

Sustainability of crystallographic phase of α -Glycine under dynamic shocked conditions

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ABSTRACT

High pressure experimental studies on amino acids have gained incredible attention over the past few years in variety of research areas such as materials science, geophysics, pharmaceutical sciences, and cosmo-chemistry. On en-route to the current experimental work, one of the elegant amino acid samples of α -Glycine is preferred in order to reveal its nature of crystallographic phase stability as and when exposed to dynamic shock waves thereby subjecting it to diffraction and microscopic analyses. Considering the observed results from the various analytical techniques, it is authenticated that the title sample does not undergo any kind of structural transition under shock wave exposed situations. On the other hand, the test sample has generated a few new shoulder peaks and diffraction peaks under shocked conditions. The outcome of the attained phase stability is well substantiated by the previous static high-pressure reports.

1. Introduction

Over the years, it has been witnessed a persistent progress in trapping relatively stable materials under highly fluctuating environments such that those materials attract a great deal of technological importance maintaining a consistent demand so that shock wave experiments can provide substantial outcomes of the nature of materials under shocked conditions. Moreover, impact of dynamic shock waves on materials has been recognized as one of the simplest means to quickly access high pressure states of a material as compared to the shock compression and static high-pressure experiments so that shock wave impact analysis can reinforce the process of identifying the apt material for every specific application that is required for the modern day. In addition to that, under shock wave exposed conditions, majority of materials undergo crystallographic phase transitions that can be either reversible or irreversible phase transition [1–3]. On the one hand, in recent years, shock wave induced technique has been frequently used to understand the polymorphic nature of materials of both bulk and nano

size. On the other hand, it has provided an alternative way to find the high shock resistant materials for engineering applications [4–7]. As far as it is known, high pressure studies on amino acids are being tagged as one of the hot research topics in recent years even though they have almost 100 years of existence in the literature of high pressure [8–10]. Moreover, high pressure experiments of amino acids have provided key insights to better understand the formation of living organisms on the earth and several publications have been made on this topic [8–12]. Shock wave assisted synthesis of amino acids has recently opened a new branch in the research area of high-pressure amino acids on which very few publications have been made so far [13,14]. As part of shock assisted synthesis of amino acids, understanding the phase stability has materialized already as a prominent research topic as it has lots of academic interest and industrial applications. Hence, in the present experiment, the simplest amino acid of glycine (α -phase) has been chosen in order to assess the crystallographic structural stability under shock wave exposed conditions, since it has polymorphic phases such as α -phase ($P2_1/n$), β -phase ($P2_1$), γ -phase ($P3_1$), monoclinic ($P2_1$),

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tetragonal ($P4_3$), orthorhombic ($P2_12_12_1$), δ -phase (Pn), ζ -phase and X-phase ($P2$) [15–17]. Among the above-mentioned phases, α , β , and γ phases are the primary phases and these three phases are only possible to be accessed in the ambient pressure and the remaining phases are considered as high-pressure phases. In addition to that, among all the above-mentioned glycine phases, α -glycine has maximum research publications which include the experimental results of high pressure due to its potential applications [18–22]. According to the previously obtained assessments, the thermo-dynamical stability performance and high-pressure compression observations reveal that the crystallographic phase stability of glycine phases is of the order of $\gamma > \alpha > \beta$ [18,19,22]. As per the data available to date, α -glycine has been subjected to the pressure regions such as 8.7, 23, and 50 GPa for which no phase transition has been observed [18,22]. No investigated research article has been found for α -glycine under dynamic shock wave loaded conditions which reflect its phase stability.

In this context, it is prolific to perform the phase stability assessment of α -glycine under shocked conditions by employing diffraction and microscopic methods. It is imperative to carry out the present experiment under shocked conditions such that it would bridge the gap between understanding the phase stabilities of amino acid crystals and the role of hydrogen bonds.

2. Experimental section

Commercially available (Sigma Aldrich) α -glycine (99%) was purchased which was in fine powder form that has been utilized as such in order to carry out experiments of shock wave impact. To start with, the obtained powder has been divided into five equal parts out of which one has been kept as the pristine and the rest of four for shock wave loading. Shock wave of Mach number 2.2 has been used to measure the crystallographic structural stability under dynamic shock wave exposure situations. The shock tube working methodology and shock wave loading procedure have been presented in our previous publications and also provided in the supplementary section [7,23]. One shock pulse has the transient pressure of 2.0 MPa and transient temperature 864 K. Subsequently, 50,100,150 and 200 shock pulses have been loaded on four different title samples in the chronological order so as to be sent for diffraction and microscopic analyses.

