RESEARCH ARTICLE

Growth, High Resolution X-Ray Diffraction, Z-Scan, Laser Damage Threshold, Etching of Maleic Acid Single Crystals by Slow Evaporation Method

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ABSTRACT: Single crystal of Maleic Acid (MA) were grown by the slow evaporation technique at room temperature has been employed for the growth of non-linear optical MA crystal. The full width at half maximum (FWHM) of the diffraction curves is 13 arcs, which is close to that expected from the plane wave theory of dynamical X-ray diffraction. The laser damage threshold is directly related to the impurities present in the crystal and the measured higher laser damage threshold indicates the suitability of the crystal for device fabrication. The Z-scan measurement with 632.8 nm laser pulses revealed that non-linear refractive index of the crystal is in the range of 3.29 x10^{-12} m^2/W. The chemical etching studies were carried out to study the shape and distribution of etch pits in the grown crystal.

KEYWORDS: Characterization, z-scan, HRXRD, Laser Damage Threshold.

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1. INTRODUCTION

Nonlinear optical materials can produce upper values of the original frequency and finds application in emerging optoelectronic technologies optical computing, laser remote sensing and so forth [1-5]. Hence there is a great demand for synthesize the new NLO materials and grow their single crystals. KDP is among the most widely used NLO material. Many methods have been tried to improve the NLO properties of KDP crystal. The key factors for material selection depend on physico-chemical properties of the crystals, such as transparency range, damage threshold, conversion efficiency and thermal stability. It has been evidenced that organic crystals can have very large nonlinear susceptibilities compared to inorganic crystals, but their use is obstructed by their low optical transparencies, poor mechanical properties, low laser damage threshold and the inability to grow bulk crystals [4]. Laser induced damage studies on NLO crystals are obviously important, since the surface and bulk damage of the crystal by high power lasers limit its performance in NLO applications.

2. METHODS AND MATERIALS

A single crystal of Maleic Acid (Fig. 1) of size was successfully grown by slow evaporation method with the size of 9x7x3 mm^3 were obtained about 20 days [5-12]. The scope of work is to concentrate the characterization of HRXRD is to identify the crystalline perfection, Z-Scan is to identify the non-linearity of the sample, laser damage Threshold is to calculate the dislocation density and crystals structures etches pits produced on the surface of the crystal [18-21].

3. RESULTS AND DISCUSSION

The polished sample of MA was attached to a microscopic slide. Electrical contacts were made in the sample by silver painted copper wire as an electrode. Then the sample was connected in series to a dc power supply with a keithley 485 picoammeter. To measure the photo current, the sample was illuminated with the halogen lamp (100 w) by focusing a spot of light on the sample with the help of a convex lens. The applied voltage was increased from 0 to 300 volts and the corresponding photo current was recorded.

Crystal perfection of the Maleic Acid single crystal was appraised by HRXRD using a multicrystal X-ray diffractometer [5]. The well-collimated and mono-chromated MoKa1 beam obtained from the three monochromator Si-crystals set in dispersive (+,-,-) configuration has been used as the exploring X-ray beam. Fig shows the high-resolution diffraction curve (DC) recorded for MA specimen using diffracting planes. As seen in the Fig.2 the DC is quite sharp without any satellite peaks, which may otherwise be observed either due to internal structural grain boundaries or due to epitaxial layer, which may sometimes form crystals grown from solution. The full width at half maximum (FWHM) of the diffraction curves is 12 arc s, which is close to that expected from the plane wave theory of dynamical X-ray diffraction. The single sharp diffraction curve with low FWHM indicates that the crystalline perfection is very good. The specimen is a nearly perfect single crystal without having any internal structural grain boundaries [13-16].

