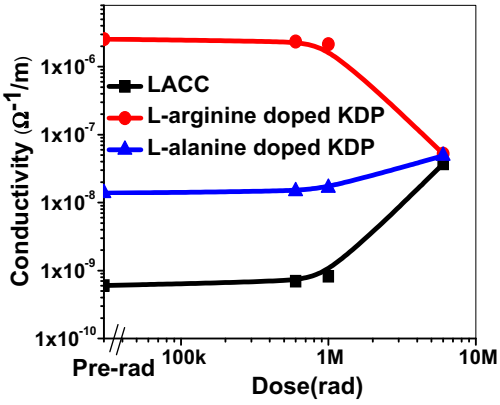

Fig. 6. Variation of σ_{dc} of LACC, L-alanine doped KDP and L-arginine doped KDP crystals with gamma radiation dose.

Table 1
DC conductivity values of unirradiated and irradiated crystals.

Dos(rad)	Conductivity $(\Omega\,m)^{-1}$		
	LACC	L-Alanine doped KDP	L-Arginine doped KDP
Pre-rad	4.92E-10	1.38E-08	2.54E-06
600 krad	6.98E-10	1.47E-08	2.35E-06
1 Mrad	1.29E-09	1.65E-08	2.12E-06
6 Mrad	3.71E-08	4.90E-08	5.24E-06

VA.7A TA Instruments DSC Q 200 in the temperature rang 30–200 °C at a heating rate of 10 °C/min in the nitrogen atmosphere (Fig. 7a). The sharp endothermic peaks at 137.5, 135.3 and 136.6 °C confirm the melting point of LACC before and after irradiation. The DSC thermograms of unirradiated and irradiated L-alanine doped KDP crystal and L-arginine doped KDP crystal are shown in Fig. 7b and c, respectively in the temperature rang 30–250 °C at a heating rate 10 °C/min in the nitrogen atmosphere. The sharp endothermic peaks at 234.6, 232.9 and 233.48 °C (Fig. 7b) confirm the melting point of L-alanine doped KDP before and after irradiation. The sharp endothermic peaks at 234.5, 235.3 and 230.149 °C (Fig. 7c) confirm the melting point of L-arginine doped KDP before and after irradiation. The sharpness of these endothermic peaks shows the good degree of crystallinity of the samples [2,3,37]. The values of the onset of melt (T_o), melting point (T_m), completing of melt (T_c), heat of fusion (ΔH) and melting range for unirradiated and irradiated crystals are tabulated in Table 2.

The DSC curves of unirradiated and irradiated LACC, L-alanine doped KDP and L-arginine doped KDP crystals showed no phase transition before melting point of the crystals. The DSC curves obtained for irradiated crystals are nearly same as that of unirradiated crystals and the melting point remain unaffected after irradiation for all samples as shown in Table 2 hence, suggesting the same chemical entity in the crystal lattice without any modification in its structure. Since there is no significant change in melting point, the quality of crystals remains unaltered after irradiation. Therefore it can be concluded that irradiation might be incapable of bringing out any damage to the structure of the crystals [17,28].

3.5. Microhardness studies

The apparent microhardness of solids depends on the applied indentation test load. This phenomenon, known as the indentation size effect (ISE), usually involves a decrease in the apparent microhardness with increasing applied test load, i.e., with increasing indentation size [18]. The polished surface of unirradiated and irradiated LACC crystals were subjected to static indentation tests at room temperature using a Vicker's microhardness tester model HMV-2 Ver 1.02 attached to a large incident light microscope. Loads ranging from 25 to 200 g were used for making indentations, keeping time of indentation constant at 10 s. The microhardness value was calculated using the equation,

$$H_v = \frac{1.8544P}{d^2}\,\mathrm{kg/mm^2} \tag{3}$$

where, H_v is the Vicker's hardness number, P is the applied load in kg, d is the average diagonal length of the indentation impression in micrometer and 1.8554 is a constant of a geometrical factor for the diamond pyramid. Fig. 8 shows the variation of microhardness with applied load for both unirradiated and irradiated LACC single crystals. From the figure, it can be seen that the microhardness was found to decrease linearly as the applied load increases for both unirradiated and irradiated crystals. However, it is observed that after irradiation the value of Vicker's microhardness corresponding to each load was found to decrease with increase in radiation dose. This is may be attributed to certain types of amorphization occurring in the material and shows that the dislocations taking place are different in crystals irradiated with different doses and substantiates the formation of isolated defect centres and weak lattice stresses on the surface [18,22,28,38]. The Meyer's index number was calculated from Meyer's law, which relates the load and indentation diagonal length,

