

Study and development of a flexible airflow sensor based on screen printing techniques

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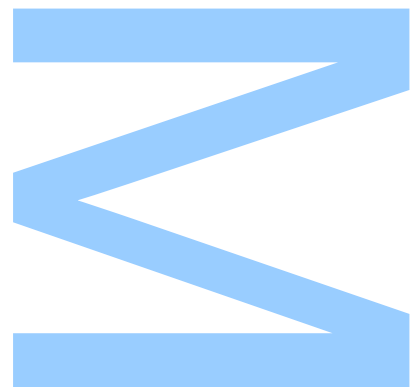
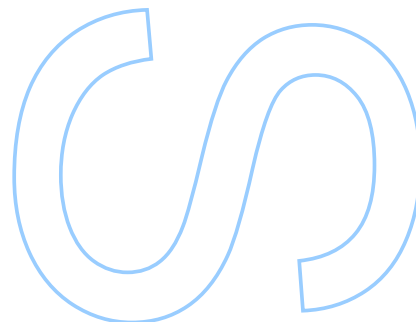
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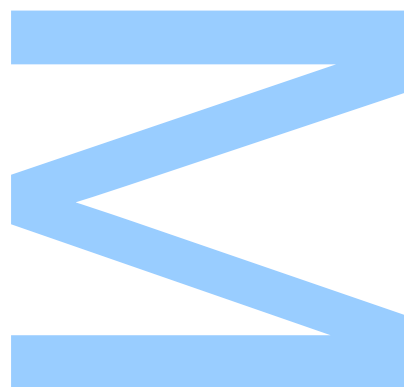
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Todas as correções determinadas
pelo júri, e só essas, foram efetuadas.

O Presidente do Júri,

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Resumo

A mudança de paradigma em setores industriais que trabalham com diversos fluídos, como o ar e a água, está relacionada com um aumento da procura por dispositivos de monitorização e controlo de fluxo. Como resultado, este panorama provocou um impacto nas projeções de mercado mundiais, que prevêem uma Taxa de Crescimento Anual Composta (CAGR) de 6,14% apenas para o mercado de sensores de fluxo, durante o período de previsão 2019-2024.

Dadas estas exigências, o presente trabalho focou-se no desenvolvimento de um sensor de fluxo de ar fabricado por técnicas de serigrafia. Este dispositivo vai ao encontro das necessidades de mercado, apresentando propriedades de transparência e flexibilidade, sendo produzido por técnicas passíveis de serem implementadas na produção industrial. O sensor proposto baseou-se em considerações térmicas, tendo a configuração de *hot-film* como princípio de funcionamento.

As geometrias da resistência e os materiais de impressão foram as componentes do sensor alteradas durante a produção do dispositivo, de forma a obter um desempenho ótimo. De um grupo de designs e materiais testados (níquel, carbono e duas tintas de prata), os resultados mais promissores para a aplicação desejada foram obtidos para um design em serpentina impresso com as duas tintas de prata, Ag-I e Ag-II. Um estudo adicional de um processo de *annealing* foi efetuado, com o objetivo de melhorar o comportamento termorresistivo. As impressões com Ag-I apresentaram os melhores resultados devido a um aumento em mais de 50% da sua resistência com este processo, um efeito que contribuiu diretamente para a melhoria da sensibilidade do sensor. O correspondente coeficiente de temperatura (TCR) medido para as amostras em questão variou entre 0,0058 e 0,0060 com um erro de 1,6%.

Para as medições de fluxo, os sensores desenvolvidos foram agregados a um controlador de corrente, uma vez que foi escolhida uma operação em corrente constante, e um tubo de vento capaz de operar no intervalo de velocidades entre 0,569 m/s e 2,506 m/s foi projetado. O modelo de sensor foi testado para diferentes parâmetros, como a resistência do dispositivo, a corrente aplicada e a posição no interior do tubo. O comportamento com a raiz-quadrada típico deste tipo de sensores foi verificado para os sensores testados, evidenciando uma maior sensibilidade para baixas taxas de fluxo. Dos sensores estudados, a melhor sensibilidade foi

obtida com o sensor moldado ao tubo, para uma maior resistência ($145,9 \, \Omega$) e para uma maior corrente aplicada (140 mA). Os valores do respetivo parâmetro b , resultante da linearização e relacionado com a sensibilidade, variaram entre 1,69 e 1,85 com um erro de 3,0%. Para além disso, os respetivos resultados de desvio apresentaram uma variação máxima de 15,5%.

O desempenho dos sensores de fluxo desenvolvidos evidenciou que o dispositivo proposto apresenta características apropriadas para aplicações onde são exigidos dispositivos robustos com implementações simples, sem a necessidade de uma elevada precisão de medida. Ainda, os métodos de fabricação envolvidos oferecem a possibilidade de uma produção em larga escala (usando técnicas rolo-a-rolo), com uma redução dos custos tipicamente associados ao desenvolvimento destes dispositivos.

Palavras-chave: deteção de fluxo de ar; sensor de fluxo térmico; sensor resistivo; *hot-film*; eletrónica flexível; eletrónica impressa; serigrafia

Abstract

The global industry on sectors that work with different fluids, such as air and water, is facing a new paradigm that results on an increasing demand for devices for flow monitoring and control. As a result, this situation triggered an impact in the worldwide market projections, that predict a Compound Annual Growth Rate (CAGR) up to 6.14% only for the flow sensors market, over the forecast period 2019-2024.

Focused on these necessities, this work relied on the development of an airflow sensor fabricated by screen printing techniques that meets the market needs, with transparent and flexible properties, and manufactured with methods capable of being scalable for an industrial production. The proposed sensor was based on thermal considerations and used the hot-film configuration as working principle.

The resistor geometries and the printing materials were the sensor components changed during the device production in order to obtain an optimum performance. From a group of tested designs and materials (nickel, carbon and two silver inks), it was a serpentine design printed with both silver inks, Ag-I and Ag-II, that presented the most promising results for the desired application. A further study was performed with an annealing process to improve the thermoresistive behavior. The Ag-I printings presented the best results due to a resistance increase higher than 50% with the annealing process, an outcome that directly contributed to an improvement of the sensor's sensitivity. The correspondent temperature coefficient of resistance (TCR) measured for the studied samples varied between 0.0058 and 0.0060 with an error of 1.6%.

Towards the flow measurements testing, the developed sensors were assembled to a current controller, due to the chosen operation mode at constant current, and a wind pipe capable to work on the velocities range between 0.569 m/s and 2.506 m/s was projected. The model of the sensor was tested for different parameters, such as the device resistance, the applied current and the position in the interior of the pipe. The square-root behavior characteristic of this type of sensors was verified for the tested sensors, evidencing a higher sensitivity for lower rates. From the studied sensors, the best sensitivity results were obtained for a higher resistance (145.9 Ω) and higher applied current (140 mA), with the sensor molded to the pipe. The values of the respective parameter b from the linearization related to the sensitivity varied between 1.69 and

1.85 with an error of 3.0%. Moreover, the respective deviation results presented a maximum variation of 15.5%.

The performance of the developed flow sensors proved that the proposed device has appropriate characteristics for applications where robust devices with easy implementations are required, without the need for a high measurement precision. Furthermore, the fabrication methods involved offer the possibility for a production at large scale (using roll-to-roll techniques), with a reduction of the costs usually associated to the development of these devices.

Keywords: airflow detection; thermal flow sensor; resistive sensor; hot-film; flexible electronics; printed electronics; screen printing

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Abbreviations

CCA Constant Current Anemometer

CTA Constant Temperature Anemometer

HVAC Heating, Ventilation and Air-Conditioning

IR Infrared

PCB Printed Circuit Board

PEN Polyethylene Naphthalate

PET Polyethylene Terephthalate

PI Polyimide

SEM Scanning Electron Microscopy

TCR Temperature Coefficient of Resistance

Symbols

α	Temperature coefficient of resistance	$^{\circ}\text{C}^{-1}$
δ	Hydrodynamic boundary layer thickness	m
δ_{th}	Thermal boundary layer thickness	m
ν	Kinematic viscosity	m^2s^{-1}
ρ	Resistivity	Ωm
ρ_0	Material intrinsic resistivity	Ωm
A	Surface area	m^2
C	Heat capacity	$\text{J}^{\circ}\text{C}^{-1}$
h	Convection heat-transfer coefficient	$\text{Wm}^{-2}\text{C}^{-1}$
I	Electrical current	A
Q	Thermal power	J
R	Electrical resistance	Ω
R_0	Electrical resistance at temperature T_0	Ω
R_s	Sheet resistance	$\Omega\Box^{-1}$
T_0	Reference temperature	$^{\circ}\text{C}$
T_{∞}	Fluid temperature	$^{\circ}\text{C}$
T_w	Surface temperature	$^{\circ}\text{C}$

u	Fluid stream velocity	ms^{-1}
V	Electrical voltage	V
W	Electrical power	W

Chapter 1

Introduction

1.1 Motivation

Flow control is widely demanded in industrial, automotive, domestic, environmental, and medical sectors. One of the main applications of flow control is related to the optimization of the energy consumption in a group of flow systems, such as heating, ventilation and air conditioning (HVAC) systems [1, 2]. A study published by Pérez-Lombard et al. [3] showed that the energy consumption of buildings in developed countries comprises 20–40% of its total energy demand, where these HVAC systems, which nowadays are considered a fundamental asset, account for almost half of that energy. The implementation of flow control devices in these systems can give valuable insights in order to reduce the amount of energy use, therefore improving the energy efficiency and decreasing the environmental impact. Furthermore, the monitoring of flow parameters can be fundamental on applications that involve specific characteristics for particular purposes of use. Respiratory and blood flow monitoring [4, 5], spirometer devices [6] or artificial ventilation [7] are some of the main areas in the medical field that require flow sensing for diagnostic purposes. Other applications of interest are related to the control of harsh environments, as the ones that can be found, for example, in nuclear power plants [8] and micro-reactors [9]. For all the applications mentioned, the determination of flow velocity, air rate and flow direction requires devices capable to maintain its structural stability under a

wide range of velocities, while offering a high degree of accuracy, fast response and low power consumption [10].

The market projections for the incoming years support the impact of flow sensors in the nowadays market, as well as in a near future. A report published by Grand View in 2015 suggests a market valuation of around USD 7.48 billion on the incoming year of 2020, with a major growth contribution of the automotive industry over the period 2012-2020 [11, 12]. New projections estimate that the flow sensors market should maintain the growing trend and reach USD 11.05 billion by 2025, mainly due to a robust growth of the water and wastewater segment resultant of the rapid urbanization expected over the forecast period 2014-2025, in particular in Asia-Pacific [13]. Markets and Markets also estimated a market growth from USD 7.2 billion in 2018 to a maximum of USD 12.7 billion by 2027, with the water and wastewater segment again accounting for the largest share of the market [14]. The flow sensors market is highly competitive, with the participation of important companies as Siemens AG, Emerson Electric AG, First Sensor AG and others [15]. The major restraints to the market growth mentioned by these publications are related to the high initial costs required to fabricate flow sensors, the costs associated to the replacement of traditional flow meters with smarter sensors and the price-sensitive market.

