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CRediT Authorship contribution statement

A.Sivakumar: Data analysis, Writing-original draft; **S.Sahaya Jude Dhas**: Visualization, Roles/Writing - original draft; **, Raju Suresh Kumar**: Formal Analysis, **Abdulrahman I. Almansour**: Formal Analysis; **Natarajan Arumugam**: Resources, **Shubhadip Chakraborty**: Formal analysis; **S.A.Martin Britto Dha**s: Investigation, Writing-review & editing.

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Assessment of sustainability on structure-optical properties of prismatic face ADP crystal at dynamic shocked conditions

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Abstract

In the present research article, we have performed an extensive experimental investigation on the structural and linear optical properties of the prismatic face ammonium dihydrogen phosphate (ADP) crystals under the impact of shock waves of various counts such that the crystals have been analyzed by X-ray diffraction, Raman spectroscopic, Ultra-violet Visible spectroscopic and optical microscopic techniques. XRD and Raman results clearly show that the shocked ADP crystals have undergone significant changes such as lattice deformations, lattice disorders giving rise to the structural disorders and significant reduction in optical

transmittance with respect to the number of shock pulses. Optical microscopic images also authenticate the formation of defective surface morphology at shocked conditions. Furthermore, we have made the comparison of the optical transmission of the prismatic face with the previously published pyramidal face ADP crystal with respect to the number of shock pulses.

Key-words: Shock waves, ADP crystal, Prismatic face, Optical transmission, Defects, Structural properties

1. Introduction

In the process of trying to get to the bottom of what is the basis of the observed changes caused by the impact of shock waves on materials, it has been identified that there are a lot of fundamental modifications which enforce drastic characteristic transformations. Much of these experimental works have come about because, in recent times, there have been witnessed a few groundbreaking results that emerge out from research groups owing to their incredible commitment. Space environmental factors can possibly affect the characteristic nature of space materials when they encounter the space condition such as gamma rays, low temperature, shock waves and cosmic rays, etc [**1-4**]. Optical devices are used in space mission for electronic types of equipment and laser operating systems to determine the efficiency of lifetime of satellites and data communication [**5,6**]. If the above-mentioned environmental factors could really affect the materials, the optical instruments are prone to undergo critical condition such that the resultant performances of the device tend to decrease resulting in deterioration of their characteristic features. Non- linear optical (NLO) crystals are used as frequency converters and for many other electronic devices such as LIDAR, SONAR, in space, military and defense technological applications [**7,8**]. High optical transmission is one of the basic requirements for the applications

of converters because it plays a crucial role in laser frequency converter devices. Hence, the stability of optical transmission has a paramount role in space, military and defense applications. To overcome the above-mentioned drawbacks and find novel suitable space materials for space applications, shock wave recovery experiment is proven to be one of the key techniques [**9,10**]. Because during the shock wave propagation on test material, it can experience a similar situation as that of the space environment exposed on the surface of the test material that can be accomplished in the indoor laboratories itself. The aftermath of shock wave impact can generate a lot of usual and unusual effects on materials. Therefore, experimental investigations on crystalline and non-crystalline materials after the shock wave exposure have been witnessed to be consistently going up such that it is positioned to be a prominent research topic in current years [**11-15**]. At shock wave loaded conditions, micro and macro-level structural defects can be developed on material surfaces. In addition to that, grain boundaries diffusion, re-crystallization and phase transformation can also occur which are highly associated with the nature of material's response to high-pressure and temperature [**16,17**]. It is well authenticated that if crystallographic structural properties are altered, all kinds of dynamic physical properties such as thermal, electrical and optical properties are found to be greatly affected. Hence, research on finding the materials of crystallographic phase stability under harsh environmental conditions has attained the position of imploding a paradigm shift to be termed as one of the top-class research themes in recent years.

Over the years, a lot of NLO materials have been found for high-power laser applications whereas potassium dihydrogen phosphate (KDP) and ammonium dihydrogen phosphate (ADP) crystals are being regarded as the legendary research materials because of their exceptional

characteristic nature of growth and physical properties [**18-20**]. Among the two crystals, KDP's crystal structure [**21**] and other physical properties such as surface properties [**21**], optical transmission [**21**], dielectric constant [**22**], thermal diffusivity [**23**] and optical band gap energy [**24**] have been extensively studied by our research group at dynamic shock wave loaded conditions. In the case of ADP crystal, the crystal structure of the pyramidal face and other physical properties have been studied at shocked conditions [**25-27**]. Sivakumar et al have demonstrated the significant improvement in the degree of crystalline nature of the pyramidal face of ADP crystal at shocked conditions and ADP's surface properties, dielectric constant [**25**], optical transmission [**26**], thermal diffusivity [**23**] are found to have enhanced. Joshi *et al* have conducted the shock wave recovery experiment on Glutamic acid doped ADP crystal and found the reduction of the degree of crystalline nature and dielectric constant at shocked conditions [**27**]. Furthermore, our research group has examined a few other technologically potential single crystals' optical properties such as benzyl [**28**], tri-glycine sulfate [**29**], potassium sulfate [**30**], copper sulfate pentahydrate [**31**] crown ether magnesiuchloride potassium thiocyanate [**32**] and found several interesting results. Based on the available literature reports, the prismatic face ADP single crystal's physical properties have not yet been reported at shocked conditions. Therefore, the prismatic ADP crystal is considered for the current investigation on the stability of structural and optical transport properties under the condition of various counts of shock waves exposed in such a way that the observed results could provide the possible key evidence on the applicability of the crystal for space applications.

