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Open Top Seeding Crystal Growth Control System

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The majority of crystal growth systems require overcoming problems related to process monitoring and continuous electronic control. In order to monitor and control such systems, high quality industrial equipment has to be used, due to its high reliability, requiring minimum long-term maintenance. In particular, reliable temperature controllers, are prevalent components which are devoted to thermal control of high temperature profiles in crystal growth systems. The following paper describes an inexpensive top seeding crystal growth system, using open-source software and low-cost electronics. This system was built in order to control and monitor the entire growth process in a Bridgman method growth system at our laboratories in Ariel University, Israel.

Keywords: crystal growth, seed growth, high temperature thermal control, top seeding method, motion control, Bridgman.

INTRODUCTION

By using inexpensive microcontrollers and electric components, coupled with rapid 3-D printing and wide Open-Source applications, the Crystal Growth community can drastically reduce the time and resources needed to manufacture such crystal growth systems. The use of industrial Single Loop Temperature Controller (SLTC) makes the whole interface with the instrument, via open-source software, both reliable and independent of the manufacturer closed source operation system. The benefits of using SLTC with accessible detailed memory addresses are associated with better supervision over the instruments via open source supervisory and control software. There is a diverse of available information on the internet about open-source supervisory systems based on PLC’s, and several Arduino based SCADA systems [1, 2], however, these systems still lack reliability and errors are still found and reported in libraries or code lines [2]. The supervisory and data storage therefore is implemented using the more reliable open-source platform – the Raspberry-Pi. Crystals possess a high degree of perfection and purity, [3] these crystals play key roles in the modern science and technology whether in their natural, synthetic or engineered form. The main problem of crystal quality control is the thermal boundary conditions [6], Thermal boundary conditions dictate the interface shape between the solid and liquid regions of the material.

Most of the time, a set of variables defines the quality of the crystal, according to its unique desired properties, and it is not always accessible, or hard to predict during the growth process. Compositional homogeneity and reduced chemical stresses were reported by [7], using a rotation of the seed or melt. To control the desired variables in these complicated systems, an accurate motion and temperature control is needed. In this article, we describe an open hardware and software design resulting in a high-performance Top Seeded Solution or Bridgman control system. The resulting system can control crystal growth in the range of room to high temperatures (limited only by refractory materials and heating elements). As proof of concept, we built and characterized the Bridgman method furnace's temperature profile and demonstrated its usefulness by growing aluminium crystals with a melting point at 660^{O}C. The requirements from this growth are a high thermal mass, low heat loss due to conduction and radiation while reaching the temperature of 750 ˚C in separated zones, with T_H, T_C that refer to the hot and cold zones temperatures, respectively. During the growth process, the molten charge is lowered in an ampoule inside the furnace temperature gradient. In the specific implementation of the Bridgman method, a crystalline oriented seed is placed right below the molten charge with its temperature slightly below the melting point of the charge. The crucible with the molten charge slowly translates from the high temperature to the solidification temperature where it obtains a crystalline structure (single crystal) oriented in the

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seeds direction. The upper zone is required to be slightly above the melting point, and the lower zone below melting. The importance of temperature profile and precise control over the process are key features of the crystal growth process.

MATERIALS AND METHODS

Temperature control

All temperatures were measured using a Watlow K24-2-505 special type K thermocouples with an accuracy of 0.4% up to 1096 ^{O}C, and Schneider Electric's Eurotherm 3216 temperature controllers. An industrial temperature controller and low limit of error thermocouple were used to ensure stable and precise control on the growth spot in the furnace. The controllers (Fig.1) switch the power supply using two Fotek SSR-25 DA Solid state relays capable of switching up to 16 [A] of heating element current [4], connected in series to a residual current device to turn off the furnace in case of an alarm or short circuit. The power was taken from the power grid via an UPS. All connections were done via [5] a supervisory unit, using RS232-to-USB converters.