1.2 Flow sensors

Flow sensors can be classified in non-thermal and thermal sensors [16]. Non-thermal flow sensors are based on a specific mechanical working principle, such as the piezoresistive effect that relies in the dragging force that acts on the sensor body due to the passage of the fluid [17]. However, these types of sensors are usually composed by moving parts that require a more complex production and are susceptible to mechanical degradation, which limits the long-term measurement [18]. On the other hand, thermal flow sensors comprise heat sources and temperature sensors, presenting a structural and electronic simplicity - due to the lack of moving parts or complex transducers - that makes them the prime approach explored by industries and reported in the literature [16, 19].

1.2.1 Thermal flow sensors

Thermal flow sensors can be used in a wide variety of flow conditions and present minimal disturbance to the flow itself [20]. In practice, thermal flow sensors can be divided in three types: hot-wire and hot-film, usually designated as anemometers (figure 1.1a), calorimetric (figure 1.1b) and time-of-flight sensors (figure 1.1c). All of these rely on the production of heat by Joule effect and in the occurrence of heat exchanges induced by forced convection phenomena, that are consequently detected by sensing elements.

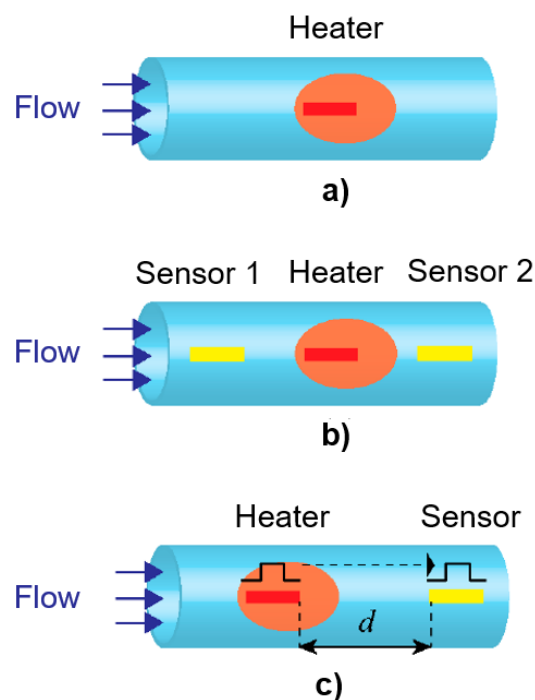


Figure 1.1: Schematic representation of the possible configurations of thermal flow sensors, a) anemometer, b) calorimetric and c) time-of-flight (from Balakrishnan et al. [21]).

Anemometers are the most simple thermal-based flow sensors [22]. This type of sensor is composed by a heater, that acts simultaneously as a heat source and temperature sensor, and eventually a room temperature sensor for monitoring purposes. The basic principle is to directly exploit the cooling effect on the heater, that results in flow-dependent changes on its temperature; hence, from the measurement of these changes it is possible to acquire information about the flow velocity. Generally, the characteristic output of anemometers follows a square-root behavior and a large measurement range is possible [16, 23]. However, they cannot detect the

flow direction, which can be a required information in specific applications, and have a fragile design [22, 24].

Calorimetric sensors principle is based in the flow induced asymmetry of the temperature profile. Typical configuration consists in temperature sensors arranged around a heater at a defined distance. Therefore, when heat is transferred from a heated element to a fluid and is transported by forced convection, a change in the temperature distribution is responsible for modifying the temperature in the sensing elements and a consequent flow-dependent temperature difference is detected between the upstream and downstream zones. The measurement of this difference allows the determination of the flow velocity. In the absence of flow, the temperature profile is expected to be symmetrical and a zero temperature difference is detected. A high sensitivity and good resolution can be obtained for a small flow range with this approach [16, 25, 26]. Also, calorimetric sensors are often preferred due to its bidirectional flow measurement, easy zero-adjusting and faster response to flow rate changes [26, 27]. A major drawback is the typical high demand of heating power associated to these devices [28].

Time-of-flight sensors measure the time passage of a heat pulse that is generated in a heater and detected by a downstream temperature sensor [16, 22]. Since the distance between the heater and sensor is previously known, the measured propagation time is dependent of the flow velocity. Usually, this type of sensor is directed to industrial applications that require measurements over a long period of time [21]. This approach is seldom mentioned in published papers, in contrast to the other two principles, that are most commonly considered in the development of flow sensors.

Thermal flow sensors can be also classified according to their physical transduction principle. Thermoresistive sensors exploit the strong dependence between the electrical resistance of a material and their temperature, which is expressed by the temperature coefficient of resistance (TCR) [16, 29]. Metals are the materials usually chosen as heaters and temperature sensors, due to their linear temperature characteristics and interesting TCR values [22, 27, 30, 31]. Semiconductors, on the other hand, present even higher TCR values, which can improve sensitivity; however, semiconductor thermistors feature non-linear characteristics - an exponential behavior -, which represents a challenge for their application as temperature sensors [21, 22]. Another set of sensors relies on thermoelectric materials and, therefore, on the Seebeck effect. This effect is known for providing good sensitivities, as well as a low offset and drift [22, 32]. These

sensors employ thermocouples - two different metals that are joined together in a closed circuit and evidence a temperature difference between its electrical junctions - connected in series (this configuration is denominated as thermopiles), responsible for generating a temperature-dependent voltage signal due to the thermoelectric effect [21]. The major drawback is that thermopiles cannot operate as heat sources [16, 22]. Thermocapacitive, thermoelectronic, pyroelectric, and frequency analog sensors are other classifications that can be found in the literature [16, 29, 33].

1.2.2 State of the art of thermal flow sensors

Hot-wire anemometers have been used since the late 1800s, mainly for fluid mechanics measurements [34]. Conventional hot-wire anemometers are composed of a small sized wire sensor (with a cylindrical structure), typically made of platinum, support prongs that elevate the wire and a probe body. Over the last years, the sensors sector in general benefited with the expansion of micro and nanotechnologies, which led to an increase of papers reporting the development of flow sensors with reduced dimensions, great performances and more intuitive electronics.

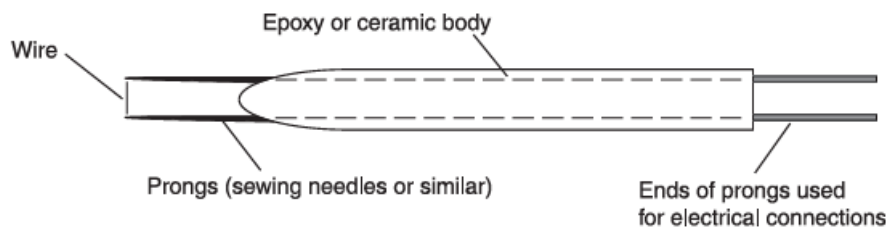


Figure 1.2: Schematic of a conventional hot-wire anemometer (from [35]).

Silicon-based flow sensors

The first thermal flow sensor based on silicon technologies was published in 1974 by van Putten and Middelhoek [36], by replacing the wire with a deposited thin film on a substrate. Since then, flow sensors have benefited with the evolution of silicon-based technologies. Jiang et al. reported a micromachined hot-wire flow sensor similar to conventional anemometers [34], consisting of a polysilicon sensing wire supported by two parallel support prongs of the same material, and tests showed that the performance can be improved when silicon-based tech-

niques are considered. In practice, due to their small geometry, the biggest advantages that micromachined sensors possess are the low energy consumption, high spatial resolution, high sensitivity and fast response [16, 22, 25].

The major advantage provided by silicon substrates is related to its capacity to sustain high temperature processes, such as microfabrication deposition techniques. However, due to the high thermal conductivity associated to these substrates, it becomes necessary to control the conduction of heat to the substrate, since it diminishes the sensor sensitivity and increases the response time [35, 37]. Moreover, the power consumption required by the sensor is reduced when the conduction of heat in the substrate is minimized [38]. Therefore, structures that provide thermal isolation are usually fabricated in order to improve the sensor's performance. This isolation can be accomplished with free standing structures, cavities, or with membranes and bridges made from insulation materials, such as silicon nitride [20, 26, 37].

Table 1.1: Overview of silicon-based flow sensors reported in literature.

Author	Configuration	Substrate	Materials	Flow range
Cubukcu et al. [38]	Calorimetric	Silicon	Amorphous Ge	0 - 5 m/s
Maily et al. [30]	Hot-film	Silicon	Platinum	0 - 20 m/s
Shin and Besser [25]	Calorimetric	Pyrex	Aluminum	0 - 20 sccm

The main disadvantage of silicon substrates is related to its mechanical rigidity, since it hinders the system integration. Furthermore, the rapid development of new applications demands devices provided with mechanical flexibility.

Flexible flow sensors

Nowadays, sensors made on flexible substrates are seen as the key of electronic innovation, provided that all of its components bend to some degree without losing their function [39, 40]. These substrates have multiple advantages when compared to silicon substrates, such as lighter weight, lower cost, and robustness, in addition to a better flexibility to absorb mechanical stress [40].

The first report of flexible electronics dates to the 1960s, when flexible solar cell arrays were made by thinning silicon wafer cells to approximately 100 μm followed by an assembling on a

plastic substrate to provide flexibility [39]. Currently, different types of flexible substrates are available, and two different approaches of fabrication can be considered: transfer and bonding of completed circuits into a flexible substrate or direct fabrication of the circuits on the substrate [39]. The desired requirements for flexible substrates are optical properties (optically clear substrates are required in applications such as the ones involving displays), thermal and thermochemical properties (working temperature must be compatible with the maximum fabrication temperature), chemical properties (the substrate should be inert against process chemicals and should not release contaminants), mechanical properties, and electrical and magnetic properties [39].

The most common flexible substrates used are polymers since they are highly flexible and can be inexpensive. However, typical polymer films can shrink by heating and cooling cycles, due to their thermal instability, and require prolonged annealing processes to minimize shrinkage [39]. Thin sheets of metal and glass can also be flexible and provide a good alternative to polymer foils given their ability to withstand high temperatures [41].

Polyethylene terephthalate (PET), polyethylene naphthalate (PEN) and polyimide (PI) are the most known polymer substrates due to their small coefficient of thermal expansion, high elastic moduli, and acceptable resistance to process chemicals [39].

Usually, polyimide substrates are the preferred choice for silicon-based applications, due to their capacity to withstand high temperature processes, in addition to their excellent chemical resistance and thermal stability [21]. Furthermore, the low thermal conductivity exhibited by these substrates minimizes the losses that can occur by heat conduction, which represents an improvement when compared to silicon substrates and enhances the sensor sensitivity [42].

Table 1.2: Overview of flexible flow sensors reported in literature.

Author	Configuration	Substrate	Materials	Flow range
Shikida et al. [43]	Hot-film	PI	Au/Cr	0 - 3000 m ³ /h
Tan et al. [10]	Hot-film	PI	Au/Cr	1,18 - 6,48 m/s

PCB-based flow sensors

Another type of sensors arises from Printed Circuit Board (PCB) technologies. Multiple advantages can be enumerated especially when these are combined with silicon-based techniques, such as the elimination of the need for wire bonding, the low cost production, as well as the very low heat dissipation characteristic of the materials used as substrates, providing a highly effective thermal isolation without the need for additional structures [26]. The conductive material is usually a copper foil and the most common substrate is glass reinforced epoxy (FR4) [29]. Flexible substrates are also available, being polyimide foil, in particular a specific foil named Kapton, the preferred one, since it exhibits a lower thermal conductivity compared to FR4 [44].

