Current Natural Sciences & Engineering
Volume 2, Issue 3, 2025, 660-667

Brief Overview of Seed Rotation Technique (SRT) Apparatus: An Effective Tool for Growing Bulk Size Single Crystals

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Received date:17/04/2025, Acceptance date: 12/06/2025

***DOI:* http://doi.org/10.63015/5c-2463.2.3**

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Abstract

Single crystals are emerging in technology effectively owing to their superior physical, optical and electronic properties. They serve as fundamental building blocks in advancing various domains, including optoelectronics, nonlinear optics, laser technology and photonics. One of the effective technique for growth of crystals is seed rotation method which has led to significant advancement in the controlled crystal growth process. In the present article, we provide a brief discussion on the seed rotation technique used in solution-based crystal growth methods to enhance the size and quality of crystals. The rotation reduces thermal gradients, inhibiting the emergence of defects such as dislocations and inclusions. It promotes convective mixing within the solution, hence reducing localized temperature variations and facilitating a more uniform thermal environment for the developing crystal. The enhanced thermal uniformity can indirectly mitigate the negative consequences usually linked to abrupt heat gradients, including strain and fault formation during growth. Rotation also facilitates enhanced crystal symmetry and homogeneity, which are crucial for the performance and reliability of crystals utilized in semiconductor, optical and advanced material applications. This article contains an overview on the seed rotation technique (SRT) apparatus and explores the impact of several parameters on crystal quality, as well as the mechanisms that contribute to defect reduction and enhanced structural integrity.

Keywords: single crystal, rotation, homogeneity, seed crystal, ACRT controller.

1. Introduction

Good quality single crystals are extensively needed for numerous optical devices, including electro-optical, nonlinear optical (NLO) and photo-refractive storage devices [1-3]. However, growing a good-quality crystal is often a tedious task. Most of the time, we lead to many disorders in the single crystals such as dislocations, grain boundaries, etc. There are several methods to grow a single crystal but, the low-temperature solution growth technique has its own superiority over the other methods due to its high-quality products [4-6]. It mainly requires an effective technique where different growth parameters like supersaturation level, phase homogeneity, temperature fluctuation, impurity formation and etc. can be controlled [7,8]. The slow evaporation solution technique (SEST) is considered to be the simplest and most cost-effective method for producing high-quality single crystals [9]. Although this technique is known as superior for growing highly crystalline products, still defects are created due to fluctuations in various parameters most of the time [10,11]. Similarly, the growth of various planes is blocked due to the unwanted nucleation at the walls of the growth vessel. Few of the unwanted growth of crystals are demonstrated in Fig. 1(a) which shows how crystal growth is hindered by the nucleation. From Fig. 1(b), one can observe the damage in the crystal base plane due to uneven nature of growth vessel which results in pits on the crystal base. In order to overcome such effects, various modifications can be implemented in the existing methods and high-quality crystals can be grown with ease. In this regard, SR (Sankaranarayanan-Ramasamy) and seed rotation technique (SRT) are considered prominent for bulk-size production of single crystals [12,13]. SR method is universally adopted to grow single crystals in a particular direction. More specifically, crystals are grown across one plane known as the unidirectional growth of single crystals [14]. Whereas seed rotation allows the growth of ingot across all defined planes. All planes will evolve in their respective directions, yielding a bulk single crystal with enhanced quality as a result of controlled parameters. Rotation ensures even exposure of the seed crystal to the supersaturated solution, facilitating uniform growth on all surfaces and reducing the risk of asymmetrical or faceted growth. In both the later said techniques, crystals are grown in a more controlled way to minimize the defect concentration and maximise the yield [15-17]. We will discuss the seed rotation technique and the effect of various controlled parameters on the growth of single crystals in this article. Details on the apparatus of the seed rotation method will be described in the forthcoming section.