On those lines, we have analyzed the stability of crystal structure and degree of crystalline nature of the prismatic ADP crystal at shocked conditions by X-ray diffraction and

Raman spectroscopic techniques. Furthermore, the optical transmittance profile has been analyzed by UV-Vis spectrometer and the stability profiles have been established with respect to the number of shock pulses.

2. Crystal growth and Shock wave loading methodology

The crystal growth details of ADP crystal have been discussed in our previous publications [**25,26**]. For the present experiment, cut and polished prismatic ADP crystals of 1mm thickness have been used. A pressure-driven tabletop Reddy tube, which was indigenously designed in our laboratory, has been made use of to generate the shock waves and the details of the shock tube has been published elsewhere [**33**]. In the present experiment, shock wave of Mach number 1.7 equivalent to the transient pressure of 1.0 MPa has been utilized for the assessment of the stability of crystal structure and optical properties of the prismatic ADP crystal. Five identical crystals have been loaded with shock pulses of 0,1,2,3 and 4, respectively. After shock wave loading, the respective crystals have been subjected to the studies of stability of crystal structure and optical transmission.

3. Results and Discussion

3.1 X-ray diffraction analysis

In the present experiment, Powder X-ray diffractometry (Rigaku – SmartLab X-ray Diffractometer, Japan) has been utilized to analyze the structural properties of the prismatic ADP crystal at shocked conditions. CuKα as the X-ray source (λ = 1.5407 Å) of radiation and 1D detector have been utilized. Before loading the shock waves on the test sample, the control test sample's XRD pattern has been recorded and the obtained XRD pattern is presented in Fig.1a.

As seen in Fig.1a, the test sample has only one strong diffraction peak at 24.461° and the corresponding diffraction position belongs to (200) crystallographic plane in the tetragonal crystal structure and the diffraction angle position is well-matched with JCPDS – 85-0815. It is noteworthy to mention that the crystal has only one diffraction plane with the specific orientation (200) and no other considerable diffraction planes have been observed.