$$P = kd^n \tag{4}$$

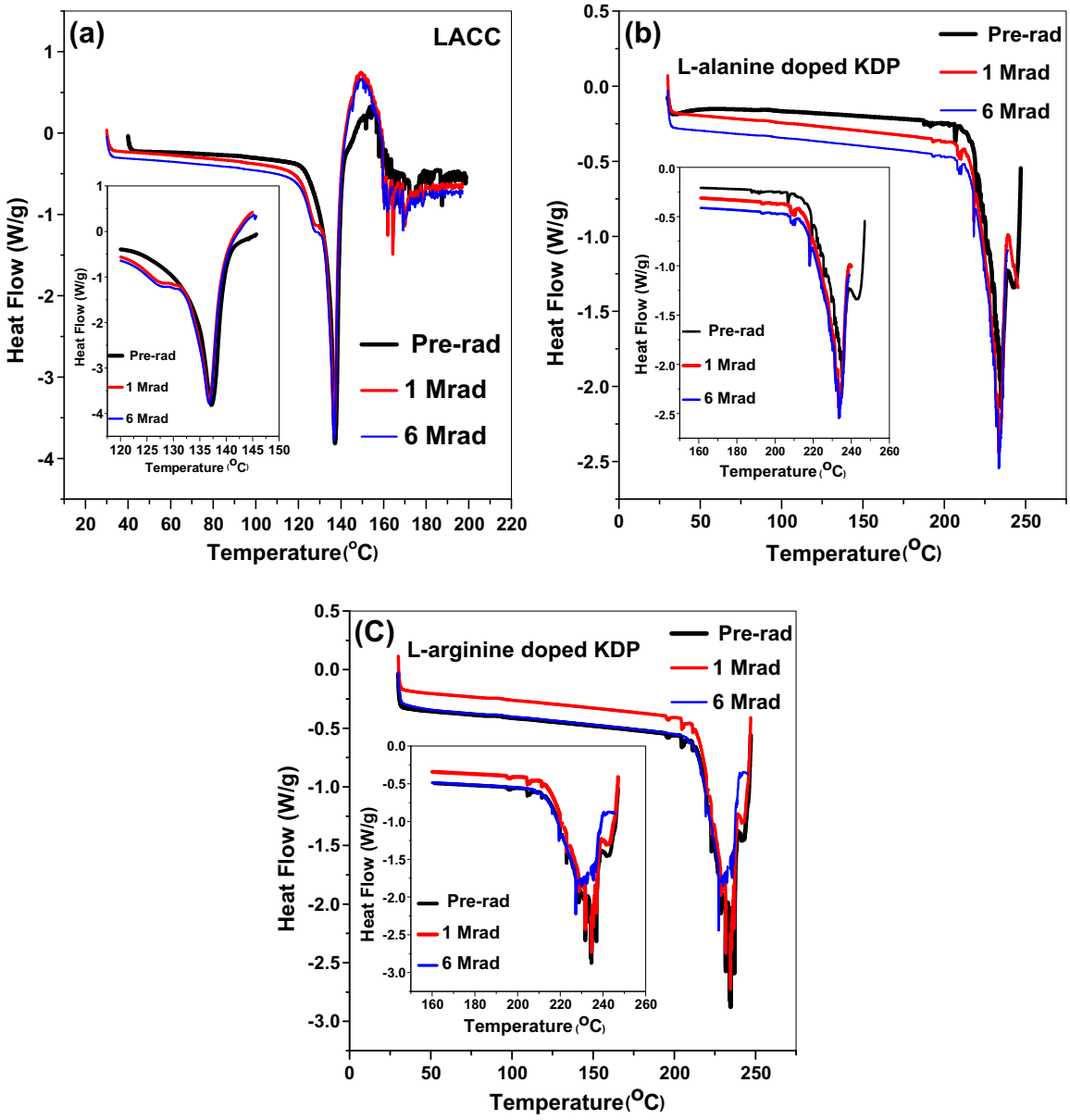

Fig. 7. DSC thermograms of unirradiated and irradiated crystals (a) LACC, (b) L-alanine doped KDP and (c) L-arginine doped KDP crystals.

Table 2
The values of T_o, T_m, T_c, ΔH and melting range for unirradiated and irradiated crystals.

	T_o (°C)	T_m (°C)	T_c (°C)	ΔH (J/g)	Melting range (°C)
LACC					
Unirradiated	114.0	137.5	152.2	152.7	38.2
1 Mrad	115.2	135.3	150.4	180.5	35.2
6 Mrad	116.3	136.6	149.0	190.9	32.7
L-Alanine doped KDP					
Unirradiated	211.9	234.6	247.1	124.6	35.2
1 Mrad	210.7	232.9	239.4	90.7	28.7
6 Mrad	212.8	233.48	240.6	78.8	27.8
L-Arginine doped KDP					
Unirradiated	209.0	234.5	247.1	177.6	38.1
1 Mrad	206.4	235.3	248.0	100.8	41.6
6 Mrad	210.5	230.2	242.3	98.5	31.8