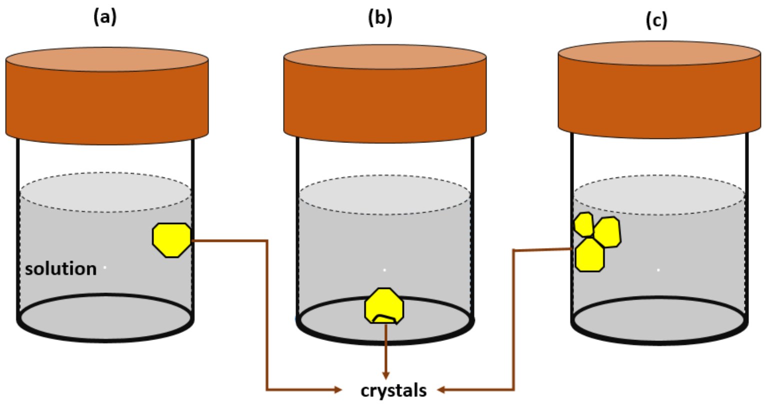

Figure 1 (a) Plane stuck on the walls of the beaker, (b) Hampered plane due to base of the beaker and (c) The formation of aggregates leading to multinucleation.

2. Design and Operation of SRT Apparatus

"SRT" is one of the most significant low-temperature solution growth technique which is used to grow high-quality and bulk-size single crystals [18-21]. Generally, parameters like supersaturation, solubility, temperature fluctuation and mechanical vibrations are significant in SEST [22]. When evaporation takes place, solute molecules settle at the bottom of the growth vessels. Therefore, the homogeneity of the solution is automatically lost, as a result of this, uniform growth of crystals is hampered. In order to overcome such difficulties, SRT is used which provides an extra parameter i.e. 'rotation' to maintain the uniform growth of the crystal. Gradually rotating the platform containing seed crystal during the growing phase facilitates a symmetrical distribution of temperature and solute concentration around the seed crystal. This will help in reducing the inhomogeneities present in the solution. In this way, it helps in maintaining a steady concentration of solute near the crystal surface by preventing local accumulation, which is crucial for sustaining constant growth rates.

Recently, we installed an 'SRT' setup at CSIR-National Physical Laboratory, New Delhi, India. The detailed setup is provided in Fig. 2 which consists of a seed rotation controller coupled with a stepper motor (controlled by using a microcontroller-based drive (ACRT controller)), a glass beaker (5 Litre) and a crystal holder with the motor shaft connecting to ACRT controller. The ACRT (which means automatic crystal rotation and translation) controller plays a vital role in the seed rotation system by offering precise, programmable control over the movement of the seed crystal through the crystal growth process. The system described here provides a maximum rotation speed of 77.9 RPM (revolutions per minute) and a minimum speed of 0.1 RPM. It has three modes of operation 1. auto mode, 2. manual mode and 3. acceleration/ de-acceleration mode. In the auto mode, the motor runs continuously for the set RPM and direction of rotation can be set either clockwise or anticlockwise. In this mode, the controller regulates the rotation speed of the seed crystal as per the set rpm, ensuring a uniform and optimized rotational motion throughout the growth period. In the manual mode, we can manually set the number of cycles, and run the motor in two different modes: fixed mode and BI direction mode. In fixed mode, the motor shaft starts running at fixed RPM in clockwise/anticlockwise direction for desired number of cycles and then motor stops. In the bidirectional mode, initially motor shaft starts running in the forward (clockwise) direction for the desired number of cycles. On completing forward cycle, shaft starts rotating in reverse (anticlockwise) direction for the same number of cycles and then motor stops. The main benefit of manual mode is user can directly control the setting parameters as per requirement in a more optimized way during the growth process. It can help in a controlled growth process and one can get better quality of the crystal. In the third mode that is acceleration/ de-acceleration mode, the speed X (RPM) of motor and time (t) for which that X has to be maintained can be set for required number of cycles. Acceleration denotes how quickly the controller increases the rotation speed from zero (or a lower value) to the set value whereas de-acceleration is how gradually it slows the movement down before stopping. It facilitates controlled and predictable movement, which is especially crucial when handling fragile or delicate seed crystals. The choice between acceleration/de-acceleration mode and continuous rotation typically depends on the desired control over convection and solute distribution. Acceleration/de-acceleration mode entails periodic variation of the rotational speed. This sporadic motion can improve convective mixing around the seed and throughout the solution, aiding in the prevention of localized supersaturation. This is often applied in the growth of crystals with complex compounds, where segregation and striation control are critical. In contrast, continuous rotation is favoured when a consistent and uniform convective flow is needed for processes like the development of high-purity single-component crystals. In this way, we can optimize the settings according to our requirement and obtain a good quality bulk single crystal useful in many applications [23-25].