3. Results and discussion

3.1. Structural properties

Powder X-ray diffractometer has been used to study the crystallographic phase stability of α -glycine under shock wave exposed conditions and the observed diffraction patterns are shown in Fig. 1. Beforehand to the analysis of induced changes in the title sample caused by the shock waves, the confirmation of the formation of the crystalline structure of the title specimen is very essential which would lead to the clear understating of the impact of shock waves. According to the reflection of Fig. 1, the obtained XRD pattern of the pristine sample is well matched with α -phase of glycine. Hence, it confirms that the formation of the host crystallographic is α -phase which has the following diffraction peaks such as (020), (–110), (011), (–120), (021) (–121), (031), (040), (111), (141), (210) and (–221). Among the listed planes, (031), (040), and (111) planes are the characteristic peaks of α -glycine (JCPDS: 32–1702) [24]. As seen in Fig. 1 by the naked eye, under shocked conditions, no signature has been found for any crystallographic phase transitions for the phases at room temperature and other high pressure glycine phases. Since α -polymorph zwitterions are known to be linked by hydrogen bonds with the formation of double anti-parallel layers, the interactions occurring between these double layers are entirely van der Waals in nature such that glycine has a relatively stable crystallographic phase under shocked conditions. On the other hand, while observing the diffraction profiles from a

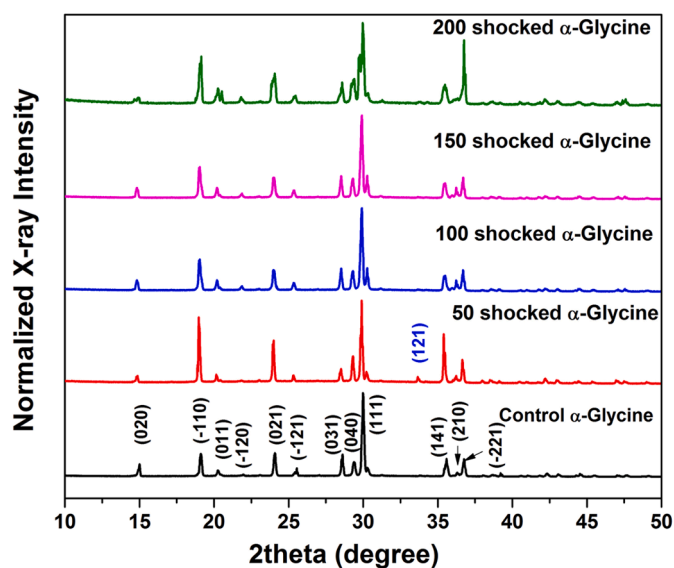


Fig. 1. XRD profiles of the pristine and shock wave treated α -glycine samples.

microscopic view, it is possible to visualize lots of changes such as peak shift, peak appearance/disappearance and formation of shoulder peaks in the mother phase of the sample. For better clarification of the above-mentioned changes and for the reader's benefits, the diffraction profiles of the zoomed-in versions are presented in Figs. 2 and 3.

According to the observed crystallographic structure of the α -glycine crystal, the cell parameters are $a=5.10$ Å, $b=11.97$ Å, and $c=5.46$ Å possessing the monoclinic system [22]. Based on the observed lattice parameter, it is clear that the b-axis is more compressible than the other axes. Hence, it has higher contribution during the experiments of pressure compression situation than the other axes. The XRD profiles of (–110) crystalline peak under pristine and shocked conditions are exhibited in Fig. 2a and a few interesting changes are observed under shocked conditions. At first, it is very clear that the test sample's XRD peaks move towards the lower angle under shocked conditions which may be due to the expansion of the hydrogen bonds influenced by the shocked waves. On the other hand, as seen in the pristine sample's XRD peak profile, there is no signature of shoulder peaks. But, under shocked conditions, there are a few shoulder peaks appearing and they are marked in blue aero-marks. In addition to that, the shoulder peak positions and peak natures have also changed against the shock pulses. At this stage, it is suspected that the hydrogen bonds may play a crucial role under shocked conditions and the position of glycine molecules might have continually changed against the number of shock pulses.