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Figure 1 MA single crystal

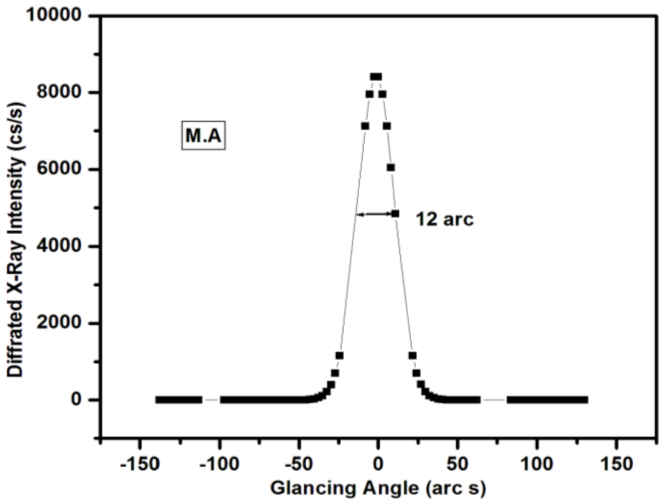

Figure 2 HRXRD spectrum of MA Single crystals

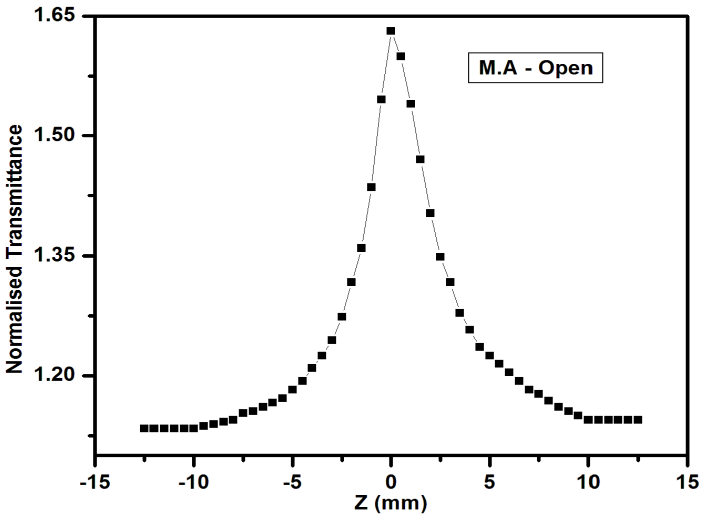

Figure 3 Open Aperture of the z-scan of MA single crystals

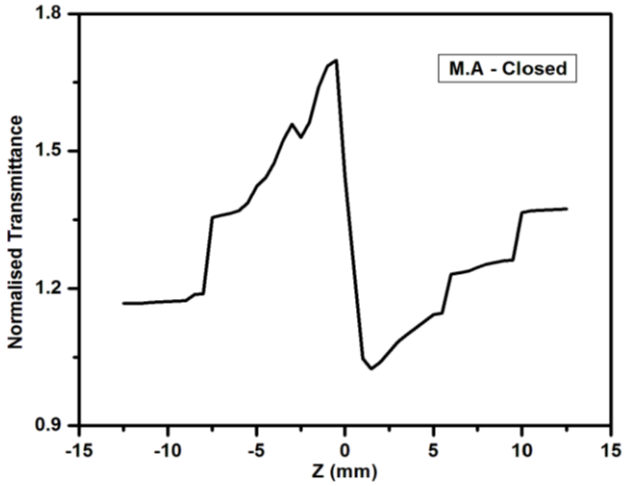

Figure 4 Closed Aperture of Z-scan of the MA single crystals

Third-order nonlinear properties of nonlinear index of refraction and nonlinear absorption coefficient of MA samples were investigated using Z-scan method. Z-scan setup with laser pulses at 632 nm operating at a 1 kHz repetition rate was employed in this study. The sample thickness used in this experiment was 2 mm. The Spot size diameter in front of the aperture (Fig.3 and Fig.4) was 1 cm with focal length (f) was 18.5 cm/ 8.5 cm. The third-order nonlinear coefficients were estimated by measuring the transmittance of a nonlinear medium through a finite aperture in the far field as a function of the sample position Z with respect to the focal plane. The transmittance change observed as a function of the sample position Z has a peak to valley shape that is characteristic of a self focusing [17]. The table 1 mentioned the Z-scan measurement values for MA single crystals.