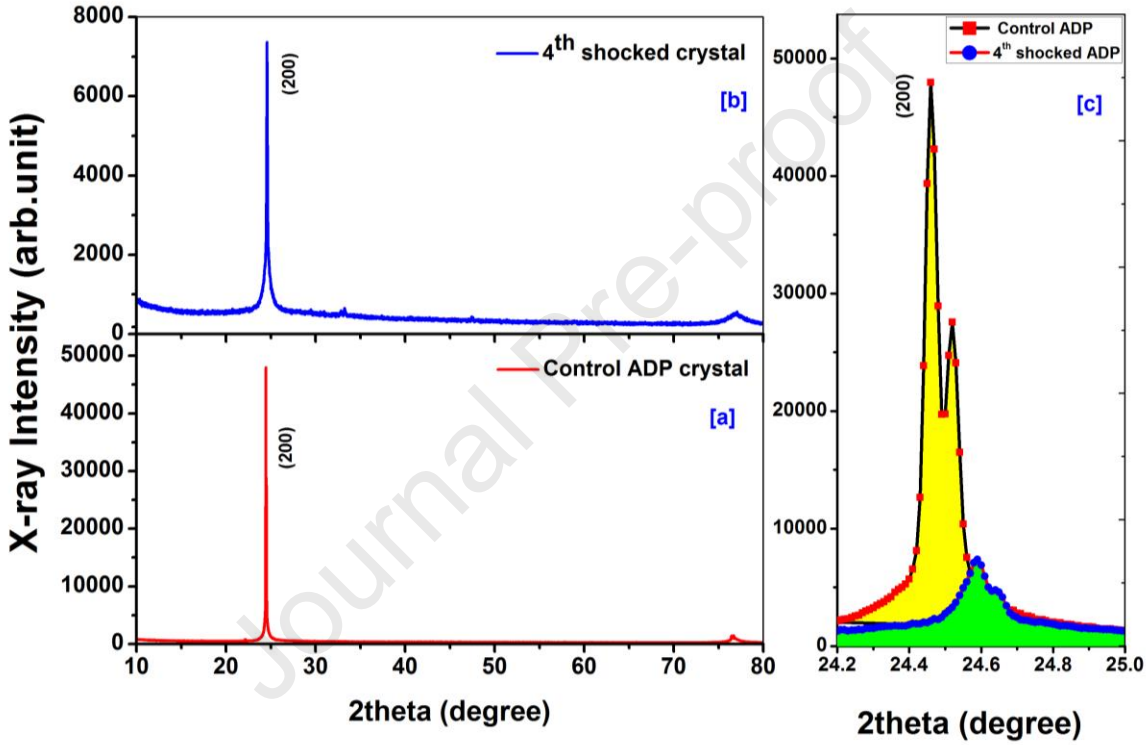


Fig.1 XRD pattern of the control and shocked prismatic face of ADP crystals

Furthermore, the observed control sample's diffraction peak (200) has very low full-width half maximum and high peak intensity which clearly demonstrate that the test sample has good crystalline nature. At shocked conditions (especially at the 4th shock), considerable changes are noticed in the intensity of diffraction peak and full-width half maximum that have been significantly altered due to the influence of shocks which have to be considered to draw the

impact of shock waves on the structural properties of the prismatic ADP crystal. As the test sample is a single crystal, the intensity of the recorded XRD pattern must be taken into the account in order to get a clear picture on the impacted changes imposed on the crystal in terms of its structure and degree of crystallinity because of shock waves. As seen in Fig.1b, the shocked sample shows the retention of (200) crystalline plane and there is no new diffraction peak observed. Hence, at the first screening of the observed XRD pattern, it is obvious that the tetragonal crystal structure has not undergone any crystallographic phase transitions at shocked conditions. KDP family of crystals undergo crystallographic phase transitions at high-pressure and high-temperature conditions [**34,35**]. Takanori Fukami et al have demonstrated the crystallographic phase transition from the tetragonal *I*-42d to the monoclinic $P2_1c$ at about 453 K [**34**]. Yan Ren et al have found the monoclinic KDP crystal at1.9 GPa [**35**]. In addition to that, it is worthwhile to note that potassium sulfate crystal undergoes the changes from the orthorhombic to the hexagonal crystal phase transitions at the first shocked conditions and retain the orthorhombic structure at the 2^{nd} shocked conditions [**30**]. But, in the present case, the substantial reduction of diffraction peak intensity is noticed which may be due to the formation of the lattice defects, changes in bonding schemes, misalignment of atomic lattice planes and deformations impacted by the shocks which have been also obtained quite similarly for the prismatic face of KDP crystal in such conditions [**21**]. Therefore, substantiation could be arrived at for the stability of the tetragonal crystal structure at post-shock conditions, but the degree of crystallinity is unstable at shocked conditions. Apart from the function of the crystal structure, the degree of crystalline nature also has the significant contribution in application point of view since most of the crystalline properties such as transmittance; thermal conductivity and dielectric constant are significantly altered with respect to the degree of crystalline nature [**23**]. Followed

by the X-ray diffraction intensity changes, the diffraction angle position also shows a considerable change at shocked conditions. Initially, the most intense diffraction peak (200) is located at 24.461° and at the 4th shocked condition; it experiences a quite higher angle diffraction shift that is located at 24.596°. The observed diffraction angle shift of the (200) plane may be due to the compression of hydrogen bonds such as N–H•••O and O–H•••O as well as crystal unit volume by the impact of shock waves [**19, 25**]. In addition to that, the title crystal belongs to the hydrogen bond family of crystals and the hydrogen bonds have high susceptible nature at high-temperature and high-pressure conditions [**36,37**]. As per the crystal structure of the prismatic face ADP crystal, N–H•••O is connected to $(NH_4)^4$ $(PO_4)^3$ and O–H•••O lies between $(PO_4)^3$ groups and the corresponding crystal packing along with (200) plane is presented in Fig.2.

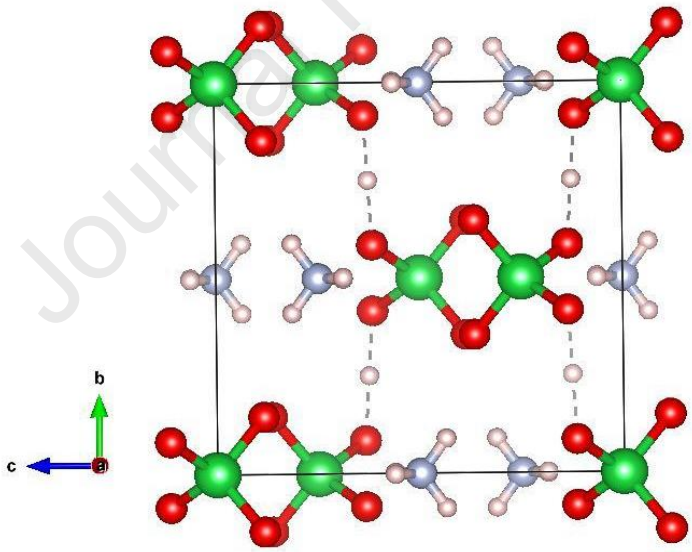


Fig.2 Crystal packing of ADP crystal along the (200) plane

As reflected in Fig.2, the prismatic face of ADP crystal comprises of alternative array of anions and cations contributing to the growth habit of the crystal as against the pyramidal face

that has two hydrogen bonds which are highly susceptible at high-pressure and high-temperature as well as at dynamic shocked conditions. In addition to that, the total bond strength of the prismatic face ADP crystal (0.7282) is significantly low compared to the pyramidal face ADP crystal (1.6341) [**38**]. So that, it may not sustain the original crystalline state with specific atomic positions at shocked conditions and as a result it may produce the displacement of anions and cations including bond length changes from the original positions which may enable the loss of crystal symmetry and enhance the deformation levels in the test sample. Due to such changes, the net degree of crystalline nature of the prismatic plane in the crystal has faced a remarkable influence in the physical properties of the crystal.

