

Investigation of nucleation parameters and third order nonlinear optical properties of triglycine sodium molybdate single crystals

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Abstract. In this paper, nucleation parameters such as induction period, interfacial tension, metastable zone width, radius of critical nucleus and Gibbs free energy change in solution-grown triglycine sodium molybdate (TGSM) single crystals are studied. The solubility of TGSM at different temperatures has been determined. The induction period and metastable zone width have been experimentally found for optimizing the growth process. Various critical nucleation parameters have been calculated from the experimentally obtained values based on the classical nucleation theory. The nucleation rate is found to increase with the growth of the supersaturation value. The third order non-linear parameters such as absorption coefficient (β), susceptibility ($\chi^{(3)}$) and refractive index (n_2) obtained from the Z-scan analysis confirm the self focusing and reverse saturation absorption nature of the grown crystals.

Keywords: nonlinear interfacial tension, nucleation, optics, supersaturation.

<https://doi.org/10.15407/spqeo26.04.450>

PACS 42.65.k, 42.70.Nq, 81.10.Aj, 81.10.h

Manuscript received 11.07.23; revised version received 02.10.23; accepted for publication 22.11.23; published online 05.12.23.

1. Introduction

Nowadays, synthesis and growth of NLO materials is essential due to their vast applications in telecommunication, electro-optic device fabrication, transducers, lasers, LEDs, optical storage devices *etc.* [1–3]. A high optical transparency is the key property of the best optical materials. Normally, organic materials exhibit fast and large nonlinear response in a wide frequency range and inherent synthetic flexibility comparable to that of inorganic materials [4]. However, organic materials have a number of downsides including poor mechanical properties, limited thermal stability and volatility. As a result, the study of semi-organic materials was prompted by the demand for novel materials with significant nonlinear optical properties, high thermal stability and superior mechanical strength. In this view, many potential semi organic materials have been investigated and reported [5–9]. As a rule, single crystals are required to ensure high quality of nonlinear optical materials for device manufacturing. In fact, they need to be highly stable against the ambient conditions and high intense light source. Therefore, knowledge about the nucleation

parameters is essential to grow large single crystals for device fabrication. A literature survey shows that the complexes of inorganic salts with amino acids are important materials for nonlinear optical applications. Hence, nucleation parameters of some amino acid based semi-organic materials have been studied and reported to improve the crystal growth process [10–13]. In general, glycine is the simplest amino acid and its complexes find applications in many NLO fields [14, 15]. Among them, triglycine sodium molybdate (TGSM) is one of the potential semiorganic NLO materials that crystallizes in non-centrosymmetric triclinic structure [16]. As no reports on nucleation parameters and third order nonlinearity of this material have been found in the literature. An effort is taken for the investigation of different nucleation parameters that are required for optimal growth. For the growth of single crystal with improved qualities, the nucleation parameters like metastable zone width and induction period have been evaluated experimentally. Since TGSM is the non-centrosymmetric crystal with SHG efficiency comparable with that of KDP, efforts have been made to confirm the existence of third order nonlinearity using Z-scan technique.

2. Materials and methods

2.1. Sample preparation and solubility measurements

TGSM single crystals were prepared from (AR grade) glycine and sodium molybdate in the molar (mass or molar) ratio of 3:1. The calculated amount of solute was added to deionized water and stirred at normal temperature until it completely dissolved. The solution was allowed to evaporate by heating till crystalline salt of TGSM was obtained. The synthesized salt was purified by repeated recrystallization process. The solubilities of TGSM were measured for various temperatures from 35 to 50 °C in the step of 5 °C. The saturation concentration of the solute was found to increase with temperature.

2.2. Metastable zonewidth

Metastable zonewidth is an important parameter of the crystallization process. The value of this parameter was measured using the polythermal method [17]. A solution of TGSM in 100 ml of deionized water was prepared at 35 °C. After this, the solution was overheated to 5 °C above the saturation temperature and it was continuously stirred to obtain homogeneous concentration. The equilibrium saturated solution prepared in this way was cooled down from the overheated temperature at a rate of 4 °C/h until first crystal nuclei became visible. The direct vision method was used for detecting such nuclei. The difference between the saturation temperature and the nucleation temperature is known as the metastable zone width. The same procedure was repeated for the temperatures of 40, 45 and 50 °C. The dependence of metastable zone width *versus* temperature is shown in Fig. 1.

