

Diffraction and microscopic studies on lithium sulfate doped L-Threonine under dynamic shock wave exposed conditions

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ABSTRACT

Scientists of shock wave research have been continuously contributing with a great deal of significant effort so as to carry forward the tempo in achieving much more applications of shock waves in the branch of solid state sciences. In the present framework, the evaluation of the structural phase-stability and morphological stability of lithium sulfate doped L-Threonine powder samples have been performed under shock wave exposed conditions and the results have been evaluated by diffraction and microscopic techniques. Based on the observed crystallographical results, it is authenticated that the title specimen retains the original crystal system ($P2_12_12_1$) with a few internal changes such as new peak appearance and disappearance of existing peaks that are observed due to the occurrence of micro-distortions and re-organization of hydrogen bond networks as well as rotational order-disorder of the lithium sulfate under shock loaded conditions. SEM micrographs clearly demonstrate the formation of cracks and breaking of particles under shocked conditions.

1. Introduction

Of late, the study of shock wave impact on non-linear optical materials has attained the status of being one of the fertile research areas in the materials science branch as a few of the materials have been already tagged to be stable so that investigating further on the behavior of materials in harsh environments would create a way forward in the development of novel structural materials for the applications of extreme conditions. Shock waves' impacts on materials can alter the physico-chemical properties including structural, optical, mechanical, electrical, and thermal properties. The study of shock wave interaction with materials is almost similar to the study of dynamic impact and high mechanical impact with the corresponding materials [1–4]. Impact

response testing is one of the primary research areas for understanding the properties of the materials and their performance under extreme conditions. Recent experimental results clearly emphasize the impact of dynamic shock waves on solid-state materials enabled by shock tubes such that it has stabilized itself as one of the efficient methods which can provide direct evidence for making convincing conclusions on the material stability under critical pressure and temperature conditions [5–7]. Interestingly, shock waves can provide real-time impact problems such as dynamic impact, high-stress, high-pressure, high-temperature and large mechanical force etc. Moreover, till this point in time, only a part of scientific knowledge is well established on the shock wave impacts on solid-state materials such that it must be enhanced so as to understand the actual outcome of the shock wave impacts on solids in such a way

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that it would provide a potential chance to understand better the usual and unusual behavior of materials against the temperature and pressure [8–10].

However, over the last couple of decades, it has been witnessed a speedy growth of shock wave research in materials which includes bulk and nanomaterials such that there have been many research papers published in this particular area with the involvement of several laboratories worldwide [1,4,8]. Our group has also made a few potential contributions to enrich the impact of shock wave research in crystalline materials and made a few contributions on this topic, particularly in electro-optical materials. Since electro-optical materials are the building blocks of modern technology and continue to be part of most electronic devices, understanding the shock resistance is highly required for device materials. Recently, we have carried out the shock wave impact test on the powder samples of potassium dihydrogen phosphate (KDP) [11] as well as ammonium dihydrogen phosphate (ADP) [12] and found a few interesting results. Note that, in the experiment of the KDP sample, a few of the diffraction peaks disappear and re-appear under shock wave exposed conditions. But no crystallographic phase transitions have been noted [11,12]. In the case of ADP samples, a rapid enhancement of the pyramidal face crystalline nature has been observed under shocked conditions whereas a massive decrease in the degree of crystalline nature has been observed in the prismatic face of ADP [12]. Followed by the KDP and ADP samples, similar experiments have been performed for benzophenone [13], glycine phosphite (GPI) [14] and tri-glycine sulfate (TGS) [15] samples whereby a few interesting results have been found in their crystallographic structures as well as crystal orientations.

