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Development of a multipurpose heat treatment apparatus favorable for the growth of solution-based large-size crystal

AVIJIT DAS*

Saha Institute of Nuclear Physics, A CI of Homi Bhabha National Institute, Bidhannagar, Kolkata 700064, West Bengal, India

Abstract. An inexpensive temperature-controlled apparatus has been developed with the intention to use it during the growth of any solution-based crystal. It consists of a metallic cell and an application-specific, stand-alone, and programmable electronic controller. That can purposely deliver unequal heating power independently to its three individual heating channels. The system was designed with the aid of an Arduino-Uno board. It was indigenously built within the laboratory with limited in-campus facilities and using locally available standard electronic modules and components. It may have diverse applications for controllable synthesis processes in scientific laboratories and its structural as well as electronic design can be modified easily according to any specific user requirement.

Keywords: heating apparatus, perovskite crystal, crystallization, Arduino, temperature Controller.

1. Introduction

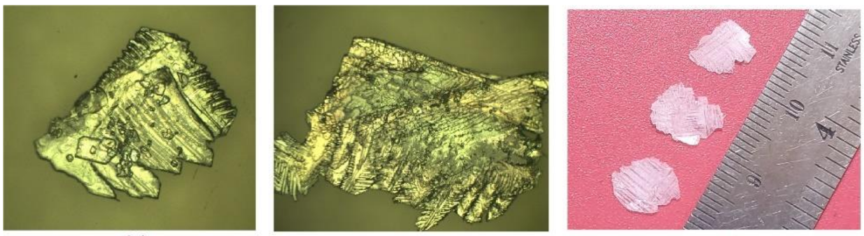
This article describes all technical details about the design of a proportional temperature-controlled apparatus focusing on its application, especially for growing large-size, low-temperature (less than 150˚C) solution-based crystals. In recent progress, 2D perovskite materials are showing remarkable properties such as long carrier lifetime, high photoluminescence quantum yield, and low defect; all these properties increase the potential for optoelectronic application. Researchers in our institute have achieved one of their goals to grow large-size (a few mm in measurement) perovskite crystals very recently using this apparatus. Though developed for this dedicated purpose, with few minor modifications this apparatus can also be deployed for other low-temperature heat treatment applications in the laboratory. Specifically, where different temperature level at different confined

*Correspondence address: avijit.das@saha.ac.in

zone inside a heating chamber has to be maintained. It is able to control desired heating power in its separate heating channels in the closed loop method, depending on more than one sensor mounted at multiple positions. Once triggered manually, it can automatically maintain a predetermined and stable heated region, which may be fixed or variable inside a metal container without any further human intervention. This device works according to a pre-programmed flow chart for several hours or days with a periodical data recording facility in a micro-SD card. Another worth mentioning point is its low development cost. This self-designed electronic controller has been built indigenously with parts and components purchase cost of around 5500 INR which is equivalent to 67 USD.

2. Literature survey and generation of the idea

To prepare perovskite crystal (Fig. 1), the precipitation method has been used by cooling of Halide Acid based (HX, where, X = Cl, Br, I) precursor solution as mentioned in paragraph 2.1.1 of the article by Jiayu Di et al.[1] This acid solution was heated to a certain temperature (<150˚C) and slowly cooled down to room temperature. In such a way the size of the single crystals reached the millimeter level (Fig. 2A of Ref. 1). This slow cooling process is very important for crystal growth. Poglitsch and Weber used such a cooling method of an aqueous solution to grow 3D perovskite single crystals.[2] They gradually decreased the temperature of an HX-based perovskite precursor solution from a temperature of around 100 °C to room temperature to obtain 2D or 3D perovskite single crystals. Bakr's group prepared single crystals by cooling 90 °C precursor solutions to room temperature at a rate of 1 °C/h as mentioned in the 'Experimental Methods' in their article.[3] Kanatzidis's group also prepared millimeter-sized plate-like crystals, but following the natural cooling method, which has been indicated in the 'Experimental Section' of their paper.[4]



(a) (b) (c)

Fig. 1. (a) and (b) Optical microscope image of single perovskite flex with 10x zoom. (c) Actual photograph of some of the large-size flexes with scale.

The cooling rate is an important factor for the growth of large-size, homologous 2D perovskite single crystals.[1] Rapid cooling leads to multiple nucleation sites with the formation of a large number of small crystals.[1] Therefore, strict control of the cooling rate is important for obtaining high-quality, large-size, and single crystals.