2.3. Nucleation kinetics

Estimations of induction period for different supersaturation ratios give an idea of an appropriate value of induction period to grow high-quality single crystals at controlled nucleation rates [17]. The solution with the supersaturation ratio of 1.2 was prepared and kept at room temperature.

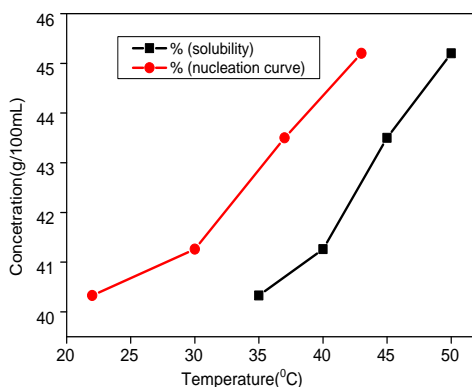


Fig. 1. Dependence of metastable zone width vs temperature.

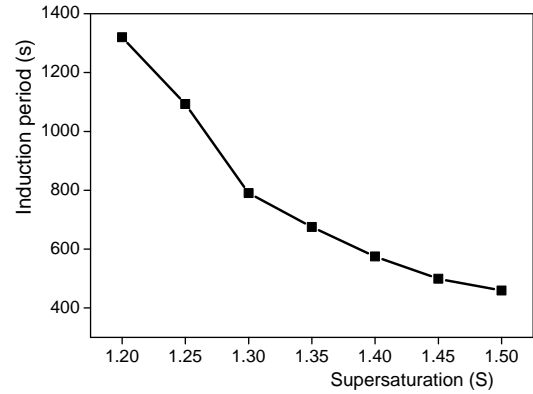


Fig. 2. Dependence of induction period vs supersaturation ratios.

The time required to grow a critical nucleus to a detectable size is negligibly small compared with the time between achieving supersaturation and appearance of a crystal of a detectable size. Hence, the time required to grow first sparkling nuclei in an undisturbed supersaturated solution is taken here as the induction period (τ). The induction period was found for various supersaturation ratios such as 1.25, 1.3, 1.35, 1.4, 1.45 and 1.5. The obtained results are presented in Fig. 2. The expression for induction period is written as follows [18–20]:

$$\tau \propto \exp \left[\frac{\Delta G^*}{kT} \right], \quad (1)$$

$$\ln \tau = \ln B + \frac{\Delta G^*}{kT}, \quad (2)$$

$$\Delta G^* = \frac{16\pi\gamma^3}{3\Delta G_v^2}. \quad (3)$$

Here, B is the constant, γ is the interfacial tension and ΔG_v is the bulk energy change per unit volume given by

$$\Delta G_v = -\frac{\Delta\mu}{V}, \quad (4)$$

where

$$\Delta\mu = kT \ln S. \quad (5)$$

The crystal volume is found from the following relation:

$$V = \frac{\text{Molecular weight}}{\text{density} \times N_A}. \quad (6)$$

The supersaturation ratio is $S = C/C^*$, where C and C^* are the actual and the equilibrium concentration of the solution, respectively.

The interfacial-tension γ is found from the experimental values of induction period. Its value is used to calculate various critical nucleation parameters. According to the classical theory of homogeneous nucleation,

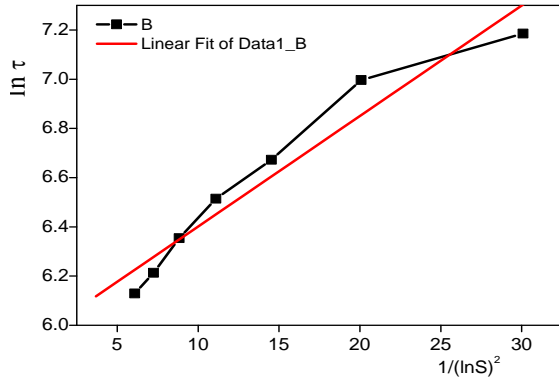


Fig. 3. Dependence of $\ln \tau$ vs $1/(\ln S)^2$.