3.2 Raman Spectroscopic analysis

Raman spectroscopic study has been performed to get deep in-sights on the structural properties of the test sample and the obtained Raman spectra of the control and shocked samples are presented in Fig.3. As it is known, ADP crystal is built by $(NH_4)^+$ and $(PO_4)^{3-}$ groups and it has two types of hydrogen bonds that are N–H•••O and O–H•••O which hold the major contribution to the structural properties of ADP crystal.

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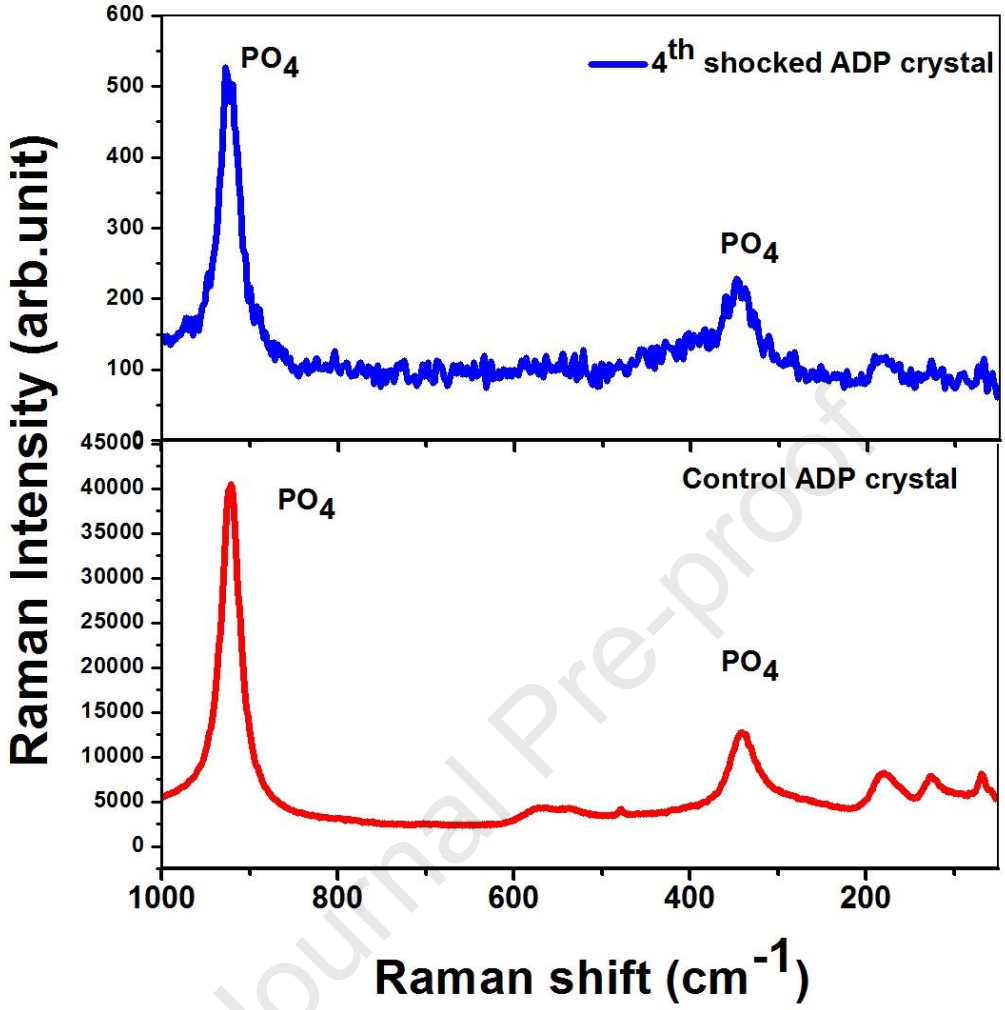


Fig.3 Raman spectra of the control and shocked prismatic face of ADP crystal

As seen in Fig.3, the control sample's Raman spectrum has the intense typical characteristic of PO_4 tetrahedral Raman bands which are located at 923 cm^{-1} (bending mode) and 339 cm^{-1} (stretching mode) which are well-matched with the observations of the previous publications [**39,40**]. The observed characteristic PO_4 Raman bands of the control sample clearly demonstrate that they belong to the tetragonal crystal structure. Furthermore, the appearance of the intense Raman bands shows that the test sample has good crystalline nature i.e. without any

surface defects. Under shock loadings, substantial changes are identified in terms of peak intensity and peak splitting of PO_4 Raman bands and the zoomed-in version of the Raman bands are presented in Fig.4 for a better visibility.

