




X-ray diffraction and optical spectroscopic analysis on the crystallographic phase stability of shock wave loaded L-Valine

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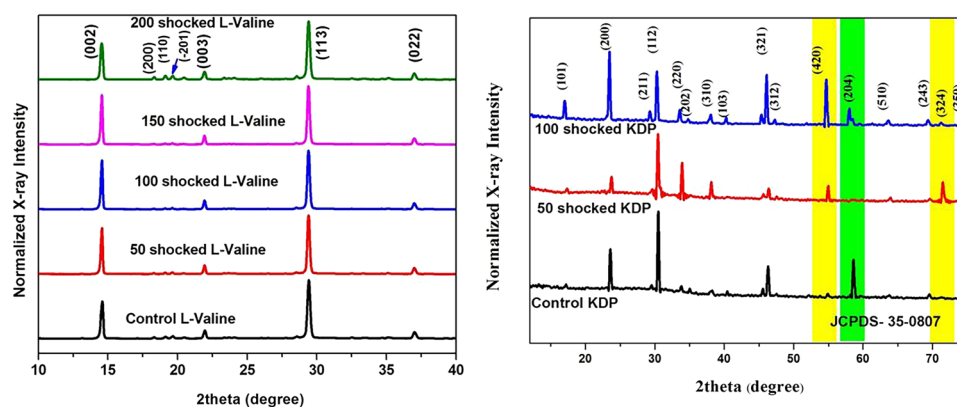
ABSTRACT

Most of the materials undergo phase transitions under shock-loaded conditions while a few of the materials experience lattice deformations. Such phase transitions and lattice deformations significantly affect the efficiency of the second harmonic generation of nonlinear optical materials. In the present context, we have undertaken the investigation of the powder samples of L-Valine ($C_5H_{11}NO_2$) which is one of the efficient nonlinear optical materials. In order to assess the crystallographic phase stability under shock-loaded conditions, shock-exposed samples and the virgin sample were directed to undergo the process of characterizations by employing X-ray diffraction, optical spectroscopy and microscopic studies. Based on the above-mentioned analytical results, it is established that the title sample exhibits the behavior of high shock resistance and the observed diffraction results have been compared with the diffraction results of potassium dihydrogen phosphate (KDP) such that the outcomes could be substantiated.

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GRAPHICAL ABSTRACT



Introduction

Over the past few years, there has been a cascade of promising prospects recognized in the field of ‘the impact of shock waves on materials’ such that the researchers could derive intense interest due to the obtained flamboyant results which pave the way for identifying materials of high stability. According to the previous publications on crystalline materials dealing with shock wave impacts, it has been well recognized that during the shock wave-loaded conditions, both bulk and nano crystalline materials tend to undergo phase transition as well as the process of lattice deformation [1–3]. On the other hand, based on the recent reports, very few materials are identified to have the tendency of possessing high shock resistance which fulfills the high industrial requirements whereas the ever-increasing demand for these materials provides a lot of room for further rigorous research such that an ample number of candidates could be found [4, 5]. Hence, the current researchers working on shock waves have been persistently delivering their significant contribution which propels the prospect of finding high shock resistant materials that are typically used for thermal protecting systems, aerospace vehicles, sensors, electronic devices, and nuclear power plants applications [6–8]. On the one hand, shock recovery experiments on

molecular crystals have provided a variety of information about the molecular arrangement and stability of crystal structures under static and dynamic high-pressure conditions [9, 10]. On the other hand, shock wave impacts on crystalline materials have offered plenty of unknown behavior of materials and their crystal structures that are yet to be materialized. Hence, from the shock wave recovery experiments, it is possible to draw new structural theories and mechanisms which would lead to a better understanding of the material properties under shock-loaded conditions so that it would open a new possible era emerging to be the next phase of high-pressure materials science. Therefore, in recent years, there has been witnessed a rapid increase in the number of publications that have added substance to the understanding of the role of shock wave resistant materials such that an alternate approach could emerge to assess the structural stability of materials suitable for extreme environmental applications [6–8, 11].

From the available research findings, it has been clearly shown that when a shock wave propagates into a single crystalline material it may create various kinds of defects in the crystal lattice that would create a significant variation in its physical properties, especially structural and morphological properties. Nowadays, nonlinear optical (NLO) materials have become prominent prospects in creating modern

technology because many industrial applications including optoelectronics, microelectronics, frequency converters, terahertz wave generation and high-power laser applications are very much dependent on these materials [12, 13]. Over the past 50 years, the research on nonlinear optical materials has resulted in well-defined research publications which promote materials of high efficiency that are classical for the above-mentioned applications. But the actual problem lies in the ability to find the relevant and existing materials which have the tendency to withstand the abrupt changes arising because of the environmental conditions of high pressure and high temperature even though they have good efficiency in terms of harmonic generation. In this context, experiments on high-pressure phase stability may help the device engineers to choose the right candidate for the above-mentioned applications in terms of structural stability and efficiency. Hence, the high-pressure experiments on solid-state materials always remain to be a special topic in the current research interest [14, 15].