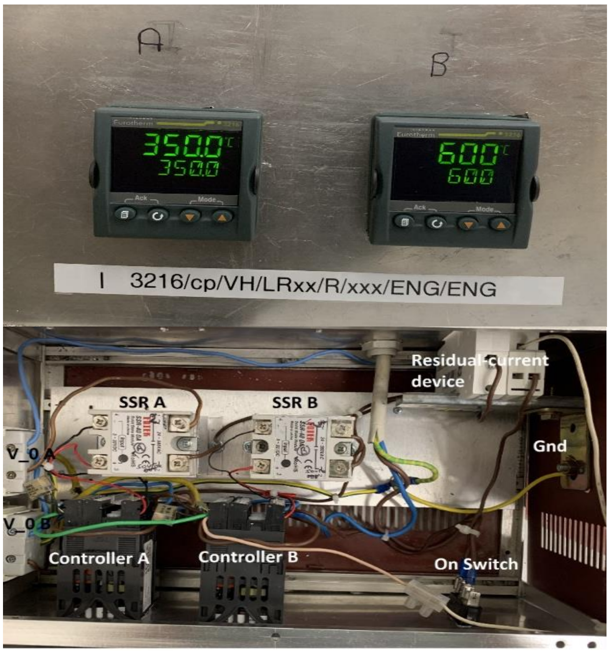

Fig.1. Temperature control box front panel and internal components

Motion control

Temperature control is handled according to the temperature profile of the furnace and stable melting zone temperatures, as the motion of the sample along the furnace is handled using a stepper motor. A NEMA 17 stepper motor is used characterized by 1.8˚ per step. Pulse Width Modulation of the control signal to the motor driver is applied resulting in smooth rotation of the motor and consistent translational or rotational movement to the charge and grown crystal. The load determines the number of steps per revolution as the torque to the crystal decreases with the increase in the number of steps. Speed of growth in the typical range of mm/hour depends on the move of the temperature gradient [6]. The Arduino platform continually moves the motors according to the last instruction (rate) stored in the microcontroller memory. The interaction between the Arduino and the actual movement of the system is done by the Raspberry-pi's Top Seeding software. The key objective of the control algorithm is to move the sample with precise accuracy through the crystallization zone to optimize the growth conditions and prevent undesired defects [3]. The flowchart described in Fig.2 illustrates the control decisions that manage the Arduino software.

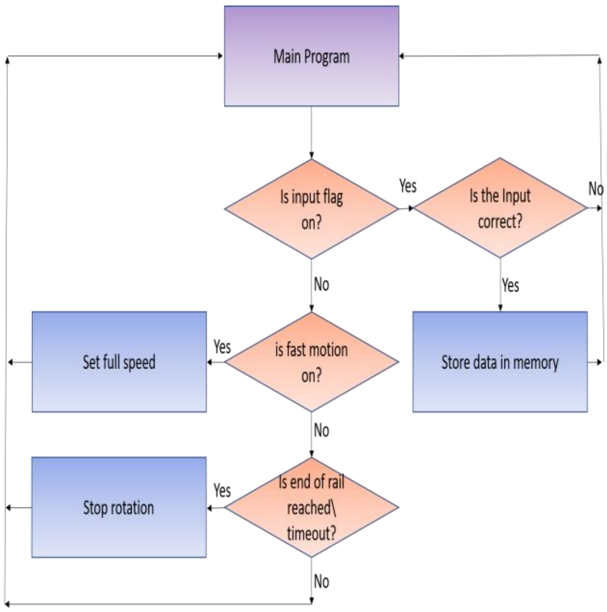

Fig.2. Flowchart of the algorithm to control the motion of the crystal inside the furnace

The Arduino board and the motor driver have been placed in a costume-built box using Open Scad and a 3-D printer is shown in Figs.3 and 4. The control over the growth variables is done semi-automatically as the vertical motion can be controlled manually to move across the rail using a switch.

