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Development of microcontroller based thermogravimetric analyzer

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ABSTRACT

Thermogravimetric analyzer (TGA) is an analytical technique extensively used to study the thermal decomposition of inorganic solids, their kinetics, reaction mechanisms, chemical property, structure of intermediate and final products and synthetic conditions. This paper reports the development of microcontroller based thermogravimetric analyzer. The TGA has been setup with additional features like controlled gas delivery system suitable for the study of decomposition (gas–solid interaction) in various atmospheres like air, oxygen, hydrogen, etc. The displacement of the spring in the system due to sample weight loss or gain on heating can be measured by using Infrared (IR) grating assembly and also by using cathetometer (least count is 0.001 cm).

1. Introduction

Thermogravimetry (TG) is used to obtain valuable information about reaction mechanisms, kinetic parameters and also to explain the stages of the reaction which are taking place in time sequence or temperature range. From the literature survey extensive work on nano material characterization has been published using TGA with automatic recording devices [1–3]. This paper describes the development of microcontroller based spring type thermogravimetric analyzer. In this thermobalance sample weight loss can be directly measured by measuring contraction or extension of helical phosphor-bronze spring (20.0063 cm/gm) of the type described by Mac Bain and Bakr [4,5]. Thermogravimetric analyzer can also be used to study the decomposition or gas–solid reaction in various atmospheres and in the preparation of nanocrystallies.

2. Microcontroller based thermogravimetric analyzer

The spring type thermogravimetric analyzer set up (Fig. 1) consists of Thermobalance, Furnace, IR grating assembly, Microcontroller assembly and Personal Computer.

2.1. Thermobalance

Sample weight loss can be directly measured by using a suitable helical phosphor-bronze spring (20.0063 cm/gm) of the type described by Mac Bain and Bakr. The principle used in this system is the extension or contraction of the spring, which follows Hook's law. The mass of the sample varies on heating due to chemical reaction, results in proportional extension or contraction of the spring.

The phosphor-bronze spring with the sensitivity of 20.0063 cm/gm, supplied by M/s thermal syndicate is used in the present studies. The spring has been calibrated with known weight and the maximum load permissible (2 gm). The quartz bucket (100–200 mg) containing the specimen is suspended at one end of the spring using a fine quartz fiber (50–100 mg). The spring and sample bucket are enclosed in a Pyrex tube or quartz tube with gas inlet and outlet (Fig. 1). The gas inlet tube has coils at the bottom to preheat the gas. The lower coil has small holes through which gas emerges and there after rises in the tube without turbulence. The outlet tube leads the gas through an indicative bubbler to the atmosphere in a suitable manner.

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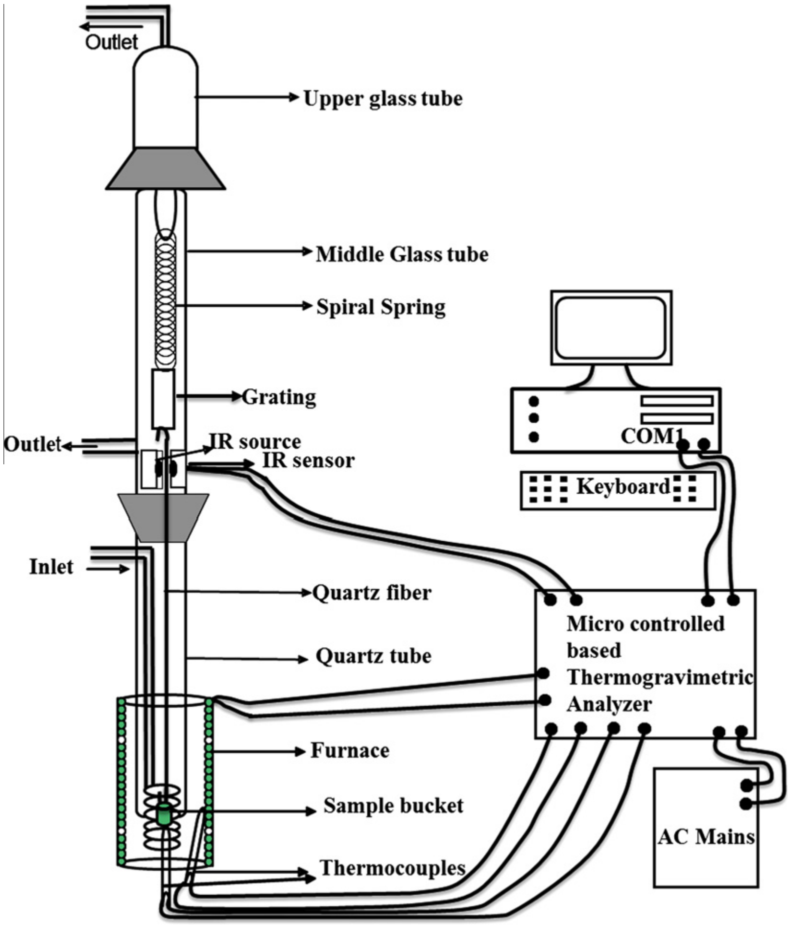