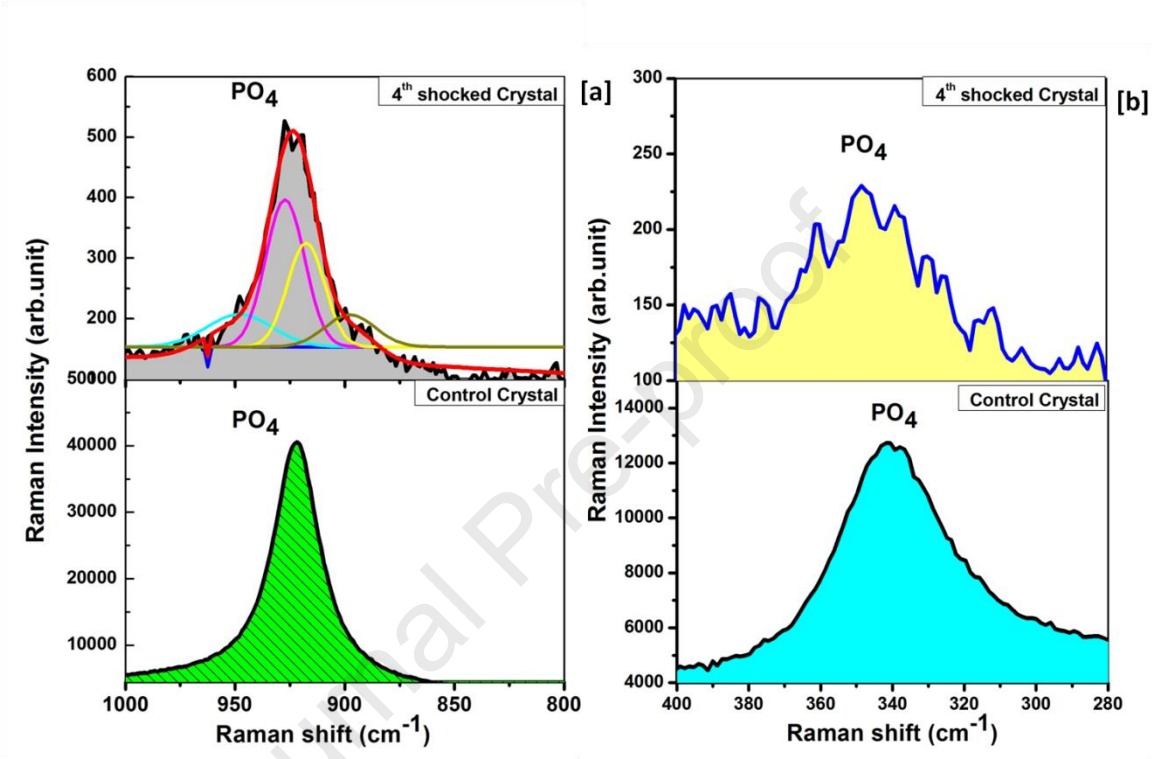
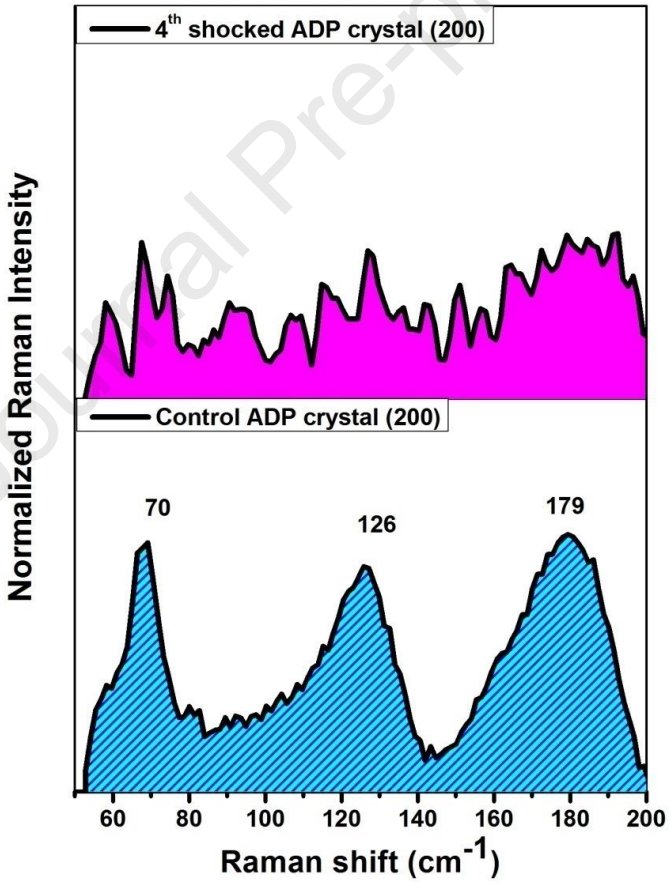