In this present framework, we have selected the powder samples of lithium sulfate doped L-Threonine to assess the stability of the crystallographic phase as well as crystalline nature against the impulsion of shock waves. The strong reason behind choosing L-Threonine for the investigation is that it is one of the potential non-linear optical (NLO) crystals which belong to the hydrogen-bonded amino acid family of crystals [16–18]. Lots of information on pure and doped L-Threonine crystals have been reported in the past several years [19,20]. For example, Elberin Mary Theras et al., have reported the lithium sulfate doped L-Threonine crystal growth details and its functional properties such as optical and magnetic properties and found that the doped samples significantly altered the functional properties compared to the pure L-Threonine crystal. But, the in-depth knowledge of this material is yet to be explored at high pressure and high temperature. Many more experimental investigations are highly required to understand better the crystal structure and its properties. On the other hand, there are only a few publications available in the area of high energy irradiation and high-pressure conditions. Conformational phase transitions in L-Threonine have been reported at high-pressure regions i.e. at 2.03 GPa by Silva et al. [21]. Nico Giordano et al. have noticed the crystallographic phase-transition from $P2_12_12_1$ to $P2_1$ at 18 GPa [22]. A complete crystalline to the amorphous state phase transition of the single crystal of L-threonine is caused by Li^{3+} ion irradiation (50 MeV) at 1×10^{12} ions cm^{-2} [23]. To date, based on the literature reports, an assessment of crystallographic phase stability on lithium sulfate doped L-Threonine under shocked conditions is yet to be reported.

The core aim of the present work is to explore the shock waves' impacts on NLO materials such that the assessment of the structural phase stability of lithium sulfate doped L-Threonine has been considered. In order to obtain the required results, XRD and SEM techniques have been employed. Moreover, the title sample belongs to the hydrogen-bonded family of materials that are highly crucial to be utilized for shock wave impact studies. On the other hand, the dopant material of Li_2SO_4 undergoes the rotational order-disorder process at high temperature and pressure conditions [24,25] such that it was strongly expected that significant changes might appear both in crystal structure and surface morphology in the test sample under shock wave exposed conditions as it happens in the ion irradiation experiments and static high-pressure compression experiments [21–23].

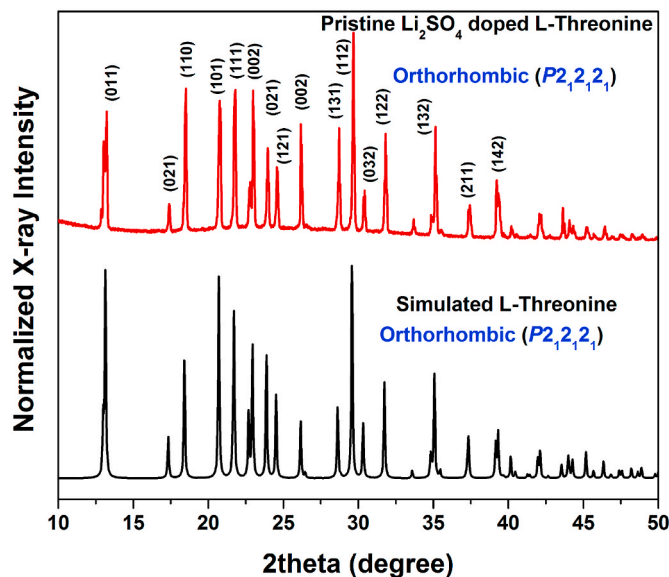


Fig. 1. XRD pattern of simulated L-Threonine (generated by CIF file: CCDC – 129494) and the observed pattern of pristine Li_2SO_4 doped L-Threonine.

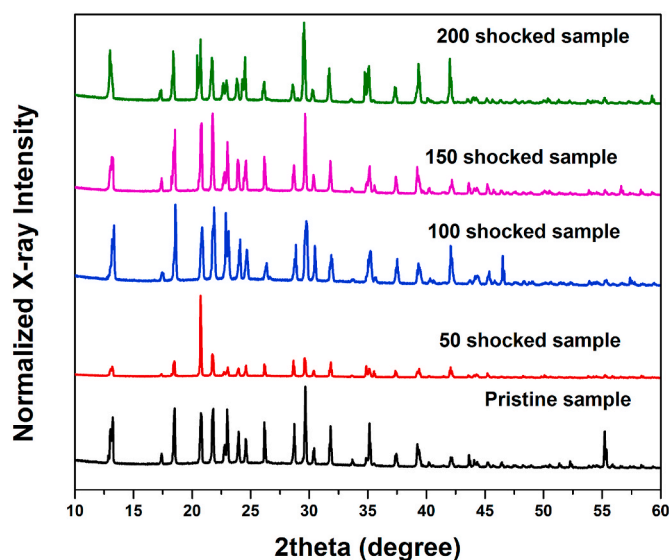


Fig. 2. PXRD patterns of the pristine and shock wave exposed Li_2SO_4 doped L-Threonine samples.