Fig. 1. The spring type thermogravimetric analyzer set up.

Provision has been made to insert a thermocouple and position its junction at the vicinity of the bucket (sample) to measure the sample temperature.

2.2. Furnace

A tubular furnace constructed is of length 30 cm with inner diameter 5 cm is mounted vertically and is capable of vertical motion so that the specimen can be positioned in the constant temperature zone of the furnace. The furnace can attain a maximum temperature of 1000 °C. A required (2 °C/min, 3 °C/min, 4 °C/min, etc.) linear rate of heating is obtained by using microcontroller based PID controller.

The block diagram of microcontroller based thermogravimetric analyzer is shown in Fig. 2, the description is as follows.

2.3. IR-grating assembly

The displacement of spiral spring is determined by using grating, IR source and IR detector (Q9874/9929 hp make) (Fig. 1). The grating hanged to lower end of the spring (Fig. 1) moves up or down between the IR source and detector, pulses generated by IR detector, as line of the grating crosses. Number of pulses generated multiplied by distance between alternate dark line gives the total displacement of the grating.

The output pulses from the IR detector are of negative value (0 to −V volts). Hence this output is connected as an input to an inverting amplifier with high gain and output voltage will be limited to 4.7 V using zener diode. The pulses are then passed through two inverting buffers (7406) to limit the pulse voltage to 0–5 V and reduce jittering (Fig. 3) and is connected as an input to pin RC_0 ($timer_1$/$counter_1$) of the microcontroller to count the number of pulses generated, inturn number of lines moved up or down thus the displacement.

2.4. Programmable AC voltage controller

The circuit schematic of programmable AC voltage controller (Fig. 4) is designed to control the rms voltage applied to the furnace (load) and hence the temperature of the furnace. The DAC is used to set output voltage of the AC voltage controller, inturn DAC input is varied by microcontroller PORTD.