Table 1.3: Overview of PCB-based flow sensors reported in literature.

Author	Configuration	Substrate	Materials	Flow range
Glatzl et al. [2]	Calorimetric	FR4 epoxy	Copper	0 - 6 m/s
Petropoulos et al. [44]	Hot-film	Kapton	Copper and Platinum	0 - 10 SLPM

Printed flow sensors

The previous topics focused on silicon-based techniques. However, over the last years, this technology has evolved into a complex process, reaching an ultimate fine resolution of 13 nm, and demanding a huge investment for mass production from the companies of the field, which is often an unsustainable requirement [45]. Besides that, the base material and the packaging issues limit the application range [46]. In order to overcome these difficulties, other techniques must be explored, such as printing technologies.

Printed electronics is a type of technology dated back prior to the 1950s that is based on the adaptation of conventional printing techniques as the means to manufacture electronics devices and systems [45]. In more recent decades, great advances had been made due to the development and maturity of nanomaterials [41]. Several materials at a nanoscale can be easily formulated into suitable inks by dispersing the fine powders of nanomaterials either in a solvent or in an aqueous solution, giving the printed patterns and structures the conducting, semiconducting or dielectric properties of interest [41].

In printed technologies, the functional materials of a device or system are directly printed as patterns onto the substrate through an additive fabrication process, in contrast to the numerous complex steps usually required in the development of silicon-based devices [41]. Thus, this technique offers a very simple process to produce integrated electronic systems, significantly lowering the production costs and often being environmentally friendly, since there is no need to use high temperature processes and/or high vacuum processes which results in low energy consumption and reduction of material waste [39, 41, 47].

The major drawbacks of printing electronics concern the lack of accuracy and resolution capability when compared to lithography techniques, as well as the absence of surface smoothness often obtained from silicon-based deposition methods [41]. Therefore, it is necessary to find the applications that can benefit from the use of printed electronics; when the aim is to get cost-effective sensors without the requirement of higher performances, printing technologies are the ideal choice since large size and mass fabrication at low costs can be achieved, providing ideal aspects for industrial applications [41].

It is common to find reports that merge printing techniques with flexible substrates, especially when the focus is the fabrication of robust, flexible and cost-effective sensors.

Table 1.4: Overview of printed flow sensors reported in literature.

Author	Configuration	Substrate	Materials	Flow range
Moschos et al. [46]	Calorimetric	PET	Silver and carbon inks	0 - 25 SLPM
Offenzeller et al. [17]	Time-of-flight	Glass	Silver and carbon inks	20 -70 $\mu\text{L}/\text{min}$

1.2.3 Concepts of thermal flow sensors

In order to understand how thermal flow sensors behave, this section presents the basic thermal and electrical principles behind their operation. For the particular case of anemometers, the characteristic response expected by theoretical considerations and the possible operation principles that arise from it are also discussed.

Thermal and electrical principles

As mentioned before, thermal flow sensors are developed with a basis on the Joule heating effect. This effect describes the phenomenon where heat is generated from the flow of an electric current through a resistor. The electrical power (W) dissipated by the resistor due to this effect can be expressed by a well-known expression

$$W = I^2 R = \frac{V^2}{R} \quad (1.1)$$

where I is the current, R is the resistance and V is the potential difference between two points.

Furthermore, a given material resistance shows a temperature-dependent response that arises from the fact that atoms from the material start to vibrate when heat is applied to it. This behavior is the key to acquire the characteristic output of thermoresistive flow sensors. The desired operation of the sensors is based on the linear variation of resistance with temperature, following the expression

$$R = R_0 [1 + \alpha (T - T_0)] \quad (1.2)$$

where R_0 is the resistance at a reference temperature T_0 and α [$^{\circ}\text{C}^{-1}$] is defined as the temperature coefficient of resistance (TCR), an intrinsic property of the material. In a conductor, for example, the vibration of the atomic structure causes collisions between the large number of free electrons (responsible for the flow of current), which in turn increases the resistance. As a result, the TCR must have a positive value.

The overheat ratio can be defined from the previous expression as

$$\frac{R - R_0}{R_0} = \alpha (T - T_0) \quad (1.3)$$

representing another form to describe the temperature changes through the resistance variation of the material and vice-versa.

For sensors based in thermal transduction principles, it is thus required a high resistance variation within a fixed temperature range in order to guarantee a high sensitivity. Furthermore, these sensors should evidence a long-term operation and provide reliable results. Therefore, some conditions must be satisfied when choosing the basis material for the thermal flow sensor,

such as a high value of TCR, stability at high temperatures and good resistance to oxidation [35].

These considerations are valid for all types of thermal flow sensors that include thermoresistive elements. The characteristic response, however, differs between them. Since this work focus on the development of a hot-film sensor, it is relevant to understand how the heat transfer theory predicts the output behavior of this type of flow sensor.

Anemometers behavior predicted by heat transfer principles

The energy balance of an anemometer can be written as [35]

$$C \frac{dT}{dt} = W - Q \quad (1.4)$$

where C is the heat capacity of the resistor, W is the electrical power supplied to the resistor, and Q is the thermal power exchanged between the resistor and the surroundings. For flow measurements, heat exchanges phenomena are, as mentioned before, predominantly due to forced convection. Therefore, conduction and radiation effects are negligible; in addition, free convection phenomena can be ignored with the increase of flow velocity [35, 48].

In a mathematical perspective, the overall effect of convection can be expressed by the Newton's law of cooling,

$$Q = hA(T_w - T_\infty) \quad (1.5)$$

where h [$\text{W m}^{-2} \text{ }^\circ\text{C}^{-1}$] (when heat flux is given in Watts) is defined as the convection heat-transfer coefficient, A is the surface area of the heating device, T_w is the temperature at the surface of the heating device and T_∞ is the temperature of the fluid. The heat-transfer coefficient shows a dependency on the viscosity of the fluid, since it influences the velocity profile and the energy-transfer rate in the region near the surface of the heating device [49].

From the conjugation of the Newton's law of cooling with the velocity of the fluid stream, u , as well as with the steady-state energy balance of the hot-wire, it is possible to obtain the following expression, known as the King's law [35],

$$I^2 R = \frac{V^2}{R} = (a + b\sqrt{u})(T_w - T_\infty). \quad (1.6)$$

Thus, the characteristic response of an anemometer is expected to follow a square root behavior according to the flow velocity. An important aspect to retain from this equation is that each sensor requires an extensive calibration, that must be repeated every time the flow temperature changes [23]. In fact, the coefficients a and b are constants determined by the calibration process. Furthermore, a represents the heat transferred due to conduction to the support and to the fluid, whereas b is related to the convection effect [23]. Therefore, the sensor must be designed in order to minimize a with respect to b .

Moreover, the non-linearity of this calibration curve simultaneously reflects the main advantage and disadvantage of anemometers in terms of its sensitivity; although a high sensitivity can be achieved for low flow rates, a substantial reduction in the sensitivity occurs with the increase of the flow rate [50].

Notice that the previous expression results from mathematical manipulations involving the geometry of a conventional hot-wire anemometer, which consider that the heater length is infinitely long compared to its ratio [35, 43]. For a more convenient result, the equation 1.6 can be derived with a dependency in u^n , where the exponent n depends on the geometry of the anemometer and can take different values according to the operation mode or type of flow, differing from the square-root behavior. Jiang et al. [34] tested a micromachined hot-wire for both constant current and constant temperature operations (that will be discussed in the next topic) and obtained exponents of 1 and 0.6, respectively. On the other hand, Mailly et al. [30] reported an exponent of 0.45 for a laminar flow regime, whereas for turbulent flow the value obtained was 0.8.

Principles of operation

The result from King's law can also be related to equation 1.2 and, therefore, a dependency in the resistance also arises. From the consequent result, it is patent that the stream flow velocity can be related to one single variable. If the electrical current is held constant, the presence of a fluid flow disturbs the initial equilibrium state, causing temperature changes in the resistor and, consequently, in its electrical resistance. This operation mode is denominated as constant current anemometer (CCA). In contrast, if the electrical resistance of the resistor is held constant, its electrical current or voltage varies according to the flow velocity. In this case, the mode is designated as constant temperature anemometer (CTA).

For operations in the **CCA** mode, the cooling effect promotes a decrease in the sensor's electrical resistance whenever the material **TCR** is positive. Following Ohm's law, the potential difference across the resistor also decreases and its measurement gives an insight about the velocity behavior. In this operation, a high sensitivity is feasible only for a limited flow range [22].

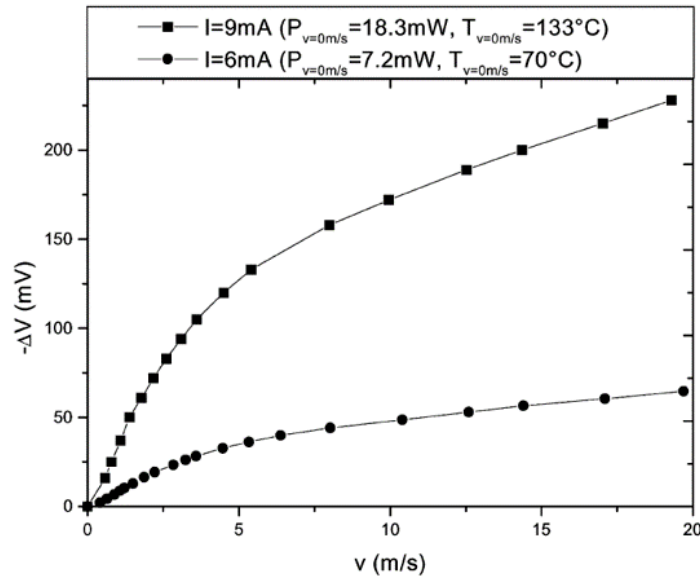


Figure 1.3: Example of the characteristic output of a hot-film sensor operating under two different values of constant current. The results show a decrease in the potential difference across the sensor resistor with the increase of flow velocity, and from the slopes of the curves it can be seen that a higher value of applied current enhances the sensitivity of the sensor (from Mailly et al. [30]).

When operating under the **CTA** mode, the sensor is connected to an external controller, responsible to establish a constant temperature between the heater and its surroundings [35]. In this case, the tendency of the sensor to cool under the effect of a flow and, therefore, to decrease its resistance (again, for a positive **TCR**), causes the controller to apply more current or voltage to maintain the desired resistance. The respective signal can be used as an output quantity, since it is dependent of the flow velocity.