In our previous experiments, investigations have been carried out to unearth the structural phase stability of technologically important NLO materials such as potassium dihydrogen phosphate (KDP) [16], glycine phosphate (GPI) [18], triglycine sulfate (TGS) [19], benzophenone [20] and ammonium dihydrogen phosphate (ADP) [17] under shocked conditions which have brought about some spectacular results. Interestingly, in the case of the KDP powder samples, it has been observed the disappearance of peak (204) and appearance of (324) at the 50 shocked condition whereas the opposite results have been observed at 100 shocks [16]. In the case of the ADP sample, the pyramidal phase gains the degree of crystallinity against the number of shock pulses [17]. Another interesting observation is that benzophenone has high shock resistance even at 100 shocks loaded conditions as reflected by diffraction peak neither appearing nor disappearing, even though it has a low melting point (48 °C) [20]. In the present context, the polycrystalline sample of L-Valine has been chosen for the analysis of the nature of behavior in terms of shock resistance. It is one of the simplest aliphatic amino acid crystals having a good efficiency of a second harmonic generation that is 1.44 times greater than that of KDP crystal [21]. A few reports are already available on its crystal growth and physical as well as chemical properties under ambient and

high-pressure conditions [21, 22]. Hermínio da Silva et al. subjected the title sample to high pressure up to the range of 6.9 GPa and found the loss of the degree of crystallinity as evidenced by Raman spectroscopy [23]. The same research team has also investigated the title sample at high-temperature environments up to 423 K and observed no indication of phase transition and re-orientation of L-Valine molecules [24]. Igor Fedorov et al. have investigated the phase stability of crystalline L-Valine at high-pressure regions up to 20 GPa and the ab initio calculations reveal a significant compression of the c-axis [25]. Furthermore, the test crystal has been subjected to a low-temperature (120 K) environment wherein no significant change has been detected [26]. Based on the above high-pressure research findings, it is worthwhile to subject the sample for shock wave recovery experiments so as to understand better the crystallographic phase stability of L-Valine.

In the present research article, the phase stability of L-Valine powder samples has been analyzed by exposing them to shock waves and thereafter subjected to X-ray diffraction, optical spectroscopic and microscopic techniques in such a way that the present work may trigger the opening of the floodgates of understanding the role of shock waves in amino acid crystals with which many more materials can be identified for specific applications.

Experimental section

The powder sample of L-Valine measuring 25 g with the assured purity of 99.99% was obtained which was utilized as it was for the required experimental studies on the shock wave impact through loading with a different number of shock pulses. The sample was subdivided into five equal parts such that one part has been kept as the control sample while the rest of the samples were made use of for the shock wave impact analyses. For the experimental analysis, a shock wave measuring Mach number 2.2 was preferred such that four different set of shock pulses namely with the counts of 50,100,150 and 200 shocks were exposed on those four samples, respectively. The shock waves were generated utilizing a semiautomatic Reddy tube. The reported findings of [27, 28] have accounted for the expanded versions of the methodology for the working of a shock tube and the shock wave loading procedure. The shock waves

emanated from the shock tube had the Mach number 2.2 possessing the respective transient pressure and temperature of 2.0 MPa and 864 K. After the completion of the shock wave impact procedures, the shock wave loaded samples and the control sample were fixed to undergo the respective characterizations of powder X-ray diffraction, diffused reflectance spectroscopy, and scanning electron microscopy.

Results and discussion

X-ray diffraction studies

The powder XRD technique is one of the most prominently used non-destructive analyses to examine the crystallographic structural properties of solid-state materials. Hence in the present investigation, the X-ray diffraction technique has been used to assess the crystallographic phase stability of the title sample under shock-loaded conditions and the observed X-ray diffraction patterns of the control and shock-loaded samples are presented in Fig. 1a. Before analyzing the shock wave induced variations on structural properties of L-Valine sample, it is essential to notify the phase formation and phase purity of the title sample. The diffraction peaks (Fig. 1a) of the control sample such as (002), (200), (110), (-210), (003), (113), and (022) are identified to be well harmonized with the reported XRD pattern of L-Valine crystal (JCPDS: 22–1930). Hence, it could be confirmed that

the test sample has crystallized in the monoclinic crystal system with the space group $P2_1$. L-Valine has the lattice parameters of $a = 9.71 \text{ \AA}$, $b = 5.27 \text{ \AA}$, $c = 12.06 \text{ \AA}$, and $\beta = 90.8^\circ$ such that each unit cell has four molecules ($Z = 4$) [29].

As per the crystallographic structure, the unit cell of L-Valine has two non-equivalent molecules and the length of the c-axis is higher compared to the other two axes and the corresponding crystallographic structure is portrayed in Fig. 2. Hence, under shock wave loaded conditions, it is suspected that a significant lattice compression might have occurred due to the longer c-axis while shock waves propagate through the sample.

