

# TRIZ AND HDC SAPPHIRE GROWTH PROCESS

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*The application of Theory of Inventive Problem Solving (TRIZ) into sapphire single crystal growth process provided thorough analysis of a problem as well as introduced two conceptually new solutions. The innovation of the Bagdasarov – Horizontally Directed Crystallization (HDC) by resolving the pollution of the observation window improves the control over the process. The increased accuracy of the control leads to supreme quality of the resultant sapphire single crystal. The ultimate goal of betterment is the complete automation of the growth process.*

*Key words: TRIZ, Sapphire single crystal, HDC Bagdasarov method, Automation, Innovation*

## INTRODUCTION

After inventing of TRIZ methodology by Genrikh Saulovich Altshuller in late 1950's the innovation process became structured and Ideal Final Result (IFR) oriented [01], [02]. Many new patents are made with the use of Altshuller's contradiction matrix [03], [04] that helps to resolve technical contradictions. The proper understanding of the quintessence of the physical phenomena and their effective exploitation in technical systems also provides a firm platform for innovations [05]. This holds true especially for material physics. The material physics of sapphire single crystal reveals a promising future for application of this material in nanotechnologies [06]. Especially, titanium doped sapphire single crystal indicates a great potential in a production of wafers for solar cells, LEDs, and other nano-applications in a semiconductor industry [07], [08], [09]. The transparent form of sapphire single crystal called leuco-sapphire is finding its way in optical applications and photonics [10], [11].

The promising future of the sapphire single crystal applications brings a rising demand for a complete automation of the production process of this synthetic mineral. The Central European Institute of Technology (CEIT) in Slovakia addresses the automation issue by innovating of the Bagdasarov - Horizontally Directed Crystallization (HDC) process. The proposed solutions in this paper

were made by utilizing the TRIZ methodology into a production of sapphire single crystal. All described activities were part of industrial project focused on research and development of technology for the production of sapphire single crystal (hereinafter referred to as Sapphire Project).

## METHODOLOGY

For a proper identification of areas for possible improvements of the Bagdasarov apparatus the thorough analysis of entire HDC system has been performed. The analysis of the technological process of the sapphire single crystal growth revealed the bottle neck process that prevents its full automation. This process is the observation of the crystallization front (CF) during the growth process. The crystallization front is the boundary region between the single crystal and the melted aluminum oxide. The gradual sedimentation of the opaque alumina film onto the surface of the observation window diminishes the transparency of the window.

This way the main function of the observation system, the transmission of the information about the position and shape of the crystallization front, is corrupted. Since the polluting of the window is a continuous and permanent process, it is very important to find a suitable and effective solution.

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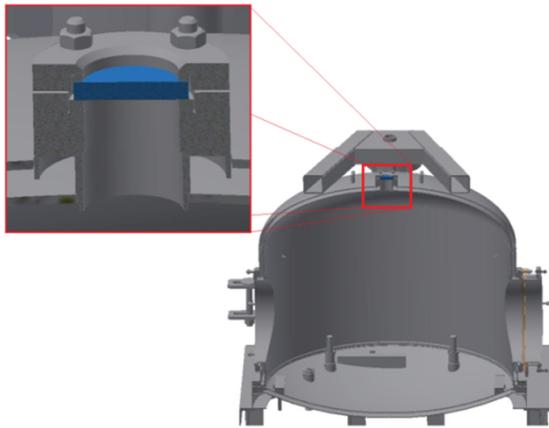


Figure 1: The location of the observation window, HDC Bagdasarov process

During the analytical phase the function-cost analysis defined relationships between individual parts of the system - Figure 2.

