Control System for Heating Substrates in Manufacturing and Characterization of Gas Sensors

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***Abstract*—Keeping substrates surfaces at high constant temperatures during transparent conducting oxides thin film deposition is essential in manufacturing gas sensors by the spray pyrolysis technique. This work describes the assembling of a heating system for this purpose, as well as the development of dedicated hardware and firmware, including a proportional-integral-derivative controller and user interface for its use. The hardware consists of a printed circuit board containing an analog-to-digital converter for temperature measurements acquired by thermocouples and a microcontroller that coordinates the entire process running a finite-state machine.**

***Keywords*—gas, sensor, heating, controller, PID**

I. Introduction

One of the numerous applications of transparent conductive oxides (TCOs) thin films in the technological industry is in gas sensors [1]. A known type of solid-state gas sensor is the tin dioxide semiconductor [2], characterized by large changes in its surface resistance [3]. Additionally, other semiconductors such as ZnO and In_2O_3 are also used in this kind of application [4].

Spray pyrolysis is a popular deposition technique, having its quality and cost-effectiveness as advantages over similar processes [5]. Furthermore, the influence of the pyrolysis temperature on the resistance and sensitivity of Tin(IV) Oxide (SnO_2) films is pointed out by [6]. Thus, the importance of a system that delivers a controllable temperature surface for the manufacture of gas sensors by this approach.

II. Methods

A. Heating base assembly

A simple, low-cost heat transducer can be found in the form of a sheet metal containing an electrical resistance. In this project, such element specified for 220 V and 350 W was utilized, supported by a base made out of ceramic refractory blocks interleaved by layers of glass wool and fixed by heat wrap as shown in Fig. 1. Four K-type thermocouples were placed below the heating element near its center and in contact with it for temperature measurement.

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Fig. 1. Assembled heating base.

This setup allowed the system to reach temperatures of up to $480\ ^{\circ}\mathrm{C}$, which is in accordance with typical substrate temperatures observed in spray pyrolysis as reported by [4], [7], [8].

B. Hardware and software development

In order to acquire the thermocouples voltage readings, the 16-bit analog-to-digital converter (ADC) LTC2439-1 was chosen for its high resolution capability and 16 input channels. The ADC readings are transmitted to a microcontroller through SPI communication protocol, and its circuit implementation is shown in Fig. 2.

Thus, since K-type thermocouples present a sensitivity of about $48\ \mu\mathrm{V}/^{\circ}\mathrm{C}$, a micropower band-gap voltage reference of $1.2\ \mathrm{V}$ is provided by an LM385B to the ADC, allowing a resolution of $18\ \mu\mathrm{V}$ to be achieved. Furthermore, the ADC is also set for measuring the room temperature from a LM35 sensor, in order to enable cold junction compensation.

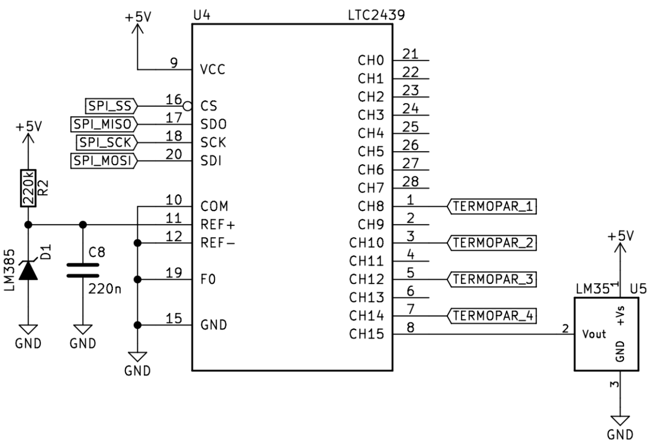

Fig. 2. ADC circuit schematics.

A printed circuit board (PCB) was designed to integrate all the necessary connections to an ATmega328P microcontroller running a custom compiled Optiboot bootloader at $14.7456\ \mathrm{MHz}$, as shown in Fig. 3. On this board, connectors are provided for plugging in a solid state relay (SSR) module, a 16-key matrix keyboard and an LCD. Furthermore, a shift register IC that allows an extension of the ATmega328P's I/O pins, and an access connector to the USART interface were added. Complete circuit schematics and PCB layout are available in [9] for reproduction of this setup.

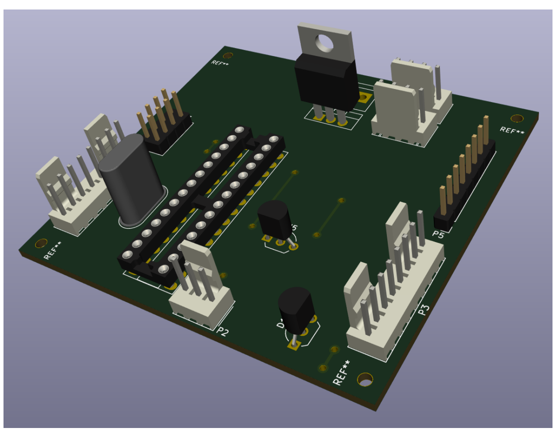

Fig. 3. Printed circuit board.

Software can be uploaded to the microcontroller's reprogrammable Flash program memory through USART using the In-System Programming technique. In this particular project, dedicated firmware written in C language and compiled using the AVR-GCC Toolchain was implemented, containing a finite state machine that goes through three states. In the first one, the user may specify input settings such as temperature and time for the system to operate. Followed by that, there is a transition state in which the controller starts to act until the system stabilizes on the specified temperature. From then, an operation state starts, maintaining the surface at the set temperature for the established time.