After careful assessment of Fig. 1, a contradiction to our prediction is observed such that the samples have not undergone any of the crystallographic phase transitions and lattice deformations as well as compression of the unit cell. All the diffraction peaks have been reproduced maintaining the original positions of diffraction angle under shocked conditions irrespective of the number of shock pulses. Moreover, the peak intensity of the diffraction pattern is also not significantly altered by the exposure to shock waves. But, in the previous experiment on the KDP samples, strong new diffraction peaks appeared, and a few peaks disappeared under shock-loaded conditions [16]. Also, the peak positions and the diffraction peak intensity have been significantly altered against the number of shock pulses and the corresponding diffraction profiles are shown in

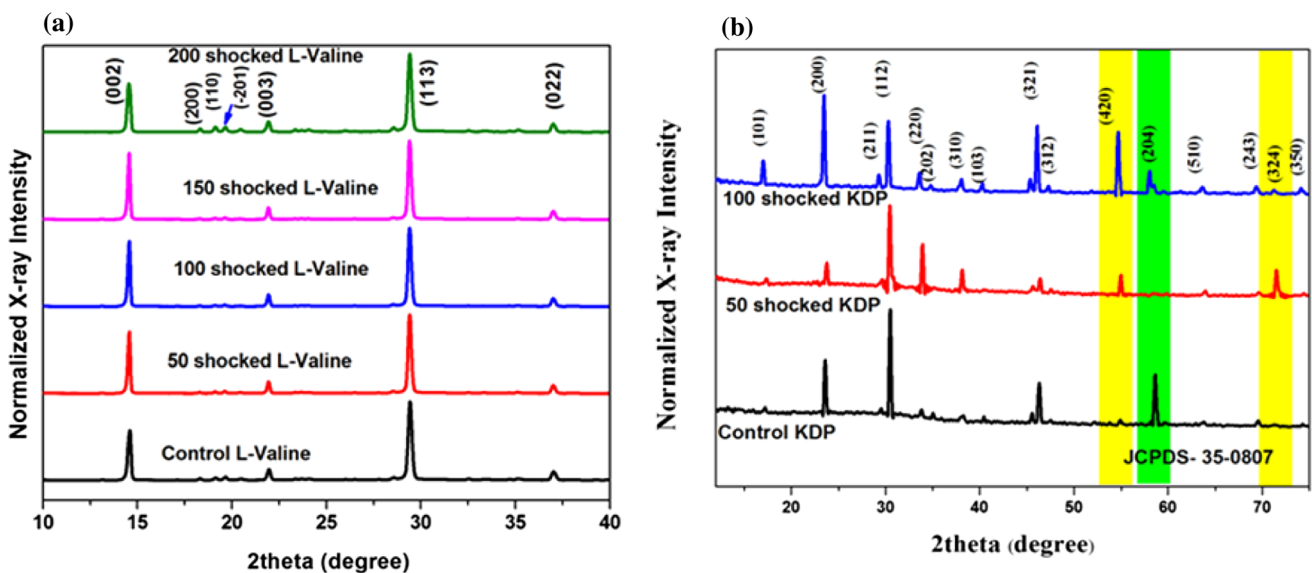


Figure 1 XRD patterns of the control and shock loaded samples **a** L-Valine **b** KDP sample (adopted from Ref. [16] with permission).

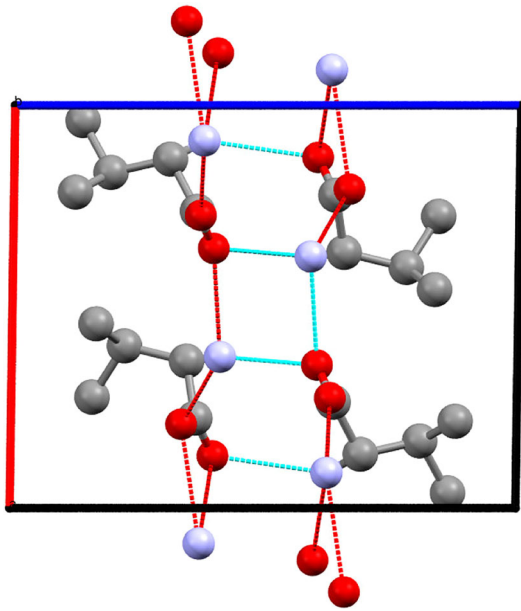


Figure 2 Molecular packing of L-Valine crystal (Red ball-oxygen, gray ball- carbon, light blue- nitrogen, H atoms are omitted for image clarity).

