



## Tuning of lower to higher crystalline nature of $\beta$ -L-Glutamic acid by shock waves

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### ABSTRACT

Understanding the crystallographic phase stability and solid – state new phases as well as their functions of materials under shock wave loaded conditions always generate a great deal of special enthusiasm among the researchers due to the emergence of lots of known and unknown behaviors of materials. Hence, such kind of shock recovery experiments is becoming one of the most promising research areas in materials science branch. In the present course of work, we have made systematic investigation for the assessment of crystallographic phase stability of  $\beta$ -L-Glutamic acid crystal under shocked conditions employing X-ray diffraction technique. From the diffraction measurements, it has been observed that the test materials turn from a lower crystalline nature to a higher crystalline nature due to the occurrence of dynamic recrystallization and reduction of structural complexity of the title material. Based on the diffraction assessments, it is confirmed that the test sample has not undergone any crystallographic phase changes even though it has several polymorphs.

### 1. Introduction

In recent years, the majority of shock wave recovery experiments conducted on solids have come up with significant changes enforced on them such that it has been observed crystallographic phase transitions and other structural related properties which have been very effectively documented by several researchers [1,2]. On the one hand, in dynamic shock wave-loaded conditions, maintaining the crystallographic phase of materials is a highly challenging task since majority of the materials undergoes phase transition behavior under shocked conditions [3–5]. On the other hand, in some cases, a few of the materials show different structural characteristics rather than the original structural signatures. In such cases, the identification of the original phase of the shocked sample is another added head-ache in solid-state science since lots of results are observed under shocked conditions. Hence, there have been witnessed lots of experimental studies to understand the relation between shock pressure and phase stability of the materials of both bulk and nano size [6–11]. Moreover, assessment of the phase stability of materials under shocked conditions is one of the standout fundamental

researches in materials science which is highly required so as to unravel the mysteries of materials under high pressure and temperature and also to find the right materials which can actually fulfill the industrial requirements to the maximum possible extend.

Few of our earlier publications have come up with the experimental results of crystallographic phase stability of technologically important non-linear optical crystals such as ammonium dihydrogen phosphate (ADP), potassium dihydrogen phosphate (KDP), Glycine phosphate (GPI) under shocked conditions which have revealed significant changes in structure and structure-related properties [12–14]. Going through a few highlights of the past publication results, interestingly in the case of ADP crystal, peak intensity of the pyramidal face (101) is significantly increased due to the stimulation of preferred orientation by the impact of shock waves [14]. In the case of KDP crystals, a few diffraction peak intensities have completely dropped while a few diffraction peak intensities have increased [14]. Surprisingly, both crystals have not shown any crystallographic phase transition under shocked conditions. Followed by ADP and KDP crystals, of late, while examining the benzophenone sample for crystallographic phase stability under shocked

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conditions, it has been found that benzophenone has high shock resistance behavior even though it has low melting point [15]. On the other hand, reversible phase transition has been witnessed in potassium sulfate crystal with respect to the number of shocks [3]. Furthermore, crystallographic phase transition has been observed in titanium oxide ( $\text{TiO}_2$ ) and cobalt oxide ( $\text{Co}_3\text{O}_4$ ) nano crystalline materials [16].

In the present course of work,  $\beta$ -L-Glutamic acid poly crystal has been chosen to the shock wave recovery experiments in such a way that the assessment of phase stability for  $\beta$ -L-Glutamic acid sample could be achieved by experimenting it under dynamic high pressure and temperature. The current research work is undertaken due to two prime reasons i.e. Firstly, Glutamic acid is one of the important amino acid crystals and it has variety of crystallographic phases such as  $\alpha$ -L-Glutamic acid (Orthorhombic- $P2_12_12_1$ )  $a = 7.012 \text{ \AA}$ ,  $b = 8.762 \text{ \AA}$  and  $c = 10.273 \text{ \AA}$ ,  $\beta$ -L-Glutamic acid (Orthorhombic- $P2_12_12_1$ )  $a = 5.139 \text{ \AA}$ ,  $b = 6.879 \text{ \AA}$ ,  $c = 17.246 \text{ \AA}$ ,  $\text{D}$ -Glutamic acid ( $P2_12_12_1$ ), and DL-Glutamic acid (Orthorhombic  $Pbca$ )  $a = 9.124 (2) \text{ \AA}$ ,  $b = 15.505 (7) \text{ \AA}$  and  $c = 10.629 (4) \text{ \AA}$  [17–20]. In  $\alpha$ -L-Glutamic acid crystal packing, four zwitterionic  $\alpha$ -GLU molecules are present per unit cell and each  $\alpha$ -GLU zwitterion forms four hydrogen bonds with the neighboring molecules wherein three of them originate from the amino group and two adjacent carboxylate oxygens and the carbonyl oxygen while one from the carboxyl OH (totally, three  $\text{H}\cdots\text{COO}^-$  bonds and one  $\text{H}\cdots\text{COOH}$  bond) whereas in the case of the  $\beta$ -L-Glutamic acid, four molecules are present per unit cell and the hydrogen bonds such as  $\text{O}\cdots\text{O}$  take place between the carboxylic and carboxylate units while  $\text{N}\cdots\text{H}\cdots\text{O}$  bonds are created between the ammonium unit and an oxygen atom in the carboxylic and carboxylate units. In the case of DL-glutamic acid, molecules are in the zwitterionic form and the crystal network is stabilized mainly by  $\text{N}\cdots\text{H}\cdots\text{O}$  and  $\text{O}\cdots\text{H}\cdots\text{O}$  interactions occurring between the molecules of the amino acids themselves and water molecules. The hydrogen bonded materials are suspected to undergo the structural transitions under high pressure and high temperature conditions [17,18].

