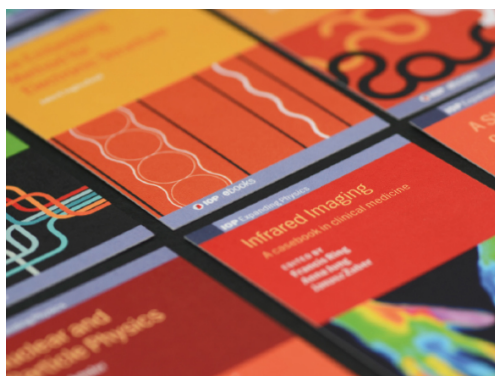


PAPER

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PAPER

Analysis of mechanical behaviour of L-arginine hydrobromide monohydrate (LAHBr) single crystal grown by unidirectional growth technique

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Keywords: x-ray diffraction, crystal growth, laser, mechanical stability, nanoindentation

Abstract

A single crystal of L-arginine Hydrobromide Monohydrate (LAHBr) with excellent crystalline perfection was grown by a modified way of unidirectional technique. The criteria behind this modified version were to reduce the temperature gradient of the solution which will enhance the quality of the single crystal. The formation of the crystal was confirmed by calculating lattice parameters from powder x-ray diffraction technique. The quality of the single crystal was analyzed by high-resolution x-ray diffraction which suggests that single crystal was free from grain boundaries and have the value full width half maxima is 9.36 arc second. Owing to high crystallinity, the single crystal was further underwent mechanical analysis using nanoindentation technique. Its structural, as well as physical transformation have been assessed by laser and shock damage threshold techniques. The presence of surface defects was examined by scanning electron microscope (SEM) and it is in tune with the high resolution x-ray diffraction method.

Introduction

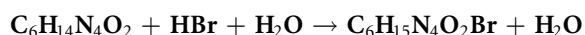
It is the demand of the time when our technology has switched to photons from electrons for the transmission of information. In this view, many new materials with unique optical properties have been analyzed to fulfill the current day to day requirements [1]. In search of innovative materials, a new class has been found i.e. semi-organic materials in which a polarizable organic compound made abound with the inorganic host material. Such type of materials is mechanically more stable than organic materials and also having high laser damage threshold value [2]. The semi-organic materials can be easily grown in three dimensions by solution growth technique which further can be cut and polished for application usage. But the main drawback associated with this technique was its inefficiency in growing bulk size crystal. Also, there has been a high probability of spontaneous nucleation during the growth period which ruins the quality of single crystal [3]. Using conventional growth method, it was found that the single crystals of different orientation are grown along with different morphology. Before making any fabrication of devices, it is necessary to obtain a good quality and bulk size single crystal along a particular direction [4, 5]. In this direction, a technique has been discovered to grow unidirectional crystal from a solution called Sankaranarayanan- Ramasamy (SR) method. This method has 100% efficiency to convert entire amount of solute into crystal and thus, avoid the wastage of solution. The direction of grown crystal was decided by suitably mounting the seed in the neck of ampoule.

L-arginine is an amino acid which has the tendency to crystallize in non-centrosymmetric structure and have anisotropic nature. It means that the crystal of these compounds shows different behavior in different directions. So, it is worthwhile if the crystal will grow through this technique. Because through this technique it is possible to grow crystal in a direction that shows high efficiency. There are many compounds of L-arginine that have been reported who show non-linear optical property like L-arginine phosphate (LAP), L-arginine trifluoroacetate (LATF), L-argininium bis(trifluoroacetate) (LABTF), L-argininium phosphite (LAPI), L-arginine 4-nitrophenolate 4-nitrophenol dihydrate (LAPP), and many more [6–10]. Such properties were motivated the researchers to grow and characterize the new compound of this family of crystals. In addition to that, L-arginine family materials are having good second harmonic efficiency than that of standard potassium dihydrogen phosphate (KDP) [11] single crystals.

In the present work, LAHBr single crystal having dimension 4.5 cm length and 1.5 cm diameter has been grown by using SR method in (001) direction. The present report mainly deal with the mechanical stability of LAHBr compound. The grown ingot was subjected to high resolution x-ray diffraction which reveals the presence of grain boundaries present in the the crystal. Further, Photoluminescence technique is also employed on LAHBr single crystal to identify the crystal defects. Whereas, nanoindentation provides the mechanical strength of the grown crystal, against different loads. Laser damage threshold of the title compound was examined by using Nd:YAG laser as the source. The detailed discussions were given in the forthcoming sections.