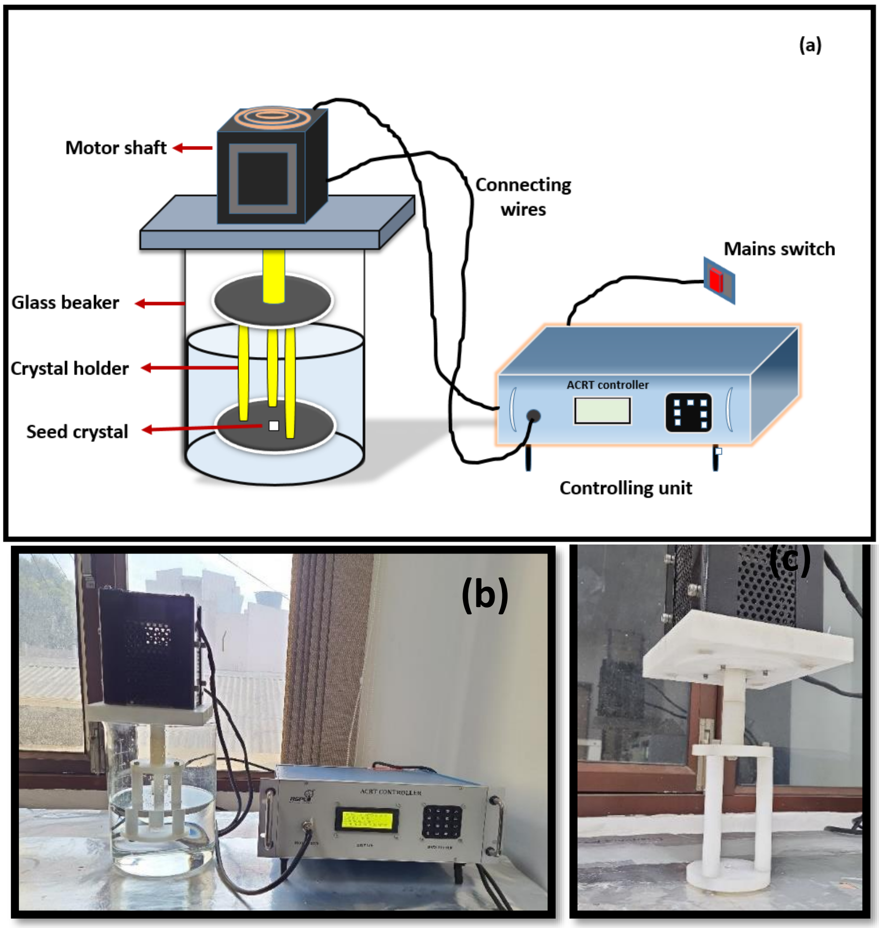

Figure 2 (a) Schematic diagram of seed rotation (SRT) setup, (b) Complete setup of SRT at CSIR-NPL, New Delhi and (c) Crystal holder with seed mount on the platform.