Hence under shocked conditions, there are plenty of possibilities for crystallographic phase transition to occur. Secondly, few researchers are trying to understand the origin of the lives in the universe by the impact of shock waves on amino acids experimentally because lives began by the simplest molecules which are the combination of elements such as carbon, hydrogen, oxygen, and nitrogen [21–25]. In addition to that, it is one of the important materials which have the capability of second harmonic generation which is used for photonic applications [26,27]. Based on the first reason, it has been found in a few reports on Glutamic acid crystal under static high-pressure experiments such that Luz-Lima et.al have measured the phase stability of  $\alpha$ -L-Glutamic crystal at high-pressure region up to 7.5 GPa using Raman spectroscopy and found some interesting changes such as peak shifting and peak broadening due to the occurrence of disorder enforced by pressure. Similar results have been reported [28] and a few more related experiments have been carried out by the same research group and have come across almost similar results [29]. Followed by  $\alpha$ -L-Glutamic crystal,  $\beta$ -L-Glutamic crystal has also been subjected to high-pressure experiment up to 21 GPa by Luz-Lima et.al who have observed significant changes in the Raman spectra [30]. Victor et al. have reported the pressure-induced phase transitions in DL-Glutamic acid crystal exposed up to 12.4 GPa and obtained three different conformational phase configurations occurring at 0.9 GPa, 4.8 GPa and 12.4 GPa, respectively [31]. It has been realized from literature survey carried out based on the above-mentioned reports wherein no information has been found on the crystallographic phase transition from one phase to another known phase. But, the parent crystal systems in the samples have been affected by the pressure-induced deformations in hydrogen bond networks so that such changes have led to the different molecular and atomic configurations.

Based on the static high-pressure results,  $\beta$ -L-Glutamic acid crystal has been identified as an ideal material for investigation under shocked conditions due to the possibility of obtaining phase transition caused by its weak hydrogen bonds and weak intermolecular interactions. Hence,

the investigation on phase stability of  $\beta$ -L-Glutamic acid crystal has been carried out under shocked conditions by employing diffraction and spectroscopic techniques whereby the expected phase transition is like that of a static high-pressure experiment [30,31].

## 2. Experimental sections

Commercially purchased  $\beta$ -L-Glutamic acid powder sample (Sigma Aldrich Company with 99.98% phase purity) has been used for the shock wave recovery experiments. Before conducting the experiments, the test sample has been finely grained using a mortar and thereafter the sample has been equally divided into five equal parts in such a way that four specimens have been utilized for shock wave-loading and one specimen has been kept as the control sample. The shock tube working methodology and shock wave-loading procedure have been discussed in the previous publications [15]. In the present experiment, we have used shockwaves of Mach number 2.2 which has transient pressure and temperature 2.0 MPa, 864 K, respectively. Subsequently, 50,100,150 and 200 shock pulses have been loaded successively on the four specimens and sent for XRD measurement. A Powder X-ray diffractometry (PXRD) [Rigaku – Smart Lab X-Ray Diffractometer, Japan-  $\text{CuK}\alpha_1$  as the X-ray source ( $\lambda = 1.5407 \text{ \AA}$ ), with the step precision of  $\pm 0.001^\circ$ ] has been utilized for this study.

## 3. Results and discussions

### 3.1. XRD diffraction results

Powder X-ray diffractometer has been utilized to evaluate the phase stability of the title sample under shocked conditions and the observed XRD profiles are presented in Fig. 1. It is essential to confirm the phase purity as well as the phase formation of the test sample before observing the shock wave-induced changes occurring in the test sample. Hence, systematic comparison of the control sample has been carried out with the observed XRD profile and the standard XRD profile of  $\beta$ -L-Glutamic acid i.e., powder XRD pattern (JCPDS: 32–1701) and the corresponding figure is presented in Fig. 2.