Fig.4 Zoomed-in versions of the PO_4 Raman bands for the control and shocked prismatic face of ADP crystals

As seen in Fig.3a, ADP crystal's characteristic Raman band of asymmetric stretching PO_4 has a single peak without peak splitting that could be noticed. But at shocked conditions, the asymmetric stretching PO_4 Raman band exhibits a remarkable reduction of peak intensity and peak splitting which clearly emphasize the formation of the distorted tetrahedral PO_4 and hence the prismatic face of ADP crystal adopts a new structural configuration with different bond lengths and bond angles along with different atomic sites which may induce the distortion of the

193 tetragonal crystal structure and the resultant is observed as a decrease in the degree of
194 crystallinity for the after-shock specimen which is in good agreement with the results of XRD.
195 In addition to that, the symmetric Raman band of PO_4 at 339cm^{-1} shows the similar results as that
196 of the asymmetric PO_4 at shocked conditions. Hence it is clear that, at shocked conditions,
197 structural distortions have occurred in ADP crystal due to the instability of the tetragonal crystal
198 structure [**41**]. In addition to that, the lattice Raman modes must be analyzed for a better
199 elucidation of the actual crystal structure of ADP crystal and hence, the lattice Raman modes of
200 the control and shocked crystals are presented in Fig.5.



201

202 Fig.5 Lattice Raman modes of the control and the prismatic face of ADP crystal

As seen in Fig.5, the control crystal has well-defined three lattice Raman modes at 70 cm^{-1}, 126 cm^{-1}, and 179cm^{-1}, respectively and the lattice Raman band positions are well corroborated with the results of the previous publications [**39,40**] such that the Raman modes corresponding to the lattice vibrations of the crystal are due to the absorption or emission of optical phonons. At shocked conditions, the initial signatures such as lattice Raman mode pattern and intensities are destroyed which are the non-debatable proofs for the formation of the distorted tetragonal crystal structure at shocked conditions. On the other hand, there is neither crystallographic phase transition nor mixed phase formation has occurred at shocked conditions.

3.3 UV-Visible spectral studies

For non-linear optical materials, wide optical transmittance window and high optical transmittance are the major requirements for the industrial applications and hence the crystal growers have taken significant effort to grow crystals of high optical transmittance. But, while suggesting a particular functional material for industrial applications, the sustainability of the optical properties should also be measured at high-pressure and high-temperature environments for a better understanding of the scale of workability of the material. If the optical transmittance window and optical transmittance percentage are changed by the external parameters such as high-pressure and high-temperature or by sudden dynamic impact, the output of the electronic devices will vary significantly. In the case of the materials of second harmonic generation, the efficiency of the harmonic generation is significantly altered as and when the surface defects and structural defects are induced by shock waves so that we have measured the stability of the optical transmittance at shocked conditions and corresponding optical transmittance profiles are presented in Fig.6 wherein the observed pattern of optical transmittance is found to be well-

matched with the experimental data of the previous publications [**26**]. As seen in Fig.6, the control sample has 36 % optical transmittance at 500 nm.