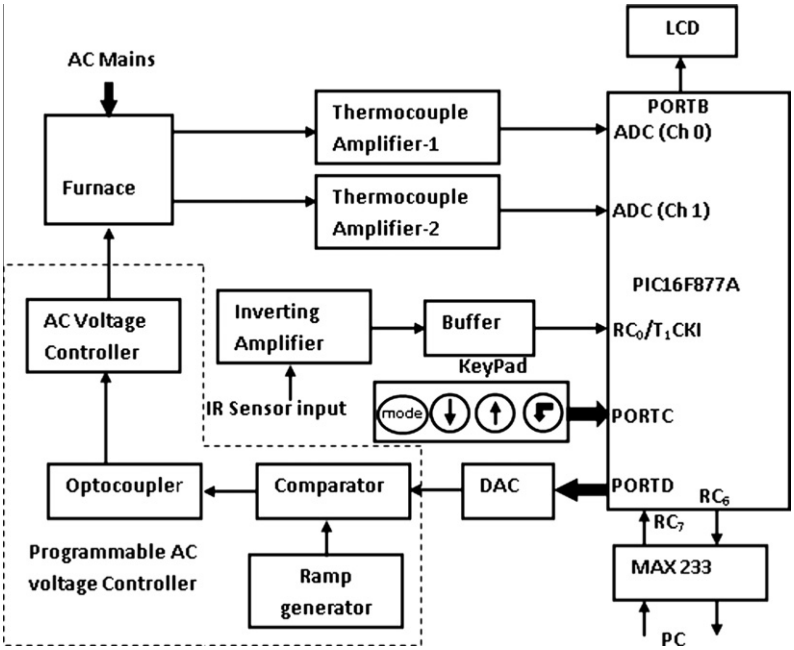

Fig. 2. Block diagram of microcontroller based thermogravimetric analyzer.

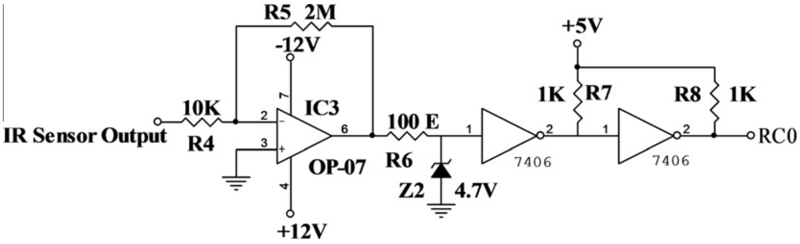

Fig. 3. IR sensor output amplifier circuit.

The triggering pulses for the TRIAC should be in synchronization with the zero crossing reference of the AC mains. A zero crossing detector is constructed to generate the pulses at zero crossings of the AC mains (Fig. 4). The output of step down transformer (16-0-16 V, 500 mA) is rectified using the centre tap full wave rectifier constructed using the IN4007 diodes. The rectified output (pulsating DC) is fed to the base of the transistor Q_1 (BC 547). The pulses at the zero crossing of the AC mains are obtained at the collector of the transistor. The zener diode Z_3 that has a reverse breakdown voltage of 10 V is used to limit the output of the transistor to 10 V.

The zero crossing pulses are applied to the capacitor C_2 (Fig. 4). Since the time constant in the charging path of the capacitor is very low, the capacitor is instantaneously charged to 10 V. When the capacitor charged to a peak value of 10 V, the transistor Q_3 (BC 547) provides the discharge path through the potentiometer P_4. The discharge time is suitably adjusted by varying the potentiometer P_4. The discharging of the capacitor is desired to be linear, for this purpose a polystyrene capacitor (C_2) is used. The instantaneous charging and the controlled discharging of the capacitor results in the generation of a ramp signal. The ramp signal at collector is fed to the transistor Q_2 (BC 547) which is an emitter follower.

The output of the ramp generator is fed to the inverting input and DAC output is fed to the non-inverting input of the comparator (LM301-Ic5) (Fig. 4). The leading edge of the comparator output varies with DAC output and this output is used to trigger the TRIAC and hence load voltage varies.

The output pulse of the comparator is fed to the optocoupler which provides isolation between the low power triggering circuit and the high power TRIAC circuit (Fig. 4). The AC mains supply is applied across the TRIAC BT136 in series with the load (furnace). The triggering of the TRIAC is done with respect to the leading edge of the gate pulse. The position of the leading edge of the pulse is controlled by the microcontroller.

The DAC 0808 is used as the digital interface between the microcontroller and the programmable AC voltage controller circuit to control the output rms voltage across the furnace (load). The 8-bit digital input required for the DAC is provided by PORTD of microcontroller. A 10 V supply is