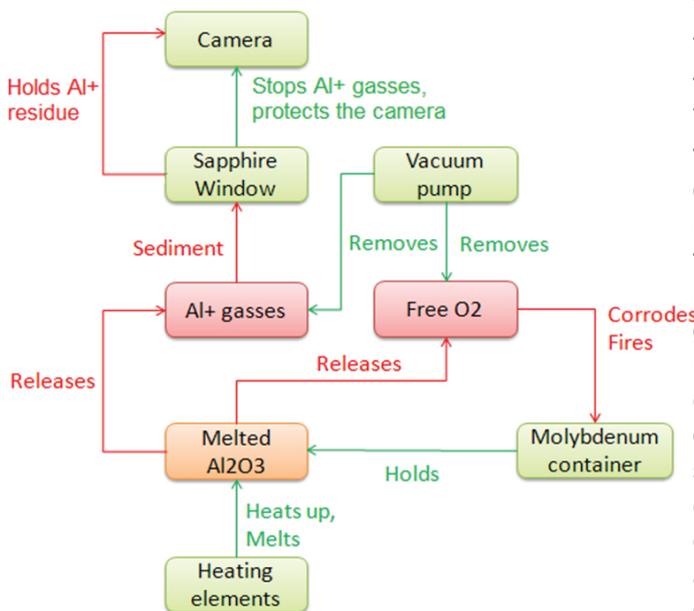


Figure 2: Function-cost analysis

The nine window analysis uncovered the system development in time as well as subsystem-system-supersystem bonds - Figure 3.

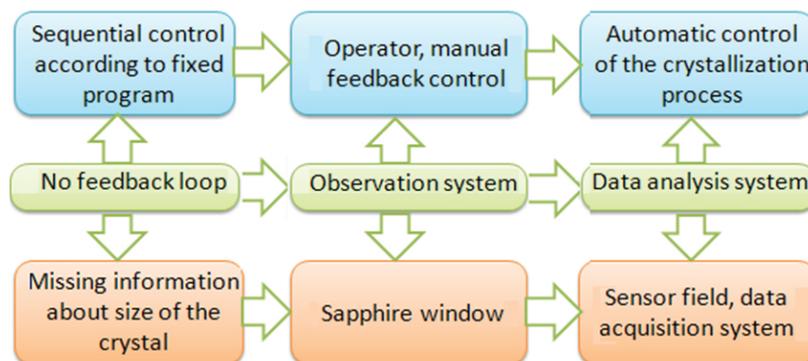


Figure 3: Nine window analysis

After thorough analysis and identification of the critical area, the synthesis of the technical solution took place. The smart little people model combined with ARIZ (algorithm of inventive problem solving) resulted in several working concepts. From the set of obtained solutions, two the most promising concepts were elaborated into detail:

- The concept of mechanical shield
- Laser ranging concept

The former one was constructed and implemented in Bagdasarov oven for sapphire single crystal growth from melt. The latter one was tested under the laboratory conditions. The lifetime calculation of the protected observation window verified the efficiency of the TRIZ methodology application in the problem solving.

**PROBLEM FORMULATION**

The use of ARIZ-86C requires precise formulation of the problem:

The Bagdasarov method of sapphire single crystal growth utilizes heating elements for melting of aluminum oxide (Al<sub>2</sub>O<sub>3</sub>). The Al<sub>2</sub>O<sub>3</sub> is placed in a boat shape container which is slowly shifted through the Hot Zone - Figure 2. As soon as the melted aluminum oxide reaches the sapphire germ, the crystallization process starts. The boundary between melt and created crystal, the crystallization front, is observed from above via observation window. Despite near vacuum pressure conditions during the crystallization process, some free aluminum atoms sediment onto cooler inner surfaces of the oven. The opaque aluminum deposit pollutes the observation window. Because of this nontransparent film on the observation window the feedback from the position and shape of the crystallization front is restricted and gradually cancelled.

Therefore the inventive task is to ensure the observation window protection with minimal changes to the system.