Experimental method

Apart from the conventional method, a new design of SR method has been used to grow crystal in order to improve the crystalline quality of the sample. Initially, L-arginine Hydrobromide Monohydrate (LAHBr) was synthesized by taking stoichiometric ratio of commercially available L-Arginine (molecular weight 174.20 and melting point 200 °C) and hydrobromic acid (molecular weight 80.91) and then dissolved in a double-distilled water. The chemical scheme for the present synthesis is given below:



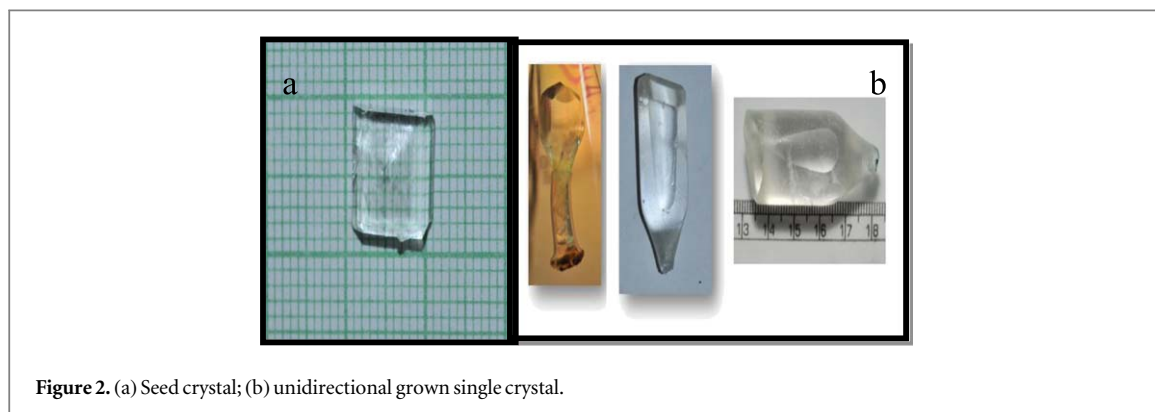
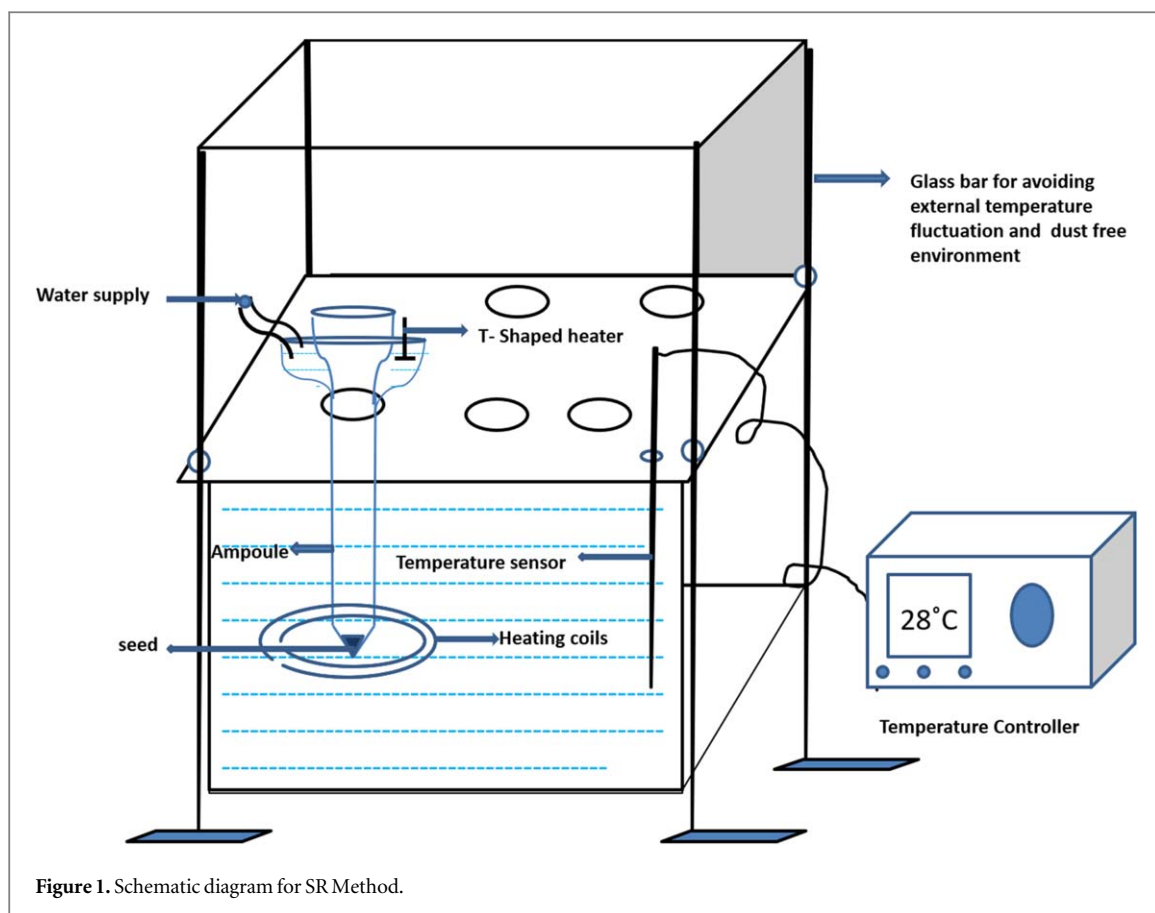
Further, a seed crystal has been developed by a slow evaporation method and then a V-type shape has been given to the seed so that it can fit into the neck of the ampoule. After that, ampoule was kept in a glass container filled with water and set at a temperature of 23 deg. C. The heating coils were set inside the tub to provide the proper temperature. Further, the temperature was controlled and continuously monitored by temperature controller along with the help of a temperature sensor. In the top part of the ampoule, a cup-like design has been made. The reason for such design was to avoid multi-nucleation as there was some temperature difference between the top and bottom of the ampoule. In order to reduce that temperature difference, the cup was filled with water and T shaped heater has been induced in it for maintaining the temperature. By reducing the temperature difference sudden formation of crystallites have been avoided. Figure 1 shows the schematic diagram of the setup.

