

# Distributed Control System for Crystal Growth

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**Abstract:** Distributed system for control over the crystal growth process is presented. The main advantages of the system are its low cost, ability to recover after power failure, an application of standard ISaGRAF software environment and available low-power PC-Controller. One of the option of the system is an ability to control symmetry and dynamics of the heat filed. This option is the key factor for the progress in growth of nonlinear optical LBO crystal.

## 1 INTRODUCTION

Crystal growth systems with various control loops (rotating, pulling, weighting of the crystal etc.) have been developed for industry since the middle of last century. Now there are a lot of commercially available growth stations with modern control systems (CS). Our approach in elaboration of CS is based on ability to nonuniformly heat the crystallization domain and therefore to apply nonsymmetric stationary or dynamic heat field. This provides additional parameters to control over heat-mass transfer processes which are always have been considered as a key factor in the growth of high quality crystal.

The base of our CS is a PC-controller I-7188 and remote input-output modules of I-7000 series (produced by ICP DAS Company). The use of the modules of I-7000 series provides quite cost-effective reliability. They are not unique. A lot of similar modules are produced by other companies, for instance by Advantech. Also we have designed home-made modules with controllers of crystal rotation and pulling, as well as load-commutator for control of the heat field.

## 2 HARDWARE

Here we consider one example of operating CS for the growth of nonlinear crystal LBO ( $\text{LiB}_3\text{O}_5$ ). Fig. 1 presents the scheme of the growth station which consists of three-zone heating furnace, balance sensor, pulling and rotation drives, contact-meter

and main controller I-7188EG. Temperature control is realized by three-zone Eurotherm regulator through the separated RS-485 bus. For that reason additional serial ports for I-7188EG were added by introducing mezzanine board X511. A main feature of the CS is the presence of the load-commutators which may switch segments of the heating zones through solid state relays according to the program. The feedback signal for thermoregulator is provided by four parallelly connected thermocouples placed around heating zone. This was found to be enough for stable regulation of temperature in the wide range of periods of switching (1 sec - 20 min).

## 3 SOFTWARE

Each growth station with individual IP-address has its own controller with onboard DOS-compatible "Mini OS7". In that way any standard programming language may be used to realize a project. In our case we were concentrated on the logic of crystal growth process, so the I-7188EG controller with built-in ISaGRAF 3.xx system was used. ISaGRAF system implements the following functions: data reading signal preprocessing, realization of control algorithms, communication between modules and HOST computer.

A program loaded in the controller is performed with cycle 0.2 sec. Crystal growth parameters are slow and have a very wide dynamic range. For instance, the growth speed may vary from 0.001 to hundreds of grams/hour.

While a parameter is low-rate changing, the controller specifies some time interval and calculates



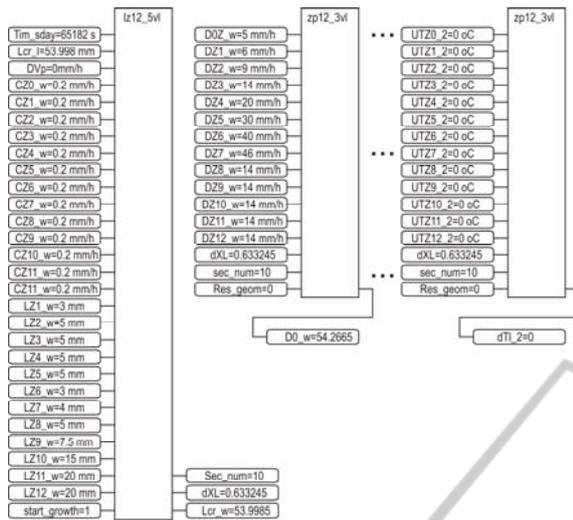


Figure 2: Block diagram of the piecewise-linear crystal geometry master.

#### 4 USE OF THE CONTROL SYSTEM. EXAMPLE OF LBO CRYSTALS GROWTH BY KYROPOULOS METHOD

Kiropoulos method consists in the growth on seed crystal slightly dipped in the melt (or the melt-solution). The growth proceeds towards the melt by a smooth decrease in temperature at small temperature gradients, which makes it possible to grow high quality crystals. This method is widely used for production of large sapphire crystals of any crystallographic orientation, with an extremely low dislocation density of less than  $1000 \text{ cm}^{-2}$ .