2. Experimental section

2.1. Crystal growth

The growth details of the title crystal, its structure and other physical properties have been detailed in the reported article [20]. For the present work, the title crystal has been subjected to gentle grinding so as to get fine powder form of the test samples that have been used for the shock wave impact study. Totally, five samples have been made from the prepared powder and out of that, one sample has been kept as the pristine sample and the remaining four samples have been sent for the process of shock-wave loading. The required shock waves have been generated by a pressure-driven table-top shock tube. The transient pressure and the transient temperature of one shock pulse have been fixed as 2.0 MPa and 864 K, respectively. The detailed experimentation of the shock tube and its working process has been reported in the previous publication [26] as well as it is also provided in the

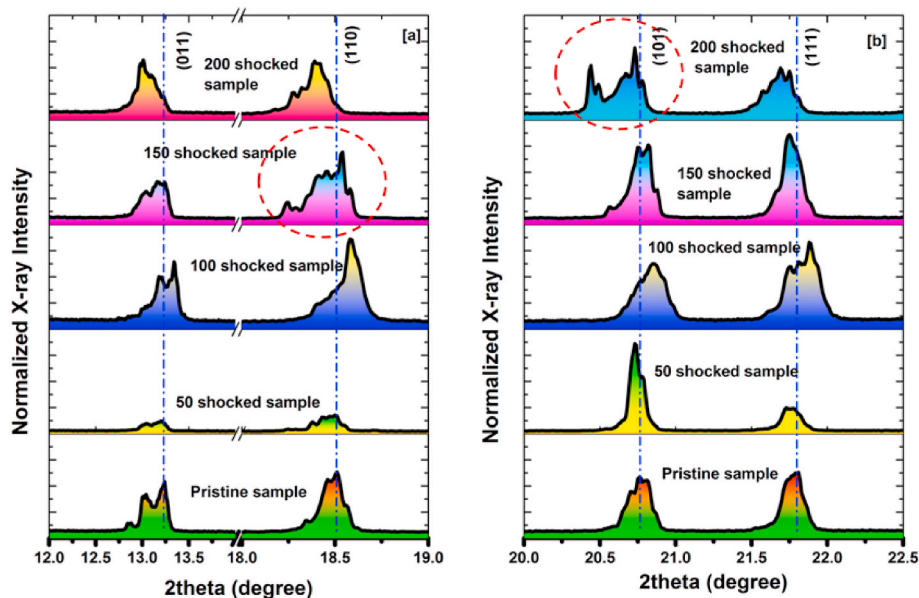


Fig. 3. Zoomed-in versions of PXRD patterns of the pristine and shock wave exposed Li_2SO_4 doped L-Threonine sample (a) $12\text{--}19^\circ$ (b) $20\text{--}22.5^\circ$.

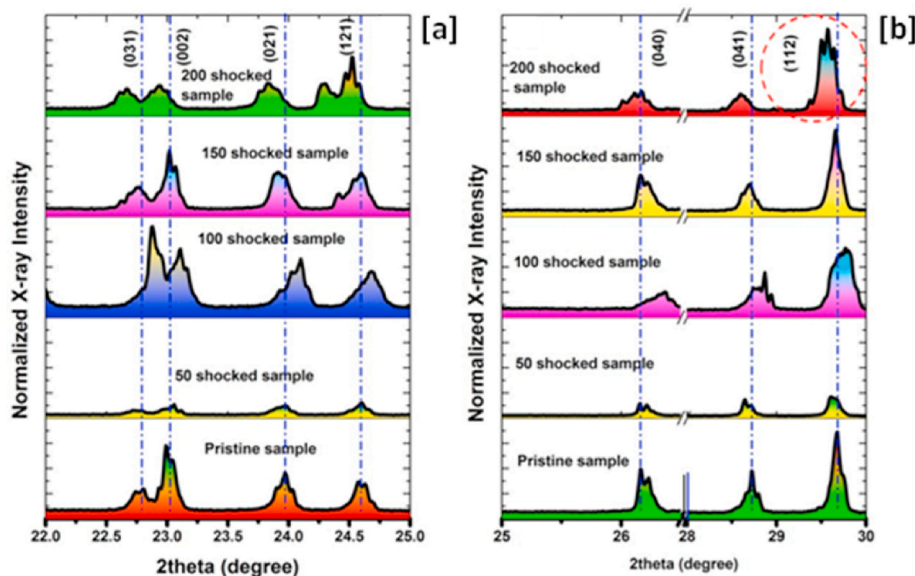


Fig. 4. Zoomed-in versions of PXRD patterns of the pristine and shock wave exposed Li_2SO_4 doped L-Threonine sample (a) $22\text{--}25^\circ$ (b) $25\text{--}30^\circ$.