In the case of (021), at the loading of 200 shocks, three distinct shoulder peaks appear at the lower angle region. Moreover, in the (021) plane, b-axis has the largest length as compared to a and c-axes. Hence, at this point, the b-axis might undergo significant compression at the shocked condition in such a way that there would be an increased length for the hydrogen bonds occurring in $\text{CH}\cdots\text{H}$ and $\text{NH}\cdots\text{O}$ for both the axes of a and c [22]. Moreover, the α -hydrogen atoms present in amino acids are triggered to form $\text{CH}\cdots\text{H}$ bonding with the atoms of neighboring nitrogen. Furthermore, $\text{CH}\cdots\text{H}$ hydrogen bonds have less bond energy (few kJ mol^{-1}) that may be the reason for the appearance of significant number of shoulder peaks at the shocked condition. Besides, it is strongly expected that during the compression of the b-axis, variations are induced in the stacking of the interlayer of glycine molecules.

Followed by the observation of the (–110) and (021) diffraction peaks, similar results are also reflected in (–130), (031), and (040) as well as in (041), (210), and (–221) which are presented in Fig. 3a and b, respectively. It is remarkable to note that the planes of (031) and (040) bring about the formation of considerable number of shoulder peaks

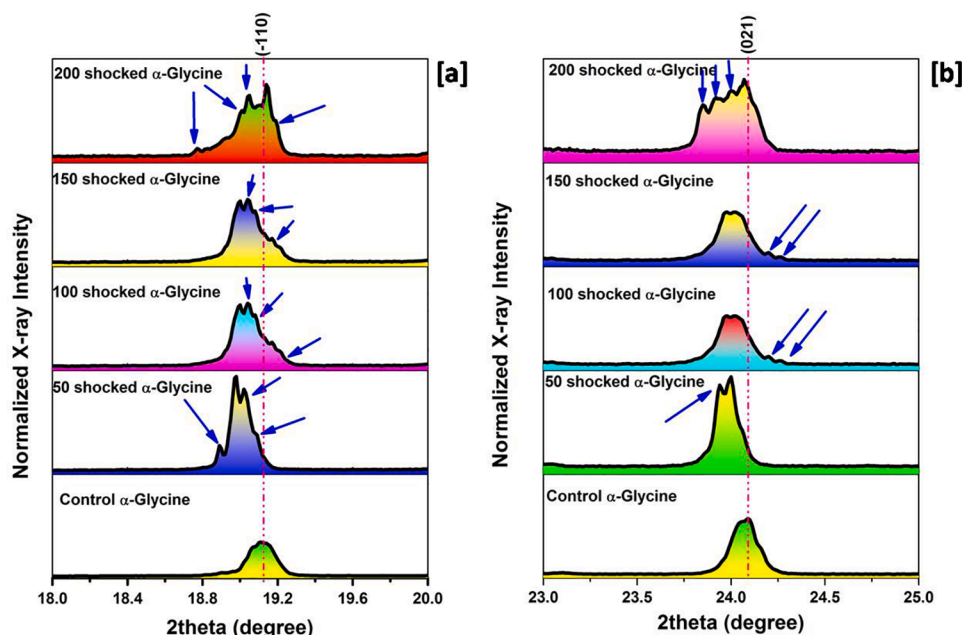


Fig. 2. The zoomed-in XRD profiles of the pristine and shocked α -glycine samples (a) 18–20 (b) 23–25 degree.