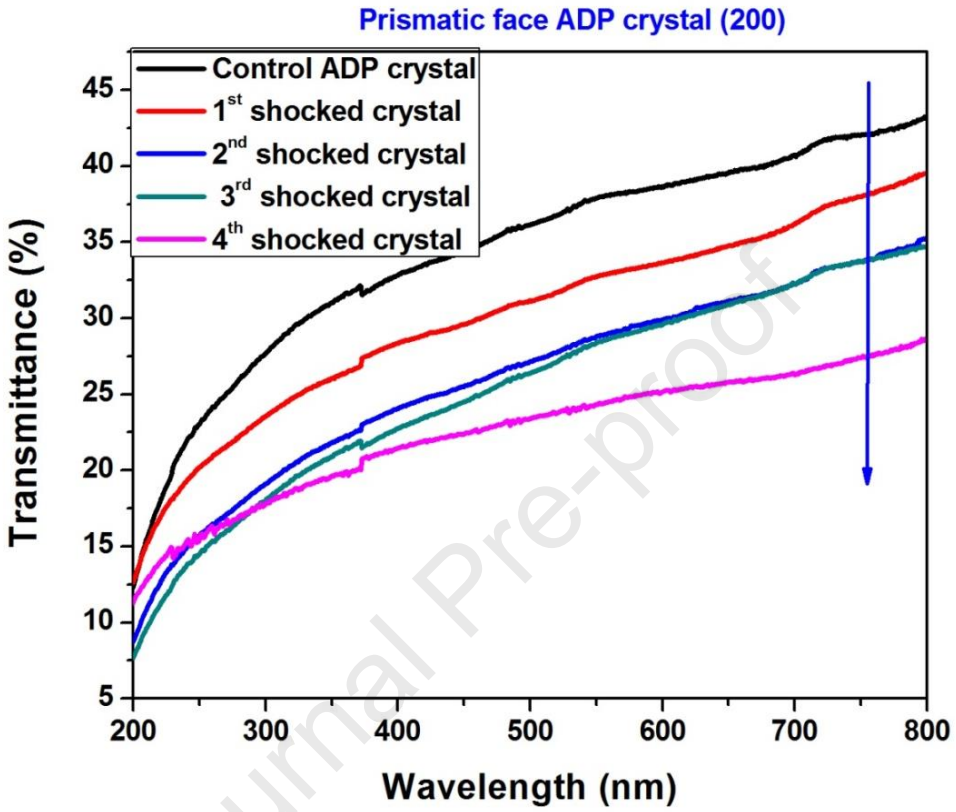


Fig.6. The profiles of optical transmittance of the control and shocked prismatic face of ADP crystals

At shocked conditions, the optical transmittance has linearly reduced with respect to the number of shock pulses and the corresponding plot is portrayed in Fig.7. The downward trending of the optical transmittance of the test crystal may be due to the formation of lattice deformations, distortion of crystal structures and surface defects. Similar results have been observed for potassium dihydrogen phosphate [**21**], tri-glycine sulfate [**29**] at shocked conditions. For a better understanding of the optical properties of ADP crystal in terms of its

sustainability with respect to the faces, we have made a systematic comparison of the percentage of optical transmittance for the prismatic and the pyramidal faces at shocked conditions and the corresponding plots are depicted in Fig.7a and 7b.

As seen in Fig.7a, optical transmittance of the (200) plane for the crystal has reduced with respect to the number of shock pulse whereas optical transmittance of the (101) plane has increased which has been previously reported [**26**]. The prismatic face of ADP and KDP [**21**] crystals' optical transmittance stability profiles are presented in Fig.7b. As seen in Fig.7b, both the crystals follow the downward trending percentage of optical transmittance with respect to the number of shock pulses. But KDP crystal shows very fine changes in the optical transmittance while compared to the prismatic face of ADP crystal. For example, the face of the KDP crystal shows the reduction of optical transmittance from 58% to 55.9% at the 4^{th} shocked condition. Whereas the prismatic face of ADP crystal shows the reduction of optical transmittance from 36% to 23.9% at the 4^{th} shocked condition and hence, we consider that the prismatic face of KDP crystal has little higher shock resistance than that of the prismatic ADP crystal.

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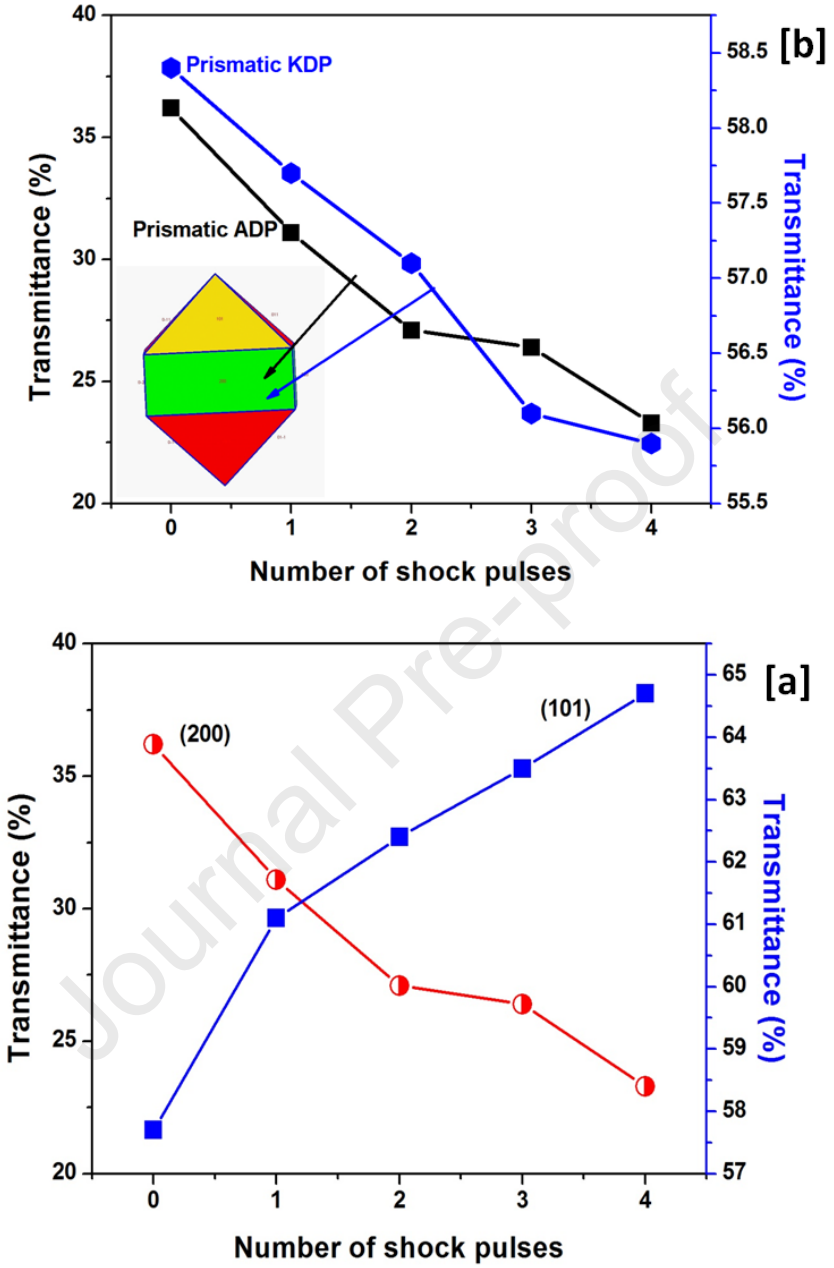