$$\ln \tau = \ln B + \frac{16\pi\gamma^3 V^2 N_A^3}{3R^3 T^3 (\ln S)^2}, \quad (7)$$

where

$$k = R/N_A. \quad (8)$$

According to Eq. (7), the dependence $\ln \tau$ vs $1/(\ln S)^2$ is a straight line and the value $\frac{16\pi\gamma^3 V^2 N_A^3}{3R^3 T^3 (\ln S)^2}$ can be estimated from the slope (m) of this line as shown in Fig. 3. It can be seen from this figure that the value of induction period decreases with the increase in supersaturation ratio. Since $\ln B$ is the temperature dependent parameter, the interfacial tension can be found from the slope of the graph in Fig. 2 as follows:

$$\gamma^3 = \frac{3R^3 T^3 m}{16\pi V^2 N_A^3}. \quad (9)$$

The change in ΔG between the crystalline phase and the surrounding liquid phase drives the crystallization process. For rapid crystallization, $\Delta G < 0$ [21]. The free energy associated with formation of nuclei is given by

$$\Delta G = 4\pi r^2 \gamma + \frac{4}{3} \pi r^3 \Delta G_v, \quad (10)$$

where r is the radius of the nuclei. The first term expresses the formation of a new surface, while the second term represents the chemical potential (μ) difference between the crystalline phase and the surrounding liquid phase.

The nucleation free energy corresponds to the condition $d(\Delta G)/dr = 0$ [22]. Hence, the radius of the critical nucleus has been calculated from the following expression:

$$r^* = -\frac{2\gamma}{\Delta G_v}. \quad (11)$$

The nucleation rate J is the number of critical nuclei formed per unit time in a unit volume and it is expressed as

$$J = A \exp\left[\frac{-\Delta G^*}{kT}\right]. \quad (12)$$

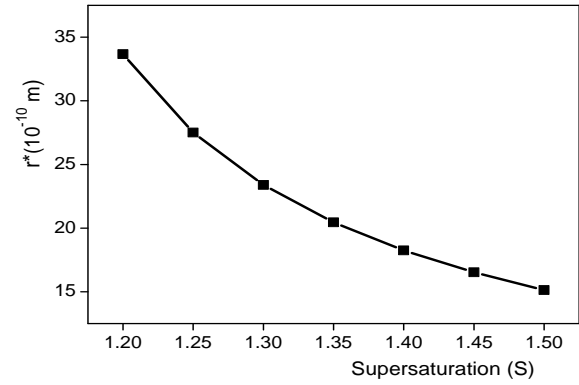


Fig. 4. Dependence of critical nucleus radius vs supersaturation ratio.

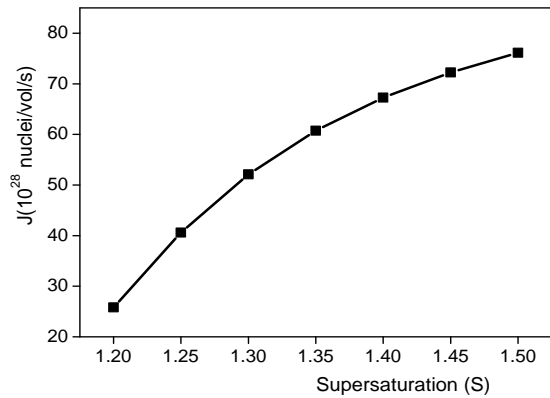


Fig. 5. Dependence of nucleation rate vs supersaturation ratio.

The number of molecules in a critical nucleus is expressed as follows:

$$i^* = \frac{4\pi(r^*)^3}{3V}. \quad (13)$$

Variations of the radius of the critical nucleus and the nucleation rate with supersaturation ratio are plotted in Figs 4 and 5.

2.4. Nonlinear optical measurements

Second harmonic generation (SHG) efficiency of TGSM has been analyzed using the Kurtz–Perry powder method by irradiating with 1064 nm Nd:YAG laser emission. The SHG efficiency was obtained to be 0.83 times that of KDP single crystals as reported by Amasingh Bhabu *et al.* [16].