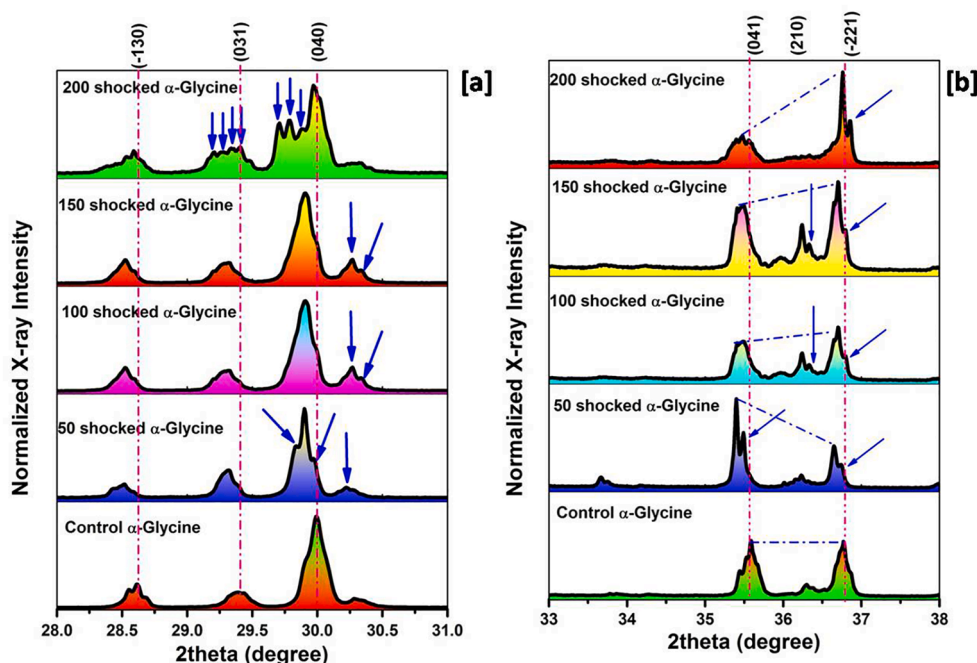


Fig. 3. The zoomed-in XRD profiles of the pristine and shocked α -glycine samples (a) 28–31 (b) 33–38 degree.

enforced by the shock waves. On the one hand, the (-130) , (031) and (040) planes are the characteristic peaks of α -glycine wherein none of the characteristic peaks have undergone any kind of peak shift. The lattice dimension of the b-axis' (11.97 \AA) length is almost two times higher than that of the a-axis (5.10 \AA) and c-axis (5.46 \AA) and hence the b-axis is highly favorable for the occurrence of lattice compression and lattice deformations by the influence of pressure than that of the a and c axes which is well-established in the static pressure experiments as well [25–27]. So that all of these characteristic diffraction peaks have experienced the formation of shoulder peaks as well as the reduction in the intensity of peaks which might be because of the suppression of the b-axis while exposed to shock waves which result in lattice compression

and lattice deformations [25–27]. The appearance of the shoulder peaks at the exposure of the shock waves might be because of the occurrence of deformations and dislocations in the crystal lattice. Interestingly, in Fig. 3b, it can be visualized the intensity ratio changes of (041) and (-221) in the X-ray scattering. Moreover, it could be noted that both (041) and (-221) belong to the rich planes of the b-axis. At the pristine stage, both the peaks have an equal amount of intensity whereas at the 50 shocked condition, (041) peak intensity has increased but (-221) peak intensity has reduced which may be due to the changes occurring in the nature of the hydrogen bonds wherein slight conformational changes are witnessed [10,16–18]. Furthermore, (-221) peak intensity has increased linearly at 100, 150 and 200 shocks loaded conditions.

Particularly at 200 shocks, (-221) has a higher intensity than that of the (041) plane, however from the observed results, it has become very clear that the anisotropic lattice compression has occurred under shocked conditions due to the enforced changes in the contribution of hydrogen bonds with respect to the crystallographic axis. Interestingly at the 200 shocked condition, the (210) crystalline plane has completely disappeared that may be the prime reason for the increment in the intensity of the (-221) plane which could be based on the lattice orientational effect taking place under shocked conditions [28] and it authenticates that the (210) plane has very less shock resistance. Fig. 4a presents the diffraction angle shift of major diffraction peaks against the number of shock pulses and the observed straight line between the points indicates that no significant peak shift is observed under shocked conditions.

Among the two planes, the (040) is comparatively quite a stable plane (Fig. 4b) against the impact of shock waves since it has a low peak shift deviation area. Hence, the prismatic α -glycine sample (040) could be suggested for manufacturing a variety of glycine compounds. Fig. 5 represents the crystal packing of α -glycine with the hydrogen bonds and the van der Waals bonds interactions. On the other hand, the characteristic peaks of α -glycine such as (-130) and (040) undergo peak shift against the number of shock pulses.

As a comparison is drawn between the pristine and 200 shocked α -glycine samples with respect to the standard XRD pattern, there is a significant scope for the understanding of the stable nature of the phase of α -glycine such that the corresponding XRD profiles are presented in Fig. 6. From the observed results, it is clear that the XRD patterns of the pristine and 200 shocked samples of α -glycine have the same crystal structure which confirms that α -glycine has relatively quite stable phase while hit by the shock waves and quite a lot of high-pressure research publications also support the observed result [18–22].