A literature survey shows that in most of the cases, no apparatus type or process has been mentioned clearly, either for annealing at some fixed temperature such as at 75 °C [5] or, at 100 °C [6] or for a slow cool-down method.[3] In this paper, attention was mostly focused on designing a dedicated apparatus along with its control electronics for very precise management of solution temperature. The total cool-down period has been kept flexible and easily editable in embedded C code. It has three individual and separately controllable heating channels with two heater cartridges connected to each channel via two individual power MOSFETs.
A heating current regulator circuit designed for this purpose is versatile in nature. That is an advantage of using this design concept for constructing any other apparatus used for the purpose of crystal growth or as any other investigating tool, commonly used in research laboratories that require temperature-controlled activities. It can also provide different heating power to different parts of any metallic body through its individually controlled separate channels. It is one of the worth mentioning advantages that may be used in a much wider perspective.

3. Materials and methods

3.1. The concept of a metallic container

To achieve high-quality, large-size single crystals, a very slow and controlled cooling rate is very essential. [2,3] A gradual decrease of the halide acid-based solution was done from around 100 °C to room temperature with a constant rate of approximately 1 °C per 15-minute time interval. To provide a uniform heating environment to a 50 ml glass beaker, an aluminium solid cylinder (Fig. 2.a) was fabricated at the workshop of our institute in Kolkata to accommodate the beaker inside it. The mechanical drawing with dimensions is shown in Fig. 2.b. Vertical drills of proper diameter were done on the periphery and at the bottom of the cylinder to insert six heater cartridges and a PT-100 stainless steel probe. The internal diameter of the aluminium container was decided in such a manner so that a 50 ml glass beaker, mostly available in the local market can fit inside easily with 3-4 mm free space around its periphery (Fig. 2.c). I have used rotary vane vacuum pump oil to fill up this gap. The oil, when heated up; in turn, conducts heat to the liquid (approx. 20-30ml) inside the beaker. It serves the purpose of uniform heat distribution to the solution kept inside the beaker. Rotary Vane vacuum pump oil has a very high boiling point and does not create any such vapour at 100 °C that can contaminate the solution. The aluminium container was insulated with a thick layer of foam around it and a small piece of glass on the top (with a little gap to release the acid fume) to avoid undesirable heat loss and thus provide uniform thermal stability to the liquid sample under test. The slow cool-down process was performed inside a chemical hood with a running exhaust fan.

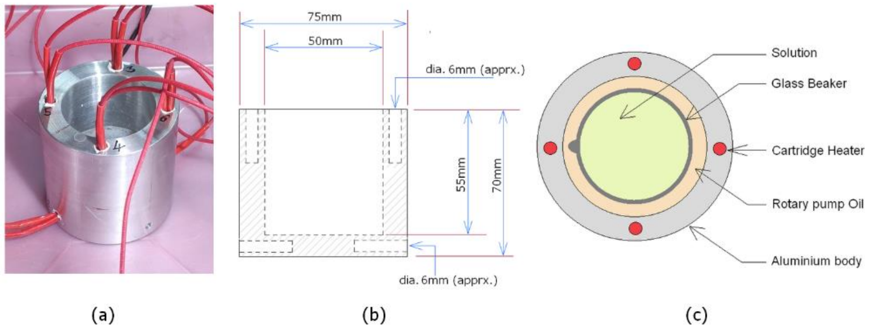


Fig. 2. (a) Actual view (without the insulating jacket) with inserted heater cartridges.
(b) Mechanical drawing of the Aluminium container. (*Not according to scale*).
(c) Schematic top view of the metallic container. (*Not according to scale*)

3.2. Electronics Modules and Components used

Six numbers of 12v 40W Ceramic Cartridge Heaters with one meter of silicone-insulated high-temperature wires were used to heat up the aluminium container. Such heaters consist of a cylindrical stainless steel tube of length 20mm and diameter of 6 mm.

A couple of PT100-S waterproof 30mm stainless steel pole probe (RTD) temperature sensors (henceforth to be mentioned as Sensor-1 and 2) of 4mm diameter are liquid immersible probes, suitable for precision air or liquid temperature measurements. The cable material is PTFE Silver plated copper. It is capable to measure temperature in the range of -200 to 420 deg C. RTD sensors contain a resistor that changes resistance value as its temperature changes, basically a kind of thermistor. In this sensor, the resistor is actually a small strip of Platinum with a resistance of 100 ohms at 0 ºC. PT type of RTD is much more stable and precise compared to thermocouples and is very popular for its accuracy, repeatability, and stability.

A single output enclosed SMPS power supply of 12V DC, 8.5A, 102 Watt (Model: LM100-20B12, Make: Mornsun, China) was used to supply heating current to six numbers Cartridge Heaters.

