

Practical and low-cost solution for the temperature control of a substrate heater for thin film deposition

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Abstract—The paper focuses on finding a suitable practical approach to measure and maintain the temperature of the substrate at a desired temperature, imposed by the type of the sputtering and the type of the desired thin film structure. The resulting solution is suitable for a wide range of experimental equipment built for the instable reactive magnetron sputtering process. The aim is to increase the stability and to enhance the repeatability of the process in discussion. By stabilizing and controlling the temperature of the substrate, the presented system contributes to the complex task of sustaining a controlled environment for a successful sputtering. The paper presents in detail the design steps of a simple resistive substrate heater assembly. The main topics include the implementation of the control mechanism on a low-cost embedded platform, featuring wireless connectivity and data transfer through different communication channels using TCP packets, as well as the incorporation of IoT services.

Keywords—resistive substrate heater; thin film; low-cost embedded solution; temperature control; IoT system

I. INTRODUCTION

Over the last few decades, the DC magnetron sputtering process has been widely used to create various thin film coatings [1]. By combining sputtered metallic particles with different reactive gas molecules, a wide variety of useful thin film surface coatings can be created, featuring enhanced mechanical or optical properties [2]. Depending on the composition and the structure, these coatings can be utilized in different industrial applications.

The basic principle of the reactive sputtering process is quite simple, implying the deposition on the surface of a substrate of different compounds formed in a vacuum chamber with a controlled atmosphere. The compounds are formed in the presents of reactive gases and energized sputtered particles. The particles result from the sputtering process in which inert gas ions bomb the surface of the metallic target. The atmosphere needed is ensured by the adequate control of the admission and evacuation of the different inert and reactive gases used [2, 3].

Having sad that, the macroscopic approach reviles a more complex functioning, making the compound forming and the deposition process highly sensitive to the variation of the various process parameters. Furthermore, it can be stated that

the process parameters are highly interdependent [2]. This fact implies that the modification of a given parameter has an effect on other process parameters. Thus the various control loops are needed. In the case of the majority of the thin film types, without external interference the sputtering process is highly unstable.

Numerous studies have been conducted highlighting that in order to achieve a stable and repeatable process, it is mandatory to create and sustain a stable environment [1, 4-7]. This is achieved by measuring and controlling as many process parameters as possible in order to ensure the conditions necessary to obtain the required stoichiometry of the thin film [2].

Our current field of study focuses mainly on the production and the evaluation of TiON, TiN and TiAlSiN surface coatings. In the case of these thin films, early results revealed that the temperature of the substrate has a direct effect on the structure of the thin film deposited. This phenomenon is emphasized especially in case of the TiAlSiN.

II. EXPERIMENTAL EQUIPMENT

The sputtering equipment consists of an octagonal vacuum chamber with an internal volume of 73l. The evacuation system is formed by an Alcatel Pascal 2021SD rotary vacuum pump, an Osaka Vacuum TH520 turbomolecular pump and a custom made butterfly valve situated in the evacuation duct for the control of the evacuation capacity.

The vacuum levels at the different stages of the equipment are measured with a PKR251 and an MPT100 full-range pressure gauge from Pfeiffer Vacuum, two custom made Pirani gauges and a high resolution CMR365 capacitive pressure gauge. The inert Ar gas, the reactive N₂ or O₂ gas is introduced in a controlled manner through separate Aalborg DFC26 mass flow controllers. Therefore the admission and the evacuation of the different gases is measured and controlled in order to sustain the desired composition of the atmosphere [2]. The energy needed to form the plasma containing the bombing ions is ensured by three independent high voltage 3kW DC power supplies developed for the equipment in discussion. The power supplies are connected to three different magnetrons.

A series of various measuring and control units are connected to the vacuum chamber, in order to measure and control as many process parameters as possible. Among these units the remarkable ones include a Leybold-Heraeus QM210 quadrupole mass spectrometer, custom developed control devices, such as the deposition rate monitor, the substrate heater controller, the optical spectral analyzer, the dynamic pressure controller [1], the various pressure measuring units, the polarization voltage meter, the cooling management unit, as well as a centralized supervisory and data acquisition application serving data management and high level control tasks.