supplementary section. In the present experiment, the shock wave of Mach number 2.2 has been chosen such that subsequently, 50, 100, 150 and 200 shock pulses have been loaded on the respective samples with an interval of 5 s between each shock pulse. Thereafter, the shock-loaded samples have been sent for the studies of diffraction as well as microscopic techniques.

3. Results and discussion

3.1. X-ray diffraction results

Rigaku-II X-ray powder diffractometry has been used to assess the crystallographic structural changes as well as modifications in the degree of crystalline nature for the title samples under shocked conditions. Diffraction angles from 10 to 80° have been measured and the step increment of diffraction angle has been fixed as 0.02° such that the observed XRD patterns are shown in Fig. 1 and Fig. 2.

As shown in Fig. 1, the powder XRD patterns of the simulated L-Threonine and the pristine Li_2SO_4 doped L-Threonine are found to be well corroborated with each other except with a fractional diffraction angle shift which is due to the incorporation of dopant in the host crystal structure [20]. Hence, it confirms that the test sample belongs to the orthorhombic crystal system with the space group $P2_12_12_1$ and the lattice dimensions are $a = 5.11 \text{ \AA}$, $b = 13.60 \text{ \AA}$, $c = 7.66 \text{ \AA}$, and $V = 532 \text{ \AA}^3$. It could be noted that the values of the mentioned lattice dimensions are obtained from the previous publication [20] which is well corroborated with the literature reports [16,18]. The fine and sharp diffraction peaks of the test sample clearly indicate that Li_2SO_4 doped L-Threonine has good crystalline nature. The XRD patterns of the pristine and shock-wave loaded samples are presented in Fig. 2. As seen in the XRD patterns of Fig. 2, on the one hand, there is no significant structural change such as crystal-crystal phase transition or crystal-to-amorphous phase transition observed. On the other hand, there are a lot of internal structural changes that can be observed such as diffraction peak

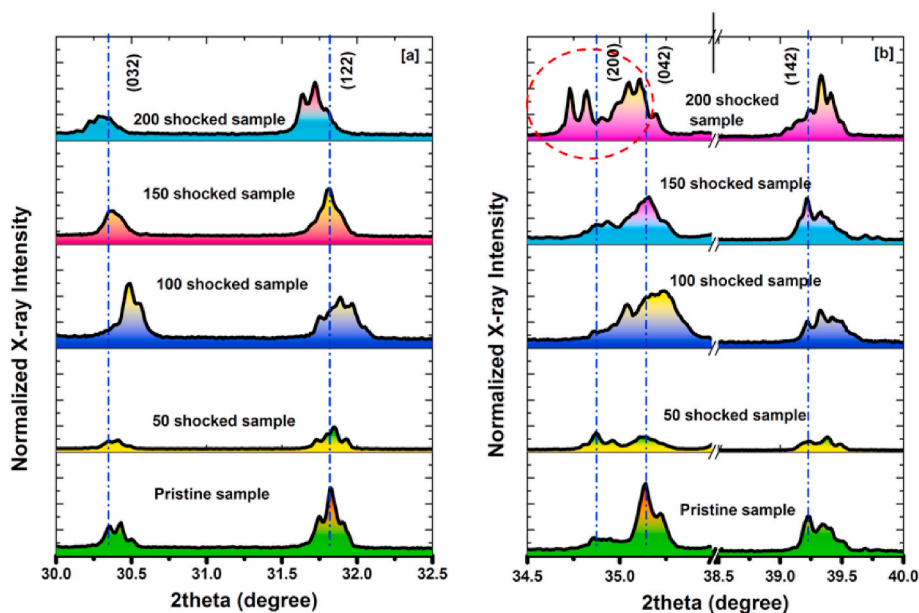


Fig. 5. Zoomed-in versions of PXRD patterns of the pristine and shocked Li_2SO_4 doped L-Threonine sample (a) $30\text{--}32.5^\circ$ (b) $34.5\text{--}40^\circ$.