Concepts of boundary layer

One of the most important concepts in fluid dynamics is designated as hydrodynamic boundary layer and was firstly defined by Ludwig Prandtl in 1905 [52]. The basis of this concept is that when a fluid flows along a wall, a region with large gradients in the flow velocity emerges, and

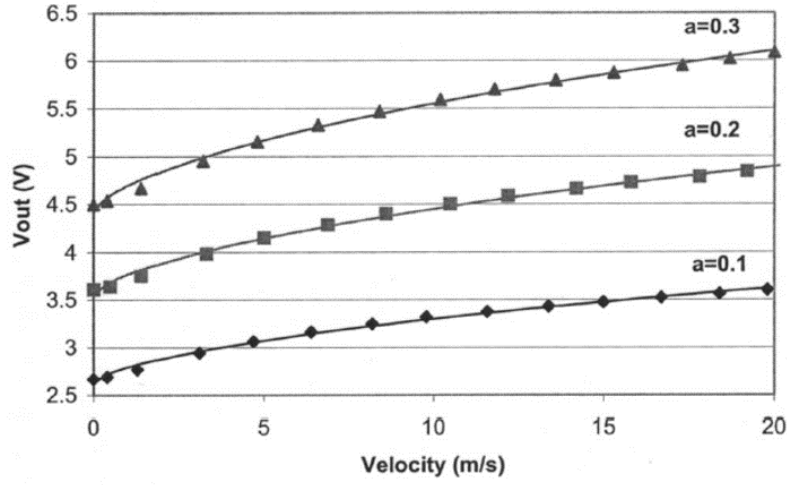


Figure 1.4: Example of the characteristic output for a hot-wire sensor operating under different values of constant temperature. The curves are distinguished by the correspondent overheating ratio. An increase of flow velocity is represented by an increase of potential difference in the sensor resistor (from Chen et al. [51]).

the boundary layer is defined as the thin layer where this region is confined. Inside the boundary layer the fluid is always dominated by viscous effects (even if the fluid is characterized by a small value of viscosity), whereas in the outside region the fluid can be considered inviscid [49].

The boundary layer in conditions of steady state for a laminar and incompressible flow can be deduced for the particular geometry regarding an anemometer. From the incompressible Navier-Stokes equation, the boundary layer thickness is given by the following expression [23]

$$\delta \propto \sqrt{\frac{\nu x}{u}} \quad (1.7)$$

where ν stands as the kinematic viscosity, x is defined as the distance from the leading edge of the wall, and u is the free stream velocity, i.e., the velocity far from the sensor wall.

It is also important to notice that, for most fluids in the presence of heating devices, an additional thermal boundary layer builds up due to temperature gradients [23]. This is the case of flow sensors based on thermal principles, that comprise heat sources and temperature sensors. As in the case of the hydrodynamic boundary layer, the majority of heat transfer phenomena are confined within the thermal boundary layer, which thickness is given by [49]

$$\delta_{th} \propto \frac{\delta}{Pr^{\frac{1}{3}}} \left[1 - \left(\frac{x_0}{x} \right)^{\frac{3}{4}} \right] \quad (1.8)$$

where δ is the thickness of the hydrodynamic boundary layer, Pr is the specific Prandtl number of the fluid, x_0 is the distance of the heating device in relation to the leading edge of the wall, and x is the distance from the heating device.

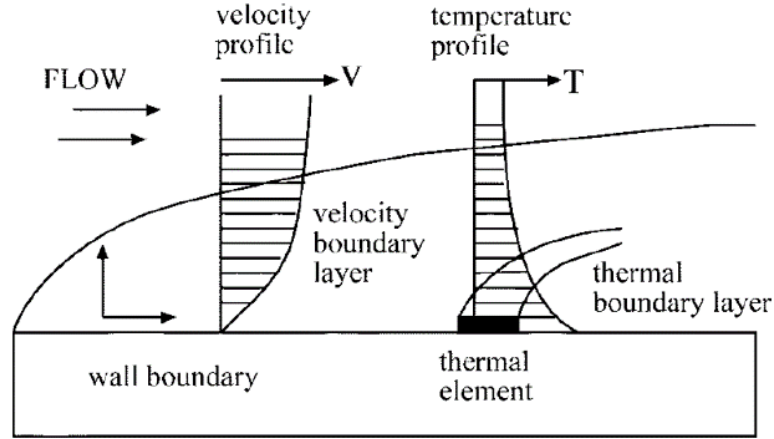


Figure 1.5: Schematic representation of the hydrodynamic boundary layer and thermal boundary layer. The vertical position where the boundary layer ends is usually chosen as the coordinate point where the velocity/temperature becomes 99 percent of the free stream value (from Liu et al. [20]).

In an anemometer, the convective heat transport is dominated by the thermal boundary layer. As a matter of fact, since the convection phenomenon is related to the heat conduction through the layer of fluid adjacent to the heating surface, from the Fourier's law of heat conduction [23, 49],

$$Q \propto \frac{\Delta T}{\delta_{th}} \propto \sqrt{u} \Delta T. \quad (1.9)$$

This result collapses to the one in equation 1.6. For thermal flow sensors with more than one element, the previous square-root behavior is still valid whenever the thickness of the boundary layer is much smaller than the distance between the elements of the structure [23]. This guarantees that the heat loss is dominated by heat diffusion along the boundary layer whilst heat diffusion directly between the elements is less significant. In contrast, when the heat transport is not dominated by the boundary layer, the output does not follow the same square-root behavior [23]. This is, in fact, the principle behind calorimetric sensors.

1.3 Objectives

The work presented in this thesis was part of a project developed by CeNTI - Centre of Nanotechnology and Smart Materials, whose purpose was the creation of a smart flexible label for the monitoring of several parameters in industrial equipment and/or processes. One of these parameters focused on the air control, in particular the measurement of flow velocity. By bearing in mind the proposed objective and the respective required conditions, this thesis main goal was the development of a screen printed flexible hot-film airflow sensor by taking advantage of the CeNTI resources.

Chapter 2

Device development: fabrication and characterization techniques

This chapter explores the principles behind the fabrication and characterization techniques involved in the production of the airflow sensor. The first section describes the fundamental properties of the screen printing technique, the printing method chosen to manufacture the resistors integrated in the sensor. In the second section, the thermal and electrical characterizations prosecuted to evaluate the performance of the manufactured resistors are explored, as well as the information that can be provided by scanning electron microscopy.

2.1 Fabrication techniques

2.1.1 Screen printing

Printing techniques can be divided into two categories, whether a mask is employed or not [41]. Screen printing is one of the techniques that relies on a mask, being the most simple printing technique and the most widely used at an industrial level [41]. When compared with other printing methods, screen printing can obtain thick films more easily and has an easier way to control the ink paste movements during the printing operation, in addition to the high aspect ratio of printed films [41, 45]. Besides that, it can be used in a wide range of substrates and, therefore, a very large variety of ink pastes types are available [47]. This technology is also easily adapted

to roll-to-roll techniques allowing the mass production of devices and systems. In contrast, the primary disadvantage is that the printable ink must have a high viscosity (above 10000 cP), which requires a treatment with additives that usually decrease the electrical properties of the materials [41]. Another disadvantage is its low resolution, which limits its usage in the development of certain types of electronic devices [41].

In this technique, a plate consisting on a screen mesh, composed of fabric threads, with a printing pattern of uniform thickness is used. The most important parameters of the screen mesh correspond to the number of fabric threads per centimeter and to the respective thread thickness. These aspects define the open screen area, which contribute for the thickness of the printed film [47]. Furthermore, the design pattern can be obtained by UV exposure of non-image and image areas covered by a screen emulsion; the film emulsion is hardened on all non-image areas whereas the image areas are not hardened and can be removed during developing [47]. The thickness of the stencil that defines the print image also has direct influence establishing the thickness of the deposited layer.

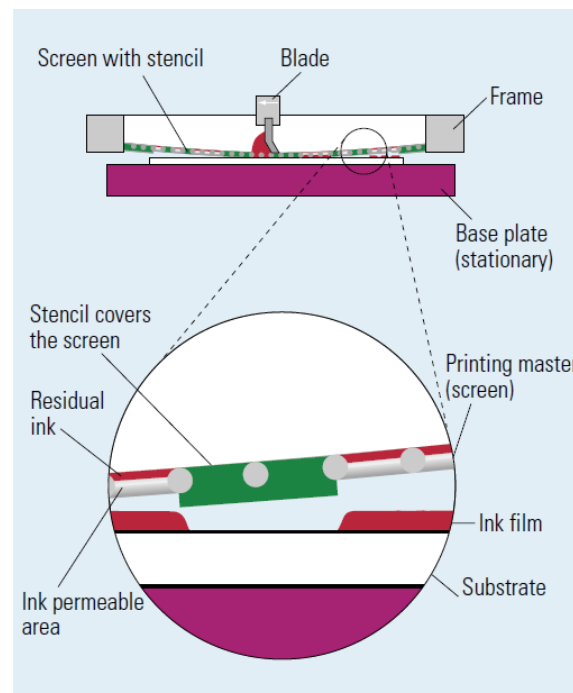


Figure 2.1: Schematic diagram of a screen printing deposition (from [47]).

During the printing process, a blade, usually named as “squeegee”, moves across the screen, applying pressure to the ink paste and forcing it to penetrate through the open mesh and to deposit onto the substrate placed under the screen [41, 47]. As a result, the exact pattern of the

screen is replicated by the printed ink onto the substrate. A schematic representation of how screen printing deposition is processed can be found in figure 2.1.

In practice, the screen printing technique can be divided in three distinct methods: the flat-to-flat method, where the printing plate and the substrate are both flat, and the ink is transferred onto the substrate by the movement of the squeegee; the flat-to-round method, that combines a directional synchronous movement between a flat printing plate and a rotating cylinder with a stationary squeegee responsible for transferring the ink onto the substrate; and the round-to-round method, where the printing plate is also cylindrical and moves synchronously with the printing substrate and the impression cylinder [47]. The rotary screen-printing technique, that makes use of the flat-to-round method, allows a high printing output at low cost, being suitable for mass production in large-scale, and is characterized by fast printings and a limited resolution [45].

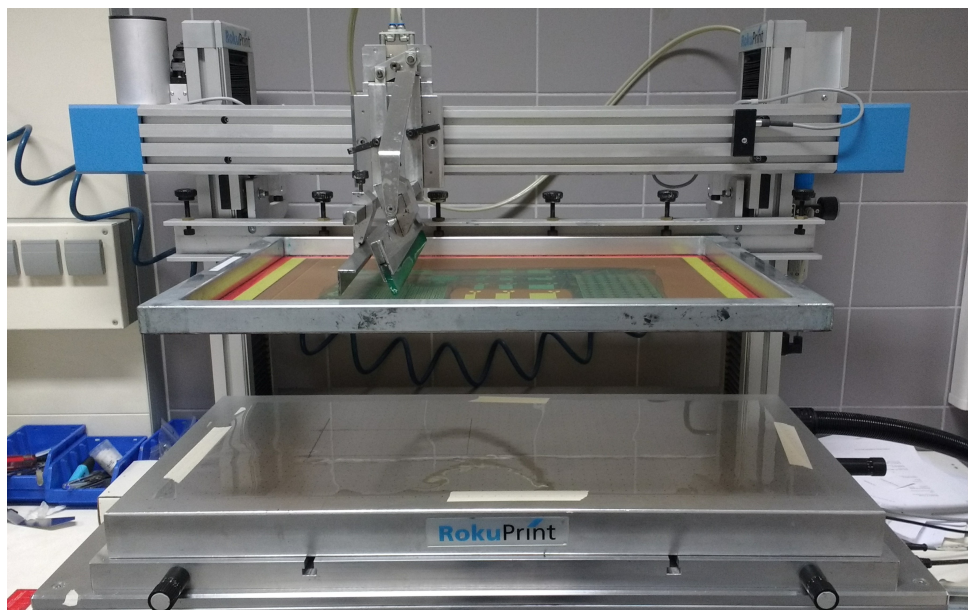


Figure 2.2: RokuPrint semi-automatic screen printing available at CeNTI installations.