3.2. Morphological studies

SEM investigations of shock wave induced samples can provide the growth kinetics of the subjected specimens. Hence for the present investigation, scanning electronic microscopic technique (SEM) has been carried out on α -glycine samples for a better understating of the role of shock waves on surface morphology and the observed micrographs are depicted in Fig. 7. As seen in Fig. 7a, the pristine sample has similar to that of hemihedral surface morphology. But, actually as per the crystallographic phase of the α -glycine, it has to be the prismatic morphology along the b-axis [29–31]. While under the exposure of shock waves, the observed changes reflected in the surface morphology of the specimen in accordance with the number of shock pulses are displayed in Fig. 7(b–e).

Under shocked conditions, the formation of well-defined surface

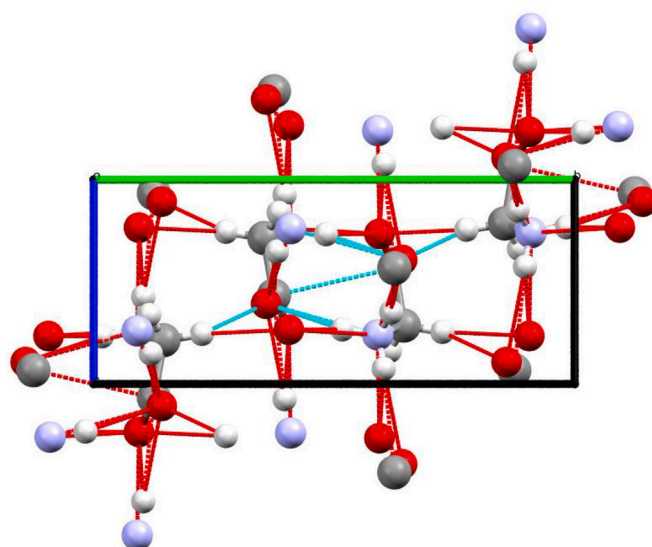


Fig. 5. The crystal packing of α -glycine (generated by CIF: CCDC 1,957,087).

morphology of the α -glycine samples has been observed such that the trigonal prismatic plane (010) is clearly visible. While the specimen is loaded with shocks, changes are induced in molecular polarization which could give rise to the preferred crystal orientation such that the resultant (010) and (011) planes are clearly observed. According to the previous publications and attachment energy theory [30,31] the (011) crystal plane has the fastest growth direction along the b-axis. Fig. 7d clearly shows the hemihedral surface morphology with the (011) orientation. It could be observed from the images of SEM that the specimen has a stable structure whereas it undergoes a few modifications in its surface morphology because of the induced dynamic re-crystallization occurring along the preferred orientations caused by the shock waves [32,33].

4. Conclusion

The final convergence is drawn to bring about the specific results of the present experimental findings that arise from the impacted shock waves which are based on the assessment undertaken for the phase stability of α -glycine in accordance with the number of shocks applied i. e., 50,100,150 and 200 counts. X-ray diffraction profiles indicate that the specimen's crystallographic phase is relatively stable against the impulsion of shock waves. On the other hand, it undergoes a few