For temperature sensing, two MAX31865 RTD Platinum Resistance Temperature Detector (RTD) Modules were used to sense PT-100 output. The MAX31865 is an easy-to-use resistance-to-digital converter (15 bits) optimized for platinum resistance temperature detectors (RTD). The high-precision delta-sigma ADC converts the ratio of the RTD resistor to the reference resistor into a 15-bit digital output.

A Micro SD Card reader/writer module was used for transferring data to and from a standard SD card. This module has an SPI interface (with MISO, MOSI, SCK, CS pins) which is compatible with any Micro SD card and it uses 5V or 3.3V power supply which is compatible with Arduino UNO board that I have used.

A blue backlight high contrast LCD 1602 display had been used with 16×2 white-colored characters (of 5x8 = 40 dots per character). An Inter-Integrated Circuit (I2C) adapter that was directly soldered onto the pins of the display board, facilitates occupying only two pins of Arduino digital port pins rather than using eleven pins in case of parallel data transfer mode.

A UNO R3 CH340G ATmega328p development board has been employed as the main control device that is the low-cost version of the popular Arduino UNO R3. It is assembled with the CH340 USB-to-Serial converter chip. It has 14 digital I/O pins available, among which there are six Pulse Width Modulated (PWM) outputs that can be used to generate variable analogue output voltage equivalent to an 8-bit digital value. I have used three PWM pins (3, 5, and 6) for controlling power to six cartridge heaters. Pin-3 generates PWM waveform of 490 Hz whereas 5 and 6 generate a 980 Hz waveform. Though, this frequency difference does not affect the heating power at its final output. The schematic diagram of the control circuit has been shown in Fig. 3.

MOSFET IRF540 was selected as the main power regulating component. It can deliver more than twenty-ampere drain current (with proper heat sink) to the load even at quite heated up condition.

A high-speed switching transistor 2N2222A has been used associated with every MOSFET that provides 12v gate voltage at the same frequency as the 5v pulses generated from a particular PWM output pin of the Arduino board.

3.3. Design of Electronic Circuit and Arduino C Programme

The heating power regulation is done by continuous comparison of temperature between a target set point (SP) and the current temperature (that is called process value or PV) of the metallic container. With the feedback data which is the temperature of the metal body sensed by sensor-1, it constantly compares the difference (also called error value) between SP and PV. The feedback signal has been taken from sensor-1 mounted inside the metal body for quicker response to the corrective action. Sensor-2 generally responds late as the oil temperature variation takes comparatively more time to change.

With the variation of the width of pulse width modulated (PWM) waveform of heating current (that may be referred to as the control variable), it increases or decreases the duty cycle to minimize the error value. The average output voltage of a PWM waveform depends upon its duty cycle. In practice, to fulfill the basic need of our application, users have decided the decrease the set point at every 15 minutes of intervals. Though the data collection has been made at every 15 minutes, we have noticed on the LCD screen, that the PV is following the SP even at a very short span without any significant overshoot or undershoot. Thus a coherent control of the temperature was observed almost throughout the range and the difference between the SP and the PV always lies within ±0.5 ºC. The manipulated variable which is the heating power is adjusted according to proportional control to the difference (error value) between the SP and PV. As the difference decreases, the circuit takes corrective action by reducing the heating power to keep the PV close to the SP as much as possible.

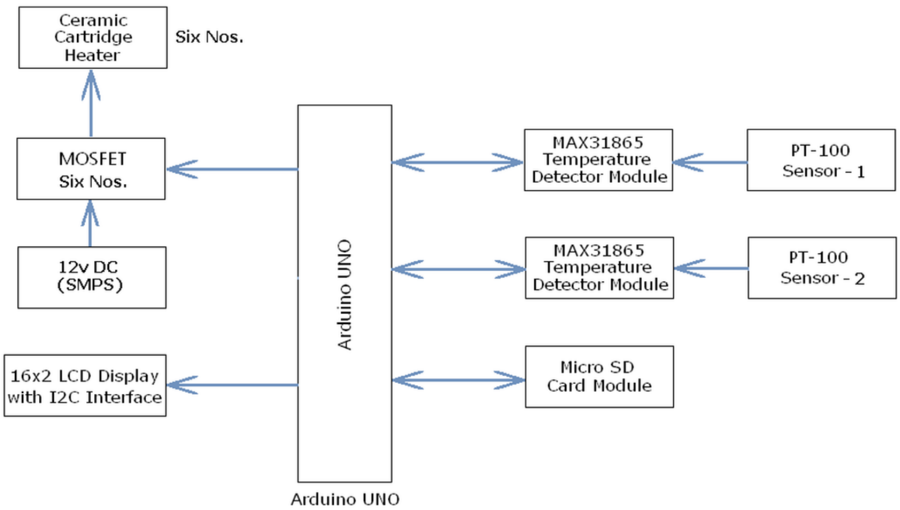


Fig. 3. Block diagram of the control electronics with modules and components.