In this thesis, the printings were performed with a RokuPrint screen printing that worked according to the flat-to-flat method, as can be seen in figure 2.2. When in operation, a blade is firstly responsible for spreading the ink along the mesh, and then a rubber squeegee applies pressure in the ink paste to deposit onto the substrate supported by a vacuum system. The distance between the mesh and the substrate during deposition as well as the pressure applied by the squeegee are parameters adjusted according to the ink to be used.

Usually, a screen mesh has a group of designs prepared to be printed. On many occasions, it is only intended to print a specific pattern, thus an aqueous emulsion can be used to obstruct the open area of the mesh pattern and preclude the passage of the ink paste through it.

Electronic properties of screen printing inks

One of the aspects to have in consideration during the fabrication of devices based in printed electronics techniques is related to changes that can occur between the properties of the ink and the printed structure. These changes are a direct consequence of chemical reactions that take place in the ink during printing, and drying and/or curing processes [41]. The manipulation of a number of variables such as ink material selection, as well as the parameters of pre-printing and post-printing processes is a common strategy to achieve the desired electronic properties.

Inks with certain electric properties must show compatibility with printing components and applications, in particular the printing method to be used and the substrate to be printed on. The viscosity of the ink is one of the most relevant parameters to take into account, since it is necessary to match the desired thickness of the printed layer with the ink viscosity; the more viscous, the thicker the layer will be [41]. These inks can be solvent based or aqueous based, property that also influences the printing. Concerning the substrate, one of the main properties to consider is the surface energy: a high surface energy substrate means the surface is hydrophilic and the printed ink will have strong adhesion, whereas a low surface energy means the surface is hydrophobic and the printed ink will have less lateral spread, improving the resolution [41].

Pre-printing processes can promote or prevent the spread of printed inks on the substrate in order to obtain the desired shape [45]. The principle behind these treatments is the change of the substrate surface energy. For example, plasma treatment of substrate surfaces is the most widely method used to create a hydrophilic surface and promote ink movements and adhesion [41, 45].

Post-printing processes can be either physical and/or chemical treatments done after the printing process in order to improve electrical properties of the printed materials. These treatments are responsible for the solidification of the ink and the removal of residual solvents or additives [41]. The drying treatment required after the printing is usually performed with an oven or by curing with a UV lamp. In some cases, high-temperature treatments are not suitable and other

specific treatments can be employed, such as laser curing or microwave curing [45].

2.2 Characterization techniques

In order to develop sensors with a high performance, it is necessary to explore the behavior of its components when under specific conditions. This section focus on the fundamental considerations behind the characterization processes required to study the response of the printed resistors.

2.2.1 Thermal and electronic characterization

For the presented work in particular, where print resistors that evidenced linear behaviors, as expressed in equation 1.2, were desired, it was convenient to study the relation between resistance and temperature. Therefore, this characterization was performed by heating the resistors in a hot-plate (ScanSci IKA C-MAG HS7) and measuring the consequent resistance value with a multimeter (Agilent 34410A). The respective setup is presented in figure 2.3.

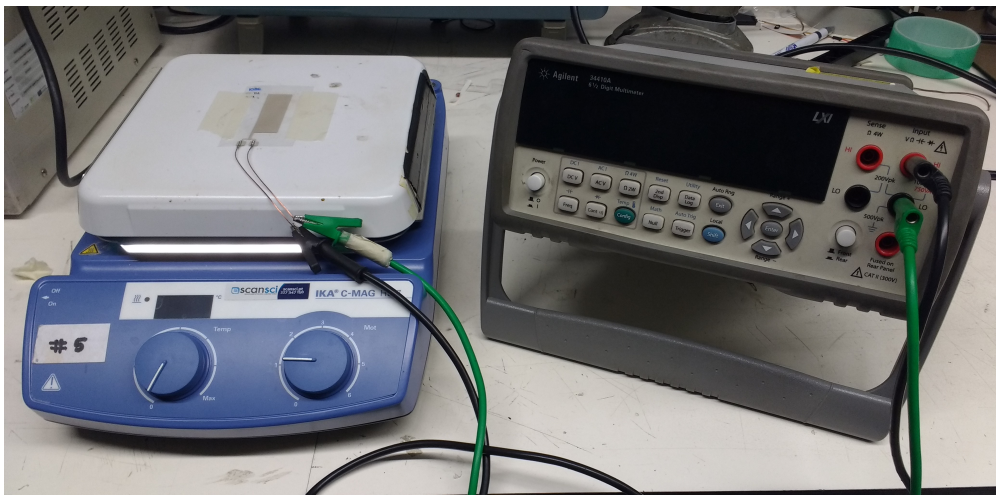


Figure 2.3: Experimental setup to measure the response of the resistance in function of an applied temperature.

Furthermore, to develop the flow sensor according to the hot-film principle, it was necessary to choose which of the operation modes would be applied. In this work, the option focused on an operation under constant current due to its implementation simplicity. Thus, for this condition, it was necessary to inspect how the resistor temperature would increase in relation to the room

temperature for different values of applied current. This characterization followed the apparatus shown in figure 2.4, where a power source (HQ Power Adjustable DC Power Supply) was responsible for supplying the desired current. It is important to bear in mind that this process did not guarantee a constant current; therefore, the final output merely provided an estimate insight of the overtemperatures associated to a range of current values.

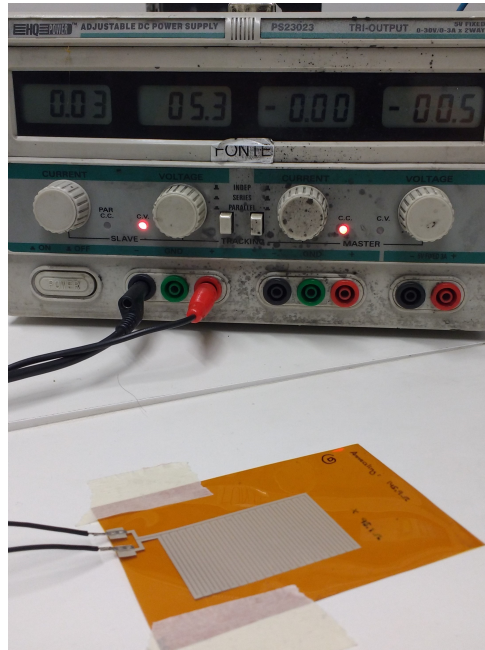


Figure 2.4: Experimental setup to measure the temperature behavior in the presence of a current flow along the resistor.

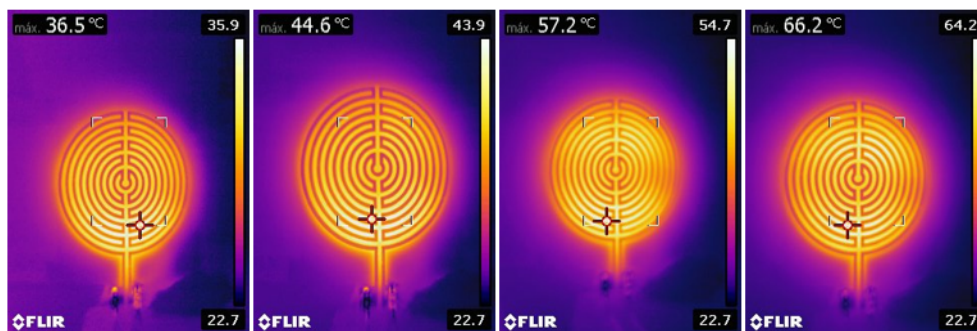


Figure 2.5: Thermographic images taken with the FLIR camera for a sample subjected to a heating process by Joule effect. It is possible to differentiate the temperature difference between the resistor and the substrate, as well as how the heat is distributed in the resistor.

The resultant temperature variations in the above characterization processes were measured by infrared (IR) thermography with a FLIR camera (FLIR E60). This thermography is based on the fact that bodies emit infrared radiation when its temperature is above absolute zero, being

this radiation dependent of the temperature. A set of thermographic images taken during the characterization of a sample subjected to different values of current are present in figure 2.5.

2.2.2 Scanning Electron Microscopy (SEM)

The Scanning Electron Microscopy (SEM) was the imaging technique chosen to visualize the printed samples at a micrometer scale. As in other similar techniques, a SEM microscope uses a beam of electrons to interact with the solid material (penetration depth of a few microns), resulting in a combination of produced signals that can give information about the topography and composition of the sample surface [53].



Figure 2.6: SEM microscope available at Faculty of Sciences of University of Porto.

This characterization process was performed to evaluate the features of the printed films, in particular to understand how the resistivity could be influenced by them. As will be discussed in the next chapter, it is expected a dependency in the grain size, grain number and quantity of ink binder present in the film. The obtained conclusions are merely qualitative, but are relevant to the improvement of the resistors performance.

Chapter 3

Design of the resistive sensor: results and discussion

The principal study behind the development of the sensor focused on the choice of the resistor design and respective material that could offer interesting performances. This chapter explores the evolution in the production of the resistors to comprise in the airflow sensor. Through the following sections, the thermal characterization results are discussed for a set of tested designs and materials, and the outcomes of an annealing process are also debated. At last, the electrical characterization for the chosen resistors is presented.

3.1 Design testing system

In order to choose an adequate sensor design and respective material, it is important to have in consideration the expression of thin film resistance

$$R = \frac{\rho}{t} \cdot \frac{l}{w} \quad (3.1)$$

where ρ [Ωm] refers to the bulk resistivity of the material, t is the thickness of the film, l is the length of the film and w is the width of the film. When the thickness of the film is uniform, a parameter known as sheet resistance, R_s [$\Omega\Box^{-1}$], can be defined such that the relation $\rho = R_s \cdot t$ is valid.

The previous expression can be related to the equation of resistance 1.2 in the following way

$$\Delta R = R_0 \cdot \alpha \cdot \Delta T. \quad (3.2)$$

Thus, if the interval ΔT is fixed, a higher value of resistance variation is obtained with an increase of α and R_0 . For a specific material, since α is an intrinsic property of the material, i.e. does not change according to the chosen design or the reference resistance, an increase of ΔR is obtained with the increase of the length of the film and the decrease of its width and thickness.

Moreover, these results can be connected to the heating phenomenon of Joule effect. From the analysis of equation 1.1, it is predictable that, for a fixed value of power, a lower resistance requires more current when compared to a higher resistance. Considering that small currents are desired when working with electronic devices, this aspect reinforces the discussion above. From the evaluation of these aspects, it is necessary to guarantee a balance between the required high resistance and the resources available for characterization purposes, since some limitations are imposed.

In the screen printing technique, it is usually difficult to effectively control the film thickness, whereby it is common to change the resistors length and width in order to change its resistance. Since the open screen area of the mesh, the stencil and the formulation of the ink paste are responsible for defining the thickness of the printed films, the resistance of the film can be changed by printing multiple layers, which results in a decrease of resistance values.