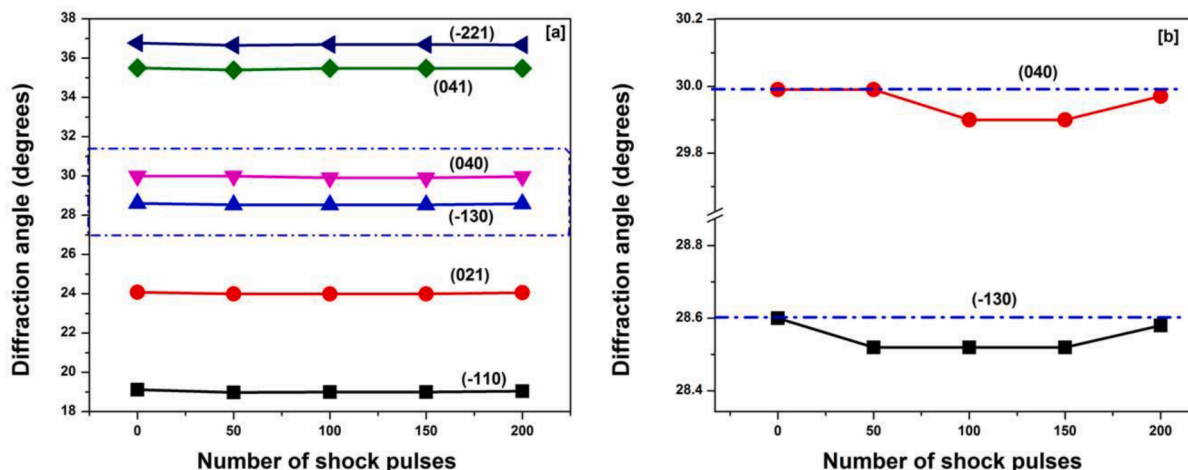


Fig. 4. Diffraction peak shift profiles of the pristine and shocked α -glycine samples.

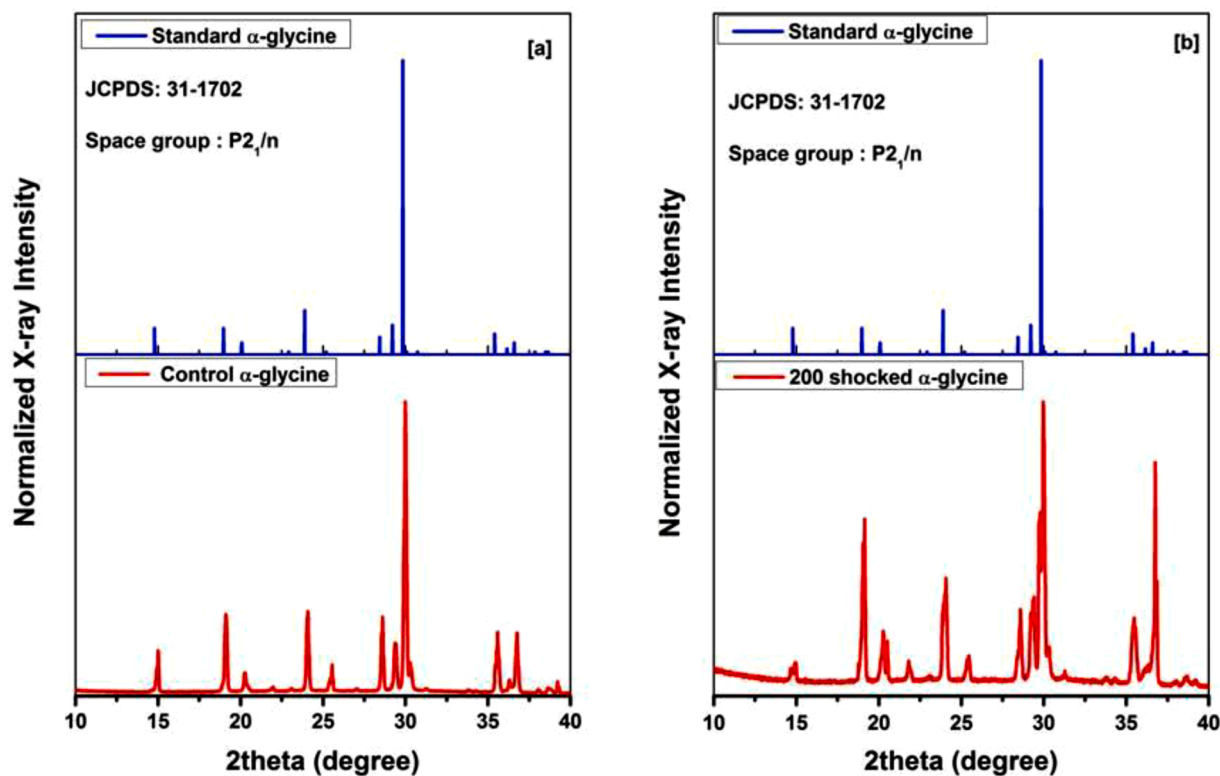


Fig. 6. Comparison of the XRD patterns of α -glycine samples (a) the pristine Vs standard pattern (b) 200 shocked sample Vs standard pattern.