A UNO R3 CH340G ATmega328p development board has been used as the main controller. Among its six PWM output pins, three (pin-3, 5, and 6) have been used to generate PWM pulse trains to the MOSFET gates. Each of these pins is connected to a pair of MOSFET gates via two 2N2222A high-speed transistors (Fig. 4 & 5). ATmega328p generates digital pulses of height 5v while the MOSFET (IRF540) needs more than 5v at its gate to operate the device with sufficient drain current. A transistor 2N2222A serves this purpose. While connected to a +12v supply, this transistor provides 0 to 11.5v switching pulses at the gate of the MOSFET.

The rectangular PWM wave of varying duty cycles switches the MOSFET on and off, and accordingly, regulates the current flow through each heater cartridge. Thus a variable duty cycle, in turn, controls the output power smoothly.

PT-100 Platinum-based sensor, that I have used, is like a variable resistor, whose resistance varies according to the environment temperature. At 100 °C it shows a resistivity of 138.5 Ω and at 0 °C it is 100 Ω. An electronic module board consists of an IC MAX31865 used here that is compatible with 2, 3, and 4-wire RTD sensor connections.[7] It supports SPI-compatible interface system (SPI modes 1 & 3) with Serial Data In (SDI), Serial Data Out (SDO), Clock (CLK), and Chip Select (CS) lines to communicate with any Microcontroller board, such as Arduino UNO was used. Adafruit Industries published a free-to-use open-source library repository available at github.com website for PT100/P1000 RTD Sensors using IC MAX31865, which I have downloaded and used as a header file in our Arduino code.[8]

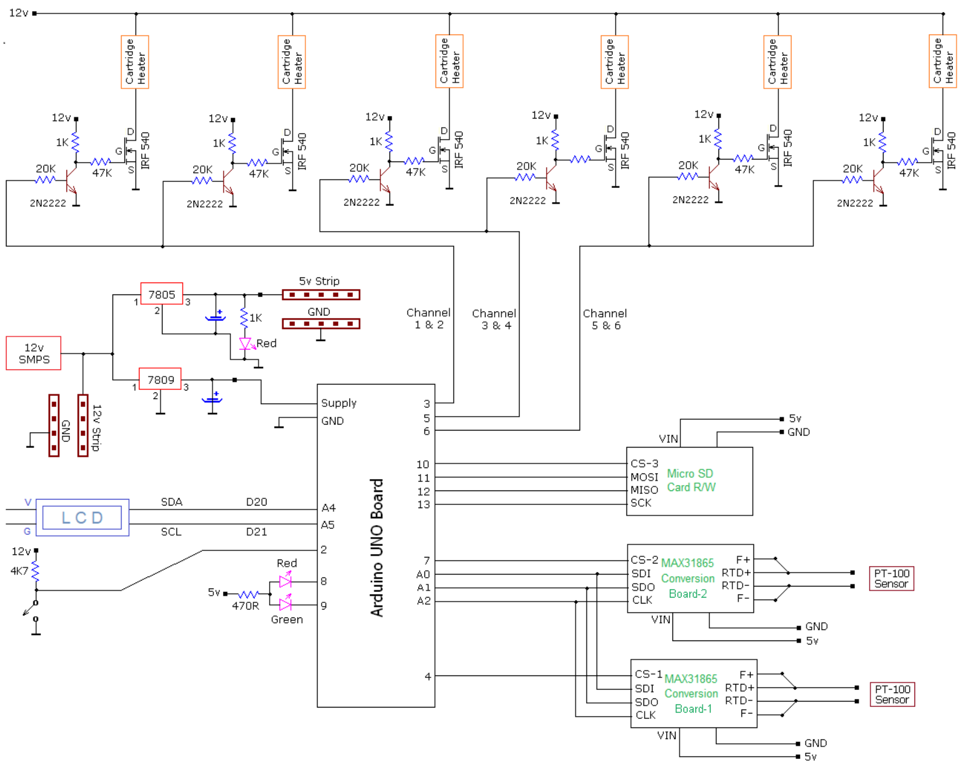


Fig. 4. Circuit diagram of the electronic control board.