LBO single crystal is a well-known material used in nonlinear optics (NLO) for the last 20 years. Due to good operating performance and relatively high nonlinear coefficients LBO crystals are widely used for frequency conversion in the visible and near UV regions. These crystals are of special interest now since they may be used in the laser systems of extremal intensity (<http://www.extreme-light-infrastructure.eu/>). Growth from molybdenum oxide fluxes has brought a considerable progress in LBO growth technology but the crystals with large high quality parts were still unavailable until recent time.

LBO growth furnace has 3 heating zones. The bottom and middle zones are composed of 8 heating elements. Their connection is realized through two separate load commutators governed by the control system. Each one allows to simultaneously switch on any of the heating elements in any sequence and

for any time period (Kokh et al., 2009).

First advantage of such a system is the possibility of intensive but noncontact mixing. It is very important since homogeneity of badly-miscible LBO and the flux seems to be a key factor to large size and high-quality crystal growth. The melt homogenization procedure is realized by prolonged period between switchings (more than 15 minutes). In this case, extremely high radial temperature gradient produces intensified thermogravitational convection. A convective pattern at the surface of the melt (Fig. 3) clearly indicates a direction of the prevalent flow which will be turned relative to the crucible after next switching of the heaters.



Figure 3: Convective flows on the top of the melt-solution during homogenization procedure.

In order to grow larger crystals the direct scale up of the setup faces some serious problems. For example, for large crucibles ( $>100 \text{ mm}$  in diameter) it is difficult to maintain axisymmetric heating and hence to fix a coldest point on the melt surface at the geometric center of the crucible. Otherwise distorted heat field can lead to highly asymmetric crystal growth which results in the formation of defects.

The CS allows to set the corrections to switching time for any group of the heaters. Seeding process is carried out in quazi – stable heat field when period of switching is  $\sim 3 \text{ sec}$ . In that case no temperature oscillations are observed in the melt. Usually our furnaces need delay corrections less than  $0.5 \text{ sec}$  for some heaters to adjust surface temperature distribution to axisymmetric one. This process is controlled by observing the movement of small pieces of LBO ( $\sim 1-2 \text{ mm}$ ) thrown onto the melt surface at a temperature of  $10-15$  degrees higher than crystallization temperature (Kokh et al., 2010). Step-by-step adjusting of time for each pair of heaters is done until a crystal piece stops to move and dissolves right in the crucible center.

Large period of switching may result in the considerable temperature oscillations ( $\sim 10^{\circ}\text{C}$ ) in crystallization region. Until recently such conditions were considered as unfavorable for crystallization process. However our results have proved this opinion to be incorrect (Kokh et al., 2012). The progress of LBO growth under nonstable temperature regime has resulted in the crystals (Fig. 4) suitable for fabrication of world largest 65 mm in diameter optical elements for high energy laser applications (Mennerat et al., 2011).

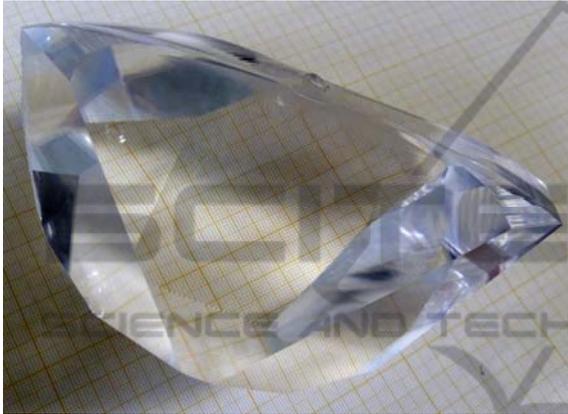


Figure 4: LBO crystal suitable for production of  $\varnothing 65\text{mm}$  nonlinear optical element; weight: 1290 g, dimensions: 149x131x83 mm.

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<http://www.extreme-light-infrastructure.eu/>

## 5 CONCLUSIONS

Elaborated distributed control system provides reliable growth process. One of the options of the system is an ability to control symmetry and dynamics of the heat field. By the example of LBO crystals, this option was shown to contribute in the progress of growth technology.

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