III. THERMAL MANAGEMENT OF THE SUBSTRATE SUB-ASSEMBLY

The substrate heater and the thermal management represent an important unit in the construction of the automated sputtering equipment. The development work is divided into two parts: the design of the substrate heater sub-assembly and the development of the low-cost, microcontroller based temperature control unit.

A. The resistive substrate heater

There are many different approaches and concepts to choose from [8-10] when designing a substrate heater. In order to design and build a heater unit suitable for a specific sputtering equipment, there are many aspects that need to be taken into consideration. The most notable aspects include the volume and shape of the vacuum chamber, the dimensions of the useful coating surface also known as the substrate, the heating method and the working temperature which is mainly determined by the type, composition and structure of the thin film formed.

The sputtering equipment presented earlier and the type of thin films studied imposed several design guide lines or constrains. In accordance with these, the sub-assembly has to hold a silicon and a steel substrate with a surface area of 1.44cm² each, the two substrates have to be easily removable and the working temperature needs to be adjustable from 200°C to 600°C.

The resulting substrate sub-assembly (Fig. 1) uses resistive heating method and consists of a fixed and a removable part. The fixed part is mounted inside the vacuum chamber having the primary role of providing a mechanical platform for the heater strip and making the necessary electrical connections. The materials used include stainless steel for the fastening mechanism, high purity copper for the electrical lines and ceramic isolators for mounting the copper plates to the base assembly. All the electrical power cables and high current couplings have a minimum cross section of 100mm².

The removable part, also known as the boat, features a stacked multilayer structure (Fig. 2). The heating element, through which the controlled current (i_{heater}) flows, is an 80µm thick, 80mm long and 20mm wide molybdenum (Mo) strip with a surface area of 0.016cm² and with a resistance of 13.87mΩ measured at room temperature. Molybdenum was used, because it has a high melting point of 2623°C, low electrical resistance, mechanical stability at high temperatures and low thermal expansion. With the presented configuration,

under real conditions the heater can withstand a continuous temperature of 700°C. On the side of the strip facing the magnetron and implicitly the plasma, a precision machined steel slab is placed, serving as a plain platform for the thin silicon (Si) substrate and is 0.4 mm thinner than the steel substrates on which the thin film coating is deposited.

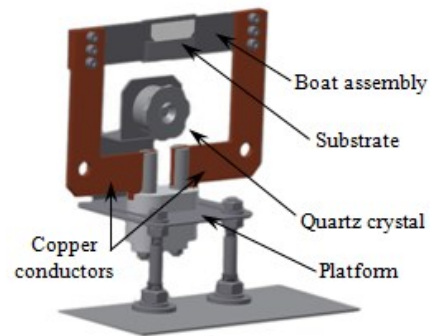


Fig. 1. CAD model of the resistive substrate heater sub-assembly

The thermocouple type temperature sensor used to determine the temperature of the substrate is placed into a 1.5mm diameter bore situated on the upper side of the steel slab. The bore has a 6mm depth, assuring the necessary contact surface for the sensor and protection against unwanted deposition inside the bore. When the boat is dismantled and taken out of the vacuum chamber, the sensor is also removed.

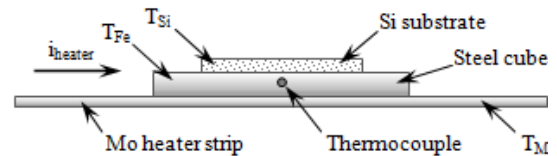


Fig. 2. Simplified structure of the boat assembly

The different layers are strongly held together by two 0.8mm thick nickel clamps. Nickel clamps were used, because at the working temperature of 200°C-600°C the metal retains sufficiently its mechanical properties. The two clamps are electrically isolated from the steel slab, therefore the current flows through the molybdenum strip and not through the nickel clamps and the substrate itself. The presented boat assembly is fastened to the electrical terminals of the sub-assembly's fixed part with six, stainless steel M3 bolts, keeping the contact surfaces from unwanted deposition. The removable part is prepared in a clean environment, after which it is placed into the vacuum chamber, reducing the time in which the chamber is opened and exposed to unwanted contamination.