**IDEAL FINAL RESULT**

This leads to four possible Ideal Final Results:

1. The polluting exhaust is not produced at all – the window stays clean;
2. Although the polluting exhaust is produced it does not reach the surface of the window;
3. Despite the polluting exhaust is produced and it reaches the window it does not stay there – the polluting particles leave the surface of the observation window;
4. Even though the polluting exhaust is produced and it sediments on the window, the transparency of the window remains unaffected – the sediment itself becomes transparent;

The further solution search has to converge to any of the listed ideal final results (IFR).

**SOLUTION SEARCH**

From the problem formulation it is evident that there is a conflicting couple – observation window and Al+ exhausts. In terms of TRIZ terminology, the product is the window and the instrument is represented by Al+ exhausts.

The conflict zone is the inner surface of the observation window during the high temperature inside the Hot Zone, with the presence of aluminum oxide.

Aiming the first ideal final result (IFR1) we can state the following technical conflicts:

*The technical conflict (TC1):*

- The temperature inside the Hot Zone is high, the sapphire is produced, but Al+ exhausts pollute the inner surface of the window - Figure 4

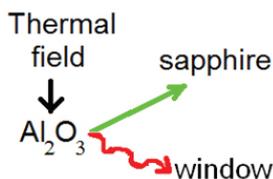


Figure 4: Technical Conflict TC1

The technical conflict (TC2):

- The temperature inside the Hot Zone is low, the window stays transparent, but the sapphire is not produced - Figure 5

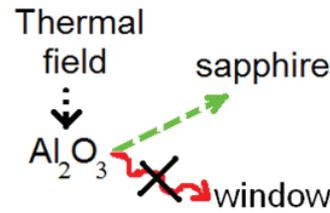


Figure 5: Technical Conflict TC2

To preserve the main function of the system, the sapphire production, the first technical conflict has to be favored. To resolve the technical conflict it is necessary to introduce X-element that will conserve the sapphire production while:

1. Preventing the production of exhausts (IFR1) – either by making retentive outer layer of the Al<sub>2</sub>O<sub>3</sub> melting charge, which does not affect the quality of the sapphire -Figure 6,

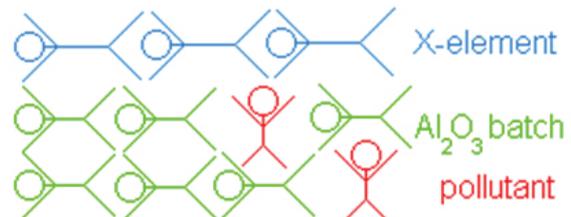


Figure 6: Smart Little People model (1)

or by strengthening the bounds between the surface atoms, such that no pollutant leaves the batch -Figure 7

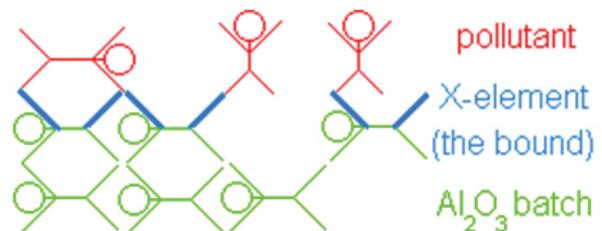


Figure 7: Smart Little People model (2)

2. Preventing polluting exhausts to reach the surface of the observation window (IFR2) – either by making the pollutant atoms heavy, so they fall back down into melting charge (due to gravity) - Figure 8,

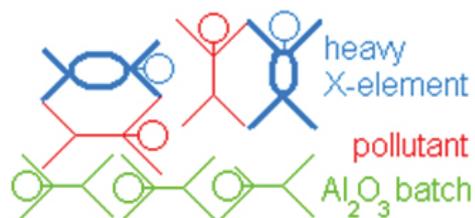


Figure 8 Smart Little People model (3)

or by changing the direction of propagation of pollutant atoms such that they miss the observation window – concepts presented below;

3. Protecting the observation window from the sedimentation of the pollutant (IFR3) – either by disabling the pollutant atoms/ions to attach to the surface of the window - Figure 9,