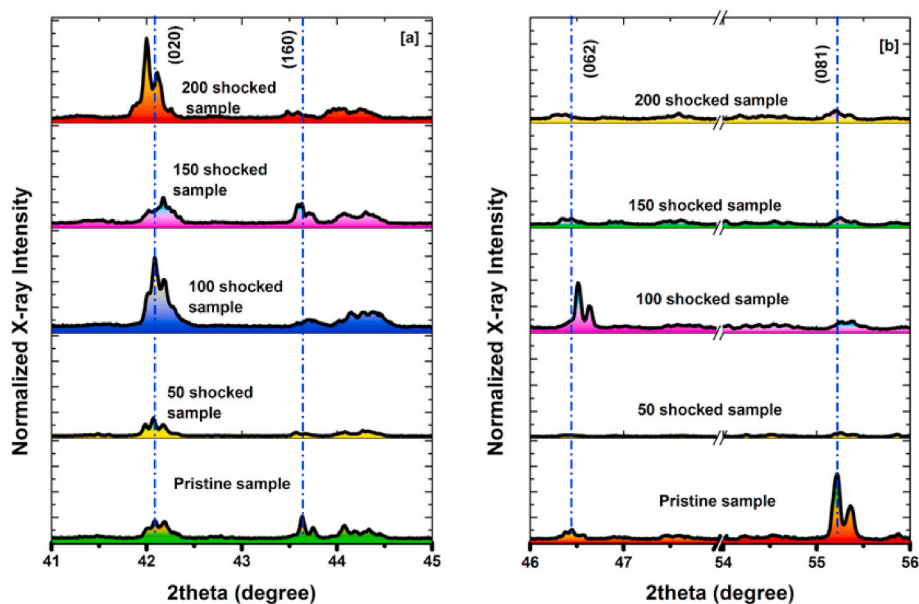


Fig. 6. Zoomed-in versions of PXRD patterns of the pristine and shocked Li_2SO_4 doped L-Threonine sample (a) $41\text{--}45^\circ$ (b) $46\text{--}56^\circ$.

intensity variations and appearance/disappearance of diffraction peaks as well as diffraction peak shifts under shocked conditions.

Zoomed-in versions of the XRD patterns of the pristine and shock wave-exposed samples are provided in Figs. 3–6 so as to ensure better visibility of the above-mentioned changes under shock wave-exposed conditions and also it would lead to a better understanding of the shock wave impacts. As seen in Fig. 3a, the pristine sample's diffraction peaks of (011) and (110) are found to have relatively high normalized X-ray intensity wherein both the peaks have a few shoulder peaks due to the generation of integral grain-boundaries and slip planes [27]. But at 50 shocked conditions, the intensity of the (011) and (101) planes have been significantly decreased by the impact of shock waves which might be due to the re-organization of hydrogen bonds in the host crystal structures and the existence of rational disorder of Li_2SO_4 under shocked conditions [3,21]. Furthermore at 100 shocked conditions, on the one hand, the diffraction peaks of (011) and (110) have experienced a

slightly higher angle shift and on the other hand, significant enhancement of X-ray peak intensity is noticed in (011) and (110) plane which may be due to the occurrence of shock wave induced dynamic re-crystallization [3]. Followed by 50 as well as 100, in 150 shock pulses exposed conditions also significant changes are observed such as the formation of several shoulder peaks in the (110) plane which is indicated by the red circular mark in Fig. 3a. Moreover, a few lower angle shifts have been observed as compared to 100 shocked conditions as well as for 150 shocks loaded sample. Similar kinds of the above-mentioned changes have been observed in (101) and (111) planes with respect to the numbers of shock pulses and the corresponding XRD patterns are shown in Fig. 3b. But in particular, the intensity of the (101) plane is considerably increased and the intensity of the (111) peak is considerably reduced which may be due to the occurrence of re-orientational changes occurring between two crystallographic planes caused by the impact of shock waves and such kind of changes have been observed in