After a span of one month, a unidirectional bulk-size single crystal was harvested which is shown in figure 2. Further, the crystal was cut and polished into a number of smaller crystals for characterization purposes.

Characterization techniques

Powder x-ray diffraction (PXRD)

The primary investigation for the formation of the compound can be done by obtaining powder x-ray diffraction pattern. This process includes phase identification of the crystalline material which reveals the unit cell parameter of the material. The diffraction pattern can be attained by interference of the radiation, passes through the compound carries structural information. X-rays are the most efficient tool for compound identification as its wavelength is comparable to the lattice parameters of the unit cell. An excellent quality crystal was crushed in the form of fine powder which then subjected to Rigaku x-ray diffractometer using CuK_α as a source to produce incident radiation ($\lambda \sim 1.54 \text{ \AA}$) with a scan speed of 4° min^{-1} . The crystallites are arbitrarily oriented with reference to the x-ray beam. Therefore, maximum reflections can be obtained from all set of lattice planes which satisfies Bragg's condition i.e. $n\lambda = 2d\sin\theta$, where λ is incident wavelength, d was spacing between planes and θ was the diffraction angle. The powdered sample has been scanned over a range of 2θ i.e. 10° – 40° , to attain diffraction planes. The observed pattern is shown in figure 3. The calculated lattice parameters is $a = 11.2042 \text{ \AA}$, $b = 8.5758 \text{ \AA}$, $c = 11.2210 \text{ \AA}$, $\alpha = \gamma = 90^\circ$, $\beta = 91.120^\circ$, $V = 1077.95 \text{ \AA}^3$ which is good in agreement with the reported literature [12, 13].



High-resolution x-ray diffraction (HRXRD)

High-resolution x-ray diffraction is an ultimate tool to assess the crystalline perfection of the crystal. A PANalytical X'Pert PRO MRD high-resolution XRD system has been used for the scanning of the crystal with $\text{CuK}\alpha_1$ radiation. The rocking curves of the crystals for the diffraction planes were recorded in symmetrical Bragg geometry using the natural facets by performing the ω scan with double-axis geometry. The monochromated x-ray beam ($\text{CuK}\alpha_1$) falling on the sample was achieved using a four-bounce monochromator Ge (220). Using a scintillation detector, the diffracted beam was detected to estimate the crystallinity of a single crystal along a particular direction which includes structural grain boundaries, dislocations, and other defects. Figure 4 represents the rocking curve of L-arginine HBr single crystal along the diffracted plane of (001).

As the figure shows that the diffracted curve (DC) contains only a single peak which suggests that the crystal is free from grain boundaries. The FWHM of the curve was found to be nearly 9.31 arc second. This value is a little higher than the predicted value for ideal crystals [14, 15]. This suggests that the defect concentration is quite low in the crystal which makes it a suitable candidate for their implementation in photonic applications since no sufficient degradation of properties is caused by these defects [16].

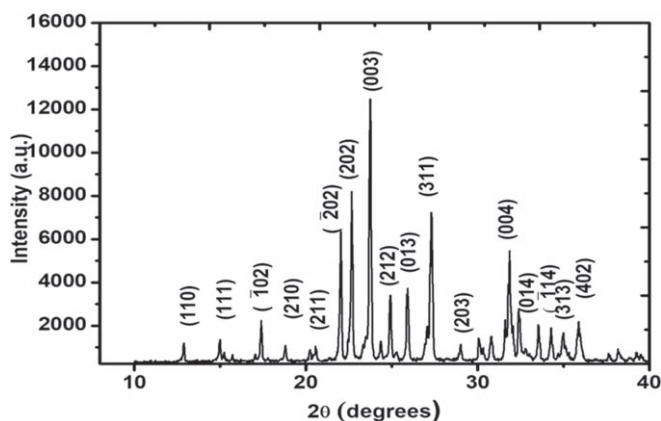


Figure 3. Powder x-ray diffraction pattern.