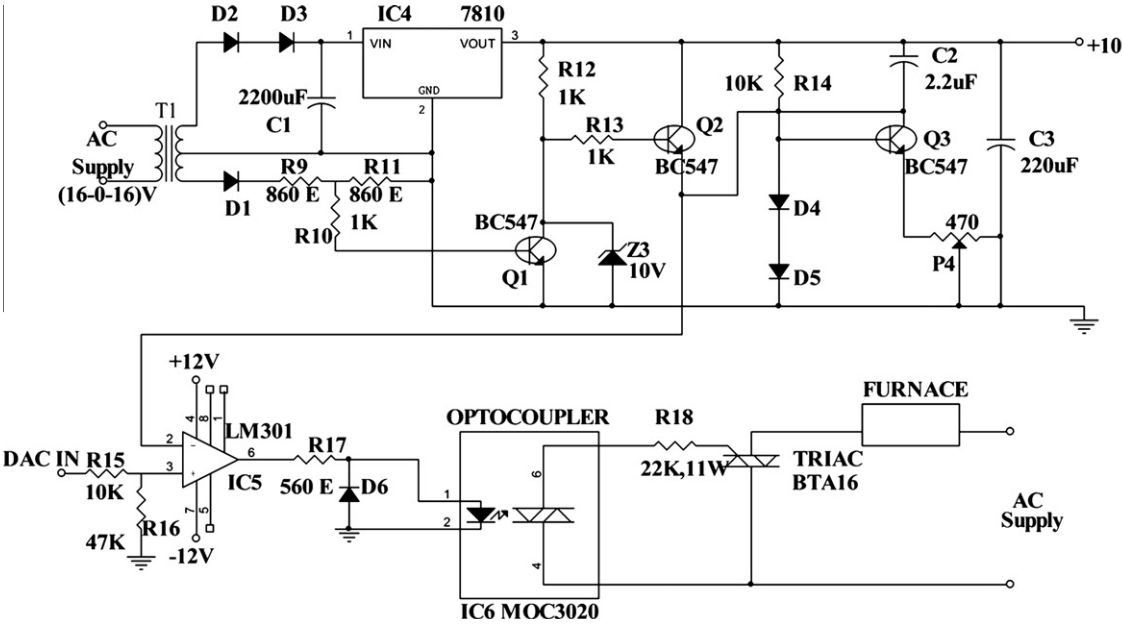

Fig. 4. Programmable AC voltage controller and +10 V regulated power supply.

used as the reference for the DAC. Hence the output voltage can vary from 0 V (00H) to 10 V (FFH) with a step size of 39.06 mV.

2.5. Thermocouple amplifier

In order to control the temperature of the furnace precisely and to study the mass variation of the substance with respect to temperature, it is essential to read the temperature of the furnace and temperature of the substance. Here the substance (sample bucket) is suspended inside the ceramic cylindrical tube over which the heating coil is wounded. The heat experienced by the substance inside the thermobalance is due to the radiation, because of which there is a temperature gradient between the walls of the furnace and the substance. In order to carry out the experiment in synchronization between the temperatures of the sample and the sample mass variation, two thermocouples are used, one to measure temperature of the furnace and other to measure temperature of the sample. In this application the maximum temperature that will be measured is around 1000 °C. PIC16F877A microcontroller used is having 10 bit internal ADC with 5 V reference. Therefore its resolution will be 4.87 mV ($\approx$5 mV).The thermocouple used is Kanthal type thermocouple which is having the resolution of 40 μV/°C and it has to be amplified to 5 mV/°C. Therefore outputs of the thermocouples are amplified by the amplifiers constructed using op-amp op07 designed for a gain of 125 (5 m/40 μ) (Figs. 5 and 6). The output of the thermocouple amplifiers are fed to channel-0 and channel-1 of ADC of the microcontroller respectively.

2.6. Microcontroller interface

The microcontroller used is the PIC l6F877A. It is a 40-pin 8-bit CMOS FLASH High performance RISC microcontroller, which has 8 channel 10 bit Analog to Digital converter. The circuit schematic of the microcontroller and its peripherals are as shown in Fig. 7. A 2 × 16 LCD display is interfaced to display temperature, displacements and time. A keypad consisting of four switches is also provided which consists of three push button switches (increment, decrement and enter) used to set parameters like maximum temperature and rate of rise in standalone mode of operation. The standalone (manual) mode or PC mode can be selected by using one more switch (SW4). Transmit (RC_6) and receive (RC_7) pins of the microcontroller are connected to receive and transmit pins of the serial port of PC via RS232C interface.