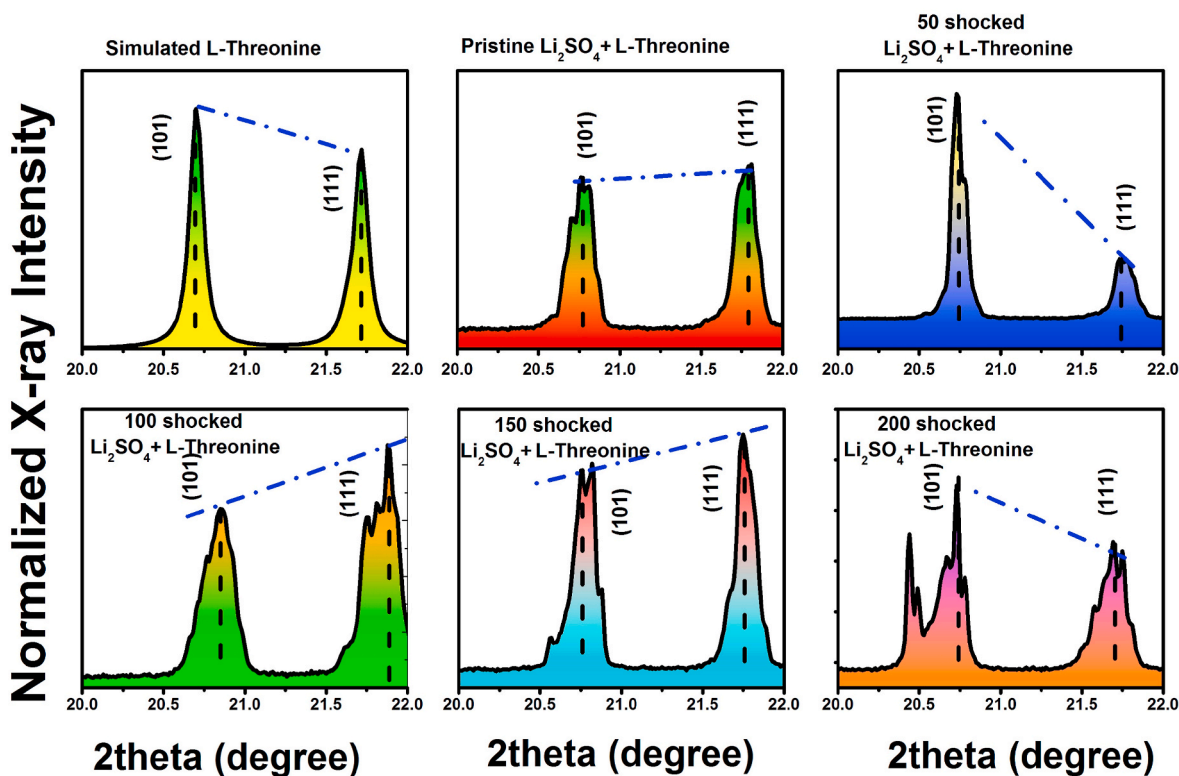


Fig. 7. Comparison of the XRD profiles of the pristine and shocked samples for the (101) and (111) planes with simulated pure L-Threonine sample.

KDP and GPI powder samples [11,14]. On the other hand, in the case of the (101) plane, one new plane has emerged at 20.44° and it could be noted that such kind of prominent diffraction plane is not present either in the pristine sample or in the pure L-threonine sample. Hence, it is clear that the formation of a new crystalline peak may be due to the occurrence of re-orientational hydrogen bonds and the new diffraction peak can be called a high-pressure diffraction peak. In addition to that, in the case of the title sample, strong compression might have developed along the b-axis because of the reduction of intermolecular cohesion and intermolecular interaction energy occurring between the amino groups and the carboxyl oxygen atoms which are clearly demonstrated by the modifications that have occurred in crystal symmetry and conformational changes [21,22].

In Figs. 4–6, zoomed-in versions of the XRD profiles of the respective (031), (002), (121), (040), (041), (112), (032), (112), (200), (042), (142), (142), (020), (160), (062) and (081) planes are presented and it is observed similar changes that have been witnessed in Fig. 3. At this stage, lots of internal structural modifications might have taken place such as distortion of the hydrogen bond networks and rotation of molecular fragmentation as well as changes in the conformational angle because of the impact of shock waves [28]. Furthermore, significant changes have been observed as that of the appearance of new diffraction peaks as well as the disappearance of the existing peaks by the impact of shock waves.