From the observed comparison analysis, it is confirmed that the test sample has  $\beta$  form of L-Glutamic acid with the orthorhombic crystal system possessing the space group of  $P2_12_12_1$  [26]. In Ref. [26] also used the  $\text{Cu K}\alpha$  X-ray source to record the XRD pattern of the sample. The observed slight shift in the XRD patterns may be due to the poor

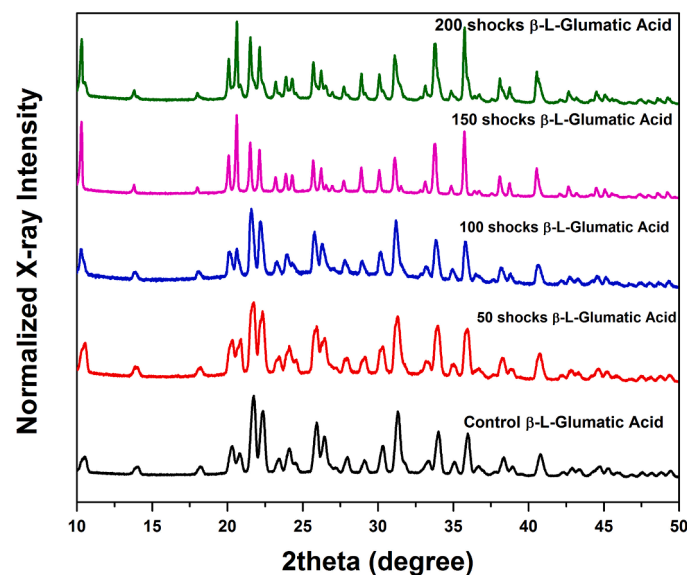


Fig. 1. XRD pattern of the control and shocked  $\beta$ -L-Glutamic acid sample.

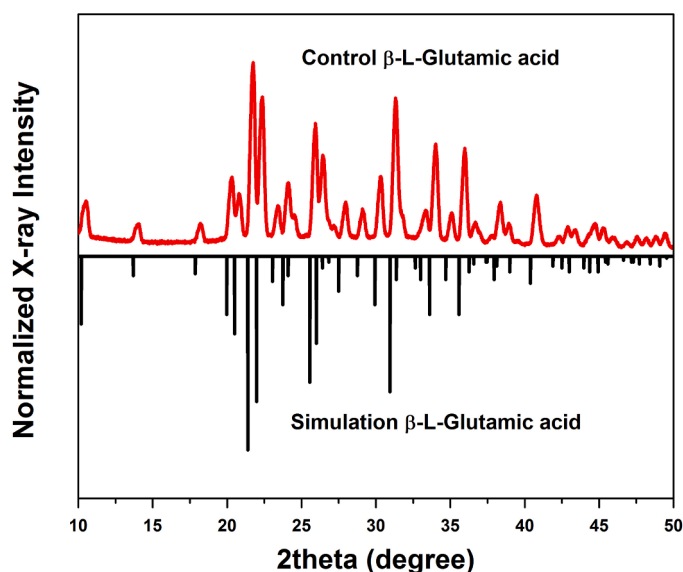


Fig. 2. Comparison of the XRD patterns of the control and the standard  $\beta$ -L-Glutamic acid sample.

crystalline nature of the control sample not due to the X-ray source. While comparing to the standard XRD pattern, it is very clear that the test sample has considerably undergone a higher angle shift and high peak broadening as well as lower peak intensity. It should be noted that, the above-mentioned parameters are the clear signatures of the lower crystalline nature of the samples. But based on the comparison results, it

is clear that the test sample has only one crystallographic face and no mixed phase is observed.

As the shock wave-induced changes are analyzed for the title sample, a couple of differences could be seen in Fig. 1 which are reflected in angular shift and full-width at half maximum of the diffraction peaks. While increasing the number of shock pulses, the diffraction peaks tend to move towards the lower angle side. On the other hand, full-width at half maximum of the diffraction peaks is reduced while the number of shocks is increased and at 200 shocks, significant reduction of peak width results in the enhancement of peak sharpness. For a better understating of these changes, selected area of diffraction angles in the XRD patterns are presented in Figs. 3 and 4. As witnessed in Fig. 3a, it is clear that the diffraction peaks such as (020) and (110) have high peak broadening due to the higher structural complexity, higher residual strain cross linked hydrogen bond formation between N-H... O and O-H...O and lower crystalline nature [30]. Under shocked conditions, there is a significant enhancement in the peak sharpness and lower angle peak shift which might be due to the contribution of elongation of hydrogen bond networking (lattice relaxation) and dynamic recrystallization. Relatively similar changes are also observed in the (110) plane under shocked conditions that might be due to a strong modification which could have taken place in the atomic structure. Followed by the (020) and (110) planes, (021), (040), (101), (111), (031), (121) and (140) planes also reflect similar changes and the corresponding XRD is presented in Fig. 3b. Moreover, it should be noted that up to 100 shocks, relatively moderate changes only appear but at 150 shocks, highly pronounced changes are observed and it may be due the thermal softening and shock wave-induced dynamic re-crystallization process. On the other hand, it is highly expected that the hydrogen bond lengths could be increased which might lead to the lower crystalline nature