2.7. Software

A firmware has been developed to receive the data with respect to the set point temperature and the desired rate of rise of temperature of the furnace from the PC serially in PC mode or from push button switches in standalone mode. The microcontroller reads the temperature of the furnace and sample through ADC of microcontroller. PID controller has been implemented to control the rate of rise of temperature according to a desired rate and to maintain the temperature at set point. Simultaneously the microcontroller transmits the temperature of the furnace and displacement of the spring to PC serially and also displays temperature, displacement and time on LCD. The flowchart for thermogravimetric analyzer is shown in the Fig. 8.

GUI based software for the PC has been developed using *C*, to send the information regarding maximum set point temperature and rate of rise to the microcontroller in PC mode and to receive the temperature of the furnace and displacement of the spring from microcontroller. The temperature and displacement thus received are stored for further processing and a graph of the temperature verses time is plotted.

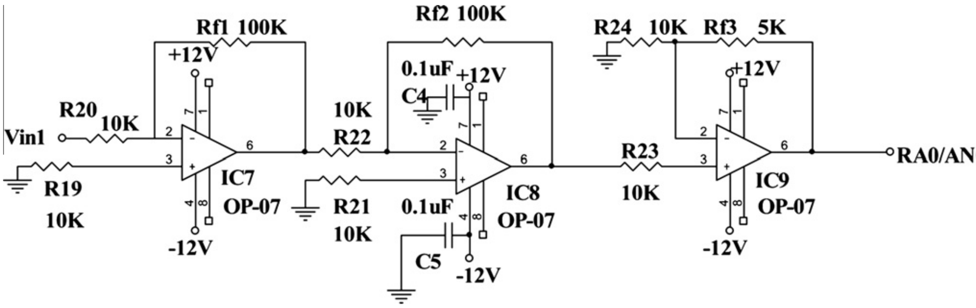

Fig. 5. Schematic circuit of thermocouple amplifier 1 (TCA_1).

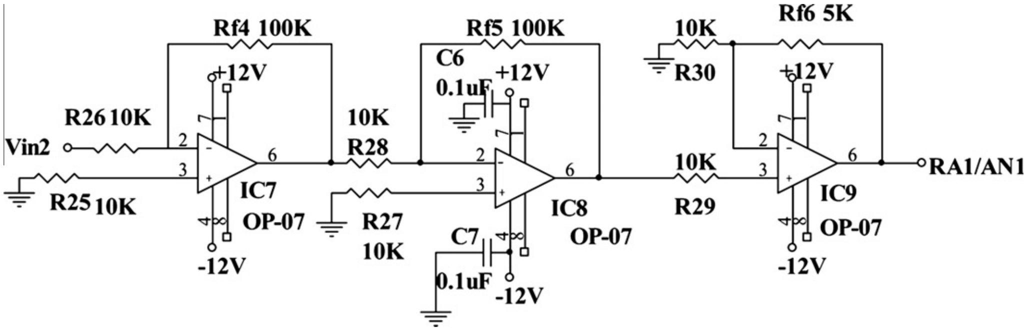

Fig. 6. Schematic circuit of thermocouple amplifier 2 (TCA_2).