As seen in Fig. 6b, there are two diffraction peaks that have appeared at 46.5° (062) and 55.24° (081). Among these two diffraction peaks, the (062) peak has very less X-ray intensity in the pristine sample and (081) has considerable X-ray peak intensity. Surprisingly, at 50 shocked conditions, the peak intensity of (062) is suppressed and again at 100 shocked conditions which have been experienced due to the rapid growth of the plane caused by the dynamic re-crystallization and compression and also by the expansion of the b-axis of the test sample. At 150 and 200 shocks loaded conditions, the plane completely disappears. Similarly, the pristine sample has the (081) plane whereas this peak completely disappears at all the shocked conditions. Under shock

wave exposed conditions, the behavior of the appearance of new diffraction peaks and the disappearance of the existing peaks is not a usual phenomenon since the majority of materials experience the lattice disorder under shocked conditions as exhibited by the non-linear optical materials as that of KDP, benzophenone and GPI [11,13,14].

Furthermore, based on the demonstration of the re-orientational changes caused by the shock wave impacts on the title sample it is quite possible to arrive at a clear understanding of the role of shock waves on materials' structural properties. Hence, it is imperative to select the crystalline planes of mid-order diffraction angles such as (101) and (111) so that the corresponding XRD profiles are shown in Fig. 7 and as seen in Fig. 7, the simulated plane (101) of the pure L-Threonine sample has quite a high X-ray intensity whereas (111) has quite low intensity compared to the (101) plane. In the case of the pristine sample, both the planes are identified nearly with the same X-ray intensity and those changes observed in Li_2SO_4 doped L-Threonine may be due to the incorporation of the dopant [20].

But at 50 shocked conditions, the (101) plane has a significant enhancement of X-ray intensity wherein the full width at half maximum (FWHM) has also increased whereas, in the case of the (111) plane, contradicting result is observed in terms of peak intensity but no peak shift change is observed. At 100 shock pulses exposed conditions, nearly the same XRD pattern and intensity ratio are observed when compared to the XRD pattern of the pristine sample. But at 100 shocked conditions, the FWHM is quite high compared to the pristine sample and an almost similar pattern is retained at 150 shocked conditions. At 200 shock pulses exposed conditions, it is clear that the intensity ratio between (101) and (111) is completely changed such that high-pressure peak of the test sample has been observed. It could be noted that such kind of peak is not present in the pure L-threonine as well as in the pristine sample. Hence, we can consider it as a high-pressure phase plane of the test sample. Such kind of formation of planes may induce significant changes in the NLO efficiencies of materials [11,29,30]. The continuous changes observed for the test sample in terms of the crystalline planes' intensity ratios against the shock counts illustrate that the hydrogen