Fig. 1b. Hence, it could be confirmed that the test sample has high shock resistance under shocked conditions compared to KDP crystals. In order to accomplish a clear and better understating of the above-proposed shock resistance, it has been investigated further such that the diffraction peaks have been analyzed in the zoomed-in version of specific diffraction angles. As seen in Fig. 3a, the diffraction profile has two crystalline peaks such as (002) and (003). It is known that the crystallographic axis of higher length is prone to undergo significant lattice compression at high-pressure compression exposure compared to the other two crystallographic axes [25]. Moreover, the $P2_1$ space group usually allows lattice compression under high-pressure conditions due to the presence of a screw axis [30, 31]. If the *c*-axis of L-Valine has experienced lattice compression the resultant intensity of the diffraction peaks has to be significantly reduced. On the other hand, surprisingly there are no deviations observed neither in peak intensity nor in peak position for both the higher angle and lower angle regions. Furthermore, it is well evident that the peak intensity ratio has also not changed. For a better understanding, it is drawn blue dotted lines between the (002) and (003) diffraction peaks which reveal that the angles subtended by the dotted lines with respect to the horizontal line are not significantly altered. Hence, it could be considered

that the crystal lattice does not undergo significant lattice compression and deformation under shocked conditions. The high shock resistance exhibited by the L-Valine crystal structure may be due to the double layers that are linked by hydrogen bonds between N–H ... O along the (001) axes [23, 24].

Followed by the analysis of the (002) and (003) crystalline planes, the (113) and (022) diffraction planes are also taken into further investigation. These two crystalline planes have slight variation in terms of the contribution with respect to both the *a*- and *b*-axes. Here also, a similar logic of Fig. 3a is utilized so as to compare the peak intensity and intensity ratio between the (113) and (022) planes and the respective diffraction profiles are displayed in Fig. 3b. It is observed similar results as that of the results reflected by Fig. 3a. Furthermore, it is essential to find the most stable crystalline plane in the L-Valine crystal even though it is by and large stable under shocked conditions so that it would be beneficial to those who are working in the orientation-dependent crystal growth for the nonlinear optical applications. Moreover, the selection of crystallographic orientation plays a major role in terms of mechanical stability and efficiency of harmonic generation [32, 33]. Hence, the (002) and (113) crystalline planes are selected, and the respective diffraction peaks are shown in Fig. 4

As shown in Fig. 4, the (002) crystalline plane has undergone a reasonable shift with respect to the lower angle side depending on the number of shock pulses. Whereas the plane (113) has not undergone any kind of peak shift under the shocked conditions which may be because of the fact that both the *a*- and *b*-axes hold the strength of the crystalline plane. At this stage, in the case of the (002) plane, hydrogen bonds may undergo slight expansion that may be up to the extent of 0.01 Å under the shocked conditions wherein the expansion value is found to have increased against the number of shock pulses [23, 34, 35]. Henceforth, it is pretty evident that the (113) crystalline plane has higher shock-resistant behavior than that of the (002) plane. Due to the higher shock resistance, the (113) plane of the crystal could be more suitable than the (002) plane for optoelectronic device fabrications. Furthermore, the comparison of XRD patterns of L-Valine and KDP crystal is validated by taking into consideration of the most primary diffraction patterns such as the (113) and (200) and the corresponding diffraction profiles are shown in Fig. 5.