In this work, third order non-linearity of the TGSM single crystals was analyzed using the Z-scan method. Non-linear refraction and absorption were measured by a femtosecond Z-scan spectrometer. In this technique, a sample is translated in the z -direction along the axis of the focused Gaussian beam from a He-Ne laser with the wavelength of 632.8 nm. Far field intensity is measured according to the sample position. By properly monitoring the transmittance change through a small aperture at the far field position. The amplitude of the phase shift was

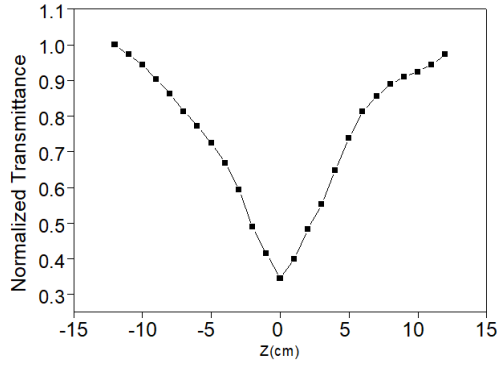


Fig. 6. Dependence of normalized transmittance vs the optical path distance obtained by the open aperture technique.

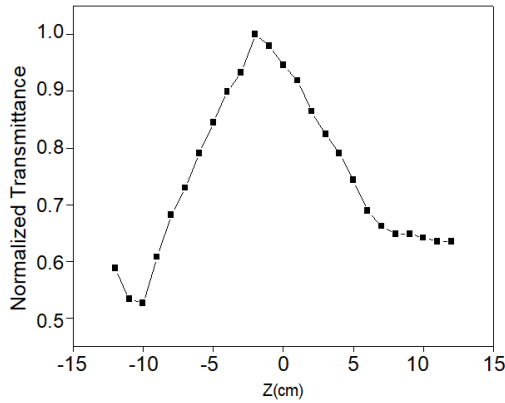


Fig. 7. Dependence of normalized transmittance vs the optical path distance obtained by the closed aperture technique.

found by observing the transmittance changes through the small apertures at the far field position (closed aperture). The intensity dependent absorption by the sample was measured by moving the sample through the focus without placing an aperture (open aperture). Both techniques (closed and open aperture) are used for measuring signal ratios, which provide the non-linear refraction of the samples. Fig. 6 shows the normalized transmittance obtained by the OA technique. The plot in Fig. 6 is symmetric with respect to zero. The minimum transmittance is observed at $z=0$. These findings correspond to two-photon absorption, which indicates that the crystal exhibits reverse saturation absorption [23].

The nonlinear refractive index of the grown single crystal was found using the following expression:

$$n_2 = \frac{\Delta\phi}{kI_0L_{eff}},$$

where $\Delta\phi = \frac{\beta I_0 L_{eff}}{Z}$ is the phase shift at the focus and

$$L_{eff} = \frac{1 - \exp(-\alpha L)}{\alpha}$$

is the effective thickness of the sample, respectively. In the last two expressions, α is the linear absorption coefficient at 632.8 nm, L is the sample

thickness, I_0 is the intensity of the focused beam, β is the non-linear absorption coefficient, and Z is the optical path distance, respectively. The non-linear absorption coefficient β is calculated from the following formula:

$$\beta = \frac{2\sqrt{2} \Delta T}{I_0 L_{eff}}.$$

The normalized transmittance for the CA technique is plotted in Fig. 7. The valley-to-peak shape of the curve in this figure implies the positive value of the refractive index and the self focusing nature of the sample. The nonlinear susceptibility is found from the following expression:

$$|\chi^{(3)}| = \sqrt{(\chi_I^{(3)})^2 + (\chi_R^{(3)})^2},$$

where

$$\chi_R^{(3)} = \frac{10^{-4} \epsilon_0 c^2 n_0^2 n_2}{\pi}$$

$$\chi_I^{(3)} = \frac{10^{-2} \epsilon_0 c^2 n_0^2 \lambda \beta}{4\pi^2}$$

3. Results and discussion

Fig. 1 shows variation of the metastable zone width with temperature. It can be seen from this figure that the metastable zone width decreases with the increase in temperature. The results of the induction period measurements are depicted in Fig. 2. The induction period decreases exponentially with supersaturation, which points to exponential growth of the nucleation rate. Hence, the number of the formed critical nuclei will increase leading to spurious nucleation. To grow high-quality single crystals, the value of the induction period as an important parameter defining the nucleation rate should be thoroughly controlled. The dependence $\ln \tau$ vs $1/(\ln S)^2$ is shown in Fig. 3. The slope of this graph is determined from its linear fit. The interfacial tension is calculated to be 0.119 mJ/m². The nucleation parameters corresponding to different supersaturation ratios are presented in Table. It can be seen from this table that the ΔG^* and i^* values decrease with supersaturation. The radius of the nucleus and the nucleation rate as the functions of supersaturation are shown in Figs 4 and 5. It can be seen from these figures that the radius of the critical nucleus decreases and the nucleation rate increases with supersaturation.