$$\mathrm{Log}P = \mathrm{Log}k + n\mathrm{Log}d \quad (5)$$

where, k is the constant for the given material and 'n' is Meyer's index or work hardening index. To calculate the value of 'n', a graph log$P$ versus log$d$ is plotted and is shown in Fig. 9 which gives a straight line; the slope of this straight line gives the value of 'n'. The calculated values of 'n' are 1.303, 1.309 and 1.217 for unirradiated and irradiated LACC crystals after 1 Mrad and 6 Mrad, respectively. The 'n' value is in good agreement with the reported value [3,4]. According to Onitsch [39] H_v should increase with P if $n > 2$ and decrease if $n < 2$ and 'n' should lie between 1 and 1.6 for harder materials and above 1.6 for softer materials. Thus LACC crystal belongs to the hard material category. Low work hardening coefficient shows less dislocation in the grown crystal since work hardening coefficient is caused by the dislocation present in the crystal.

The above results show that, after irradiation the conductivity increases in the case of irradiated LACC and L-alanine doped KDP crystals owing to the fact that more defects are created after irradiation and which is predominantly due to the increase in the movement of defects. The increase in the conductivity after gamma and ion irradiation was also observed in KDP, benzimidazole, dimethyL-amino-pyridnium-4-nitrophenolate-4-nitro-phenol crystals [17,35,36].

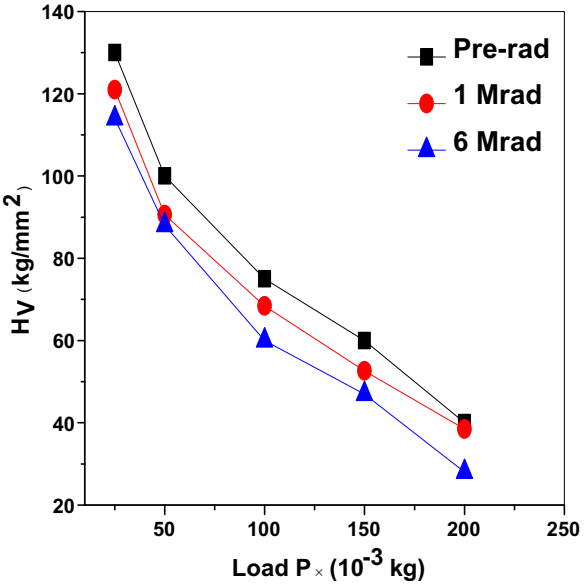

Fig. 8. Variation of microhardness values with load for unirradiated and irradiated LACC crystals.

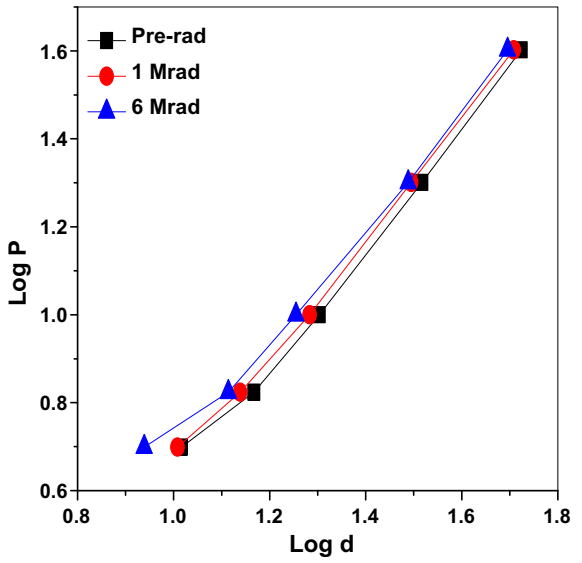

Fig. 9. Plot of log*P* versus log*d* for unirradiated and irradiated LACC crystals.

The decrease in the value of microhardness with increasing the load is in agreement with the normal indentation size effect (ISE) as observed by other researchers [3,4,18,38]. It is also observed that, the value of Vicker's microhardness corresponding to each load decreases after irradiation with increase in radiation dose [18,22,28,38]. This may be attributed to certain types of amorphization occurring in the material due to the production of dislocations and the density of dislocation may increase with increase in the total dose which intern produce weak lattice stresses on the surface.

4. Conclusions

Single crystals of LACC, L-alanine doped KDP and L-arginine doped KDP were grown by slow evaporation method at room temperature. The effects of Co-60 gamma radiation on the chemical nature, surface morphology, dc conductivity, thermal and mechanical properties of the grown crystals have been studied. There is no significant formation of intermediate chemically distinct material during irradiation. The investigation of the influence of gamma irradiation on the surface morphology of the grown crystals reveals the formation of pores and cracks on the surface. The dc conductivity of LACC and L-alanine doped KDP increases with increase in radiation dose where as in case of L-arginine doped KDP crystals, the dc conductivity was found to decrease with increase in radiation dose. The thermal analysis shows that the melting point remains unaffected after irradiation. The decrease in microhardness on irradiated LACC can be attributed to defect centers which generated after irradiation.

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