C. Control system design

As a control variable, a $4\ \mathrm{Hz}$ pulse width modulation (PWM) drives the SSR, allowing the average mains voltage to be applied to the heater's resistance in a controlled fashion according to the calculated duty cycle. In order to describe the system mathematically, an open-loop step response test was performed, in which the SSR was kept on, as the microcontroller acquired temperature measurements and sent them via serial communication to a computer that stored the test's data in a log file. From this experiment, the system's dominant pole $(s+1/49)$ was obtained by measuring its settling time of $147\ \mathrm{s}$, and a second pole $(s+1/28)$ was defined by observing the simulated response that was the most similar to the one of the real system. This resulted in the approximate transfer function presented in (1), which nearly describes the system's behavior.

$$G(s) = \frac{7.289 \times 10^{-4}}{s^2 + 5.612 \times 10^{-2} s + 7.289 \times 10^{-4}} \tag{1}$$

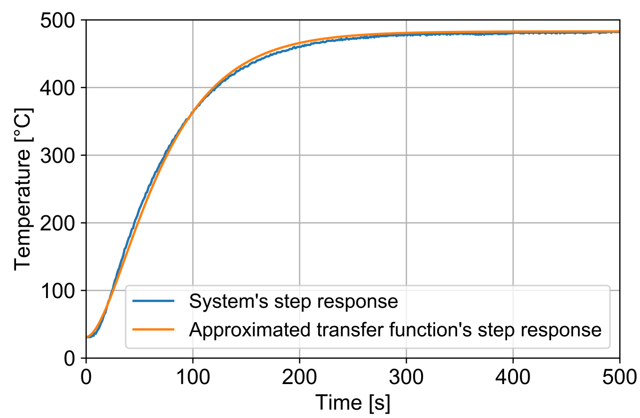

Fig. 4. Open-loop step response.

Fig. 4 shows the step response obtained from the open-loop test as well as the simulated response corresponding the approximate transfer function. As can be seen, the system has an overdamped response.

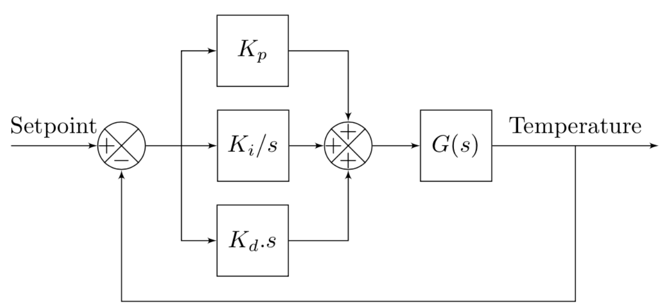

Fig. 5. Control-flow diagram.

A simplified control-flow diagram of the closed-loop system with a proportional-integral-derivative (PID) controller is shown in Fig. 5. Based on the information extracted from the experiment, two sets of control parameters were tuned by first setting the integral and derivative gains Ki and Kd to zero and increasing the proportional gain Kp until the overshoot reached the setpoint value. Then, Ki was incremented in order to correct the steady-state error, and finally Kd was set to minimize the overshoot.

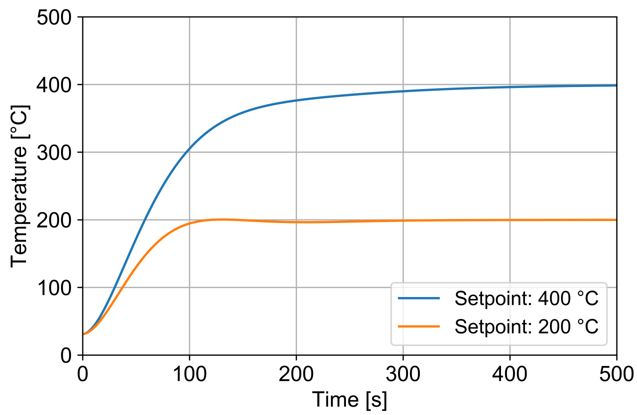

Fig. 6. Closed-loop simulated step response.

Specified to operate at temperatures of up to $350\,^{\circ}\mathrm{C}$, one of them was designed with a faster response. The other, intended to operate at temperatures near the system's limit, of around $480\,^{\circ}\mathrm{C}$, was designed with a larger settling time, since it is bounded by the system's natural maximum response. Simulation results of their closed-loop behaviors are illustrated in Fig. 6, where the controller with each set of gains was set to $200\,^{\circ}\mathrm{C}$ and $400\,^{\circ}\mathrm{C}$.

III. Results

Fig. 7 shows the complete system. The acrylic compartment, in which the heating base is inserted, was built to provide a controlled environment, without interference from external temperature or impurities present in the atmosphere. This structure contains valves for subsequent application of chemical solutions for deposition of thin films on substrates positioned on the heated surface. The user interface, constituted by the LCD and matrix keyboard, allows that the desired temperature and heating time are set.

IV. Discussion

Future enhancements in the heat transducer's thermal insulation can be made in order to achieve higher temperatures if needed. Nonetheless, many cases of thin films deposition, such as observed in [1], [2], [5] and [7], have been reported within the temperature range already attained by the structure herein presented.

Although the control system shows promising simulation results, benchmarking and adjustments will be required in the process of its practical implementation. However, building the entire system to attend the necessary requirements for manufacture and characterization of gas sensors, including

Fig. 7. Full system assembled.

the development of the hardware, firmware and the control design, is the first step towards our goals. As a future objective, measurements on commercial sensors and substrates doped with Tin(IV) Oxide will be arranged, for proper validation of the equipment that was developed.

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