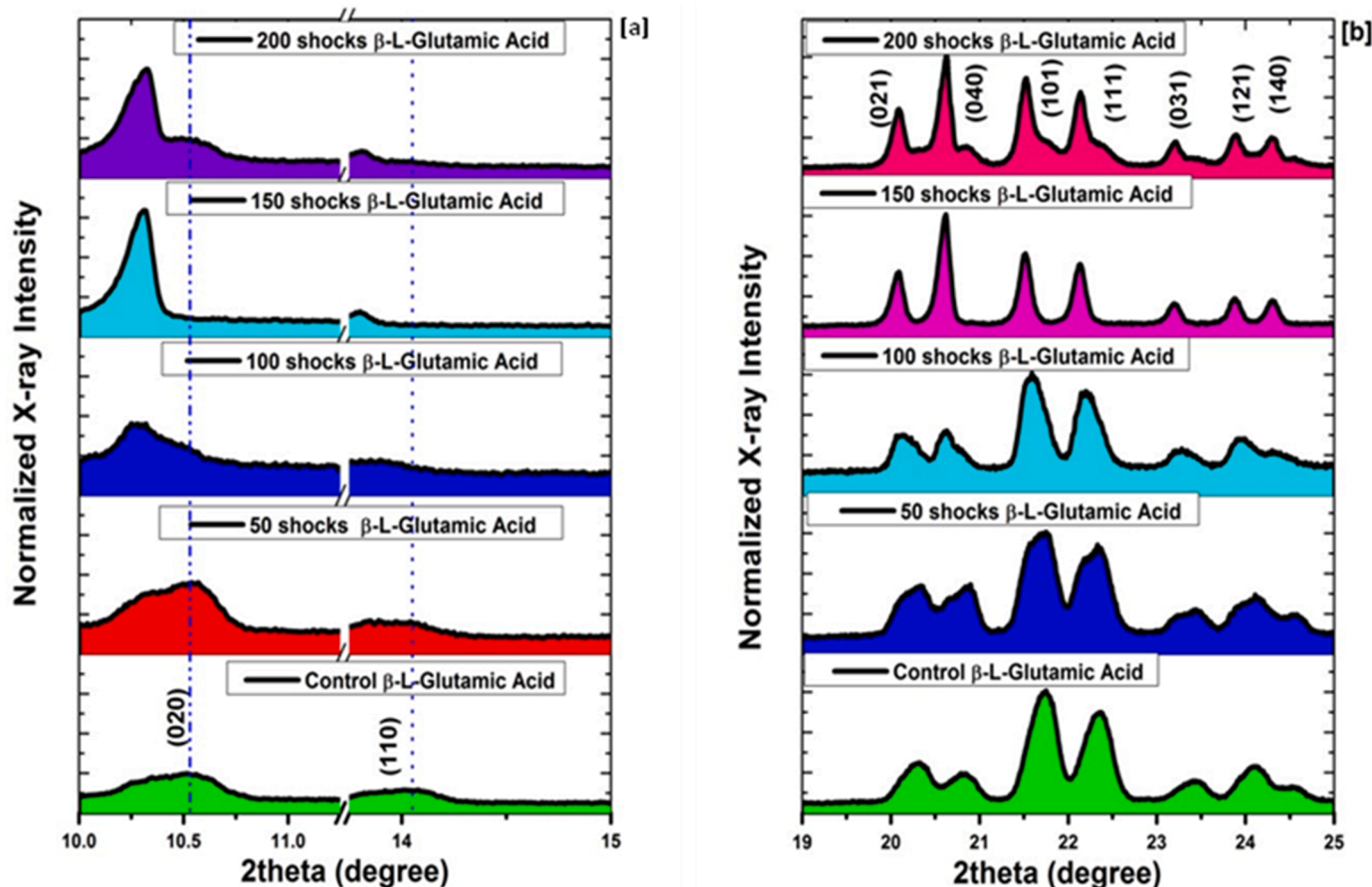


Fig. 3. XRD patterns of selected region of the control and shocked  $\beta$ -L-Glutamic acid sample [a] 10–15° [b] 19–25°.