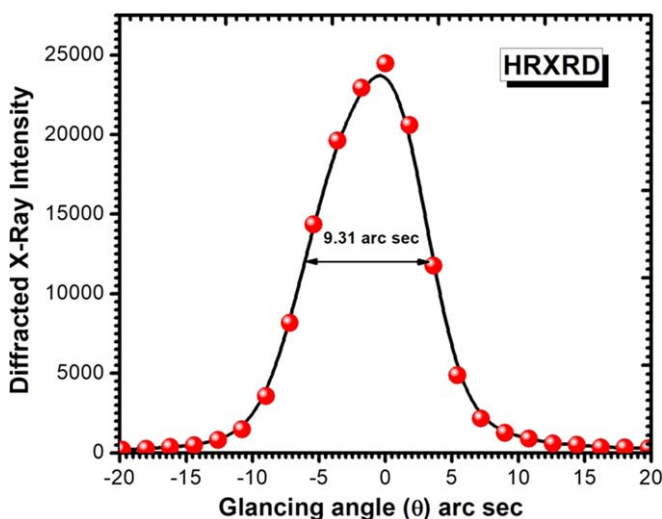


Figure 4. Rocking curve recorded for a plane (001) of LAHBr single crystal.

Nanoindentation

Applications of a non-linear optical material require interactions of materials with lasers. Laser being high energy radiations, generally tends to damage the crystal surface. The extent of these damages depends on the mechanical strength of the material. Therefore, mechanical parameters play a vital role in determining the suitability of material in gadget manufacturing. Nanoindentation is an efficient tool to extract strength parameters and detailed analysis of the material's mechanical strength at nanoscale. So, this technique was utilized to measure its hardness, stiffness, young's modulus from the load-displacement curve. In this technique, the sample was subjected to different loads using sharp Berkovich tip (three-sided pyramidal) of edge radius of 20 nm, the indenter faces angle was fixed at 65.3° from the vertical axes and connected to fully calibrated nanoindenter. The High-resolution Atomic Force Microscope (AFM) in contact mode is used to visualize the surface defects or cracks when the load is applied. Using, Oliver and Pharr's method the results were analyzed [17]. Using the recorded data, first we have plotted the load versus displacement (loading and unloading) curve using the relation

$$F = \alpha(h - h_f)^m \quad (1)$$

Where F and h_f referred to the applied force on the exposed material and final displacement where the unloading curve completes respectively. α and m are the constants. The slope of the unloading curves give Stiffness can be calculated as

$$s = \frac{dF}{dh} = \alpha m (h - h_c)^{m-1} \quad (2)$$

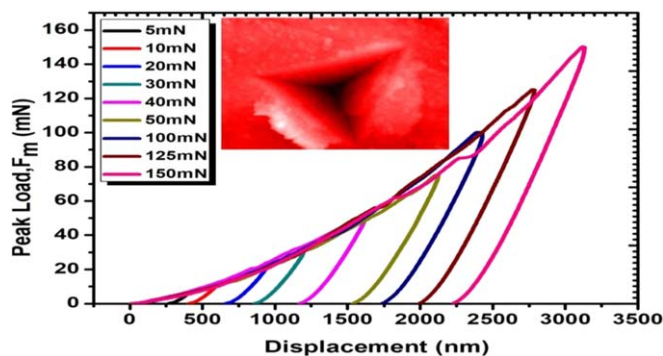


Figure 5. Load displacement curve obtained from the (001) plane of the LAHBr single crystal with inset of imprint made by 50 mN load.

Using the above value, one can deduce contact depth which can further be used to evaluate the independent hardness of the titled material using equations given below

$$h_c = h_{\max} - \varepsilon \times \frac{F_{\max}}{S} \quad (3)$$

$$H = \frac{F_{\max}}{A_p} \quad (4)$$

Where H , $F_{\max}$ and A_p stand for the load-dependent hardness, maximum load offered by indenter and projected contact area of indenter respectively. The load-independent hardness (H_0) is determined using empirical relation.

$$H_0 = Ka_2 \quad (5)$$

Where K is a constant and its value vary with indenter geometry. Further, Young's modulus can be calculated using the equation

$$E = \frac{1/\sqrt{\pi}}{2A_p} \frac{dF}{dh} \quad (6)$$

In the current study, a good quality single crystal of LAHBr was first lapped and polished to get a smooth surface. After that, the sample was subjected to nanoindentation over a load range of 5 mN to 150 mN. In this measurement, force and displacement are recorded whenever the test material is compressed by the Berkovich tip with a prescribed loading and unloading profile. Figure 5 shows a P-h curve obtained from (001) plane and the inset shows a pileup from the crystal impression made by 50 mN force.

On a detailed analysis of the curve at each load, traces of slight pop-in feature have been observed during the loading sequence. These features are corresponding to nucleation of plastic deformation during the indentation of a single crystal. In addition, no pop-out has been observed in the unloading region which is generally attributed to pressure-induced phase transformation [18]. After applying various loads, the optical image of the test material was captured and compared with a pristine surface as shown in figure 6.