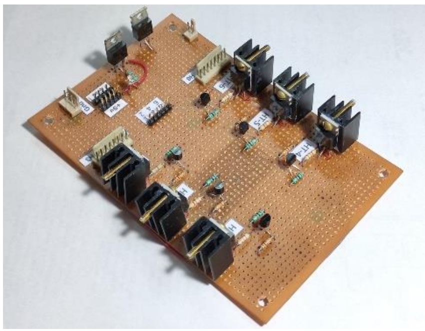


Fig. 5. Circuit board with MOSFETs and Transistors assembled.

The following line at the beginning of our C code in Arduino IDE (version 1.8.9) includes the Adafruit MAX31865 library file from the Arduino library manager.

```
#include <Adafruit_MAX31865.h>
```

Arduino board pins A0, A1, and A2 are connected in parallel to SDI, SDO and CLK pins of two MAX31865 modules. To select a specific board at a time, pin-4 and pin-7 were used for sensor-1 and sensor-2 module boards (Fig. 4).

Creation and initialization of two MAX31865 objects for board 1 and 2.

```
Adafruit_MAX31865   brd1 = Adafruit_MAX31865(4, A0, A1, A2);
Adafruit_MAX31865   brd2 = Adafruit_MAX31865(7, A0, A1, A2);

brd1.begin( MAX31865_2WIRE );
brd2.begin( MAX31865_2WIRE );
```

The following lines give us the sensed temperature with the help of Adafruit MAX31865 library files.

```
temp1 =  brd1.temperature(R_nom, R_ref);
temp2 =  brd2.temperature(R_nom, R_ref);
```

Here, R_ref is the value of the reference resistance on board that is 430.0, while using PT-100 sensor. And R_nom is the nominal resistance of the PT-100 at 0 °C is 100.0 Ω.

The Micro SD card was used to store the data inside its memory and its pins are compatible with the Arduino UNO board. It employs a 4-wire synchronous serial communication interface system called Serial Peripheral Interface (SPI) for short-distance communication. It communicates with the Arduino board in full duplex mode using master-slave architecture. The inclusion of two header files SPI.h and SD.h from Arduino library manager simplified the programming job quite a lot.

The LCD 1602 display is used to monitor the Set Point temperature (SP) assigned by the embedded code, sensed temperature by PT-100 sensors 1 and 2, elapsed time in minutes, and an 8-bit value of current PWM output 1 and 2 (value of PWM-3 is not shown only due to scarcity of free space). It communicates with the Arduino board by I2C serial bus. I2C is a synchronous, multi-target (master/slave) serial communication bus to interchange data with onboard peripheral devices. It uses two bidirectional lines, called SDA (Serial Data Line) and SCL (Serial Clock Line). Both are connected to +5v DC supply via two 1K-ohm pulled-up resistors. In our project, analogue input pins A4 and A5 have been configured to work as SDA and SCL lines. An interface address of hexadecimal 0x27 in general remains assigned by the manufacturer to such display modules. The header file LiquidCrystal_I2C.h downloaded from github.com website was included at the beginning of the C code to take advantage of this library file.[9]

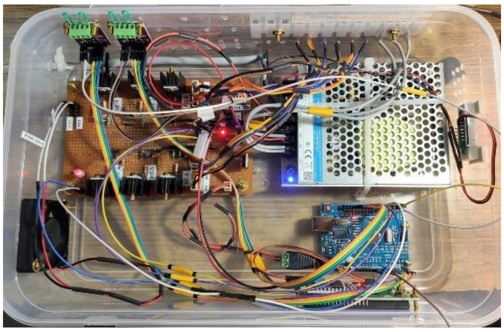


Fig. 6. Complete view of the electronics controller
with all components and modules within a plastic enclosure.

3.4. Method to use the setup

Firstly, in our application power was increased to bring the solution temperature up to around 104 °C in three steps with 10 sec. delay in between each. The temperature was increased from room temperature to 60 °C in step 1, 61 to 80 °C in step 2, and 81 to 104 °C in step 3. With the help of 'if-else' statement, I was able to keep smooth control of the linear increase of the temperature. Different 8-bit PWM values were used in every step. PWM values along with periodic heating time were determined by trial method with two/three dummy test runs to finalize the most optimum values.

Some amount of process time is consumed during accessing PT-100 interface module board for its analogue to digital conversion. It is mentioned as 'Temperature Conversion Time' in the user's manual and this delay is around 52-62 m-Sec. Apart from this, some more time delays occurred during exchanging commands and data with the Arduino board.