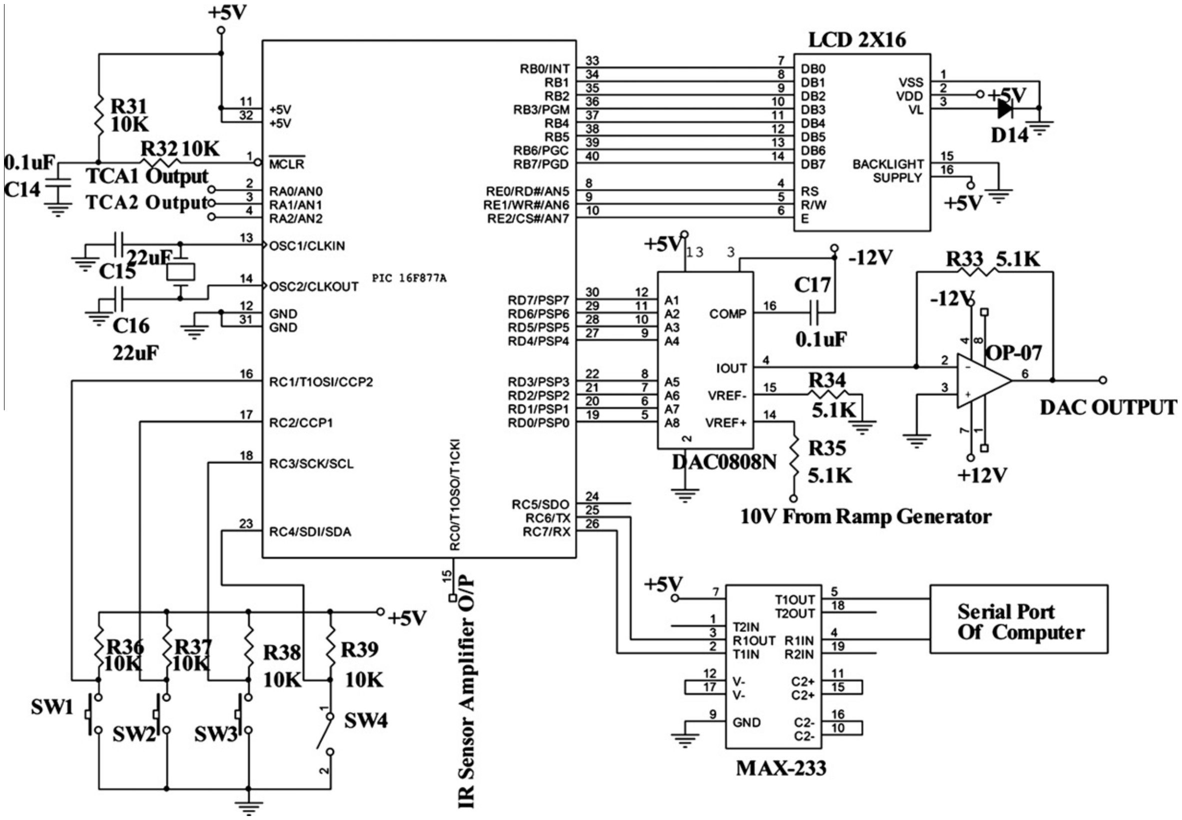

Fig. 7. Circuit schematic of hardware design of the microcontroller.