An Arduino is using embedded RS232 to USB converter and is connected to the Raspberry-pi using a USB cable. A design choice was made, in which each motor has a different Arduino as a controller. This increases the robustness and reliability of the growth system, simplifying on site repairs should one of the controllers fail. The cost of this redundancy is negligible.

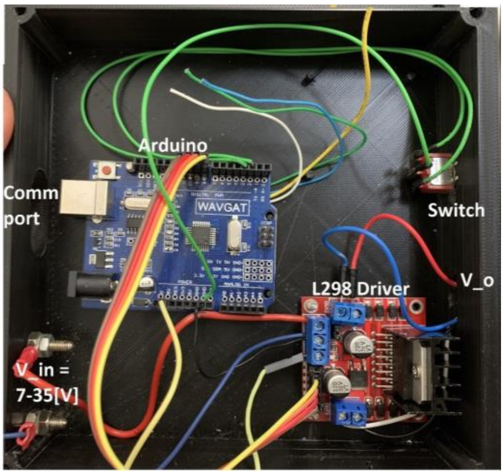

Fig.3. Arduino box and schematics drawn with Fritzing [8]

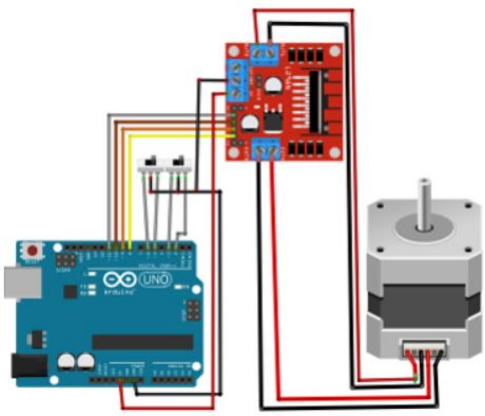

Fig..3 Electronic wiring of the Arduino Uno to L298 driver and switches used to control the stepper motor motion. A power supply of at least 7-35[V DC] and 0.8-1.5[A] is needed

Bridgeman furnace assembly

High thermal mass can be achieved using thick insolation around the heating element, and sufficient power per square centimeter of the heating element. The heating element was prepared from 2.5cm O.D ceramic tube made by extrusion to ensure even wall thickness and high tube concentricity. These features prevent thermal strains and ensure uniform isolation profile [9]. The heating coil was wrapped around a ceramic tube in different spaces to set the furnace with its unique temperature profiles of hot and cold zones. The furnace was designed with a total heating power of 1130 [W] separated for the hotter zone and the cooler zone. The two zones were connected in parallel to the power supply. The difference between these zones was achieved using different length of heating wires. The hotter zone benefits longer wire and therefore higher power per surface area, making it favourite for higher temperatures. The resulting resistances are R_C = 120 [Ω], R_C = 100 [Ω], with R_H, R_C referring to the hot and cold zones, respectively. The heating wires were covered with an alumina isolating blanket. Another ceramic tube was attached in parallel to the main tube for the measurements of the temperature along the furnace. The heating wires were connected to the power supply fitting box (Fig.5).