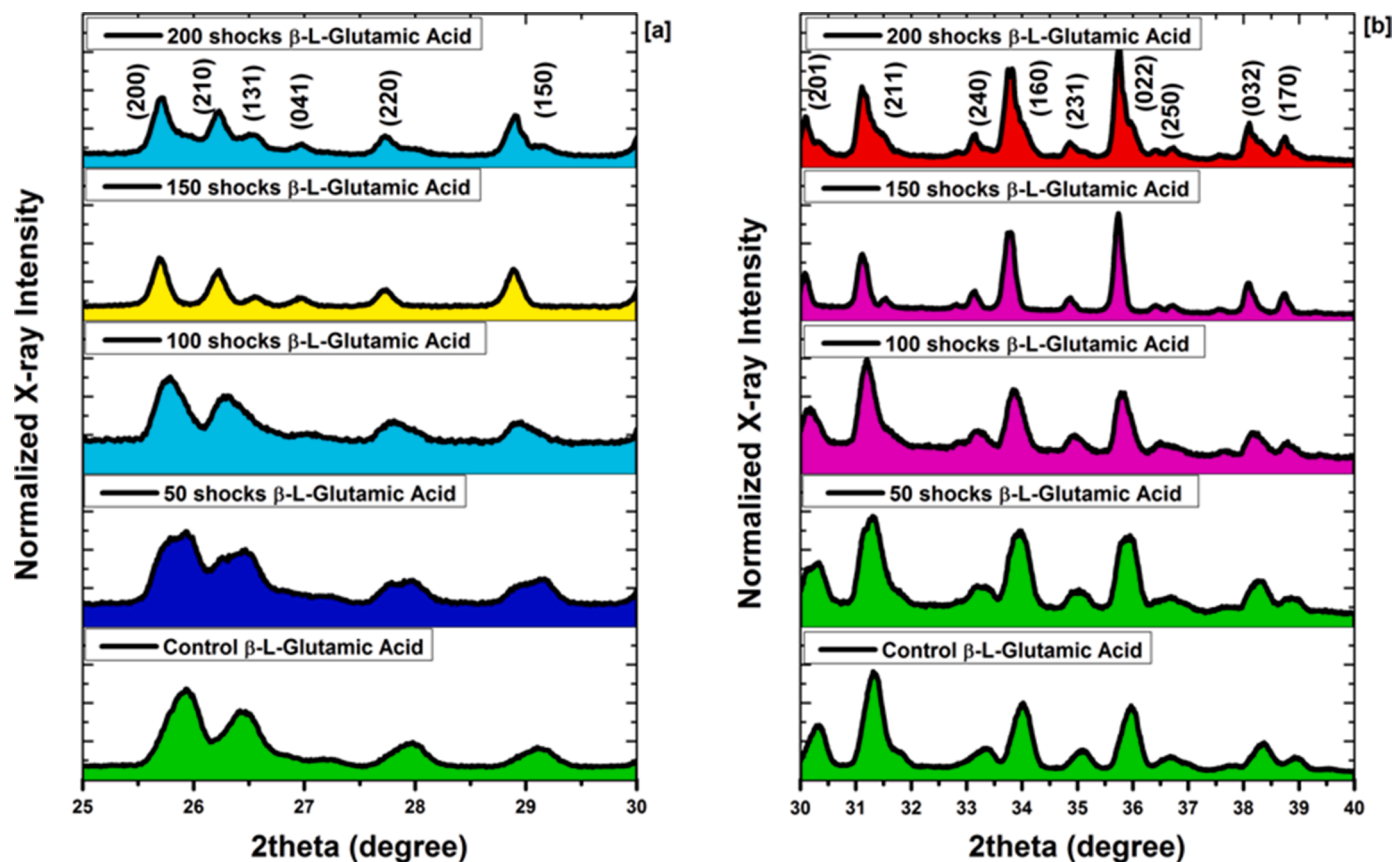


Fig. 4. XRD patterns of selected region of the control and shocked  $\beta$ -L-Glutamic acid sample [a] 25–30° [b] 30–40°.

moving towards the higher crystalline nature with the same space group while number of shocks are increased. Among the several diffraction peaks in Fig. 3b, the (040) peak shows the most pronounced changes under shocked conditions.

Fig. 4 also exhibits similar changes with respect to the number of shock pulses as it is followed in Fig. 3. Hence, it can be authenticated lucidly that the title crystal undergoes from lower crystalline nature to higher crystalline nature due to the dynamic recrystallization. For a better understanding of the peak shift of the title sample under shocked conditions, the data of Fig. 3 is considered such that plots are traced in terms of diffraction peaks positions as well as diffraction angular shift

with respect to the number of shocks and the corresponding figures are presented in Fig. 5. As seen in Fig. 5a, all the diffraction peak positions move towards the lower angle side with respect to the number of shocks. It should be noted that in normal circumstances, changes in diffraction peak shift either in lower angle or in higher angle observed in diffraction patterns are not considered as a major issue concerned with the existing changes occurring in the powder XRD spectra. However, lower and higher angular shifts observed in diffraction peaks are highly sensitive to high temperature and high pressure. Moreover, it should be proved further such that the observed changes occur in the entire crystalline system. Hence, peak positions of mid-order diffraction angle between 21