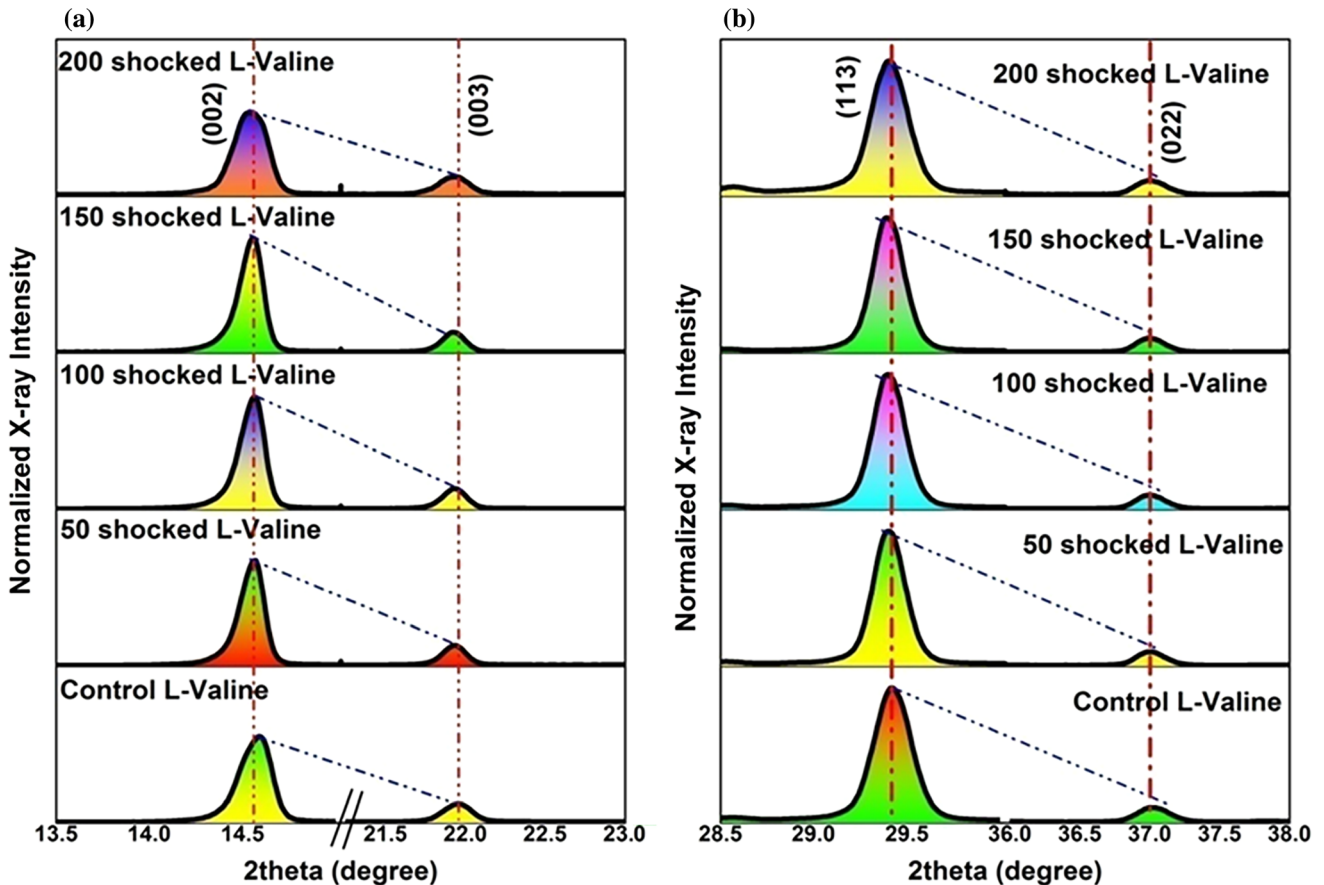


Figure 3 XRD patterns of the control and shocked L-Valine in selected 2θ ranges **a** $13.5\text{--}23^\circ$ **b** $28.5\text{--}38.5^\circ$.

As seen in Fig. 5a, the L-Valine crystalline peak (113) does not show any change either in peak shift or the intensity of peak with respect to the number of shock pulses. But in the case of the KDP sample, significant changes both in peak shift and peak intensity have been observed with respect to the number of shock pulses. Furthermore, the values of the laser damage threshold also support the claim in such a way that KDP has the laser damage threshold of 0.2 GW/cm^2 [36] whereas L-Valine has 11.11 GW/cm^2 [21]. Basically, laser irradiation and shock wave-loading process provide the extreme high pressure as well as high temperature simultaneously on the surface of the crystals under investigation. During the irradiation process and at the exposure of high number of shock pulses (or) high laser energy if materials have sustained and withstood, they are considered as highly stable materials [37–40]

Optical properties

Investigation of optical properties of NLO materials under shocked conditions is yet another interesting study that brings in a lot of new facts about the electronic structure of the materials. Furthermore, the optical transmittance window range and percentage of the optical transmittance are the non-debatable criteria that are essential in order to choose the apt materials for the nonlinear optical applications. As the selection of an appropriate optical transmittance window and a good percentage of optical transmittance are already mandatory for a material to be used for device fabrication, a couple of basic requirements can be further considered. The first one is the stability of the absorption edges of the materials and the second one is the percentage of optical transmittance at high pressure, temperature and laser irradiation conditions. In order to satisfy these two, it is obligatory to perform a systematic investigation of the stability of optical properties of the material under shock-loaded conditions so that a better

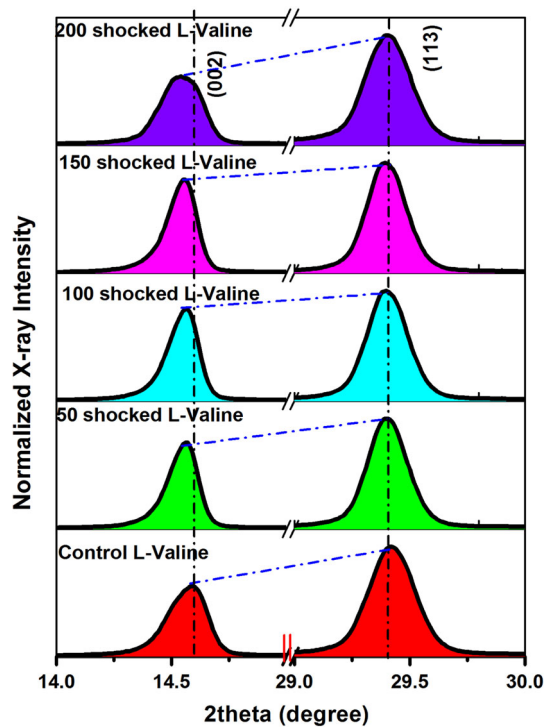


Figure 4 XRD patterns of the control and shocked L-Valine for the region 14–30°.

understanding would be enabled. Therefore, it is possible to draw the structure-properties stability profile for the test sample under shocked conditions.