The temperature of the substrate is an equilibrium temperature, determined by the balance of the input and output heat. The power balance of the presented sub-assembly is given by the following equation:

$$P_{GEN} = P_{AMB} + P_{CH}, \quad (1)$$

where P_{GEN} is the net electrical power generated by the power supply, P_{AMB} is the power loss on the connecting electrical cables from outside the vacuum chamber and P_{CH} is the total power introduced into the sputtering equipment. In the present application, it is considered that the P_{AMB} equals only the convection power (2). Having said that, based on measurements, the P_{AMB} in the current configuration takes up 15% of the total generated power.

$$P_{AMB} = P_{CONV-CU-AIR} \quad (2)$$

Taking into consideration the working parameters such as the substrate temperature and the pressure value from inside the chamber, the power distribution of P_{CH} is given by the following equation:

$$P_{CH} = P_{COND-Mo-Cu} + P_{RAD-Mo-Fe} + P_{RAD-Fe-Si} + P_{COND-Fe-Si} \quad (3)$$

where $P_{COND-Mo-Cu}$ is the conducted power of the copper power cables, $P_{RAD-Mo-Fe}$ is the radiated power from the surface of the molybdenum heater strip, $P_{RAD-Fe-Si}$ and $P_{COND-Fe-Si}$ are the radiated and conducted powers in the case of the steel slab on which the substrate is positioned. It is considered that the temperature difference between the Si substrate and the steel slab is negligible, because of the good thermal coupling. Contrary to that, the thermal coupling between the heater strip and the steel slab is quite low due to surface imperfections; therefore the heat is transferred mainly by radiation. This also assumes that the molybdenum strip has a higher temperature than the steel slab and the substrate.

The whole sub-assembly is mounted on a rotating table (Fig. 3). The table itself has an independent position controller.

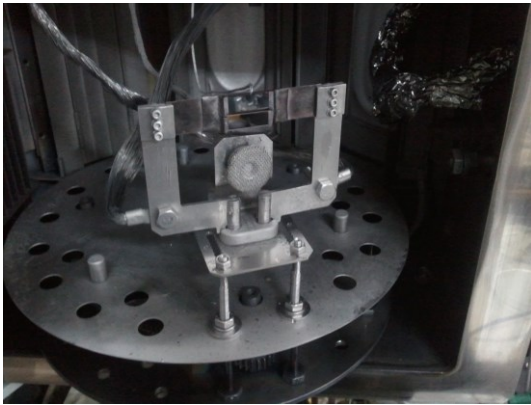


Fig. 3. Substrate heater sub-assembly mounted on a rotating table inside the sputtering equipment's vacuum chamber

With the use of the rotating table, the heater can be oriented towards the opening of the vacuum chamber in order to ease the mounting process of the removable part or boat containing the Si substrate (Fig. 3).

During the sputtering process, the table is positioned so that the substrates face one of the three magnetrons available

(Fig. 4). The heater unit houses also the Inficon 750-1005-G10 type Quartz crystal used to measure the layer thickness and to calculate the growth rate. In order to accurately measure the deposition rate, the crystal oscillator needs to be positioned as close to the substrates and the boat as possible (Fig. 3).

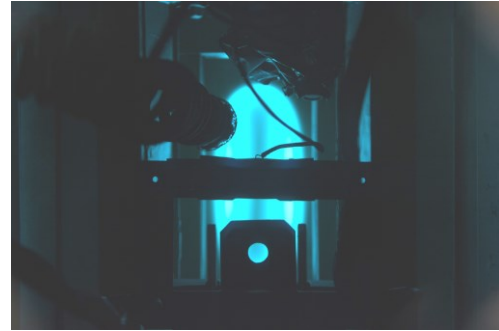


Fig. 4. Substrate heater sub-assembly orientated towards the magnetron during sputtering

B. Low-cost embedded control unit

The substrate temperature control unit (STH) incorporates the basic setup of a closed loop control (Fig 5). The presented system includes a temperature sensor for feedback, a microcontroller (MCU) based embedded system for the control algorithm and an output unit in the form a high current AC power supply. The conventional configuration is expended by a secondary feedback loop in the form of a current transducer for measuring the heater current and for introducing auxiliary control signal conditioning.