Fig.7. The profiles of optical transmittance of the control and shocked crystals (a) (200) and (101) planes of ADP crystals (b) (200) plane of ADP and (200) plane for KDP crystal

3.4 Optical Microscopic Images

Furthermore, we have obtained the optical microscopic images to get the possible insights about the surface morphological changes of the prismatic ADP crystal at shocked conditions. As seen in Fig.8a, the control crystal's surface has a flat and smooth morphology. But at the 4th shocked condition, the test crystal's surface shows significant changes in the form of surface defects and the corresponding micrograph is presented in Fig.8b. The formation of surface defects may also have the equal contribution for the reduction of optical transmittance due to scattering that is followed by the loss of degree of crystalline nature and such surface defects formation and loss of optical transmittance have been witnessed for potassium dihydrogen phosphate and Tri-glycine sulfate crystals at shocked conditions [**21,29**].

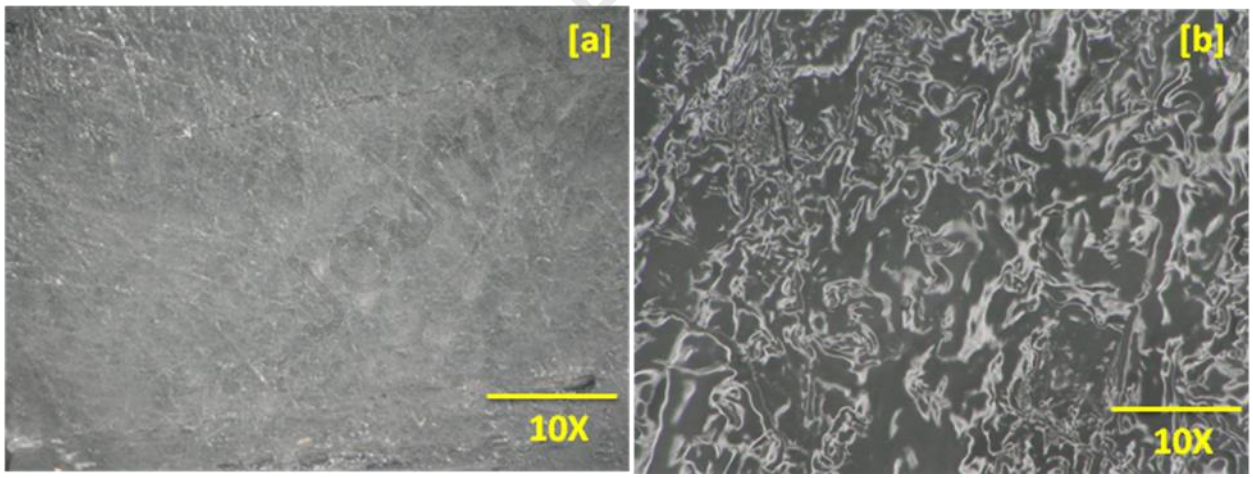


Fig.8 Optical micrographs of the prismatic ADP crystal (a) the control (b) the 4th shocked ADP

4. Conclusion

On summarizing the present research work, an organized assessment has been carried out on the nature of crystallinity and the stability of the optical transmittance of the prismatic

face ADP crystal at dynamic shocked conditions and the corresponding diffraction, spectroscopic results have been investigated.The observed X-ray diffraction and Raman spectroscopic results show the formation of the distorted tetragonal crystal structure at shocked conditions due to the noteworthy reduction of intensity of the diffraction peaks and complete modification of the lattice Raman modes. The analysis retrieved from UV-Visible spectrometer clearly discloses the optical linearly that decreases in terms of percentage in accordance with the number of shocks because of the formation of defects in the crystal structure and hence the prismatic face ADP crystal might not be a potential material for high-temperature and high-pressure environmental applications. In addition to that, optical microscopic images also provide the possible evidence for the formation of surface defects at shocked conditions. It could be noted that the prismatic face KDP crystal has high shock resistance than that of the prismatic face ADP crystal so that the prismatic face KDP crystal can be used for high-temperature applications.

Conflicts of interest

The authors declare that they have no conflict of interest.

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Compliance with ethical standards

None

Conflict of interest

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

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