In this context, the ultraviolet diffused reflectance spectroscopic technique has been performed so as to obtain the optical constancy of L-Valine samples under shock-loaded conditions and the observed optical transmittance profiles are shown in Fig. 6a. The test sample shows the absorption edges at 210 and 283 nm which are the characteristic absorption peaks of L-Valine sample [22]. Furthermore, the test sample has a transmittance window ranging from 300 to 800 nm which suits the title material for the NLO applications. In addition to that, there is no major shift in absorption edge (blue shift or red shift) observed under shocked conditions for the samples. For a better reliance on this statement, it is presented the zoomed-in version of the transmittance profiles between 200 and 300 nm in Fig. 6b. Moreover, the percentage of the optical transmittance remains stable up to 150 shocks whereas it is slightly reduced at 200 shocks that must be due to the surface changes occurring at shocked conditions. In the case of the KDP crystal, a linear red shift was observed against the number of shock pulses [16].

Furthermore, employing the Kubelka–Munk relation, the indirect optical band gap energy has been calculated and the computed optical band gap patterns of the control and shocked L-Valine samples are portrayed in Fig. 7. The observed band gap values of the control, 50,100,150 and 200 shocked samples are 5.35, 5.38, 5.35, 5.33 and 5.34 eV, respectively. Hence,

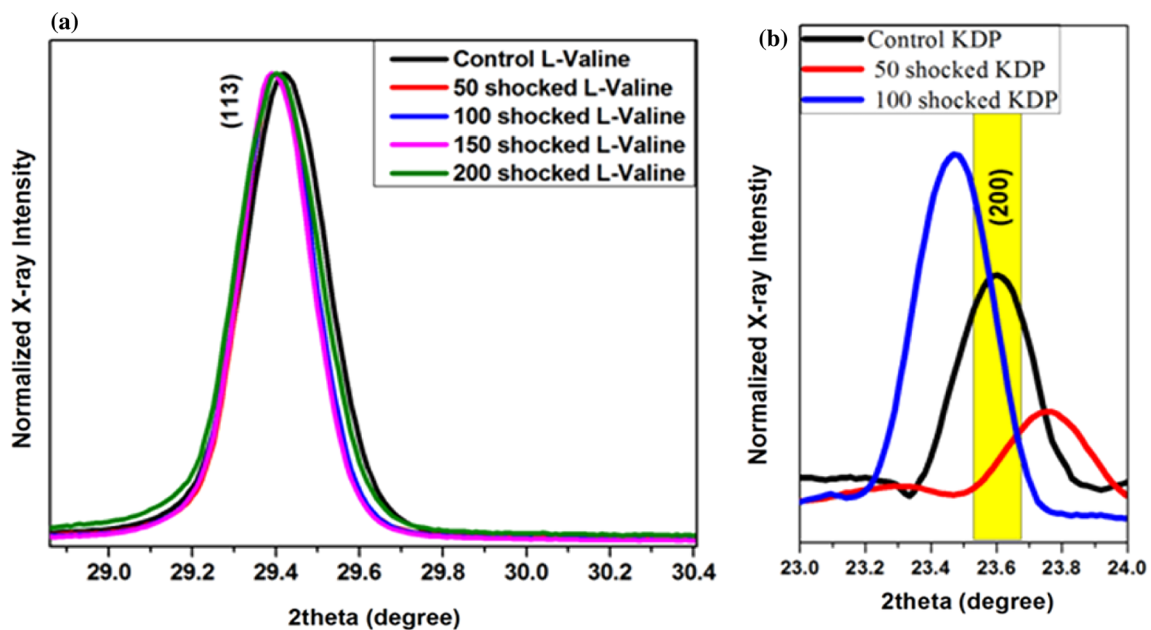


Figure 5 The XRD characteristic peak natures of the control and shocked conditions a L-Valine – (113) b KDP–(200).