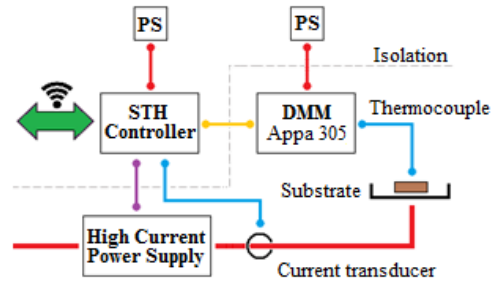


Fig. 5. Functional schematic of the electronic control system

Measuring the temperature of the substrate represents one of the key tasks of the temperature control unit. In the presented approach the temperature is measured using a K type (Nickel-Chromium / Nickel-Alumel) thermocouple. The type K is a simple, inexpensive, robust and reliable thermocouple, making it one of most common types. The sensor has a wide temperature range, an accuracy of $\pm 2.2^\circ\text{C}$, and it can be operated safely at a maximum continuous temperature of 1.000°C , making it suitable for the proposed application.

Through proper electric-vacuum coupling, the sensor is connected to an external APPA305 digital multimeter, assuring a 0.1°C temperature resolution in the range of -40°C and 1000°C . The measured and scaled temperature value is then transmitted through an optically isolated serial line to the embedded system. This step is necessary, because the

substrate sub-assembly is polarized with a 300V DC voltage; therefore the measuring line needs to be isolated from the low-voltage control side. The data transfer is initiated by the MCU. Every 600ms a new temperature sample is received from the multimeter.

With the advancements in the field of semiconductor technology, microcontroller units become more powerful, efficient and accessible. The embedded system is based on a new generation Espressif ESP-WROOM-02 type microcontroller, featuring 32bit architecture and wireless network connectivity. The microcontroller is responsible for the data acquisition, for executing the temperature control algorithm and for generating the corresponding control signal. The 75x35mm double layered printed circuit board (Fig. 6) includes the power management circuit, the microcontroller, and the EIA-232 driver/receiver circuit with the appropriate D-SUB9 connector, and a low-signal digital/analog I/O interface for an external extension card.

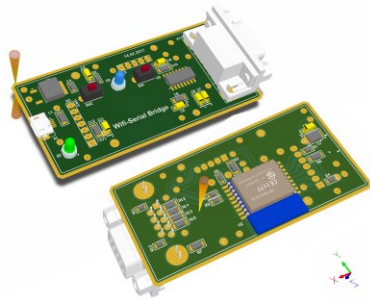


Fig. 6. CAD model of the embedded system

The output unit consists of an Ulvac Sinku Kiko 150A high current AC power supply, which is modified to receive external analog set value from the embedded system. The extension card, making the transition between different low-level analog (set value and measured current) and digital signals (different power supply states) is integrated into the power supply. The set value generated by the MCU has a 10bit resolution, which results in a 146mA resolution on the output of the power supply.

The AC current is measured using a current transformer with a 150:5 ratio and a $\pm 5A$ ACS714, Hall effect-based linear current sensor with additional analog signal conditioning circuit (Fig. 7) integrated into the extension card. The ACS714 sensor has an output sensitivity of 185mV/A and a 2.5V null point offset. The signal conditioner consists of a 2.5V level shifter, a low pass filter and a buffer stage. As a result, the 0÷150A current range corresponds to a 0÷925mV low-voltage range. The MCU features a 10bit analog to digital converter and an internal voltage reference source of 1V. Taking into consideration all the above, the heater current is measured with a resolution of 158mA.

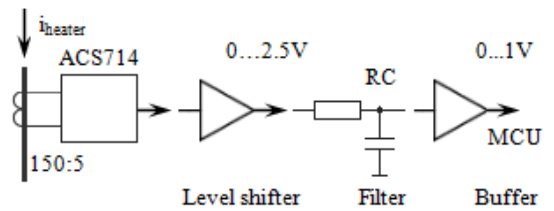


Fig. 7. Analog signal conditioning circuit for measuring the heater current

IV. MEASUREMENTS AND TEMPERATURE CONTROL

The pre-sputtering phase presumes the deposition of a thin 2-4kÅ metallic base layer on the surface of the Si substrate. The deposition of the metallic film is an exothermic reaction, resulting in an excess of a small quantity of heat energy, which combined with the energy from the plasma, increases the overall temperature of the substrate and sub-assembly (Fig. 8).