3. Working Procedure

In this technique, first we require a seed crystal which is produced using conventional slow evaporation solution technique (SEST). The seed crystal must be free from inclusions, cracks, or twin boundaries and size of the crystal should be sufficient to initiate growth (typically 3-10 mm) with well-developed natural facets. It is then lapped and polished with the help of alumina slurry to make flat at bottom. The final surface roughness after polishing must be ≤ 0.1 µm and force applied should be 1–2 N (gentle to avoid microfractures). The SRT apparatus is then used to grow the bulk size single crystal. At first, a saturated solution of the same material is prepared following the SEST procedure with the temperature range determined by its solubility curve [4,18]. The saturated solution is poured in the glass beaker with the help of funnel only after filtration in order to remove undissolved particles or dust that could act as unintended nucleation centers. Ensure the beaker in which solution is poured is thoroughly cleaned and rinsed with the same solvent used in the preparation of the solution. The solution should be slowly transferred along the side of the beaker to minimize turbulence, avoid bubble formation, and prevent localized cooling or splashing. Crystal holder made up of teflon is dipped in this solution after pasting the seed crystal on its surface as seen in Fig. 2(a). The quantity of the saturated solution in the beaker should be made such that it should cover some portion of the crystal holder as shown in Fig. 2(b). The stepper motor is paired with a seed (crystal) holder where the seed crystal is kept for crystallization. This motor rotates the seed holder in the crystallizer. The seed crystal is put in the centre of the platform and fixed with the help of adhesive to avoid disturbance. The adhesive Anabond 202 can be used which is a non-reactive material to avoid any kind of impurity in the solution. The crystal holder rotates along with the motor shaft according to the commands that motor shaft receives from the controller unit (ACRT controller) via the connector [26]. The ACRT controller controls the rotation motor settings. It has a LED display where we can see the stepper motor settings and set accordingly. This makes SRT an effective tool which can be made even more impactful by various controlled factors. The set up should be kept in a controlled environment for the growth process. The impact of various parameters during the growth part will be discussed in the next section.

4. Impact of different parameters

By utilizing seed rotation technique, various growth parameters such as growth atmosphere and rotation rates under the fixed growth rate can be optimized which will help in production of high quality bulk single crystals. The effect of rotation plays vital role in the growth part. Rotation will provide uniformity to the solution making it stable which in turn will help in the uniformity in the single crystal. Inclusions in the crystals will occur as a result of inhomogeneous supersaturation in the solution, which is why the seed must rotate uniformly to prevent stationary areas or recirculating flows. This will give crystal with minimum inhomogeneity which mainly includes dislocations, strains, grain boundaries, etc. Also, it is important to optimize the speed of the motor as the rotation speed also plays an important role in improving the crystal quality. It was found that when the solution was stirred by rotating the seed crystal at 10 rpm in the anticlockwise direction, due to stability in the solution the crystal quality was improved. Hence, the importance of optimum rates of rotation in crystal growth processes has significantly lead to improved quality. Conversely, if not well controlled, rotation can cause uneven solute concentration around the seed, leading to striations or irregular crystal shape. High-speed or fluctuating rotation in some cases can induce mechanical vibrations or misalignment, leading to growth instabilities or off-axis crystal development. Therefore, it is crucial to manage rotation parameters carefully to avoid such disruptions. In addition to this, in systems with a high solvent evaporation rate, rotation can accelerate solvent loss or disrupt the meniscus, making the process unstable. In these instances, seed rotation is less relevant, and alternative growth strategies are preferred.

Thermal fluctuations due to natural convection can also be controlled to stabilize the solution. Controlled atmosphere can be maintained by providing constant temperature to the solution. In order to maintain constant temperature of the solution, the beaker attached with crystal holder is kept in the constant temperature bath (CTB) which can help optimize the temperature as depicted in Fig. 3. The crystal can be extracted from the solution once the desired crystal size and quality are attained, following a progressive reduction or cessation of the heating process to provide controlled cooling. The crystal is then separated from the seed holder and removed from the apparatus for post-growth processing, including cutting, polishing, or characterization. The sulphamic acid crystal grown by Sonia *et al.* and corundum single crystals grown by K. Watanabe, *et al.* using the seed rotation technique are the well-known examples [27,13]. Among these, the sulphamic acid single crystal obtained using seed rotation technique possess dimensions 40 × 10 × 23 mm^3 which was produced with a seed crystal of dimensions 10 × 3 × 7 mm^3. These examples underscore the versatility and efficacy of seed rotation in improving the quality of various functional crystals in optical, electrical, and photonic applications [28,29].