The laser damage density of MA crystal was measured using a Nd:YAG laser with the wavelength of 1064 nm. A fundamental wavelength, 1064 nm, with a pulse width of 10 ns and a repetition rate of 10 Hz was used. The laser beam of diameter 2 mm was focused on the crystal. The sample was placed at the focus of a plano-convex lens of focal length 30 cm. When high intense laser beam was made to fall on the MA crystal, a slight damage *w*as observed on the surface of the crystal. During the laser interaction, the MA crystal absorbs a part of the thermal energy, this conversion of energy leads to a temperature gradient inside the crystal. The laser damage threshold is high for the crystal, whose temperature gradient is low. The surface damage threshold of the crystal was calculated using the expression (Pd) = $E/t\pi r^2$ where E is the input energy (mJ), is the pulse width (ns) and r is the radius of the spot (mm).The laser damage density of MA crystal was found to be 1.27 GW/cm^2 for (100) plane, 1.17 GW/cm^2 for (010) and 1.2 GW/cm^2 for (001) plane [22].

The chemical etching studies were carried out to study the shape and distribution of etch pits in the grown crystal. The surface of the MA crystal sample was polished and studied under a polarized. Optical microscope fitted with Motic camera. The etching studies were carried out on the grown crystals of MA using High resolution optical microscope. Acetone, dil HCl, hexane, toluene and water were used as etchants. Then the crystal was completely immersed in the double distilled water for an etching period of 5 s and 10 s and then the etched crystal was dried using tissue paper and features of etch patterns were analyzed. Fig.5 shows the crystals structure etches pits produced on the surface of the crystal for the time of etching 10s and 20s [23-24].

Table 1 Z-scan measurement values for MA single crystals.

Parameter	Value
Aperture Radius	2 mm
Optical Path Distance (Z=0)	115 cm
Effective Thickness L_{eff}	2.99 mm
linear absorption coefficient α	0.19
Nonlinear refractive index n_2	4.29 $x10^{-12}$ m^2/W
Nonlinear absorption coefficient β	0.38 $x10^{-4}$ cm/W
Linear Refractive Index n_0	1.53 (no unit)
Real NLO Susceptibility χ (3)	4.48 x 10^{-6} esu
Imaginary NLO Susceptibility χ (3)	2.69 x 10^{-6} esu
Third-order NLO Susceptibility χ (3)	4.3 x 10^{-6} esu

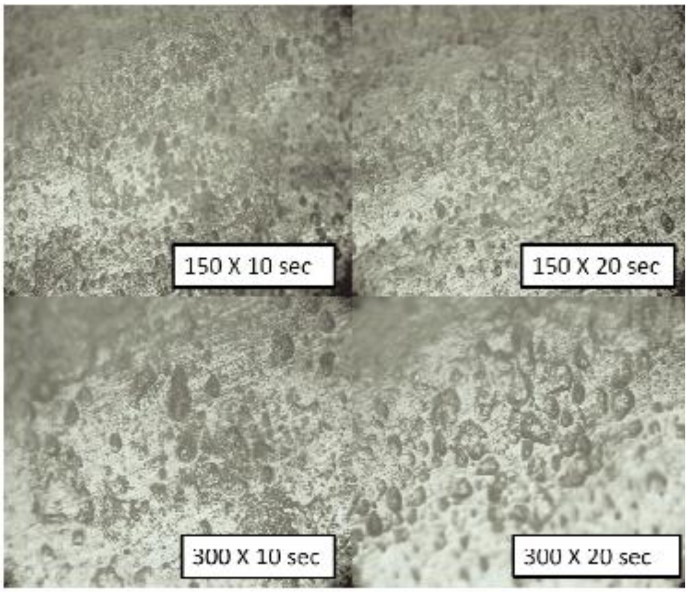

Fig.5 Etching pattern of M.A crystals

4. CONCLUSION

Single crystal of Maleic Acid were grown by the slow evaporation technique at room temperature has been employed for the growth of non-linear optical MA crystal. The full width at half maximum (FWHM) of the diffraction curves is 13 arc s, which is close to that expected from the plane wave theory of dynamical X-ray diffraction. The Z-scan measurement with 632.8 nm laser pulses revealed that non-linear refractive index of the crystal is in the range of 3.29 x10^{-8} cm^2/W. The laser damage threshold is directly related to the impurities present in the crystal and the measured higher laser damage threshold indicates the suitability of the crystal for device fabrication. The chemical etching studies were carried out to study the shape and distribution of etch pits in the grown crystal.

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