Sensor-1 is inserted inside the bottom part of the aluminium container body, which at the position of thermal equilibrium, senses the temperature that is being used for controlling the power to the heating elements in a closed-loop control system.

3.5. A brief explanation of the Control Logic

The control logic applied in the embedded C code of the Atmel Microcontroller used in the Arduino board is explained concisely with a program flow chart shown in Fig. 7.

During the cool-down process, the controller tries to decrease the temperature (sensed by Sensor-1) by initially reducing the power to zero percent. In every monitoring cycle, it continuously compares the error value between the SP and PV, with the intention to keep the offset always between ±0.5 °C. To maintain this value, it switches the power 'on' as necessary for a short period and then again switches it 'off'. The amount of power applied and its application time is proportional to the difference between SP and PV.

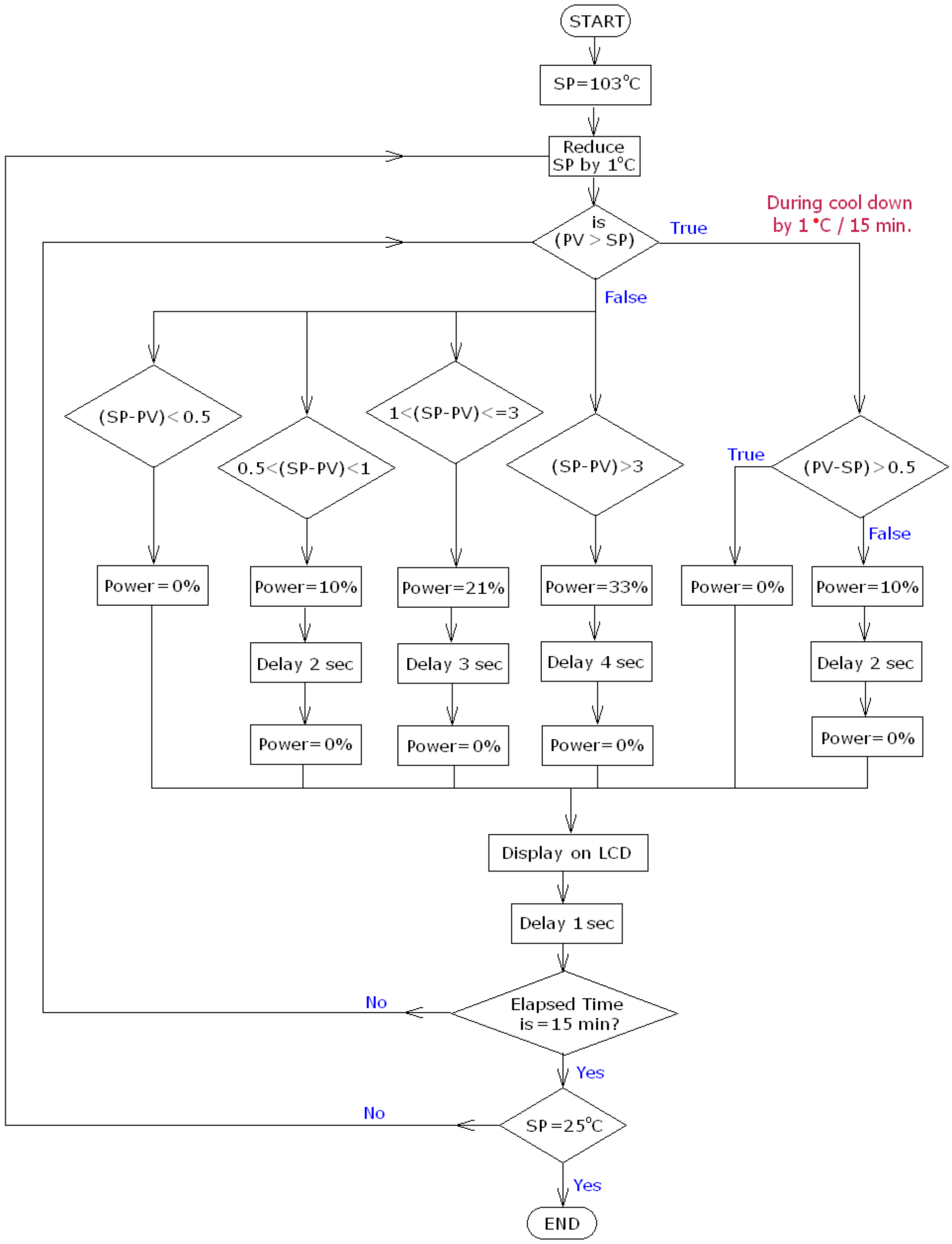


Fig. 7. Logic flow chart of the embedded C code for programming the AVR Microcontroller flash memory. It has been designed especially for cooling down process only with each step as 1°C. Heating up process logic chart is not shown here.