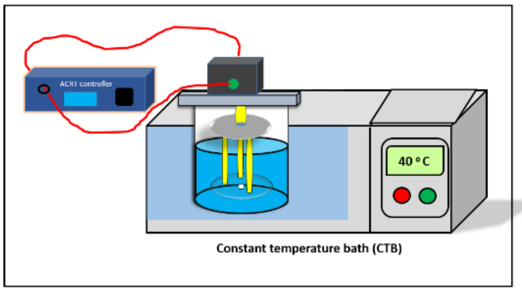

Figure 3 Constant temperature bath (CTB) to maintain growth atmosphere stable.

5. Conclusion

In conclusion, the seed rotation technique (SRT) apparatus represents a significant advancement in crystal development, especially for the production of bulk-sized single crystals with improved structural quality. The equipment facilitates controlled and uniform rotation of the seed crystal by efficiently minimising temperature and chemical gradients at the growth interface, hence decreasing the probability of defect formation and fostering uniform crystal development. Its adaptability to several crystal growth techniques and materials makes it a versatile tool for both research and industrial applications.

Conflicts of Interest

There are no conflicts to declare.

Acknowledgements

All the authors in this article are grateful to the Director, CSIR-NPL, New Delhi for providing such a facility to grow single crystals and persuading Ph.D. in ACSIR. One of the authors Ms. Kiran is grateful to CSIR, India for the financial support through CSIR- Senior Research Fellowship (SRF) and AcSIR-NPL, India for PhD registration.

References

[1] P.S. Patil, S.R. Maidur, S.V. Rao, S.M. Dharmaprakash, Crystalline perfection, third-order nonlinear optical properties and optical limiting studies of 3,4-Dimethoxy-4′-methoxychalcone single crystal, Opt. Laser Technol. 81 (2016) 70–76.

[2] L. Zhao, S.B. Li, G.A. Wen, B. Peng, W. Huang, Imidazole derivatives: Thermally stable organic luminescence materials, Mater. Chem. Phys. 100 (2006) 460–463.

[3] M. Narayan Bhat, S.M. Dharmaprakash, Growth of nonlinear optical γ-glycine crystals, J. Cryst. Growth 236 (2002) 376–380.

[4] Kiran, N. Vijayan, D. Nayak, M. Kumari, Vinod, K. Kumar, P. Vashishtha, N. Thirughanasambantham, V. Balachandran, B. Sridhar, G. Gupta, A comprehensive assessment on synthesis, growth, theoretical & optical properties of glycine zinc sulphate pentahydrate single crystal for third-order nonlinear optical applications, J Mater Sci: Mater Electron 34 (2023) 1132.

[5] B. Simon, R. Boistelle, Crystal growth from low temperature solutions, J. Crys. Growth 52 (1981) 779-788.

[6] R.A. Laudise, Techniques of crystal growth, Appl. Solid State Phys. Boston, MA: Springer US, (1970) 171-178.

[7] S.A. Gandhi, Single crystal growth by a slow evaporation technique: Concept, mechanisms and applications, CRYSTAL GROWTH (2017) 1.

[8] K. Byrappa, T. Ohachi, Crystal growth technology, Elsevier, 2003.

[9] A. Badoni, Kiran, N. Vijayan, D. Joshi, Jyoti, K. Kumar, S. Srivastava, J. Prakash, Analysis on optical, thermal and third-order nonlinear properties of L-histidine hydrochloride monohydrate single crystal, Phys. Scr. 99 (2024) 105959.

[10] W.R. Wilcox, Removing inclusions from crystals by gradient techniques, Industrial & Engineering Chemistry 60.3 (1968) 12-23.

[11] Kiran, N. Vijayan, N. Sarkar, D. Joshi, Jyoti, K. Kumar, S. Yadav, S. Das, Unveiling the optical, thermal and nonlinear behavior of guanidinium benzenesulfonate: A promising organic single crystal for NLO applications, Opt. Mater. 147 (2024) 114683.

[12] M. Kumari, N. Vijayan, D. Nayak, Kiran, J. S. Tawale, P. Singh, R. P. Pant, Investigation on unidirectionally grown < 010 > potassium acid phthalate single crystal by Sankaranarayanan–Ramasamy (SR) method for optical applications, Appl. Phys. A 127 (2021) 780.

[13] K. Watanabe, A. Iida, Y. Sumiyoshi, Growth of corundum single crystals by seed rotation method, J. Cryst. Growth. 54 (1981) 381—393.