The different mechanical parameters calculated from the load-displacement curve using the above equations are tabulated in table 1. A Contact depth versus peak load curve has been plotted to calculate the load-independent hardness of the single crystal as shown in figure 7.

From the curve, it can be clearly seen that there is an increment in the value of contact depth as peak load increases. This is due to the fact that indentation size can affect the hardness of the material. The contact depth, peak load and hardness of the material is related as

$$F_{\max} = a_0 + a_1 h_c + a_2 h_c^2 \quad (7)$$

A polynomial data has been fitted to the curve to get the coefficient values a_0 , a_1 , a_2 . The obtained value of a_2 coefficient was $2.71812 \times 10^{-5} \text{ mN nm}^{-2}$ and calculated independent hardness is found to be 1.109 GPa or 1109 MPa. This value is found to be quiet high as compared to other compounds of L-arginine such as L-arginine 4-nitrophenolate 4-nitrophenol dihydrate (LAPP), L-argininium bis(trifluoroacetate) (LABTF) [19, 20]. Thus the present compound favors the high resistance against the deformation. Further, applying the Oliver and Pharr method, Young's modulus has been extracted from the P-h curve is plotted against peak load as shown in figure 8.

The curve indicates that there is a significant decrement in the value of Young's modulus as the peak load increases. The reason for such behavior is due to the induced strain in single crystal while applying the nano

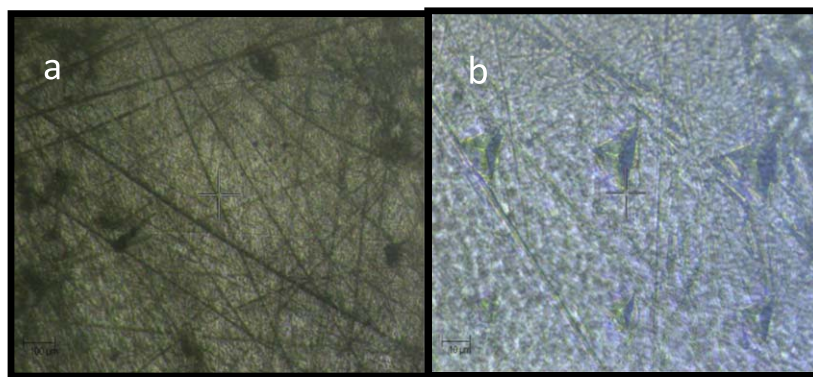


Figure 6. Optical images of the LAHBr single crystal (a) before and; (b) after indentation.

Table 1. Various obtained parameters related to mechanical properties of LAHBr single crystal along the (001) plane.

(001) Plane											
F (mN)	H _{it} (MPa)	H _V (Vickers)	E _{it} (GPa)	h _m (nm)	S [mN nm ⁻¹]	h _c (nm)	h _r (nm)	h _p (nm)	A _p (nm ⁻²)	m	
5.064	1888.483	174.894	31.459	389.575	0.064	330.41	310.595	267.642	2.68146E6	1.513	
10.062	1634.5	151.372	26.94	591.356	0.084	500.62	470.919	411.612	6.15574E6	1.462	
20.061	1279.532	118.499	22.624	933.503	0.112	798.954	755.05	669.117	1.56786E7	1.456	
30.048	1166.607	108.04	20.652	1196.732	0.132	1024.04	968.705	872.051	2.57572E7	1.419	
50.069	1061.831	98.337	18.424	1624.427	0.159	1385.565	1310.298	1183.317	4.71539E7	1.389	
75.063	941.425	87.186	15.774	2124.423	0.178	1801.718	1702.493	1556.598	7.97329E7	1.355	
100.06	986.516	91.362	15.24	2428.792	0.194	2032.111	1912.896	1760.186	1.01428E8	1.324	
125.03	944.294	87.452	14.29	2787.805	0.208	2321.783	2186.632	2017.706	1.32406E8	1.285	
150.071	901.619	83.5	13.459	3132.037	0.22	2603.188	2449.252	2259.535	1.66447E8	1.288	

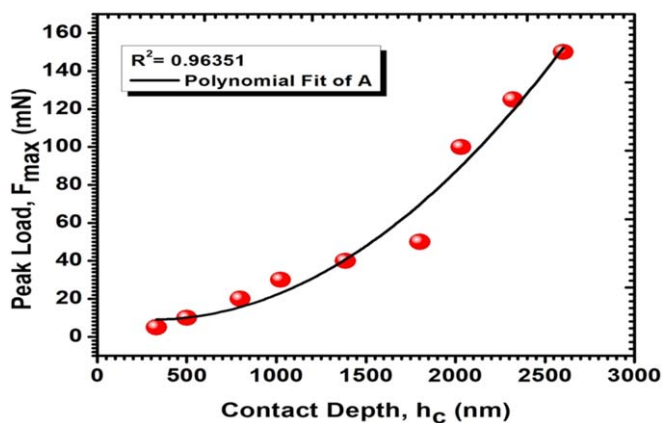


Figure 7. Curve of the peak load versus contact depth for the LAHBr single crystal.

loads. Figure 9 represents the plot of initial unloading stiffness (S) with the contact depth at peak load for the titled compound.