When reactive gases are added to the process, the deposition process becomes an endothermic one, resulting in the reduction of the sub-assemblies temperature due to heat loss (Fig. 8). In the presented process the sputtering power varied between 200W and 600W, depending on the growth rate and the actual stage of the deposition. Throughout the process the target temperature was 400°C and the heating current was maintained at a constant 53A. At the end, the coating reached an overall thickness of 25kÅ.

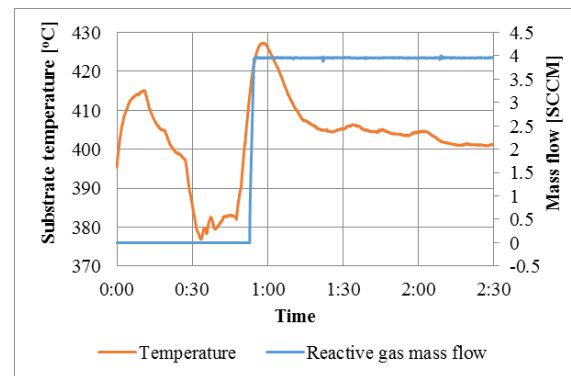


Fig. 8. Temperature deviation of the substrate in function of the sputtering stage (heating current maintained constant)

With identical sputtering parameters and source material, at different substrate temperatures different crystallization phases can be observed. This fact justifies the need for a temperature management unit. The nature of temperature deviations detected (Fig. 8) revealed a relatively slow thermal process. For the control of the process in hand, a discrete and manually tuned proportional–integral (PI) type regulator was proposed.

When implementing the regulator, it became quite obvious that a series of constraints need to be taken into consideration. One of most important constraint is that the Si substrate and the thin molybdenum heater strip needs to be protected against thermal shocks. An unwanted result of thermal stress are cracks in the substrate and possibly in the thin film deposited on the Si as well, which alters the structure of the coating. As

a further consequence, this effect most certainly reduces the possibility of processing the thin film coating with different analytical methods.

In order to reduce the thermal stress in the substrate, the regulator uses a ramped temperature reference value. The steepness of the ramp can be selected by the user from an experimentally determined interval of $1^{\circ}\text{C}/\text{min} \div 20^{\circ}\text{C}/\text{min}$.

As a measure of precaution, the variation speed of the regulator output is permanently verified, and if necessary the output is limited by overruling the controller. The variation speed of the measured current is also monitored. Based on this information another constrain is added and the temperature regulator is broaden with a secondary or auxiliary feed-back loop. As a result the controller's performance is increased and a new safety function is added in the form of current limitation. The variation speed of the current is set to $2\text{A}/\text{s}$.

The closed-loop temperature regulator was tuned using the Ziegler–Nichols method and the obtained parameters ($K_p=15$, $T_i=2$, $T_s=1.2\text{s}$) were manually fine-tuned. The performance of the control algorithm (Fig. 9) was inspected over the course of a temperature reference value change from 400°C to 500°C with the maximum ramp steepness of 20°C . The maximum deviation from the ramped reference value was 11.2°C , whilst the measured overshoot at the end of the ramping stage reached a peak value of 9.3°C . The maximum settling time observed was 149s ($t_{1\%}$).

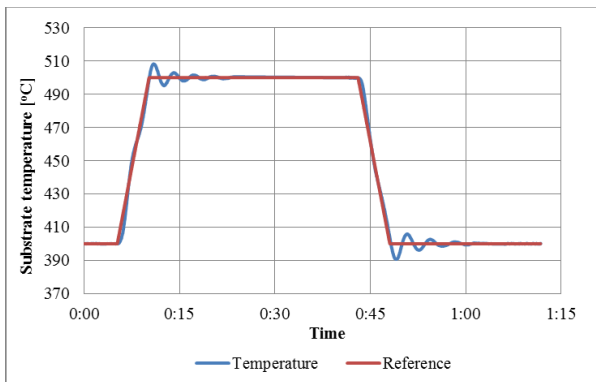
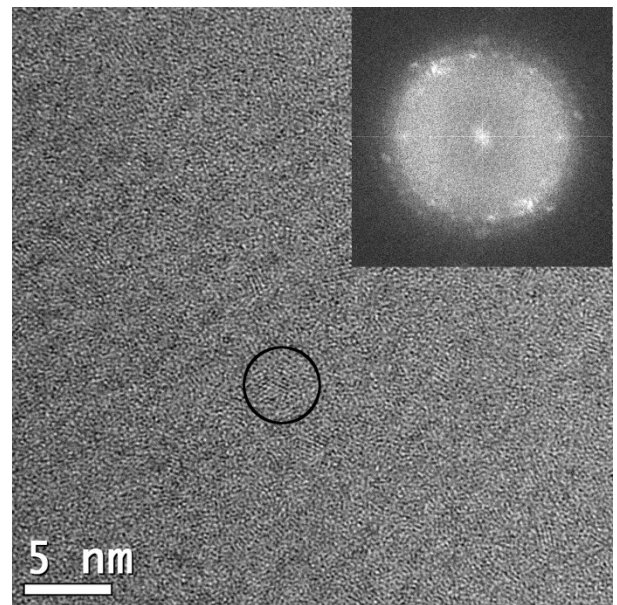