[14] K. Sankaranarayanan, Growth of large size 〈1 1 0〉 benzophenone crystal using uniaxially solution-crystallization method of Sankaranarayanan–Ramasamy (SR), J. Cryst. Growth. 284 (2005) 203-208.

[15] C. Fang, S. Liu, Y. Wen, M. Chen, N. Wang, W. Luo, Y. Feng, H. Yao, Heng-Yun Ye, L. Liu., Dynamic seed rotation growth of two-dimensional perovskite single crystals for high-performance x-ray detect, Next Materials 2 (2024) 100137.

[16] M.S. Pandian, U. Charoen In, P. Ramasamy, P. Manyum, M. Lenin, N. Balamurugan, Unidirectional growth of sulphamic acid single crystal and its quality analysis using etching, microhardness, HRXRD, UV–visible and Thermogravimetric-Differential thermal characterizations, J. Cryst. Growth. 312.3 (2010) 397-401.

[17] C.W. Lan, Recent progress of crystal growth modeling and growth control, Chem. Eng. Sci. 59.7 (2004) 1437-1457.

[18] Vikram, N. Vijayan, Ravindra, Sonia, Mahak, Apurva, Growth of KDP crystal by automatic temperature controlled seed rotation method, Asian Journal of Physics, 27 (2018) 13-21.

[19] G.H. Nancollas, The growth of crystals in solution, Adv. Colloid Interface Sci. 10.1 (1979) 215-252.

[20] S.A. Markgraf, Y. Furukawa, M. Sato, Top-seeded solution growth of LiB3O5, J. Cryst. Growth 140.3-4 (1994) 343-348.

[21] P.V. Dhanaraj, N.P. Rajesh, P. Ramasamy, M. Jeyaprakasan, C.K. Mahadevan, G. Bhagavannarayana, Enhancement of stability of growth, structural and NLO properties of KDP crystals due to additive along with seed rotation, Cryst. Res. Technol. 1 (2009) 54 – 60.

[22] A. Mersmann, A. Eble, C. Heyer, Crystal growth, Crystallization technology handbook CRC Press, (2001) 95-158.

[23] M.S. Bakshi, A simple method of superlattice formation: step-by-step evaluation of crystal growth of gold nanoparticles through seed− growth method, Langmuir 25 (2009) 12697-12705.

[24] S. Satapathy, S.K. Sharma, A.K. Karnal, V.K. Wadhawan, Effect of seed orientation on the growth of TGS crystals with large (0 1 0) facets needed for detector applications, J. Cryst. Growth. 240 (2002) 196-202.

[25] Kiran, N. Vijayan, N. Sarkar, Vinod, Jyoti, D. Joshi, Chetan, Thermal Behaviors, Hirshfeld Surface Analysis and Impact of Shock Wave on Glycine Zinc Sulphate Pentahydrate Single Crystal, Cryst. Res. Technol. (2025) 2500016.

[26] H.J. Scheel, H. Müller-Krumbhaar, Crystal pulling using ACRT. J. Cryst. Growth. 49.2 (1980) 291-296.

[27] Sonia, N. Vijayan, M. Bhushan, K. Thukral, R. Raj, K.K. Maurya, D. Haranath, S. A. Martin Britto Dhas, Growth of a bulk-size single crystal of sulphamic acid by an in-house developed seed rotation solution growth technique and its characterization, J. Appl. Cryst. 50 (2017) 763–768.

[28] D. Nayak, N. Vijayan, M. Kumari, Kiran, P. Vashishtha, S. Das, B. Sridhar, G. Gupta, R.P. Pant, Investigation on synthesis, growth, Hirshfeld surface and third order nonlinear optical properties of Urea-Succinic Acid single crystal: A potential candidate for self-defocusing lasing application, Opt. Mater. 124 (2022) 112051.

[29] A. Saranraj, S.S. Jude Dhas, M. Jose, S.A. Martin Britto Dhas, Growth of bulk single crystals of urea for photonic applications, Electron. Mater. Lett. 14 (2018) 7-13.