A linear relation can be observed between Stiffness and contact depth h_c in accordance with the relation

$$S = a + bh_c \quad (8)$$

Where a and b correspond to indentation tip rounding and reduced Young's modulus respectively. A linear plot has been fitted to the curve and the slope of the fitted line gives the reduced Young's modulus. The reduced Young's modulus was found to be $6.733 \times 10^{-5} \text{ mN nm}^{-2}$ or 67.33 GPa for LAHBr single crystal.

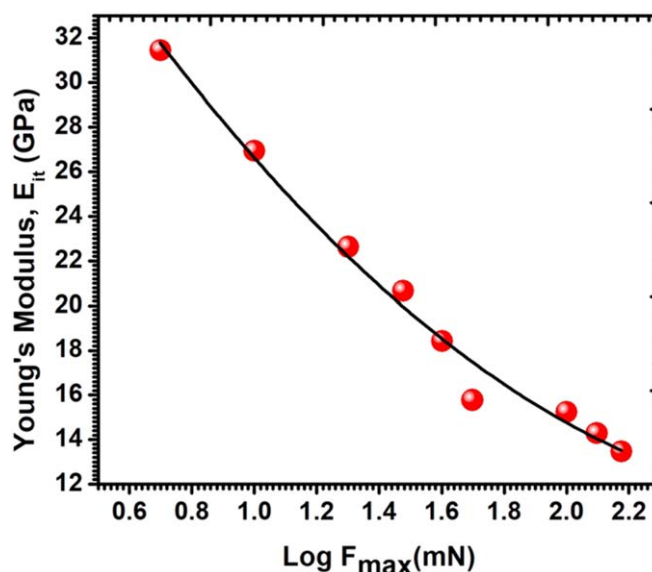


Figure 8. Variation of Young's modulus as a function of the log of the peak load for the LAHBr single crystal.

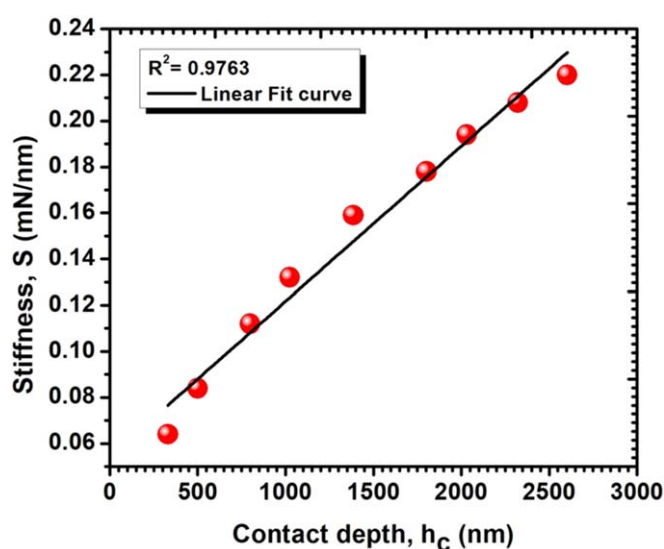


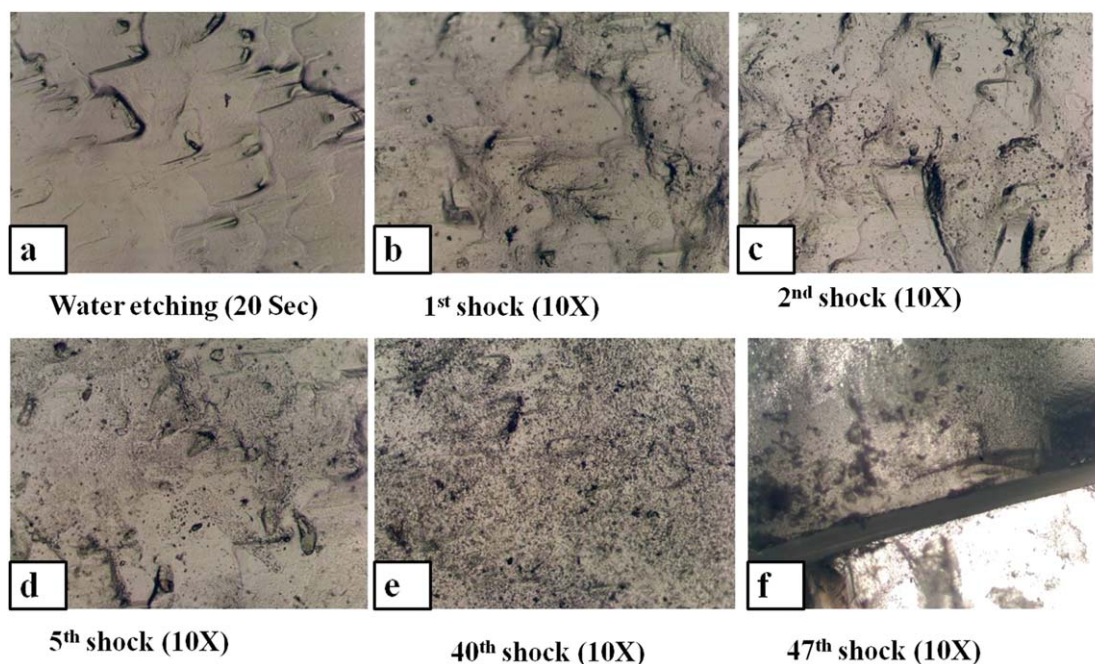
Figure 9. Variation of stiffness with contact depth of LAHBr Single crystal.