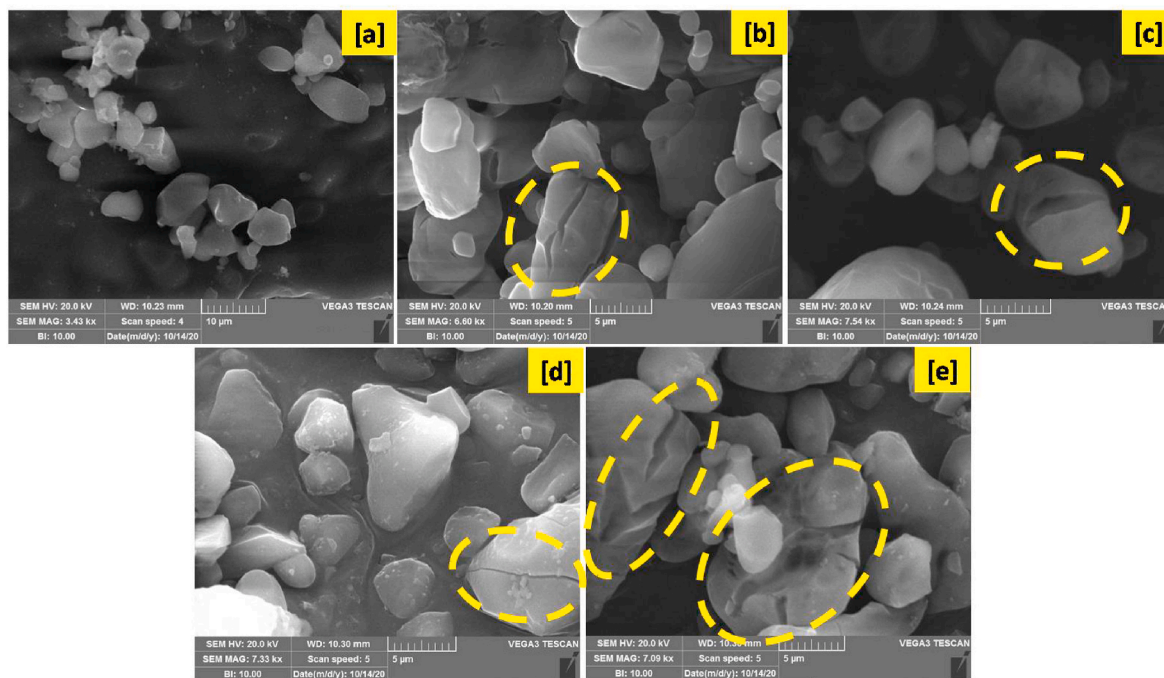


Fig. 8. SEM images of the pristine and shock wave exposed samples.

bond network is continuously changed accordingly. But no crystallographic change is observed even at 200 shocked conditions.

3.2. Morphological properties

Scanning electron microscopic (SEM) technique has been carried out to monitor the surface morphological changes of the test sample since there are plenty of possibilities for significant morphological changes to occur under shock wave exposed conditions [2]. On the other hand, in the case of optical materials, surface defects play an essential role in the harmonic generation of the samples [31,32]. Hence, surface morphology analysis is highly required under shock wave exposed conditions in order to consolidate the XRD results of the samples. In Fig. 8, the captured SEM images of the pristine and shocked samples are presented. As seen in Fig. 8a, the morphology of the pristine sample looks like that of a plate-like hollow sphere. As seen in Fig. 8a, there are no visible surface damages and deformations that are observed on the surfaces of the test sample. Hence, it is clear that the test sample does not have any surface defects, cracks and deformations prior to the shock wave exposed conditions. At 50 shock pulses exposed conditions, a significant crack is noticed and the corresponding SEM micrograph is portrayed in Fig. 8b wherein this crack might have been formed due to the shock wave impacts. During the shock wave loaded conditions, a high dynamic impact has been created on the sample such that, due to the instability of the test sample's surface morphology, it gives in to suffer a few surface modifications such as deformations, cracks, changes in shape etc. In the present case, no surface morphological shape change is observed. But some cracks are highly visible in the range of $5\ \mu\text{m}$ and the crack level is found to be increasing with respect to the number of shock pulses such that the positions of significant cracks are pointed out in yellow circles and the corresponding SEM micrographs are presented in Fig. 8. More interestingly, at 200 shocks loaded condition, the particles in the sample have experienced a significant shock crushing due to high transient pressure so that the particles' shape is entirely changed at this stage.

4. Conclusion

To draw the summary of the present investigation, the impact of dynamic shock waves on Li_2SO_4 doped L-Threonine sample has been studied in such a way that its structural and surface morphological properties have been systematically examined by diffraction and microscopic methods and the observed results are presented. Based on the obtained diffraction profiles, the test crystal does not undergo any structural phase changes even though other crystal forms coexist such as D and DL - Threonine. But, subtle changes have been noticed such as peak shift, new peak appearance and disappearance of existing peaks against the shock wave impulsion such that these changes might have occurred due to the re-organization of the three-dimensional hydrogen bond network of the host crystal structure and rotational order-disorder behavior of the dopant influenced by the impact of shock waves. SEM micrographs provide the required evidence for the formation of cracks under shocked conditions. From the crystallographic point of view, it could be noted that the test sample has relatively higher shock resistance than that of the most familiar hydrogen-bonded non-linear optical materials such as KDP and GPI samples [11,13]. Hence, Li_2SO_4 doped L-Threonine can be considered the right candidate for device fabrications. It is believed that this research work may open up a potential platform with which it is possible to understand the properties of amino acid materials under shocked conditions since, so far, a very less number of reports are only available on the impact of shock waves on materials, especially on amino acids.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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