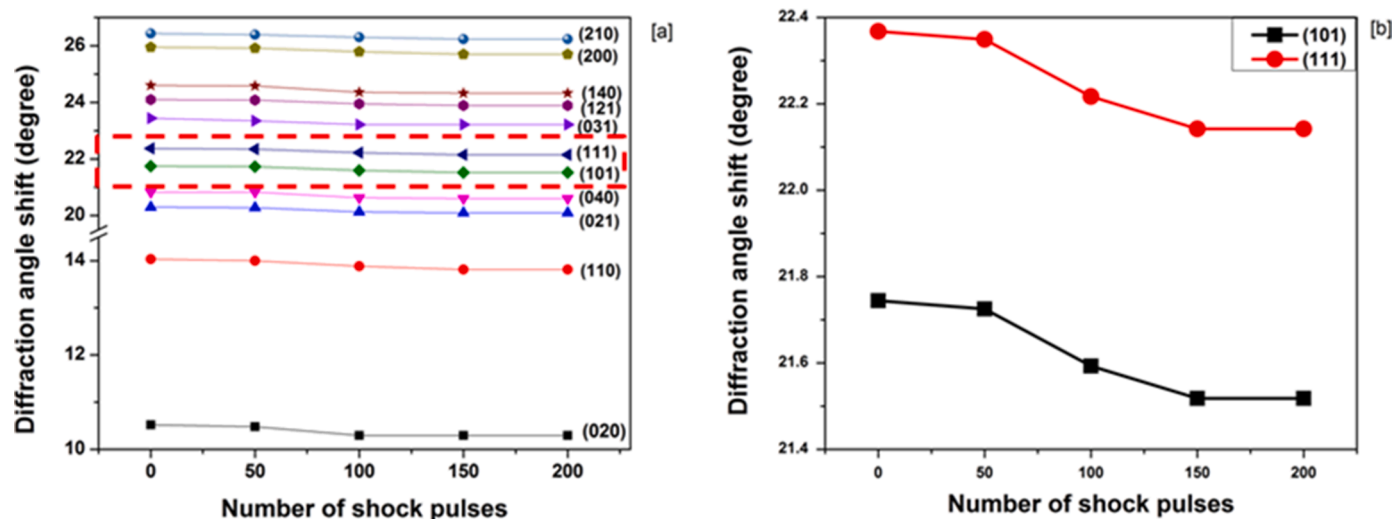


Fig. 5. [a] Diffraction peaks position Vs number of shocks [b] (101) and (111) peak positions Vs number of shocks.

and  $23^\circ$  under shocked conditions are selected and the corresponding peak positions are presented in Fig. 5b which comprehensively prove that the diffraction peak positions move towards the lower diffraction angle under shocked conditions.

Moreover, full width at half maximum (FWHM) values are also deserved to be given special attention since significant changes are observed under shocked conditions. Hence, the full width at half maximum of the (101) and (111) diffraction peaks are calculated under shocked conditions and the corresponding profile is presented in Fig. 6. As witnessed in Fig. 6, there is significant reduction observed in FWHM at 150 and 200 shocks while compared to the lower number of shocks and the control sample which is one of non-debatable signature proofs for the enhancement of degree of crystalline nature of the test sample under shocked conditions. Under shocked conditions, the internal grain boundaries may collapse due to the high transient pressure and temperature so that the crystalline nature of the test sample is enhanced. Especially for amino acids, several factors have to be considered to get a clear understating about the observed changes such as caloric effects, transition kinetics and more importantly role of hydrogen bonds and hydrogen bond lengths. As seen in Fig. 2, the control sample has higher angle shift as compared to the standard pattern. Hence, it is suggested to be one of the key factors to substantiate in such way that the control test sample has lower hydrogen bond length and lower cell volume as well as lower crystalline nature.

Under shocked conditions, the sufficient latent heat is supplied to the test sample so as to induce the recrystallization process which gradually increases while the numbers of shocks are increased. According to the literature, there are several publications available with which a better understanding of the shock wave-induced dynamic recrystallization in solid samples is pretty possible [1,2]. At shock wave loaded stage, complexity of the sample and residual stress are significantly reduced such that bond lengths may increase, especially the hydrogen bond lengths, due to the impact of shock waves. So that, the unit cell volume is expected to be increased due to the enhancement of bond lengths along with lattice parameters. Furthermore, it has been observed an interesting coincidence of the standard XRD pattern with the 200 shocks-loaded test sample's XRD pattern and the corresponding figures are presented in Fig. 7. As seen in Fig. 7, it is very clear that the crystalline peak (040) of the test sample has low diffraction intensity than that of the (021) peak whereas contrary result is found in the standard pattern. Surprisingly at 200 shocks, it is observed the coexistence of diffraction pattern and peak intensity being at the same level and for a

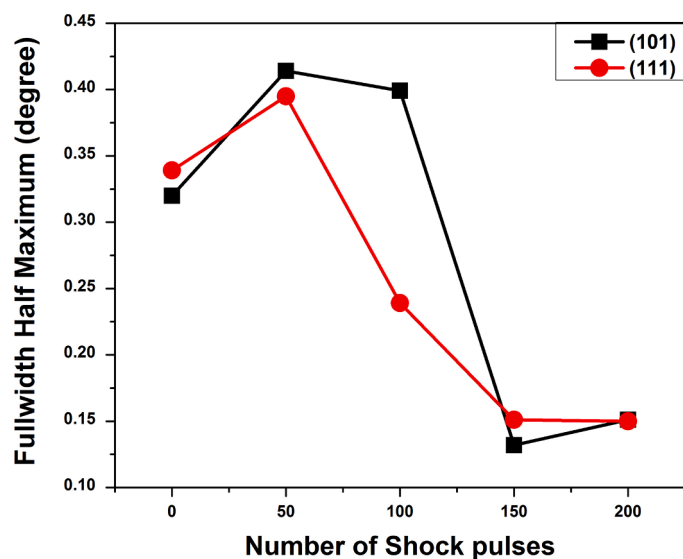


Fig. 6. FWHM values of the (101) and (111) planes under the control and shocked conditions.