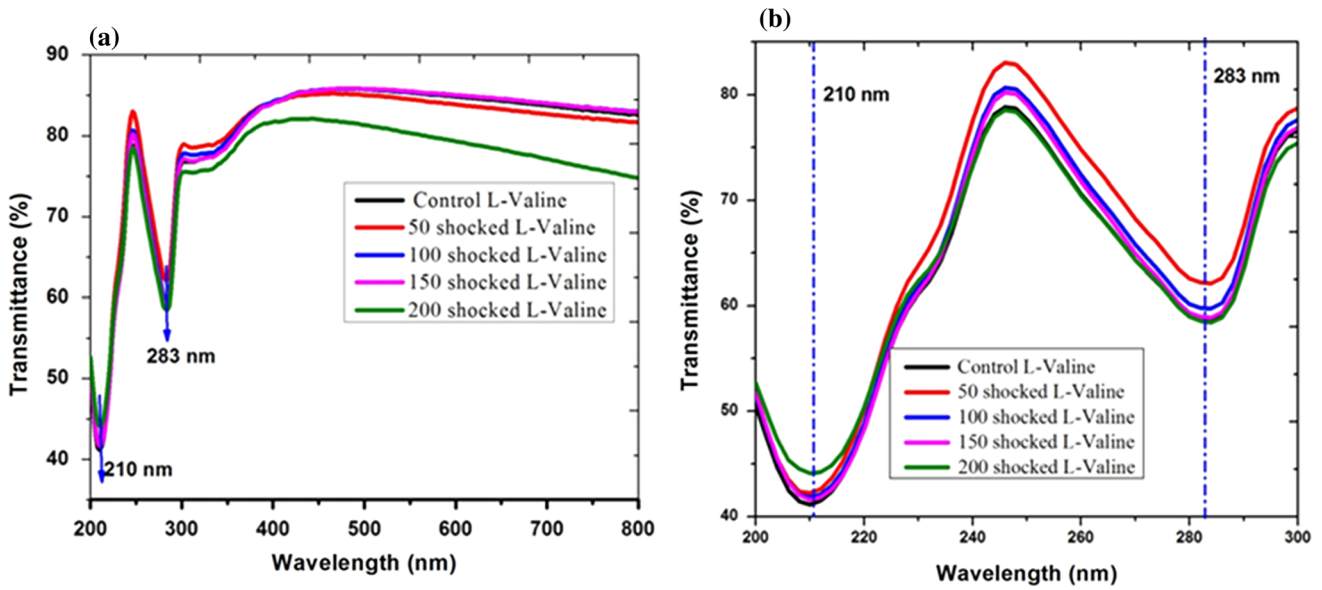


Figure 6 Optical transmittance of the control and shocked L-Valine a 200–800 nm b 200–300 nm.

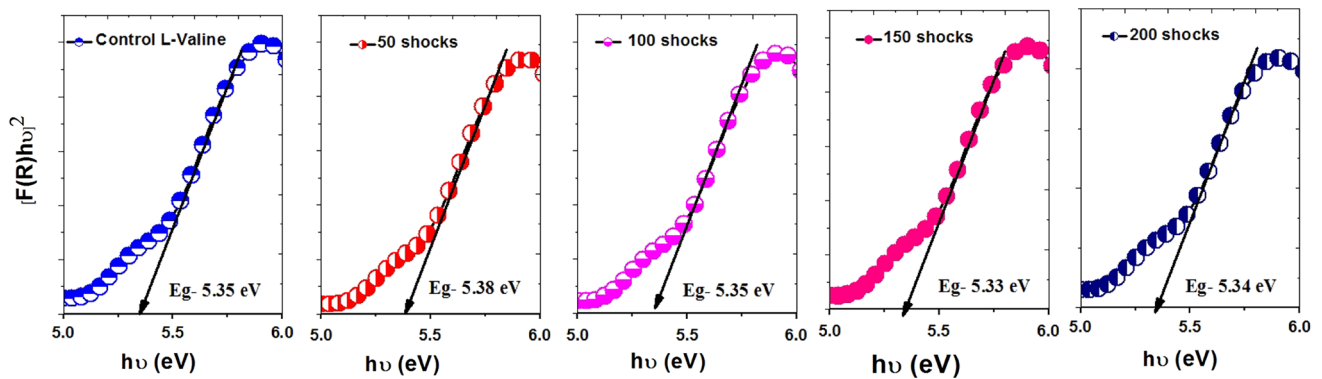


Figure 7 The values of band gap energy of the control and shocked L-Valine samples.

it confirms that the test sample has consistent electronic properties even under shocked conditions. On the other hand, the KDP sample has varying values such as 4.79, 4.55, and 4.70 eV against the number of shock pulses because of the changes occurring in absorption edges within the same phase [16]. Potassium sulfate crystal also experiences changes in the values of band gap energy such as 4.53, 3.83 and 4.57 eV against the number of shocks due to the occurrence of crystallographic phase transition [41]. But no change is observed in the L-Valine sample with respect to the number of shocks which is because of the presence of double layers linked by

hydrogen bond networks in the crystal system. The observed reduction of the optical transmittance at the exposure of 200 shocks leads to further investigation that has to be carried out to understand the mechanism behind it.