3.2 Study of the design influence on the resistance

Initially, different designs were considered in order to study if they could offer the characteristics that fulfilled the main interests, namely a relatively high resistance. In order to verify if those designs were applicable for the purpose of this work, tests were performed for resistors printed with a silver ink. Although a high resistance was desired to promote the sensor's sensitivity, this silver material was chosen due to its conductivity, since it was peremptory to assure that the resistors analysis could be performed with the resources available (for high resistances, a limitation on the power source in use for the electronic characterization was encountered).

After some preliminary tests, four designs were selected to proceed with the studies, that are presented in figure 3.1. The correspondent dimensions can be found in table 3.1.

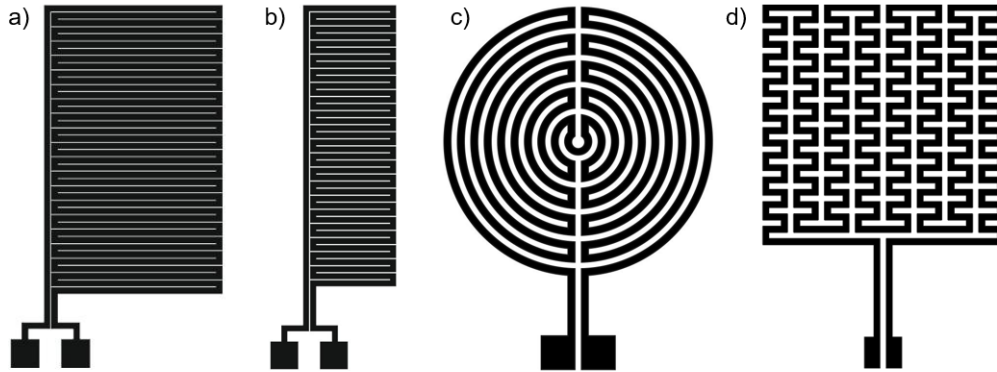


Figure 3.1: Serpentine, circular and intercalate resistor designs tested as heating elements, designated in the next topics as a) design A, b) design B, c) design C, and d) design D.

Table 3.1: Dimensions of the chosen designs presented in figure 3.1.

Design	Line width (mm)	Gap width (mm)	Total height (mm)	Total width (mm)
A	1	0.3	65	22
B	1	0.3	67	17
C	1	1	54	40
D	1	1	59	39

The forward mentioned printings were manufactured by screen printing, as represented in figure 2.2. The dimensions of the screen mesh in use were 140 x 31 μm . These values state the open screen area and are one of the parameters that define the thickness of the printed films. Therefore, the resistance values obtained in this discussion are a result of the chosen mesh.

The resistance offered by these designs was tested with resistors printed on PET substrates with a silver ink designated ahead as Ag-I. The cure drying process was performed on an oven at 120°C for 20 minutes. The PET substrates can tolerate this temperature without shrinking, hence the reason why this material was chosen for these tests. For each design, two samples were printed in order to correlate the results and assure that the obtained values were reliable. In figure 3.2 the measurements of resistance for each sample immediately after the curing process are exhibited.

As can be seen from the results of Ag-I, the designs B, C and D present very similar values of

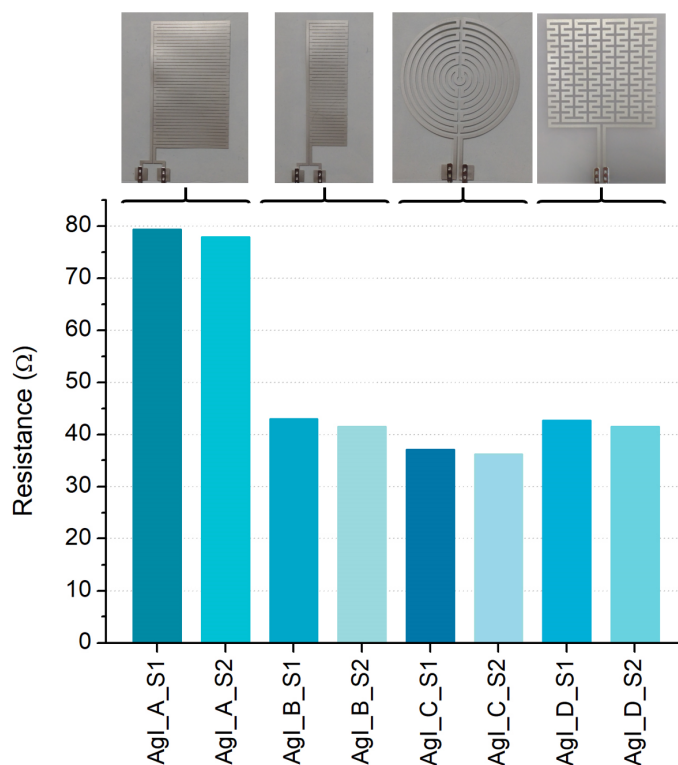


Figure 3.2: Measurements of resistance at room temperature after the curing process for samples printed with silver ink Ag-I.

resistance. Then, to study the resistance values provided by other materials and to understand how their properties could be advantageous to the sensor finality, only the serpentine designs, A and B, were printed.

3.3 Effect of different ink formulations

In order to perform a wider study of the behavior of other materials in comparison to the Ag-I ink tested previously, the resistors were printed with nickel and carbon inks. From tabulated values, it is known that the bulk resistivity of carbon is naturally superior than the one of silver, whilst the resistivity of nickel has a value between the resistivities of carbon and silver. Therefore, higher resistances were expected for these materials in comparison to the values obtained for Ag-I. To compare the results for different ink formulations of the same material, the designs were also printed with another silver ink, forward designated as Ag-II, that according to its datasheet is

capable to maintain a stable behavior at high temperatures.

The designs printed with the nickel ink were performed on PET substrates and subjected to a cure process realized at 125°C for 5 minutes. The measured resistance for each sample is listed in figure 3.3. These results show resistance values in order of M Ω , and therefore this material was not suitable for the purposes of this work. The reduction of the resistor dimensions could be an alternative to decrease the resistance values, however, given the results obtained, a substantial decrease was required, up until a resolution that is not provided by screen printing techniques. Another alternative relied on the hypothesis of printing multiple layers, but it would be necessary to print about one hundred thousand layers with the least resistive design in order to obtain a resistance near 100 Ω , which is not feasible.

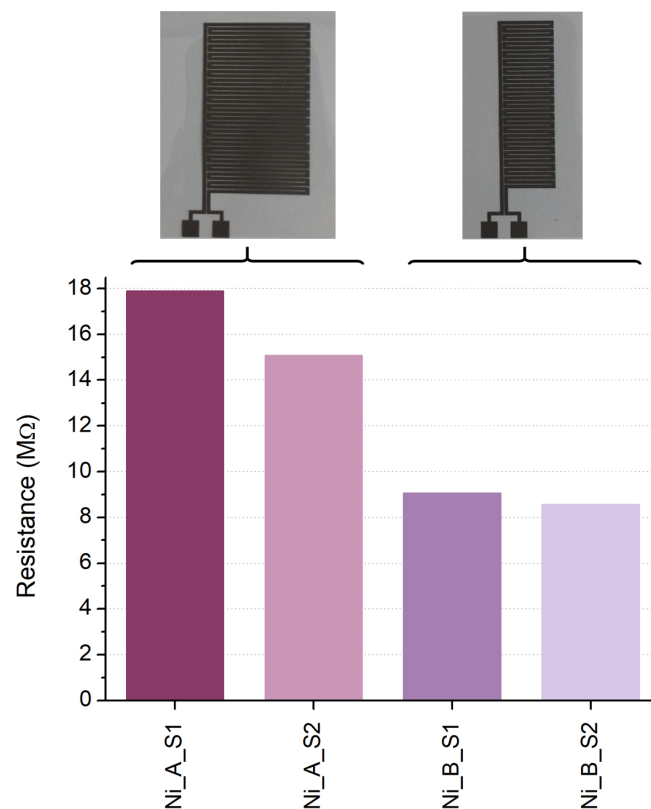


Figure 3.3: Measurements of resistance at room temperature after the curing process for samples printed with nickel ink.

Concerning the printings with the conductor carbon ink and following the results obtained above, relatively high resistances were predictable for this material. Therefore, each design was directly printed with three and thirty layers (layers dried between them with a final cure at 120°C

during 6 minutes) onto PET substrates; the relation between the resistances was then expected to decrease by a factor of 10 with the increase of layers. The measurements of resistance are exhibited in figure 3.4. Once again, the values in the order of $k\Omega$ are not adequate for the desired application. However, the decrease in resistance by an order of magnitude for a sample with thirty layers in comparison to the three layers one is an important result, since it shows that printing multilayers can be a good option to decrease the resistance in a given design (in accordance to equation 3.1), whenever it is a viable process.

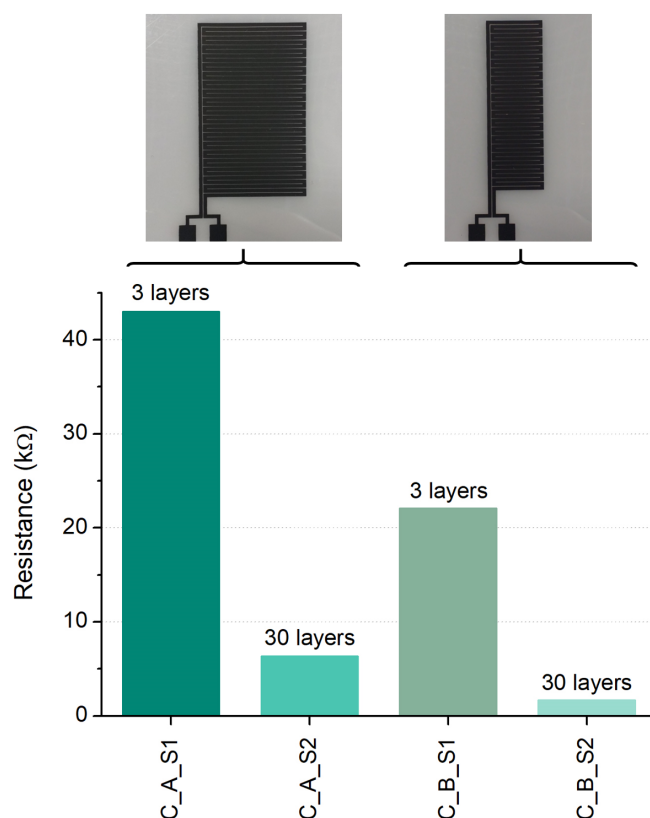


Figure 3.4: Measurements of resistance at room temperature after the curing process for samples printed with carbon ink.

To test the behavior of the Ag-II ink compared to Ag-I, the samples were printed onto PI substrates, since the cure drying process was chosen to be performed at 130°C for 20 minutes (PET substrates are not an eligible choice when cure temperatures above 125°C are required). From the results presented in figure 3.5 for this material, the resistance values are again in the order of Ω as in the case of resistors printed with Ag-I and, therefore, also meet the desired requirements.