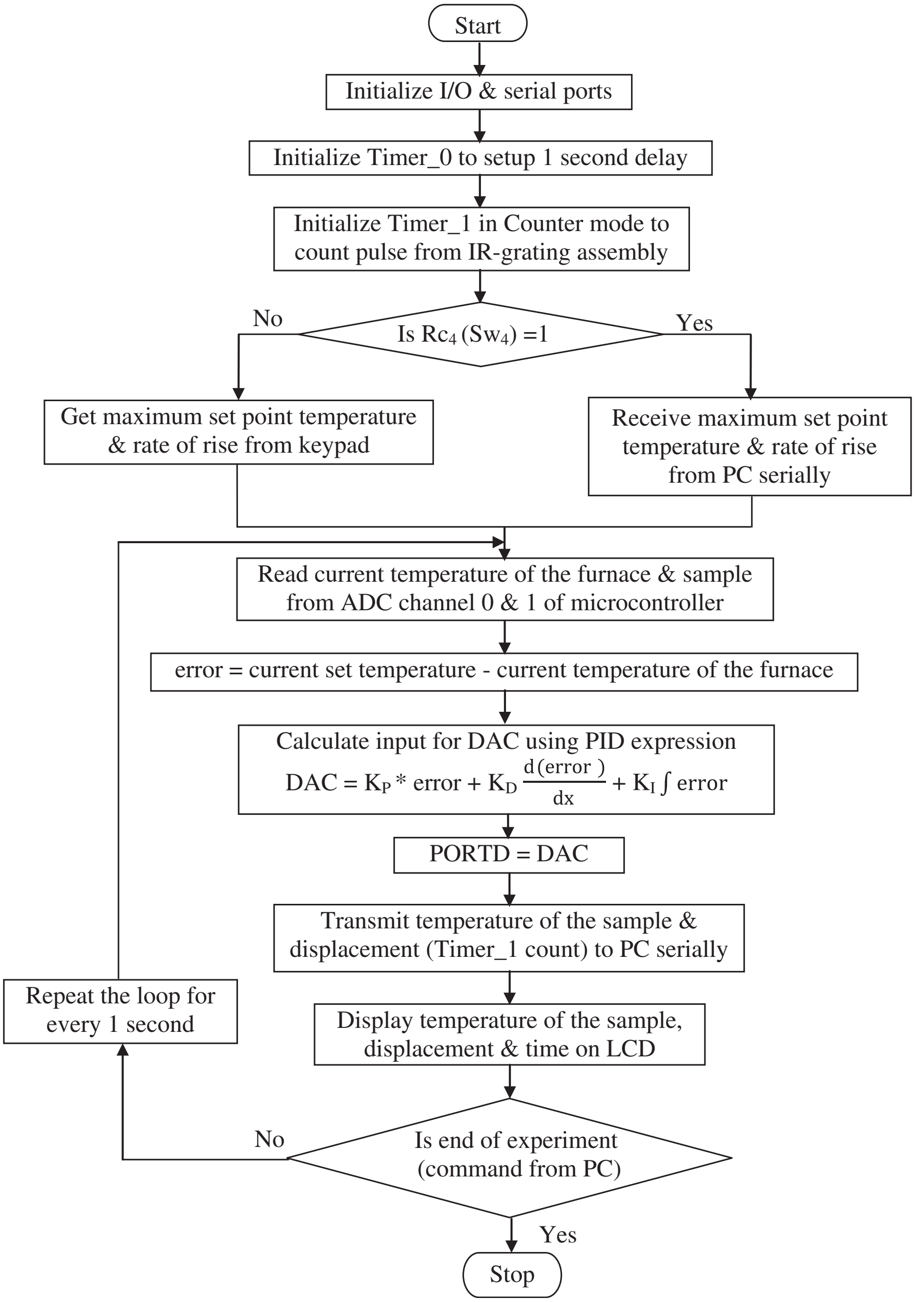

Fig. 8. Flowchart for thermogravimetric analyzer.

Table 1
Effects of varying tuning parameters.

Parameter	Rise time	Overshoot	Settling time	Steady state error
K_P	Decrease	Increase	Small change	Decrease
K_I	Decrease	Increase	Increase	Eliminate
K_D	Small change	Decrease	Decrease	None

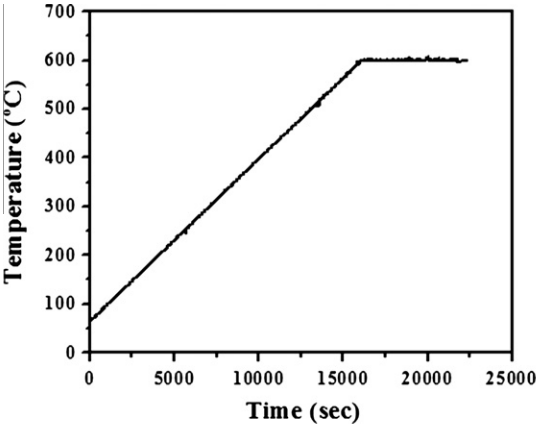

Fig. 9. Rate of rise −2 °C/min and maximum temperature −600 °C.