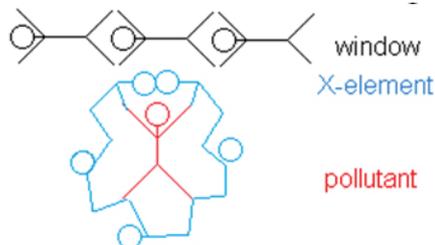


Figure 9: Smart Little People model (4)

or by making the surface of the window unattachable for pollutants (protective coating layer) - Figure 10

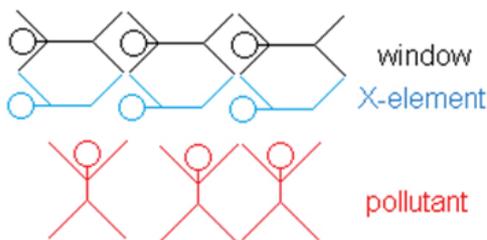


Figure 10: Smart Little People model (5)

4. Acting on sediments and making them transparent (IFR4) – e.g.: creating transparent polycrystalline alumina (TPCA) layer;

It is very important to emphasize, that pressure conditions are so low ( $P = 10^{-3}$  Pa), that only molecular flow of particles takes place inside the vacuum chamber during the crystallization process. According to this fact, many solutions including the laminar and turbulent flows of particles are retrenched.

Another restriction on the solution search is a low price and a high simplicity of the result. Therefore, many concepts, such as filtration of the charged particles by ionic sputter pump in front of observation window are disregarded.

With respect to the given criteria IFR1 has been declined, since we are not able to introduce any material changes to keep the high quality of the resultant single crystal. IFR3 was declined due to inability of current protective coating layers (e.g.: nanotechnology coating) to withstand such high temperature inside the furnace. The same holds for the additives that would disable pollutant atoms/ions to attach to the observation window

and leave the quality of the single crystal unaffected. IFR4 was disregarded due to very strict conditions needed for transparent polycrystalline alumina production. The energetic investment into creating such conditions would significantly raise the price of the final product. Therefore, IFR2 became the reachable hot favorite that we decided to aim for.

### PHYSICAL CONTRADICTION

The crystallization of the leuco sapphire is a very sensitive process when it comes to impurities. The resultant leuco sapphire has to fulfill strict quality requirements. Therefore, any substances that can affect the product quality are forbidden. Aiming the IFR2 by changing the direction of propagation of pollutant atoms, the physical contradiction shall be formulated. Since the system in this form does not contain any easily changeable elements, the ambient environment (the atmosphere inside the vacuum chamber) shall be considered as an X-element. Thus, the statement of physical contradiction (for IFR2) is:

The inner atmosphere shall be passable for the light from crystallization front to reach the observation window, but it shall be impassable for pollutant atoms to reach the surface of the window. The physical contradiction in this form can be solved with the help of separation principles.

### THE CONCEPT OF MECHANICAL SHIELD

Since the crystallization process is a slow process and there is no need in continuous observation of the crystallization front, the observation window can be covered by the mechanical shield - Figure 11.

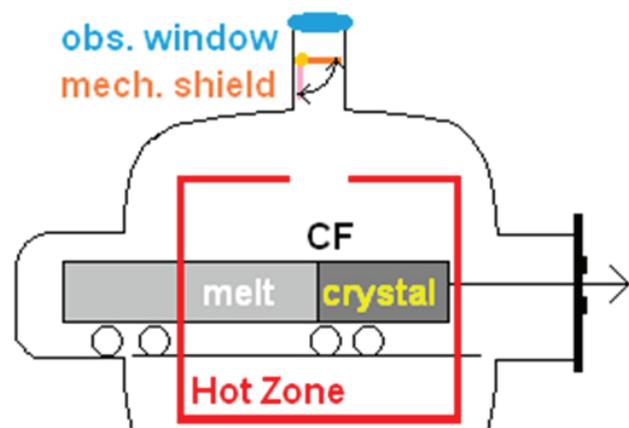


Figure 11: Conceptual solution

The mechanical shield is opened only during the CF reading periods and vast majority of time it protects the window -Figure 12.