Third order non-linear optical analysis enabled the estimation of the nonlinear parameters. The OA and CA responses are shown in Figs 6 and 7, respectively. The valley-to-peak shape of the graph obtained by the closed aperture technique evidences positive value of the refractive index and self-focusing nature of the material under study [24]. Hence, this material is capable of refocusing the beam and allows more light to get through the aperture thus providing lens functionality. The non-linear refractive index of the material is evaluated to be $5.45 \cdot 10^{-11} \text{ cm}^2/\text{W}$. The symmetric response obtained by

Table. Kinetics parameters of TGSM crystal nucleation.

| Super-saturation $S = C/C^*$ | $\Delta G^* \cdot 10^{-21}$ J | $J \cdot 10^{28}$ nuclei/vol/s | $r^* \cdot 10^{-10}$ m | i^* |
|---------------------------------|-------------------------------|-----------------------------------|------------------------|-------|
| 1.2 | 5.65 | 25.8 | 33.67 | 14.82 |
| 1.25 | 3.77 | 40.59 | 27.51 | 8.08 |
| 1.3 | 2.73 | 52.09 | 23.39 | 4.97 |
| 1.35 | 2.08 | 60.74 | 20.45 | 3.32 |
| 1.4 | 1.66 | 67.26 | 18.24 | 2.36 |
| 1.45 | 1.36 | 72.24 | 16.52 | 1.75 |
| 1.5 | 1.14 | 76.14 | 15.13 | 1.35 |

the open aperture technique indicates the reverse saturation absorption nature of the studied sample. This property is used in optical limiters such as protecting goggles. The calculated value of absorption coefficient is $4.345 \cdot 10^{-4}$ cm/W. The calculated effective value of susceptibility is $1.17 \cdot 10^{-7}$ esu. The real part ($2.92 \cdot 10^{-11}$ esu) is found to be less than the imaginary part ($1.17 \cdot 10^{-7}$ esu). This suggests the dominance of absorption over transmittance that can be seen from the larger amplitude of the valley as compared to the peak amplitude in Fig. 7 [25].

4. Conclusion

The optimized growth parameters for formation of bulk single crystals of TGSM such as interfacial tension, free energy, radius of the critical nucleus and number of nuclei per unit time in a unit volume were calculated from the experimental data. The study confirms that with the increase of supersaturation ratio, the critical nuclei size decreases and more nuclei are generated in a unit volume. This suggests that spontaneous nucleation can be minimized by reducing the nucleation rate, which is suitable for the growth of bulk crystals. The third order nonlinear optical studies of TGSM single crystals using the Z-scan technique with a He-Ne laser confirms suitability of our material for nonlinear optical devices such as optical limiters and optical switching.

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Дослідження параметрів зародкоутворення та нелінійних оптичних властивостей третього порядку у монокристалах тригліцин-молібдату натрію

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Анотація. У цій роботі досліджено ряд параметрів зародкоутворення, таких як індукційний період, міжфазний натяг, ширина метастабільної зони, критичний радіус зародкоутворення та зміна вільної енергії Гіббса, у монокристалах тригліцин-молібдату натрію (TGSM), вирощених з розчину. Визначено розчинність TGSM при різних температурах. Для оптимізації процесу росту експериментально знайдено величини періоду індукції та ширини метастабільної зони. З експериментально спостережуваних величин із застосуванням класичної теорії зародкоутворення було розраховано значення ряду критичних параметрів зародкоутворення. Установлено, що швидкість зародкоутворення зростає зі збільшенням пересичення. Значення нелінійних параметрів третього порядку, таких як коефіцієнт поглинання (β), чутливість ($\chi^{(3)}$) та показник заломлення (n_2), отримані з аналізу результатів Z-сканування, підтверджують, що вирощені кристали мають природу самофокусування та оберненого насиченого поглинання.

Ключові слова: нелінійний міжфазний натяг, зародкоутворення, оптика, пересичення.