Fig. 9. Temperature response in case of reference change ($\Delta T=100^{\circ}\text{C}$, ramp steepness $20^{\circ}\text{C}/\text{min}$)

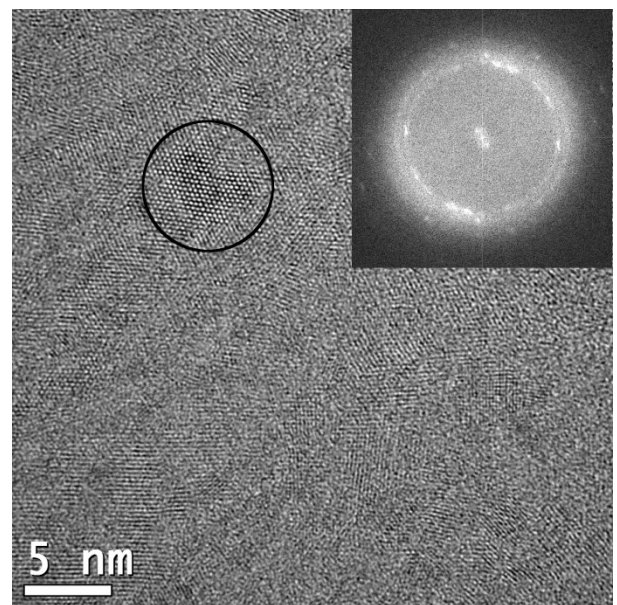
After the sputtering process, the substrate is cooled down in a controller manner, using the same ramped temperature reference method as in the case of the heating up stage. Below 100°C , the temperature controller is suspended and enters into a standby mode.

In order to investigate and present the effect of the temperature variation on the structure of the thin film, a two stage TiAlSiN coating was produced. Both stages were created using the same process parameters and the same sputtering system settings. The only difference was the temperature, which in the case of the first stage was maintained at 400°C , whilst in the case of the second stage at 500°C . During the sputtering process, the sputtering power was kept at 400W ; the dynamic pressure was maintained at a predefined value of 0.4Pa ; the mass flow of the inert Ar gas was set to 6SCCM , whilst the mass flow of the reactive N_2 gas was set to 2SCCM .

The basic setup and composition of the stages are congruent, being able to isolate TiAlN grains or crystals embedded in an intermediate amorphous Si_3N_4 matrix[11] (Fig. 10). The grains are basically areas presenting some kind of pattern, which in the case of the second stage are characterized by a more extensive surface (Fig. 10).



a).



b).

Fig. 10. Morphology of the TiAlSiN thin film sample at different substrate temperatures: a). 400°C , b). 500°C (HRTEM recording with the FFT of the image in the upper right corner and grains marked with black circle)

The differences in the morphology of the two separate stages of the presented sample are a result of the volume diffusion. The presented results were obtained with a high-resolution transmission electron microscope (HRTEM).

In case of a temperature sensor malfunction, the embedded unit automatically freezes the regulator algorithm and maintains the heating current at the last valid value until the user acknowledges the fault. At this point, in order to continue the sputtering process, the user has the possibility to convert the thermal management unit into an expert system. This means, that the output value is selected from a predefined table, containing temperature-control signal values.

The values were determined through a series of measurements without sputtering, because in different sputtering processes the intensity of the plasma varies, therefore the energy from the plasma absorbed by the sputtering process varies as well. The presented state is not an appropriate working condition, therefore it was unnecessary to extend and increase the complexity of the expert system with multiple tables for different sputtering scenarios. This means, that the temperature of the substrate may deviate from the predefined value. Over the course of a sputtering process, this value can exceed 30°C. By introducing this alternative control method, the sputtering process, including the relatively long preparation period of the sample does not have to be interrupted in case of a malfunction.