Figure 12: The mechanical shield

This concept allows both manual and automatic operation. To make a clear field of view during readings, the tube connecting the vacuum chamber and the observation window has to have a square cross-section. The prolongation of the service life of the observation window can be calculated according to the formula:

$$\eta = (1 - T_R / T_T) \cdot 100\%$$

Where [%] is the percentage of lifetime prolongation, TR is the read out time, TT is the total time of the crystallization process. In this formula, it is assumed, that the sedimentation is continuous during the sapphire growth.

If there are needed 100 readings, each lasting for 2 seconds ( $T_R = 200s$ ) and the growth process lasts for 4 days ( $T_T = 345600s$ ) then the lifetime prolongation of the window, using the mechanical shield, is  $\eta = 99.94\%$ .

Besides the simplicity and efficiency of this solution the ease of implementation has to be mentioned. The original interface for the observation window grip is utilized and the measuring method is preserved. The physical contradiction arisen from the IFR2 – the window should be transparent during the reading and covered otherwise, has been resolved by separation in time.

### THE LASER RANGING CONCEPT

The second method for resolving the physical contradiction is a separation in space:

One part of the inner atmosphere has to be passable for the light, while another part of the inner atmosphere has to be impassable for pollutant atoms.

In this case a mechanical barrier shall be introduced to separate the two regions. Knowing the fact, that the leuco-sapphire single crystal is optically transparent material for a wide range of wavelengths [12] and assuming that the laser beam will be reflected back from the crystallization front, the laser ranging concept can be formulated. Since the leuco-sapphire germ is also transparent, the protective tube can connect the germ with observation window - Figure 13. This way the tube is enclosed from both ends. Polluting atoms can sediment on the outer surface of the tube, leaving the inner surface clean.

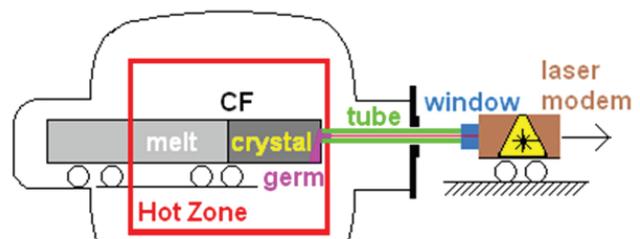


Figure 13: Laser ranging concept

The laser ranging concept is based on the horizontal laser ranging of the crystallization front (CF). The precise time measurement between the sent and received laser pulses will allow accurate localization of the CF.

Since one part of the protective tube enters the inside of the Hot Zone (temperature  $T = 2100^\circ C$ ), the tube has to be made of molybdenum.

The other end of the protective tube leaves the vacuum chamber into standard laboratory conditions (temperature  $T = 21^\circ C$ ). Thus, special sealing interface has to separate both regions and in a meanwhile allow the shifting of the boat container. The biggest advantage of this concept is a permanent measurement of the crystallization front. The novelty of the proposed solution is counterbalanced with the need in modification of the shifting mechanism as well as the use of expensive materials. The last but not least, the laser point sensor available on the market e.g.: [13], that fulfills the imposed requirements, has to be modified to fit into the system.

### CONCLUSION

The complete automation of the sapphire single crystal growth process is very feasible for the further reduction of the production costs of this unique material. Company CEIT introduces new concepts that resolve the critical issue restraining the Bagdasarov HDC growth process

from further removing of the human factor. The search for the win-win solutions avoiding unfavorable compromises requires new approaches employing creative problem solving. TRIZ methodology allows addressing such challenges and brings new insight into the solution search. The obtained concepts are contributing to significant improvement of the sapphire single crystal growth process and provide a promising platform for future innovation.