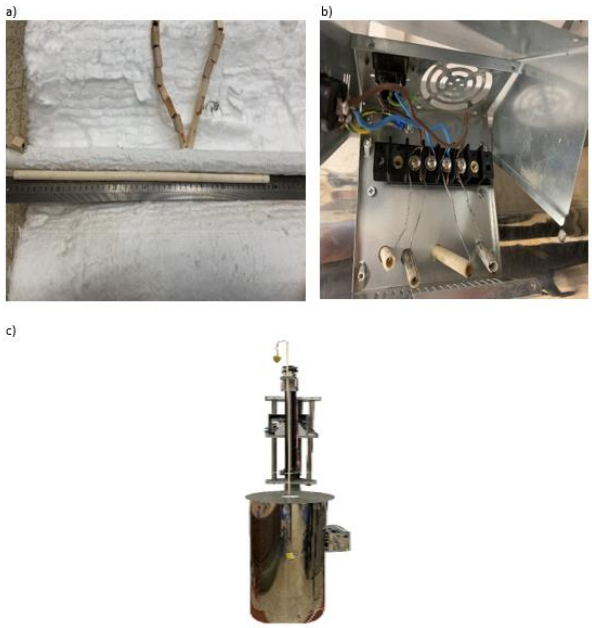

Fig.4. Assembly of Bridgman method furnace. a) Heating wired wrapped around 2.5 [mm] X 70 [cm] ceramic tube. b) The heating wires connection to the power box. c) The fully assembled system, the furnace at the bottom and translation stage above it

The motion module was attached to the entrance of the furnace with thermocouple connecting the motor's rail for temperature measurements along with the furnace. A meter was attached next to the rail to indicate the motor movement.

Open-source user interface

An open-source software called Top Seeding, based on Python programming language [10] [11], Remote terminal Unit (RTU) Modbus communication protocol [12], and matplotlib for visualization was designed to gather data from the process, generate commands and send these commands to the process. To communicate with the Raspberry-pi, SLTCs, and Arduino, an asynchronous serial communication was set via RS232 connectors for each RTU with the ability to convert signals obtained from the sensors to digital data and sending them to the supervisory system. According to [13], using asynchronous communication "silent period" of 3.5 bytes in the message limits the maximal baud rate optional, using RTU Modbus protocol (Fig.6). The User Interface was designed using Object Oriented Programming (OOP) architecture. Number of libraries were used in this software and they are all listed in Tab.1. Following the OOP architecture, the devices features, connected to the Top Seeding software, were defined as specific modules for each instrument in the system and described the access to the instrument's memory addresses.

Table 1. List of libraries and fields corresponding to the Top Seeding software

Name of Python Library	Field of attributes
Tkinter	GUI
OS	Operation system
Matplotlib	Graphics
Minimalmodbus	Modbus protocol
Pyserial	Serial communication

The object oriented nature of the software, gives the desired agility adding the flexibility of inverting objects and applying the programming paradigm to a variety of instruments and future additions.

Fig.5. The Raspberry-Pi is connected to instruments via USB ports and stored in a 3-D printed box (designed using Open-Scad)

Cautiousion was taken by blocking the operators ability of writing to non-volatile flash memory. Although the system cannot change configuration settings on the PLC's memory and initial conditions on motion control modules, this setting has been considered preferable to prevent memory failures.

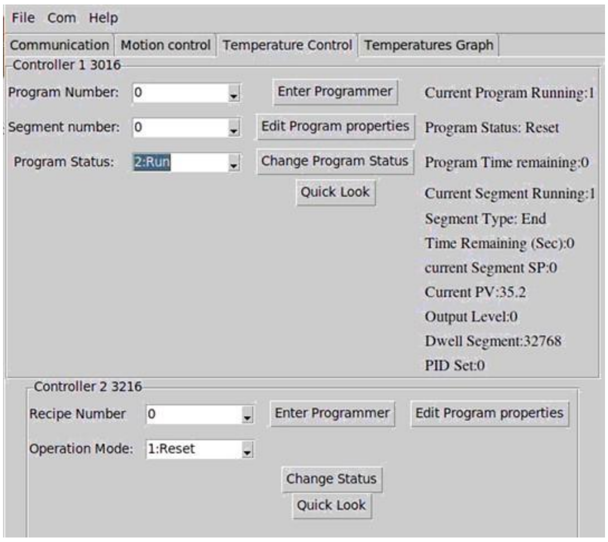

Fig.6. Top seeding software, Temperature control tab communicates with Eurotherm controllers using Modbus protocol