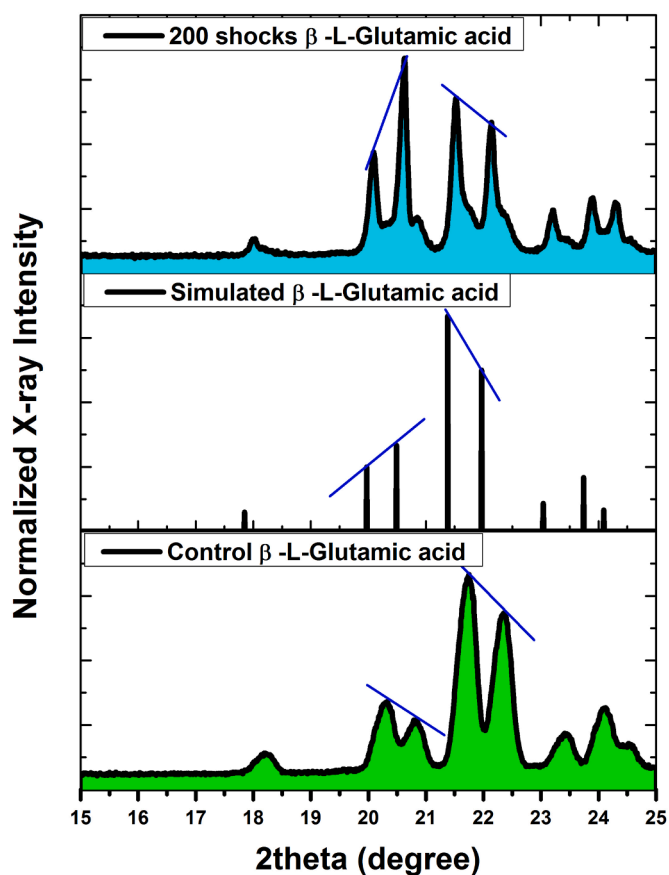


Fig. 7. Comparison of the XRD patterns of the control, simulated and 200 shocks-loaded samples.

better understanding of these changes, it is drawn a line between intensity ratios of the (021) and (040) as well as the (101) and (111). As reflected in Fig. 7, the (101) and (111) peak intensity ratio is not changed under shocked conditions which are found to be similar for the control, simulated and shock wave-loaded conditions. Note that, the acoustical shock waves can produce considerable changes in the crystallographic structures in the amino acids. For example, L-leucine exhibits conformational phase change from phase-I (low temperature  $P2_1$ ) to phase -II (high-temperature  $P2_1$ ) at the 50 shocked condition. But higher than 50 shocked conditions and up to 200 shocks, it is found that the stable crystal structure remains [32]. In the case of L-Asparagine, there is no crystallographic structural transition under shocked conditions but, several modifications are seen in the shapes of XRD peaks and their respective positions with respect to the number of shock pulses [33]. In such cases, the inter-hydrogen bonds lengths and their angles are greatly altered in a few materials that undergo phase transitions [32] while a few materials undergo structural deformations [33] and the above-mentioned changes highly depend upon the nature of the starting material (crystalline purity as well as crystallographic structure).

For a generalized conformation, it has been calculated the values of the lattice parameters of the control and shocked samples and the respective values are presented in Fig. 8 and the calculations have been accomplished by the UNITCELL software [34]. As it is in the previous results, in this case also, the values of lattice parameters are increased with respect to the number of shock pulses due to the shock wave-induced recrystallization process. Moreover, the observed values are compared with the standard values of the JCPDS database lattice parameters of  $\beta$ -L-Glutamic acid crystal and the obtained values are presented in Table 1. Based on the observed results, the values of lattice parameters of 200 shocks-loaded test sample are found to be more closely matched with the simulated XRD of  $\beta$ -L-Glutamic acid than that