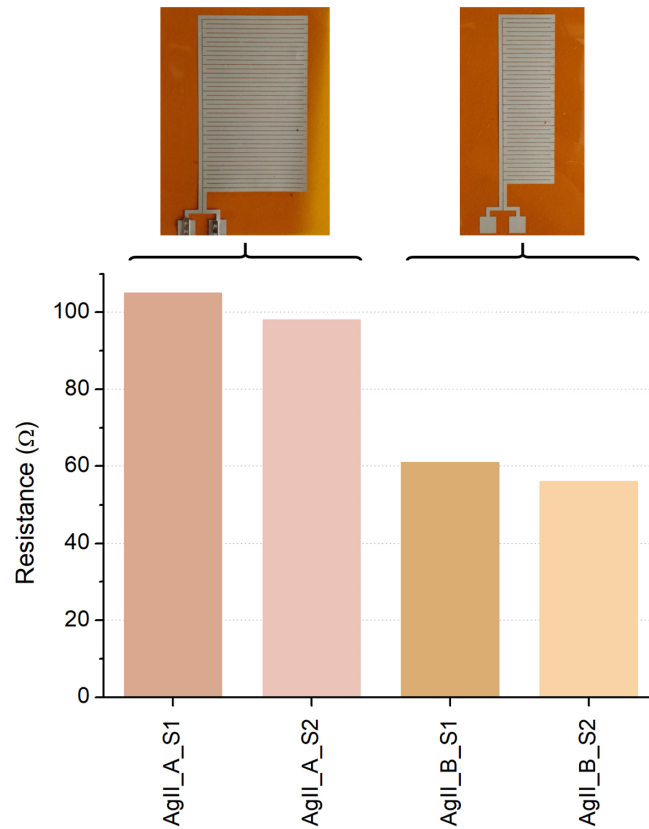


Figure 3.5: Measurements of resistance at room temperature after the curing process for samples printed with silver ink Ag-II.

From the resistance results obtained at this stage, the thermal characterization study proceeded with the resistors printed with Ag-I and Ag-II. These materials evidenced resistance values that followed the desired behavior and an in-depth study was required to evaluate what to expect from the performance (in particular, the sensitivity) of the resistors when operating as flow sensors.

3.3.1 Thermal characterization of Ag-I and Ag-II

The characterization relative to the behavior of the resistors printed with Ag-I was firstly performed six days after the printing. This information is important to retain, since the ink is subjected to chemical reactions over time, as is the case of oxidation phenomena due to air exposure. For this material, the behavior of all the proposed designs was studied. From the evaluation of the resistance variations presented in figure 3.6, registered for a temperature in-

terval ranging from room temperature until near 70°C, it can be seen that the serpentine design A evidences a higher variation, whilst the remaining designs show a very similar variation between them. This result corroborates the discussion that arose from equation 3.2; a higher variation was obtained for the design with the higher resistance value at reference temperature, assumed to be the room temperature. In table 3.2 the respective values of TCR are presented.

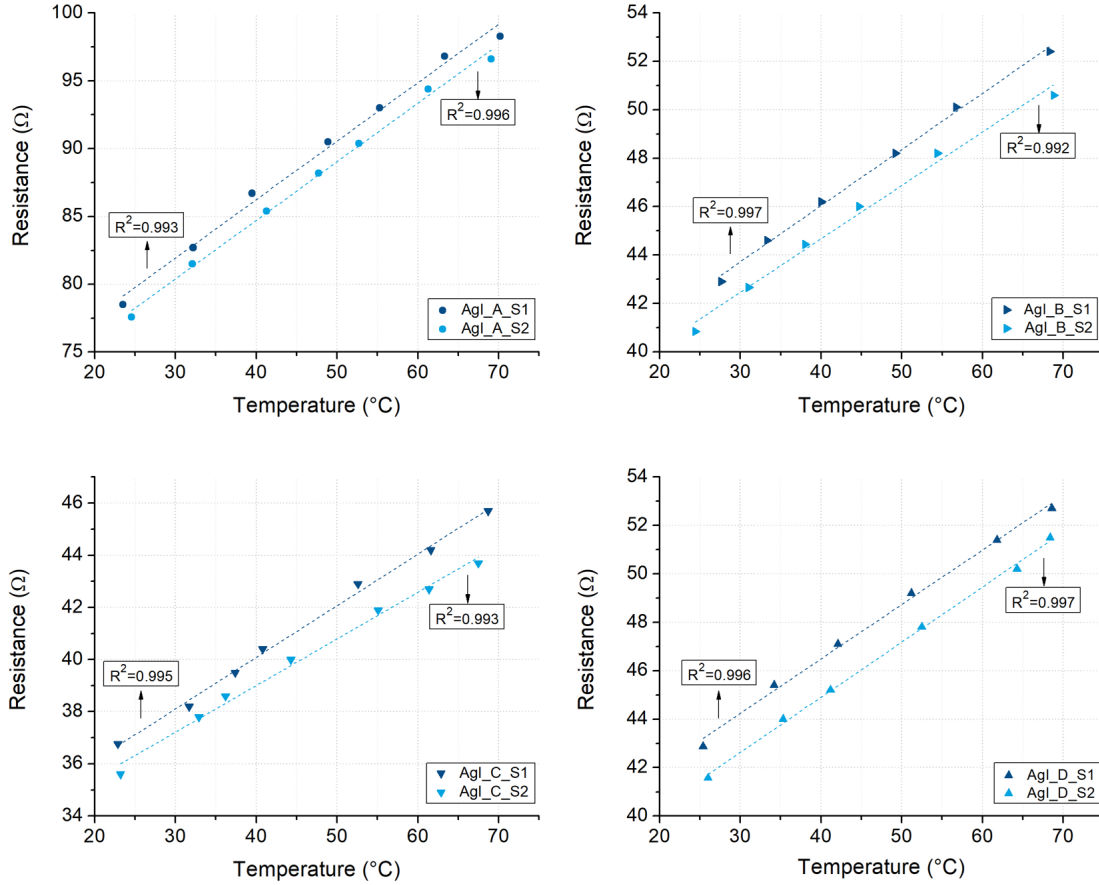


Figure 3.6: Graphic representation of the resistance response in function of temperature, six days after the printing of samples with Ag-I presented in figure 3.2.

The TCR was determined from the slope of the curve

$$\frac{R}{R_0} = \alpha (T - T_0) + 1 \quad (3.3)$$

being R_0 and T_0 the reference values taken as the first point measured (at room temperature). From the results obtained, the assumption that the TCR is an intrinsic property of the material was proved. It is necessary to retain, however, that it was required to apply an equal cure

process for all samples to have conformity in these results; different temperatures and/or times of cure can result in changes of TCR values, since electronic and chemical properties react in different manners according to the cure conditions.

Table 3.2: Values of TCR obtained from the linear behavior of the resistance with temperature, six days after the printing of samples with silver ink Ag-I.

Sample	TCR (/°C)
AgI_A_S1	0.0055 ± 0.0002
AgI_A_S2	0.0057 ± 0.0001
AgI_B_S1	0.0054 ± 0.0001
AgI_B_S2	0.0053 ± 0.0001
AgI_C_S1	0.0054 ± 0.0002
AgI_C_S2	0.0051 ± 0.0002
AgI_D_S1	0.0052 ± 0.0002
AgI_D_S2	0.0055 ± 0.0001

The same characterization was again realized forty-two days after the printing for the serpentine design A, since it was the one that interested the most given the results previously obtained. Each sample was tested twice, and the temperature range studied was extended in order to observe at which temperature the linear behavior was lost and to visualize changes that could occur in the resistance. From the results exposed in figure 3.7, it is possible to conclude that the linear regime is restricted to the interval between room temperature to near 70°C.

As can be seen in table 3.3, the TCR values measured within the linear regime show an increase in comparison to the ones obtained in the previous characterization. The reasons behind this increase can be related to oxidation phenomena and a continuous ink solvent evaporation that occurred in the days after the first characterization, and that were possibly potentiated by the heating processes performed.

Furthermore, it was possible to observe an evident loss of resistance at room temperature: the resistance after heating did not return to its initial value, showing a consecutive decrease. By comparing these results with the ones in figure 3.2, the resistance decrease is also evident. This phenomenon particularly occurred whenever the resistors were subjected to heating processes and can be again connected to oxidation effects that degrade the resistance.

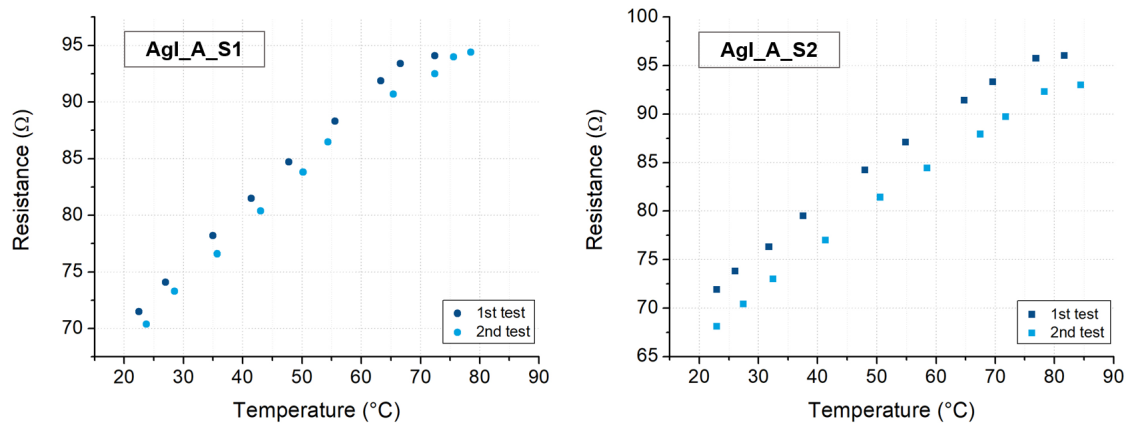


Figure 3.7: Graphic representation of the resistance response in function of temperature, forty-two days after the printing of samples with Ag-I.

Table 3.3: Values of TCR obtained from the linear behavior of the resistance with temperature, forty-two days after the printing of samples with silver ink Ag-I (R_b stands for resistance before heating and R_a for resistance after heating).

Sample	R_b (Ω)	TCR ($1/^\circ\text{C}$)	R^2	R_a (Ω)
AgI_A_S1	71.5	0.00691 ± 0.00009	0.9990	70.4
	70.4	0.0070 ± 0.0002	0.997	68.3
AgI_A_S2	71.9	0.0064 ± 0.0001	0.998	68.1
	68.1	0.0066 ± 0.0002	0.997	66.0

The characterization results for the printings with silver ink Ag-II are exhibited in figure 3.8 and in table 3.4, referring only to the serpentine design A. Although in this case the study was not so exhaustive as the one performed for Ag-I, these results were sufficient to take relevant conclusions. From figure 3.8, it is possible to observe that the linear regime stands for an interval between room temperature and a temperature between 70°C and 80°C, following a similar behavior as the Ag-I ink. The tabulated results, on the other hand, evidence a lower TCR value within the linear interval. Nevertheless, the resistance variation evidenced similar values with Ag-I, since the lower TCR is compensated by the higher values of resistance (as discussed in section 3.1). Besides that, the loss of resistance was also verified, evidencing a considerable decrease in its values due to the heating process.