RESULTS

The furnace was heated up to $T_H = 600$ [°C], $T_L = 350$[°C], motion control was set to gradually rotate downward the furnace at a constant velocity of two revolutions per minute i.e. 4 mm/min. At the end of the translation stage, a microswitch was set to stop the motor and the profile was tested again while the TC moving upwards the furnace. To reflect the effective operation of the approach, Fig.7 shows the screen of Top Seeding software temperature control tab under working conditions. The variables from the Arduino and SLTC are displayed in real-time for the user, separated to different informative tabs demonstrating the current process. In many automated infrastructures, the system also preforms active control tasks. In the present case, the growth time of a typical crystal makes it more reasonable to handle the process semi-automatically. It means that the control algorithm carried out by the motion control module and SLTC, can be intervened with at any time, using the Top Seeding software. As can be observed in Fig.7, a graphic chart illustrates the measured temperature along the furnace axis. The tab named

Temperature Control on the Top Seeding software GUI, displays its numeric value together with other variables related to the SLTC; including the type of operation, target value, output level etc. Additionally, an output logger records the entire process corresponding to UTC date and time for desired interval of seconds on the Raspberry-pi memory.

The resulting average temperature profile is shown in Fig.8. This temperature profile indicates that the growth interface should be at approximately at a depth of 35 cm from the upper opening of the furnace, yet a more detailed profile of the hottest spot in the furnace had to be taken in order to place the crystal seed exactly in the right position, where it can benefit the best growth conditions. The temperature profile indicates that placing the seed below 35cm, lowering it at a constant velocity will commence crystal growth. Once the entire ampoule passes the melting point, the higher zone is slowly cooled to the same temperature of the lower zone and annealing take place at a constant temperature of 350 ˚C.

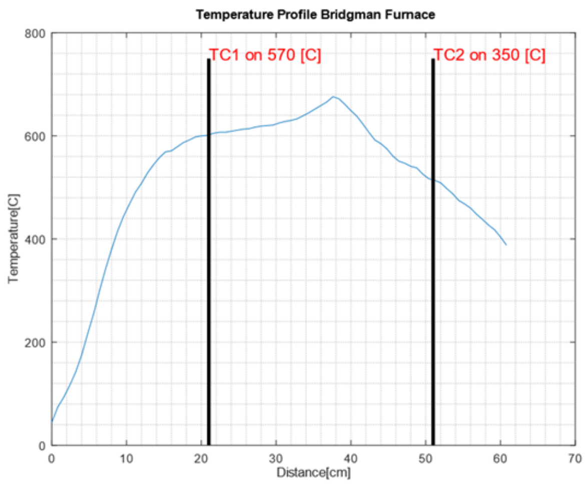

Fig.7. Measured temperature profile along the furnace axis

The temperature measured outside the ceramic tube, indicated on the efficiency of the measurements and the SLTC. It is indicated by the vertical lines on the chart. Despite the obtained results, some disadvantages characterize this system in comparison to other industrial systems. Open hardware-based setups can be unreliable due to the use of jumper wires that might disconnect. Raspberry-Pi does not meet the required system safety due to its multitasking nature and thus results long delays depending on the CPU load. This delay is unacceptable in case of high power and temperatures. Therefore, alarm commands are all handled by the industrial SLTP. Although full open hardware system is not considered suitable for hard real application, this system provides a proper behaviour on the so-called semi-automatic approach.

CONCLUSIONS

Open hardware and software became a viable option for lab control systems. They cost less than industrial devices and are supported with a wide range of literature. This article has presented a system for half open-source electronics hardware (Arduino and SLTC) and open-source software (Top Seeding) for crystal growth systems based on the Top Seeding method. The open-source device is responsible for sensing, control, and data acquisition tasks. The proposed technique represents the versatile open-source approach to the crystal growth control systems. A Bridgman furnace was built and characterized for use as a benchmark to analyse and validate the proposed solution by means of temperature profile test under real operating conditions. The experimental results reflect on the effectiveness of the system display, data exchange, and process logging.

ACKNOWLEDGEMENTS

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