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### REFERENCES

- 1) Chechurin, L., & Borgianni, Y. (2016). Understanding TRIZ through the review of top cited publications. *Computers in Industry*, 82, 119–134. <http://doi.org/10.1016/j.compind.2016.06.002>
- 2) Bušov, B., Žídek, J., & Bartlová, M. (2016). TRIZ Already 35 Years in the Czech Republic. *Procedia CIRP*, 39, 216–220. <http://doi.org/10.1016/j.procir.2016.01.191>
- 3) Palčák, F. (2017). Improvement of Dynamic Characteristics of the Car in the Light of Technological Evolution (pp. 391–397). Springer International Publishing. [http://doi.org/10.1007/978-3-319-44087-3\\_53](http://doi.org/10.1007/978-3-319-44087-3_53)
- 4) Katolický, Z., Busov, B., & Bartlova, M. (2014). Turbojet engine innovation and TRIZ. In *Proceedings of the 16th International Conference on Mechatronics - Mechatronika 2014* (pp. 16–23). IEEE. <http://doi.org/10.1109/MECHATRONIKA.2014.7018230>
- 5) Bultey, A., Yan, W., & Zanni, C. (2015). A Proposal of a Systematic and Consistent Substance-field Analysis. *Procedia Engineering*, 131, 701–710. <http://doi.org/10.1016/j.proeng.2015.12.357>
- 6) Kuchuk, A. V., Lytvyn, P. M., Li, C., Stanchu, H. V., Mazur, Y. I., Ware, M. E., Salamo, G. G. (2015). Nanoscale Electrostructural Characterization of Compositionally Graded Al<sub>x</sub>Ga<sub>1-x</sub>N Heterostructures on GaN/Sapphire (0001) Substrate. *ACS Applied Materials & Interfaces*, 7(41), 23320–23327. <http://doi.org/10.1021/acsami.5b07924>
- 7) Leroux, M., Brault, J., Matta, S., Korytov, M., Damilano, B., Vinter, B., Jaziri, S. (2015). Optical properties of Al<sub>0.5</sub>Ga<sub>0.5</sub>N/GaN polar quantum dots and UV LEDs made of them. In *2015 IEEE 15th International Conference on Nanotechnology (IEEE-NANO)* (pp. 278–281). IEEE. <http://doi.org/10.1109/NANO.2015.7388977>
- 8) Balakrishnan, G. (2015). Time-resolved photo and radio-luminescence studies demonstrate the possibility of using InGaN/GaN quantum wells as fast scintillators. *Nanotechnology*, 26(9), <http://doi.org/10.1088/0957-4484/26/9/090501>
- 9) What is nanotechnology. (2016, January 12). Retrieved January 03, 2017, from <http://what-is-nanotechnology.com/2synthetic-titanium-doped-sapphire-Al2O3.htm> 2016 Sep 27.
- 10) SINGLE CRYSTAL SYNTHETIC TITANIUM DOPED SAPPHIRE (Ti:Al<sub>2</sub>O<sub>3</sub>). (2016, January 13). Retrieved February 03, 2017, from <https://www.linkedin.com/pulse/single-crystal-synthetic-titanium-doped-sapphire-nano-technology?trk=mp-reader-card> 2016 Jan 12
- 11) Scheps, R., & Myers, J. F. (1992). Doubly resonant Ti:sapphire laser. *IEEE Photonics Technology Letters*, 4(1), 1–3. <http://doi.org/10.1109/68.124855>
- 12) Sapphire Windows. (2016, January 01). Retrieved September 27, 2016, from [http://www.baselabtools.com/Sapphire-Windows\\_c\\_88.html](http://www.baselabtools.com/Sapphire-Windows_c_88.html)
- 13) Distance sensor VDM70-10-L/87/122/160. (2015, January 01). Retrieved September 27, 2016, from [http://www.pepperl-fuchs.com/global/en/classid\\_53.htm?view=productdetails&prodid=34354](http://www.pepperl-fuchs.com/global/en/classid_53.htm?view=productdetails&prodid=34354)

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