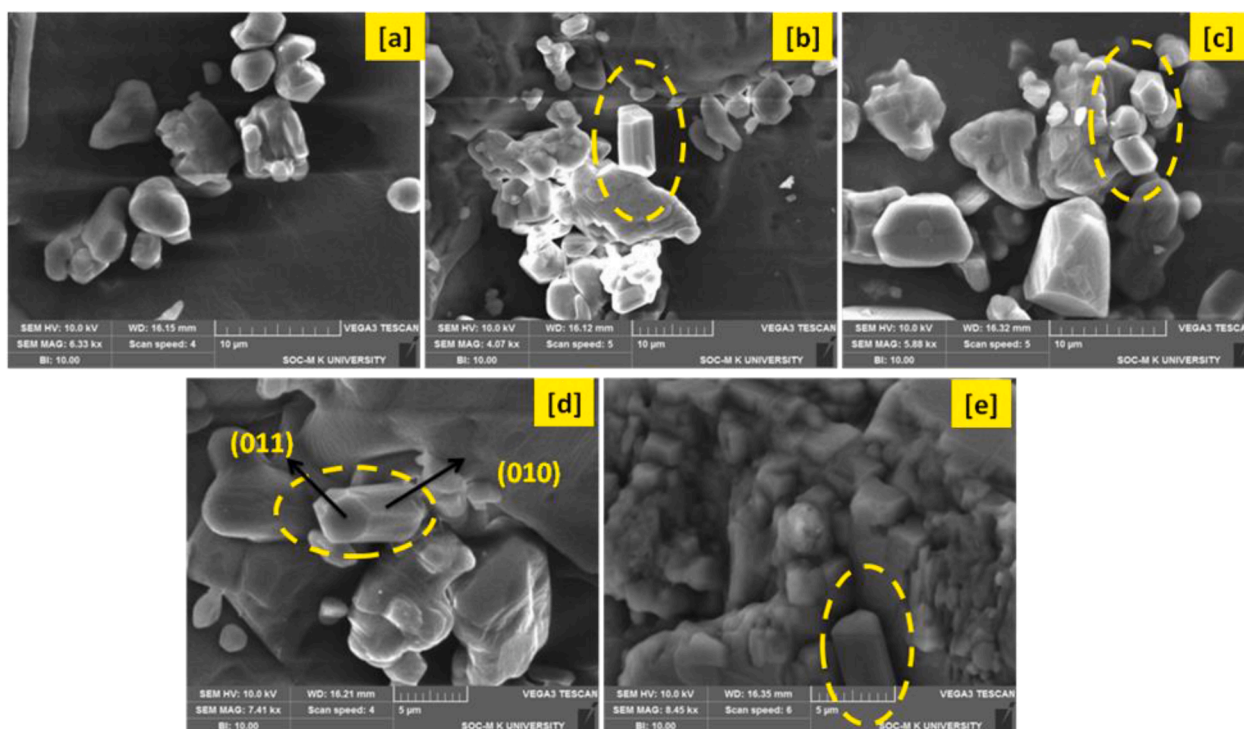


Fig. 7. SEM images of the pristine and shocked α -glycine samples (a) the pristine (b) 50 shocked (c) 100 shocked (d) 150 shocked (e) 200 shocked.

modifications in the internal structural dynamic pattern so that a few shoulder peaks appear/disappear while at the exposure of shock waves which is because of the changes occurring in hydrogen bond patterns. SEM micrographs of the pristine and shocked samples exhibit well defined surface morphology under shocked conditions due the dynamic

preferred orientation and re-crystallization. As α -glycine possesses a high stability of phase for the exposed shock waves it is suggested for the industrial applications while several publications on high pressure research also support the present experimental results. Furthermore, experiments on shock wave recovery are very much essential for a better

understanding of the feasibility of amino acids for applications which fairly rely on the materials' stability.

Compliance with ethical standards

None.

CRedit authorship contribution statement

A. Sivakumar: Data curation, Writing – original draft. **S. Sahaya Jude Dhas:** Visualization, Writing – original draft. **Lidong Dai:** Visualization, Writing – original draft. **P. Sivaprakash:** Formal analysis. **Raju Suresh Kumar:** Formal analysis. **Abdulrahman I. Almansour:** Formal analysis. **S. Arumugam:** Resources. **Ikhyun Kim:** Resources. **S. A. Martin Britto Dhas:** Investigation, Writing – review & editing.

Declaration of Competing Interest

The authors declare that they have no conflict of interest.

Data availability

Data will be made available on request.

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at [doi:10.1016/j.molstruc.2023.136139](https://doi.org/10.1016/j.molstruc.2023.136139).

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