An example of the C code used to configure the three numbers of PWM pins of the UNO board at the very initial stage. The 8-bit value in decimal is 255 which produces 100% duty cycle at each output pin. The Transistor 2N2222 remains in the fully saturated stage and its collector connected to the MOSFET gate delivers zero voltage which keeps the MOSFET in 'off' state.

```
analogWrite(ht_1_2, 255);          // PWM o/p Pin-3
analogWrite(ht_3_4, 255);          // PWM o/p Pin-5
analogWrite(ht_5_6, 255);          // PWM o/p Pin-6
```

Table 1 illustrates the relation of Arduino board PWM values with the power delivered to the cartridge heaters. Though I have the freedom to maintain different power to each of the three channels, but for simplicity, I kept the 8-bit PWM values the same for every channel and it was observed that it had demonstrated a quite satisfactory result throughout the ramp-down range.

Every second used to maintain delay in the C code is counted and accumulated within a code variable and compared with a pre-determined cycle delay time at every inner program loop. After the completion of a loop of 15 min., it decreases the set point by 1 °C. The logic has been built to ramp down the temperature up to 25 °C. Though the set point is forced to go down to 25 °C, the process value actually follows a flat region at the bottom of the curve, without any further decrease when it reaches the present room temperature.

Table 1. Different 8-bit values of Pulse Width Modulated (PWM) output and corresponding average voltage measured across the cartridge heater and current through it while connected to a 12v DC power supply.

8-bit PWM value on Arduino Pins 3 or 5 or 6	DC voltage measured across the heater (volt)	DC current measured through the heater (amp)
255	0	0
220	2.79	0.74
200	3.9	1.04
180	4.9	1.3
160	5.84	1.55
140	6.8	1.81
120	7.75	2.06
100	8.7	2.31
80	9.65	2.56
60	10.63	2.82
40	11.7	3.11
20	11.93	3.16
10	11.94	3.16
0	11.97	3.17

During the period of the actual sample preparation stage, Hydro Bromic Acid was used for crystal precipitation that is corrosive in nature. Hence it is not advisable to insert the stainless steel encapsulated sensor directly inside the acid solution during the actual cool-down process. I have adopted an indirect temperature sense method of the HBr solution by inserting another PT-100 sensor (henceforth referred to as Sensor-2) placed inside the rotary pump oil surrounding the glass beaker (Fig. 8.a).
But this indirect method of measuring the temperature of the acid solution also provides only a tentative idea about the solution temperature. To minimize the ambiguity, I performed another dummy test by filling the glass beaker with the same rotary pump oil, inserting the second PT-100 (Sensor-2) inside it, and cooling down the heater temperature (read by Sensor-1) from around 102 °C to room temperature at the rate of 1°C/15 minutes (Fig. 9.a).
In both cases, temperature values (with a precision of two digits after the decimal point) were monitored at regular intervals which were recorded on the Micro-SD card memory. And with the help of this data, two graphs had been prepared that show the variation of the measured temperature, plotted against the elapsed time (Fig. 8.b and 9.b).

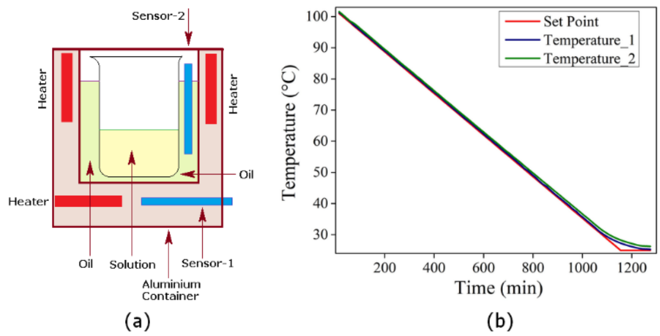


Fig. 8. (a) Sensor-2 is immersed in the oil and placed between the aluminium container and the glass beaker. That would be its position during the actual process of solution cool down. (b) Graph plotted from the data file saved in the SD card.