Laser damage threshold (LDT)

The laser damage threshold is one most important parameter that has to be considered when the system is integrating with optical components like mirrors. The Laser beam is highly monochromatic, directional and having intense energy which makes it capable to estimate the damage sensitivity of the crystal. The laser damage threshold is a destructive technique that comprises two broad phenomena absorption-driven and dielectric-breakdown damage. The sample was exposed to the Nd:YAG laser of wavelength 1064 nm [21]. The damage occurs due to the laser was totally dependent upon the material properties which include absorption coefficient, specific heat, melting temperature as well as defects present in the crystal. Also, the geometrical properties like thickness, surface morphology and homogeneity can affect the efficiency of the crystal. In the present experiment, the damage threshold value has been examined for a plane (001) against the laser exposers. From the value of FWHM, it has been cleared that the crystal carries high crystalline perfection which in result gives a better value of damage threshold in comparison to its other family compound. As the defect concentration decreases, the energy absorption also decreases which leads to an increase in the value of laser-induced breakdown. Hence, this material proves to be an efficient candidate for optical applications. The damage threshold value for 1 pulse per second and 10 pulses per second is given below in table 2.

Table 2. Laser damage threshold value for single and multiple shots.

Crystal	Damage threshold			
	At 1 pulse per second		At 10 pulse per second	
LAHBr	31.6 J cm ⁻²	3.16 GW cm ⁻²	26.2 J cm ⁻²	2.62 GW cm ⁻²

**Figure 10.** Optical microscopy images of the crystal surface (a) before applied shocks; (b) after 1st shock; (c) after 2nd shock; (d) after 5th shock; (e) after 40th shock; (f) after 47th shock.

These tabular values were further compared with L-arginine based compound for single pulse shot like L-arginine 4-nitrophenolate 4-nitrophenol dihydrate (LAPP) with a value of 2.54 GW cm⁻², L-arginine trifluoroacetate was 2 GW cm⁻² and L- arginine hydrochloride (LAHCL) was 0.011 GW cm⁻² [19, 22, 23]. By comparing the values, it was found that LAHBr has a higher value of LDT which was due to its high crystallinity as mentioned in HRXRD.

Shock damage threshold

A material may undergo physical and structural transformation due to sudden disturbances like an increase in pressure, temperature and mechanical stress. Such disturbances arise due to natural incidences such as earthquakes and volcanic eruption which lead to the generation of shock waves. Experimentally, the effect of these shock waves can be studied with the help of shock tubes developed in a laboratory [24, 25]. For that purpose, a good quality LAHBr crystal of 2 mm thickness was placed at a distance of 1 cm from the open end of the driver section and then it was subjected, initially with low strength. After that, the strength was increased to Mach number 2.4. Before the experiment, the surface of the crystal was well polished and etched with water and the etched surface is shown in figure 10(a).

Then the shock wave was exposed on the surface of the crystal and the surface was analyzed by an optical microscope as shown in figure 10(b). It is clear that the etch pattern is not affected and no crack is observed by the shock waves, but the molecules are peeled off from the surface which resulted as small circular pits. The pits are randomly scattered with irregular sizes. When the next shock waves are exposed, the pits densities are increased as the number of shocks increases in figures 10(c)–(e). Even for the 46th shock, the pit density reached the maximum but no crack was observed. But for the 47th shock, as the crystal was damaged with a crack (figure 10(f)) which is the shock damage threshold for the present crystal for Mach number 2.4. The results show that this crystal has very high shock resistivity and good mechanical stability. Hence this crystal can be used in the fast-moving and highly vibrating instruments.