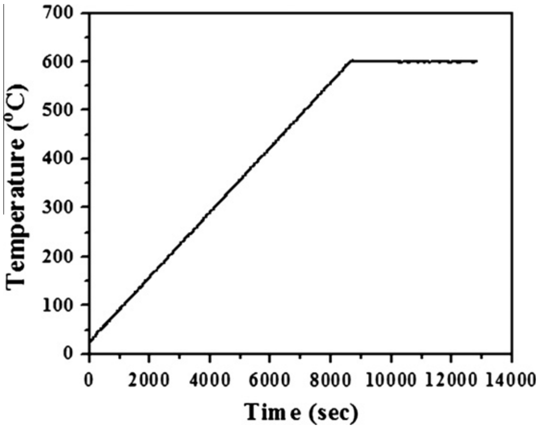

Fig. 10. Rate of rise −4 °C/min and maximum temperature −600 °C.

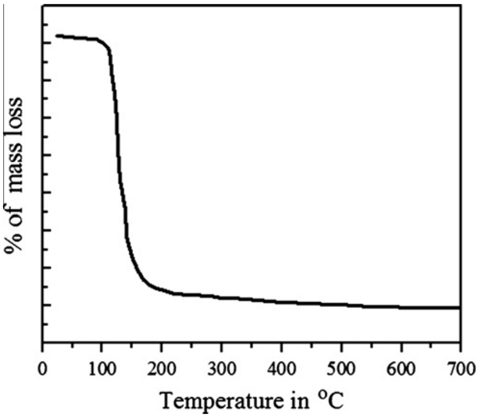

Fig. 11. Thermogram of BFO precursor.

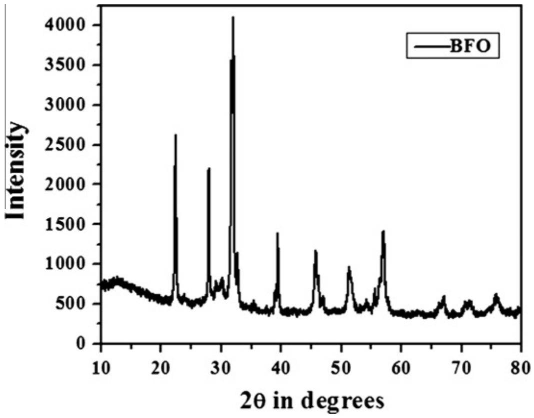

Fig. 12. XRD pattern of BFO precursor.

2.8. Tuning of PID

The PID controller parameters, the gain of the proportional, integral and derivative terms, if chosen incorrectly, the controlled process input can be unstable, i.e., its output diverges, with or without oscillation, and is limited only by saturation or mechanical breakage. Tuning a control loop is the adjustment of its control parameters to the optimum values for the desired control response.

The PID controller has been tuned by setting K_I and K_D to zero, K_P has been increased until the response overshoots. In order to reduce overshoot to an acceptable level, K_D has been increased. This decreases the settling time. Finally to obtain an error free response, K_I has been increased. The effects of varying these parameters are summarized in Table 1. The experiment is carried out with maximum temperature of 600 °C and different rates of rise of temperatures 2 °C/min, 4 °C/min is shown in Figs. 9 and 10.

3. Testing of instrument

The instrument designed has been tested by taking 50 mg of BFO precursor in the sample bucket and it was heated in air at a rate of 4 °C/min from room temperature to 700 °C. The variation in mass is also recorded for every 4 °C rise of temperature using cathetometer. It is found that the values recorded using the instrument are in good match with values recorded using cathetometer. The variation in mass of BFO precursor with respect to temperature is as shown in the Fig. 11. It can be noticed that only one highly exothermic effect with a major weight loss in the temperature range 100–400 °C and no other effects up to 700 °C. This result indicates that the ignition temperature for the combustion reaction is in between 100 and 400 °C. The XRD pattern (Fig. 12) was taken for the final combustion product. The TGA system has been calibrated for BFO precursors, the thermogram (Fig. 11) results and XRD pattern (Fig. 12) results are in good agreement with the results published [6–10].

4. Conclusions

Microcontroller based thermogravimetric analyzer has been designed and implemented. The software for the microcontroller has been developed using C language to implement the PID controller to control the temperature of the furnace and also to communicate with the PC. This facility provides the mechanism of high temperature gas–solid interactions in different gas facility and also best synthetic conditions (self ignition temperature) for preparation of nanocrystalline materials.

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