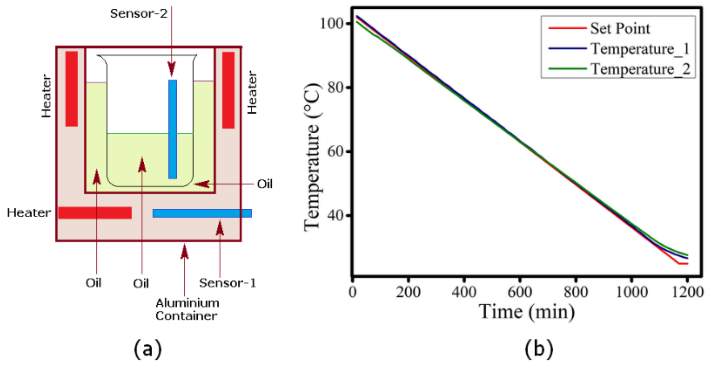


Fig. 9. (a) Sensor-2 inserted inside the oil of the glass beaker. That would give us an idea of solution temperature as the sensor cannot be dipped inside the acid solution during the actual process to avoid corrosion. (b) Graph plotted from the data file saved in the SD card.

4. Discussion on Merits and Demerits

4.1. Advantages compared to a commercial PID controller

Commercial PID temperature controllers generally possess one sensor input and one or two output relays or SSDs in their low-cost variant. Hence, such a controller does not have multiple and individual channel control options. Whereas, in our design, the controller is capable to monitor temperature from several sensors mounted at different locations and can individually control heating power in three numbers of channels with Arduino UNO and twelve numbers of channels, in case an Arduino Mega board is being used. Once the most optimum percentage of power is decided depending upon the ramp rate and total process time, it can be used afterward as a portable, stand-alone device without further connecting to a computer.

MAX31865 boards are interfaced with the Arduino board through I2C protocol. A particular PT-100 sensor board selection (called Chip Selection pin CS) was done by separate digital port pins. If any such future work requires more numbers of sensors to attach, the same number of individual CS pins on an Arduino board has to make free for use. Otherwise, a decoder IC like 74HC4514 serves the same purpose that can select any one of sixteen MAX31865 boards (maximum) using only four digital output pins of an Arduino board.

4.2. Limitations and further scope of improvement

Undoubtedly, a commercially available temperature controller that works with PID algorithm, may enhance the feature of optimal control function with its in-built autotune facility. It can optimize the control parameters in a single shot while applied to a system for the first time. But our controller needs two or three trial runs to perform at the initial stage while connected to a new heating chamber or cell. Developers, who are desirous to use such PID algorithm with Arduino board for further improvement, may include it in their code but that will surely increase the complexity with enlarged coding volume.

Arduino UNO has fourteen digital input/output pins available for use among which six can be configured to generate pulse width modulated output. Unfortunately, due to the shortage of port pins, I had to use three of these PWM output pins (9, 10, and 11) for some other purposes. Instead of using UNO, an Arduino MEGA board may also be used that has twelve PWM output pins.

The data was collected for this particular application at an interval of 15 minutes and evidence of precise control was recorded as the data file (Fig. 8.b and 9.b). But for any other application, where a more rapid ramp-up or down rate is required, it may demonstrate a little wider deviation due to insufficient soak time allowed for the temperature to become stable.

Currently, data is recorded in a Micro-SD card at a theoretically calculated time interval of the innermost loops (Fig. 6). But this cycle duration is determined partially on assumption considering the time delay added due to the analog-to-digital conversion and signal exchange of the MAX31865 module. In practice, it was observed that the elapsed time of 15 minutes calculated in the C code when counted in reality by a stopwatch has become 15 min. and 20 sec. In our application, precision calculation of the cycle duration was not very essential. Hence, I did not take any steps to improve it. But in any other upgraded appliance, the addition of a battery-powered Real Time Clock (RTC) module can avoid this limitation.

5. Conclusions

This circuit has quite enough possibility for future expansion to increase its diversity in different fields of scientific applications. Designers will have more freedom to use increased numbers of separate heating channels with multiple sensor inputs. Individual heating channels can be used for maintaining different heating zones at different parts of a cell - that may be fixed or variable temperatures, with ramp-up or ramp-down facilities. By using this instrument one can optimize the crystal growth properly. Researchers in our laboratory have prepared near about 7-8 mm size large size crystal flex by using this instrument following the precipitation method of a precursor solution. Before using this, they could have prepared few micron size crystals. One can prepare any kind of solution-based crystal with proper optimization by using this kind of application-specific and customized heat treatment apparatus.

Acknowledgment

Mr. Subhadip Chowdhury, Senior Research Fellow of our institute has performed every step of the sample preparation processes all by himself. He has helped me a lot with his suggestions during preparing this manuscript. He provided the optical microscope images and the data files. He also supplied the selected reference papers from his collection; those are relevant to decide the structural and functional design parameters of the apparatus.

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