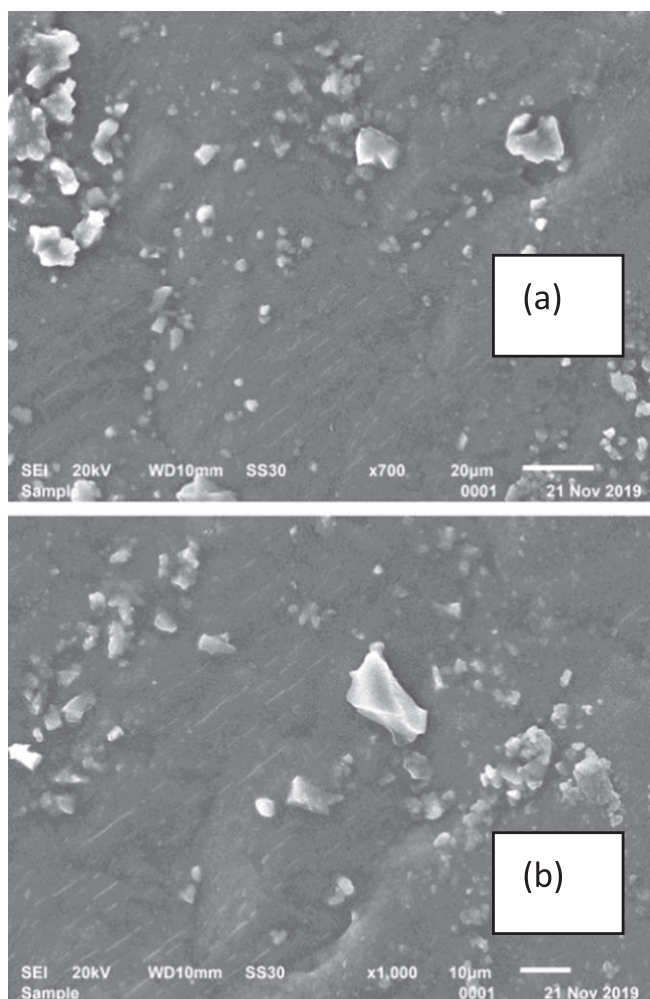


Figure 11. (a) and (b) Surface image of LAHBr single crystal by SEM.

Scanning electron microscope (SEM)

The presence of defects on the surface of the specimen was examined by scanning electron microscope. The LAHBr single crystal was taken and then the surface was neatly polished by silk cloth and then subjected for the present study. The recorded SEM image on (001) direction is given in figures 11 (a) and (b). It is clearly visible that there is no remarkable defects on the surface of the specimen. In case if the surface is defected, the defected region is propagated inside the specimen and it will induce the microcracks. It will indirectly show grain boundaries. But in the present study, we have not noticed such type of defects. This is in tune with the recorded high resolution x-ray diffraction studies.

Photoluminescence

Photoluminescence is an important technique for measuring the purity and crystalline quality of single crystals and for quantification of the amount of disorder present in them. Photoluminescence emission spectrum of the grown LAHBr crystal was recorded using an Edinburgh luminescence spectrometer under ambient conditions. The recorded spectrum is shown in figure 12. Emission spectra of the single crystal LAHBr was recorded at an excitation wavelength of 235 nm. The LAHBr single crystal exhibit high intensity PL emission at 407 nm which indicates the titled crystal is a good candidate for absorption of ultraviolet light and emission of light in violet region. The less intense bands may be due to the lattice points in crystals, which are also being confirmed by the HRXRD analyses.

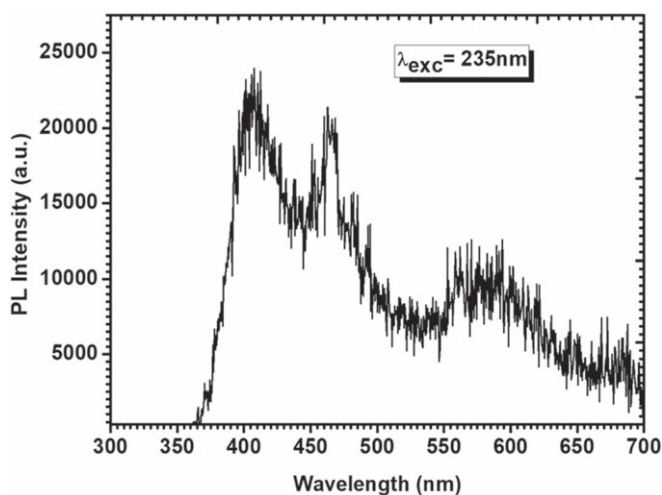


Figure 12. Photoluminescence spectrum of LAHBr.

Conclusion

A good quality single crystal has been grown by a modified way of unidirectional technique along (001) direction. The grown crystal carrying excellent crystalline perfection with the value of FWHM ~ 9.31 arc second. Further, the crystal has been subjected to mechanical analysis. The titled compound was subjected to different loads and found high resistance against the deformation. Its resistivity towards the laser was evaluated using laser damage threshold technique which reveals that the crystal could be sustained for multiple shots. Its physical transformation was observed by applying shock waves to the crystal. The presence of defects on the surface was examined by SEM and it is in tune with the HRXRD technique.

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