V. INTEGRATING THE TEMPERATURE CONTROLLER INTO THE SPUTTERING EQUIPMENT'S AUTOMATED CONTROL SYSTEM

Due to the complexity of the sputtering process and of the experimental equipment, the presented controllers and measuring units are integrated into a hierarchical multilevel process control system in order to ensure the comprehensive and adequate management of the process in discussion [3]. The result is a modular distributed system using Ethernet network for data exchange. As a result, the substrate temperature control unit represents one of the presented controllers and is integrated into the multilevel system.

By simplifying and reducing the multilevel system to only the temperature control unit, we can distinguish a field level, a control level, a supervisory level, a data acquisition level, as well as an external supervisory level (Fig. 11) [3].

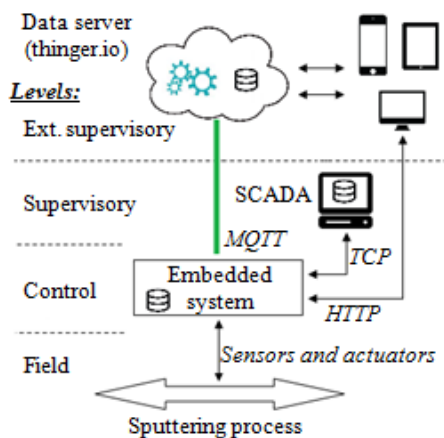


Fig. 11. Simplified structure of the multilevel control system

The inferior field level incorporates the sensors and the actuators establishing direct connection with the sputtering

process. The level contains the resistive heater assembly, the temperature sensor, the current transducer and high current AC power supply. The control level includes the microcontroller based embedded unit and all its auxiliary electronic circuits. As the name implies, this level is responsible for the local thermal management of the substrate.

The two superior levels are basically used to handle the local and remote, high level data management and process control through a wired and wireless Ethernet network. With the help of different communication channels and network services, the embedded unit features multiple connections with well-defined data packets. A TCP channel is used to establish a full access, bidirectional communication with a custom developed, centralized supervisory and data acquisition (SCADA) software [3]. The application enables the end user to monitor the behavior of the sputtering process, an implicitly the functioning of the substrate heater. With the help of the interface, the user can modify online the regulator parameters, the sampling time, the limit values, the temperature reference value and the steepness of the ramp. Being a bidirectional communication, the measured temperature, calculated current, actual reference value and the regulator output are received and plotted out on the GUI with a refresh rate of 400ms. The embedded system features an integrated web server as well, enabling unidirectional remote access for solely monitoring purposes.

With the rapidly evolving IoT systems and applications, there are many possibilities to extend the features and the services of virtually any embedded system. Some of the most significant services include remote data archiving, online data visualization, bidirectional data exchange between a remote server and the embedded system, event logging and notification management [12]. By using a cloud based IoT data server such as the thinger.io, the substrate temperature control unit benefits of all the listed services, including email notifications to defined recipients, containing status and error messages. The status messages contain information regarding the heating stage, whilst the error messages contain alerts such as sensor malfunction, high heater current, high current deviation or interrupted power circuit.

VI. CONCLUSIONS

From the start of the development work, there were some target expectation imposed that needed to be fulfilled. These included the design of a relatively simple and durable resistive substrate heater assembly mounted inside the sputtering equipment, capable of reaching 600°C. As a supplementary unit, the substrate heater is controlled by a low-cost embedded system, incorporating a series of well-defined network and IoT services. Furthermore the developed microcontroller based system is more than capable of executing a series of complex tasks. Among these tasks the remarkable ones include the measurement of the temperature and heating AC current, the execution of the thermal management algorithm and the implementation of the data exchange through wireless Ethernet network in the form of different communication channels. The presented network and IoT services tend to increase the usability of the heater thermal management unit in discussion. The presented configuration is quite modular, easy

to use, robust, and can be easily adapted for various experimental equipment. As a future development, different control algorithms and mechanisms will be developed, tested and compared in order to increase the overall performance of the substrate heater.

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