Surface morphological studies

In order to consolidate the results, along with the diffraction and spectroscopic studies, surface morphological analysis is also required for making a clear conclusion about the shock-resistant behavior of the sample against the impact of shock waves. For optical

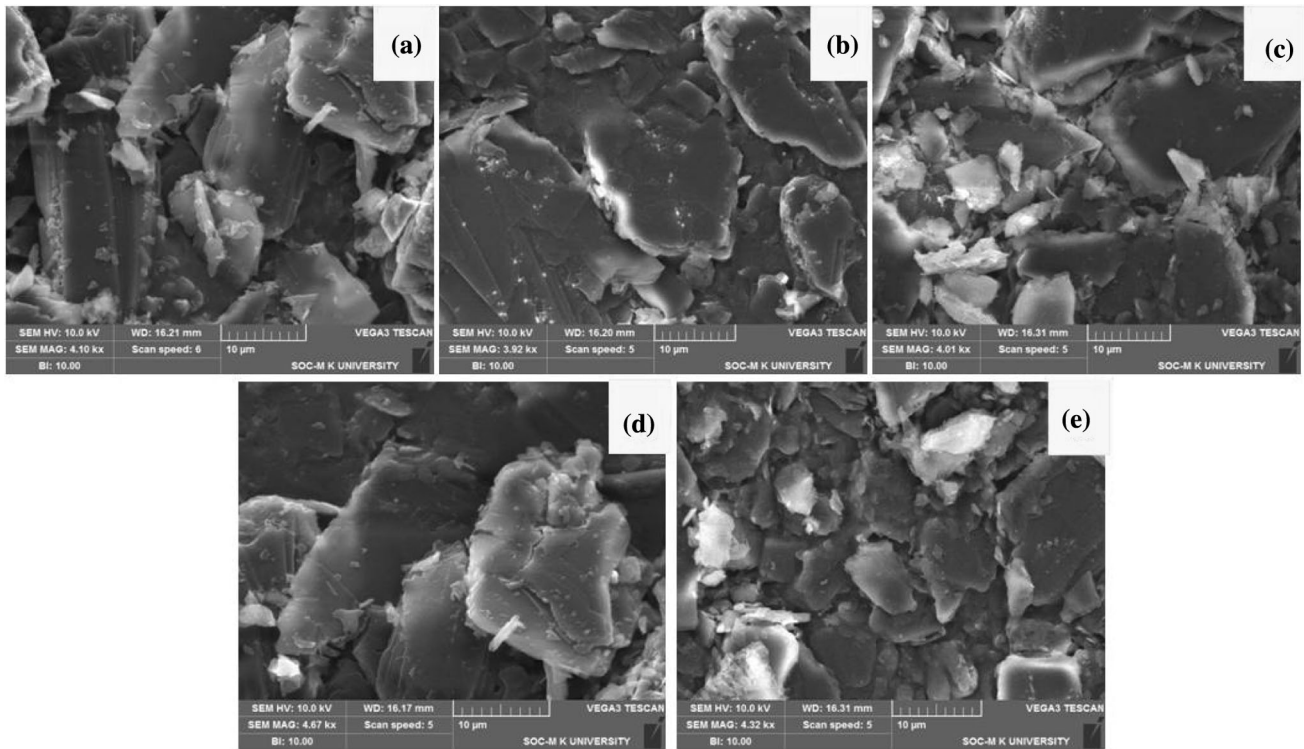


Figure 8 Microscopic images of the control and shocked L-Valine samples **a** the control **b** 50 shocked **c** 100 shocked **d** 150 shocked and **e** 200 shocked.

materials, surface morphology plays an imperative role in the applications of harmonic generation. To understand the response of the surface morphology of the shocked samples, a scanning electron microscope has been utilized. As seen in Fig. 8a, the control sample has a rectangular shape morphology which is extended along the *c*-axis. There is no substantial change observed in the surface morphology up to the exposure of 200 shocks (Fig. 8).

Conclusion

Based on the results of the present shock wave loading experiment, it is possible to draw the concluding remarks such that the title sample has been exposed to different numbers of shock pulses and the influence of shock waves on the samples has been analyzed by diffraction, spectroscopic and microscopic analyses. From the observed X-ray diffraction and optical spectroscopic methods, it is clear that the test sample has not undergone any kind of phase transition and lattice deformations. From the optical physics point of view, any kind of shift has not been observed i.e., either blue or red shift against the

number of shock pulses. Based on the overall observation, it is suggested that the test sample has higher shock-resistance than that of the KDP crystal and hence it is strongly proposed that the title crystal can be considered for the fabrication of devices that come under severely persisting environmental changes.

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Author's contribution

AS Data analysis, Writing—original draft; SSJD Visualization, Roles/Writing—original draft; LD Investigation, Writing—review & editing, VM Sample preparation; PS Formal Analysis, RSK Formal Analysis,

AIA Formal Analysis, SA Formal Analysis; IK Resources, SAMBD Investigation, Writing-review & editing.

Data and code availability

The data in my manuscript can be obtained from the corresponding author.

Declarations

Conflict of interest The authors declare that they have no conflict of interest.

Ethics approval None.

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