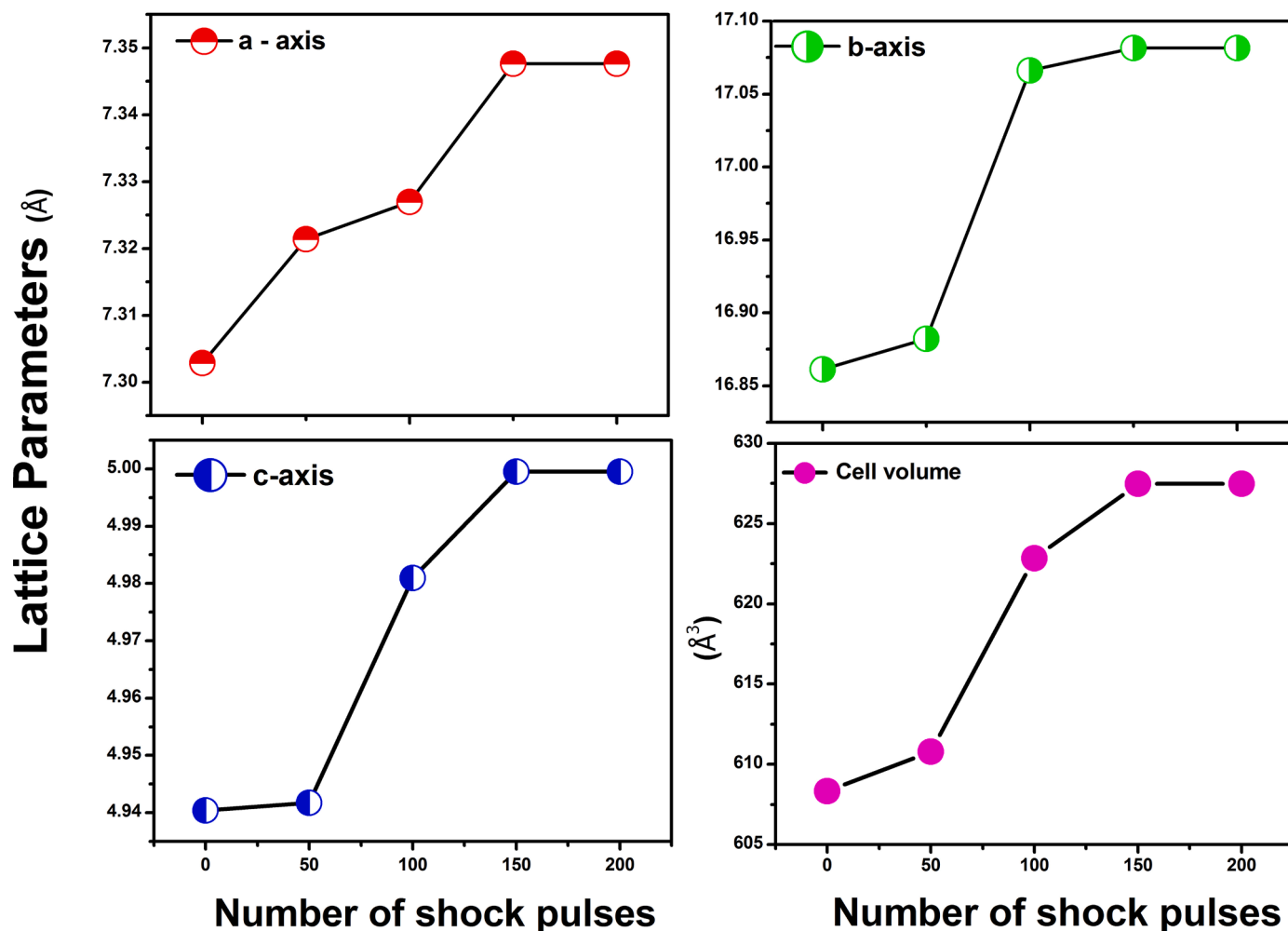


Fig. 8. Lattice parameter values of the control and shocked samples.

**Table 1**  
Comparison of the lattice parameters of  $\beta$ -L-Glutamic acid crystal.

Experiment condition	a (Å)	b(Å)	c(Å)	Cell volume (Å <sup>3</sup> )
Control sample	7.302	16.861	4.940	608
Standard Sample (JCPDS: 32-1701)	6.965	17.30	5.160	623
200 shocked sample	7.347	17.08	4.999	627

of the control sample. The overall outcome of the observed XRD results confirms that the control sample has lower crystalline nature than that of shock wave-induced sample which is due to the occurrence of dynamic recrystallization process. The average crystallite sizes have been calculated by employing the Debye-Scherrer formula and the obtained values are 28, 27, 30, 36 and 43 nm for 0, 50, 100, 150 and 200 shocks, respectively. Moreover, diffraction peak positions of the standard pattern as well as the shock wave-induced samples and peak intensity ratios are well corroborated with these results.

#### 4. Conclusion

As the concluding remarks are made for the present course of work, it has been demonstrated the phase stability of the technologically important  $\beta$ -L-Glutamic acid powder samples while exposed to a series of shock pulse-loaded conditions such as 0, 50, 100, 150 and 200 consecutively using a tabletop pressure driven shock tube. From the

experimental outcome, it has been observed that there is continuous lower angle shift with respect to the number of shocks and a significant shift is observed at 150 shocks. On the one hand, peak width is appreciably reduced and on the other hand, diffraction peak intensity is appreciably enhanced with respect to the number of shocks. Based on the changes observed in the diffraction pattern, it is strongly suggested that the hydrogen bond network expands due to the impact of shock waves and lattice parameters are also found to be increasing which in turn influence an increase to the unit cell volume and several high-pressure amino acid reports support the obtained observations. Hence, it confirms that the test sample turns from the lower crystalline nature to a higher crystalline nature based on the dynamic recrystallization mechanism and undergoes reduction of structural complexity. The major finding of the present study is that shock wave-exposed  $\beta$ -L-Glutamic acid sample has a better crystalline nature than that the control sample and the 200 shocked sample's XRD pattern is found to be well-matched with the standard crystallographic XRD data. Hence, it is strongly advocated that shock wave-induced samples could be used to carry out the crystal growth research to obtain a highly-ordered single crystal and related industrial applications of single crystals.

#### Compliance with ethical standards

None.

### CRedit authorship contribution statement

**A. Sivakumar:** Formal analysis, Writing – original draft. **Lidong Dai:** Supervision, Writing – review & editing. **S . Sahaya Jude Dhas:** Visualization, Writing – original draft. **Raju Suresh Kumar:** Formal analysis. **Abdulrahman I. Almansour:** Formal analysis. **S . A . Martin Britto Dhas:** Supervision, Investigation, Writing – review & editing.

### Declaration of Competing Interest

The authors declare no competing financial interests.

### Data availability

Data will be made available on request.

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