Thus, at this point, the encountered problematic was related to the sensor lifetime, that is compromised if resistance losses occur in a prominent way whenever the resistor is exposed to a

heating process, that is the basis of a thermal flow sensor. In order to overcome this obstacle, it was necessary to perform an annealing process that could improve and stabilize the resistor behavior, in addition to the primary cure process. The annealing is a process performed at high temperatures and that can affect the electrical properties of the ink by evaporation of the total amount of its solvents.

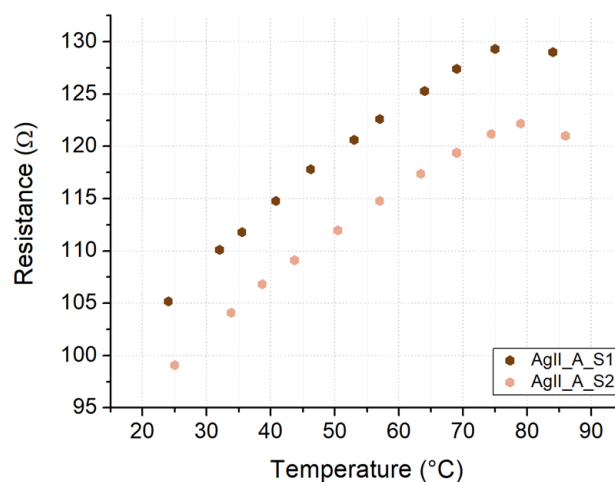


Figure 3.8: Graphic representation of the resistance response in function of temperature for samples printed with Ag-II.

Table 3.4: Values of TCR obtained from the linear behavior of the resistance with temperature, after the printing of samples with silver ink Ag-II (R_b stands for resistance before heating and R_a for resistance after heating).

Sample	R_b (Ω)	TCR ($1/^\circ\text{C}$)	R^2	R_a (Ω)
AgII_A_S1	105.0	0.0044 ± 0.0001	0.992	96.0
AgII_A_S2	98.0	0.0045 ± 0.0001	0.993	90.4

3.4 Post-printing annealing process

To study how the annealing process could be advantageous to improve the performance of the resistors, the focus was maintained on the serpentine design A and new samples were printed with the silver inks Ag-I and Ag-II. This annealing was conducted for 2 hours at a temperature of 200°C. Therefore, the chosen substrate for all samples was PI to avoid shrinkage effects. In order to correlate the results, four samples of each ink were printed, but only two were subjected to the annealing.

3.4.1 Ag-I ink based sensor

For samples printed with Ag-I, the cure process was performed during 20 minutes for a temperature of 120°C, as done previously. In figure 3.9, the results for the resistance values measured at room temperature are shown. A preliminary analysis evidences a substantial increase of resistance when the annealing is conducted (up to 53.4%), which is a condition of interest to this application. To complete these results, the resistors were evaluated according to the characterization of resistance with temperature.

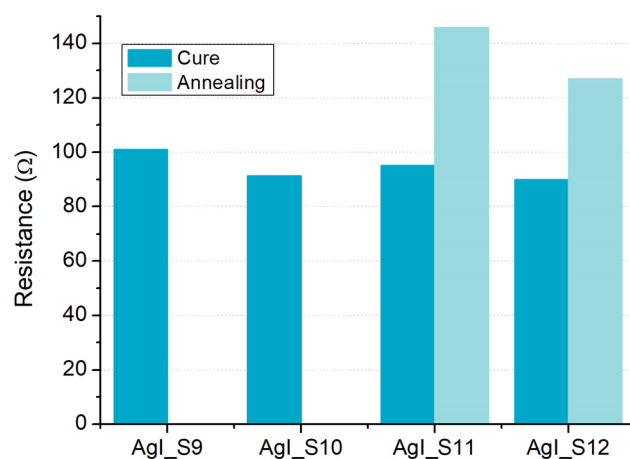


Figure 3.9: Measurements of resistance after curing and after the annealing process, when performed, for samples printed with silver ink Ag-I.

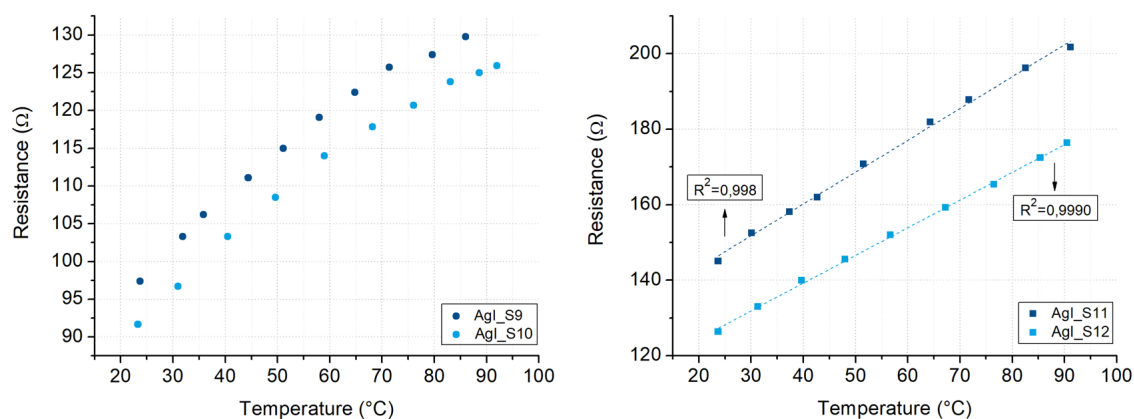


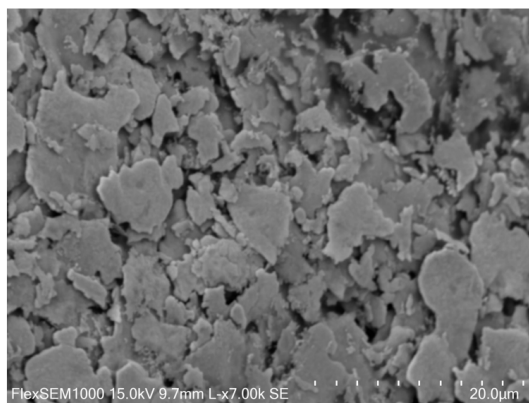
Figure 3.10: Graphic representation of the resistance response in function of temperature for samples not subjected to annealing (left) and for samples subjected to annealing (right), after printing with Ag-I.

The graphics exposed in figure 3.10 exhibit an improvement of the resistance linear behavior when the annealing was conducted. For samples not subjected to annealing it is patent a loss of the linear regime at temperatures near 70°C (as already seen before), whereas for annealed samples this regime is maintained for the entire range of studied temperatures. Furthermore, the results of table 3.5 corroborate these assumptions (the TCR values were obtained within the linear regime). In addition to these conclusions, the thermal hysteretic effect of the resistance was apparently eliminated (a more accurate conclusion could only be obtained if the resistance variation curve was determined for both heating and cooling processes).

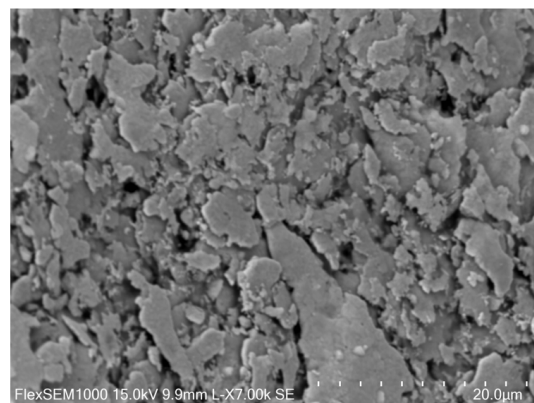
In figure 3.11, the SEM images for samples cured and samples subjected to the annealing are presented. The major difference between both images is related to the grain size and number. These results suggest that the annealing process promoted an interaction between the ink binder and the film grains, resulting in the reduction of the grain size and, consequently, in the increase of the number of grains.

Table 3.5: Comparison between the performance of samples not subjected to an annealing process and samples subjected to annealing for resistors printed with Ag-I (R_b stands for resistance before heating and R_a for resistance after heating).

Sample	R_b (Ω)	TCR ($^{\circ}\text{C}$)	R^2	R_a (Ω)
AgI_S9	91.7	0.0061 ± 0.0001	0.990	87.5
AgI_S10	97.4	0.0060 ± 0.0001	0.996	93.6
AgI_S11	126.3	0.00580 ± 0.00009	0.9990	126.4
AgI_S12	145.7	0.00600 ± 0.00009	0.998	145.7



a)



b)

Figure 3.11: SEM images of samples printed with silver ink Ag-I after a) the cure and b) the annealing.

3.4.2 Ag-II ink based sensor

For samples printed with Ag-II, the study of the annealing was conjugated with the variation of the parameters imposed to the primary cure process. This discussion seemed interesting for this topic since the ink datasheet proposes a large range of temperatures and times of cure. Thus, the chosen temperatures of cure were 130°C, 150°C and 200°C, and the time was fixed in 30 minutes for all printings.

Given the results obtained, only the ones for resistors subjected to a cure of 130°C and 200°C are debated here. From the immediate comparison between the results exhibited in figure 3.12, it is suggested that the increase of the cure temperature promoted a more effective evaporation of the ink solvents; this result is expressed by the difference between the resistance values before and after the annealing of the samples, that is particularly substantial for the cure performed at 130°C (a maximum decrease of 53.8%, compared to the 12.2% of a 200°C cure).

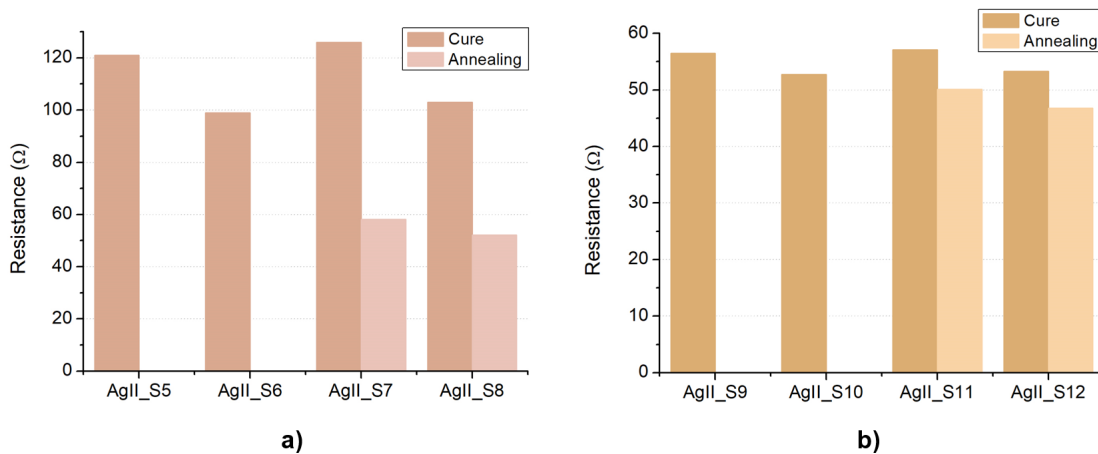


Figure 3.12: Measurements of resistance after curing and after the annealing process, when performed, for samples printed with silver ink Ag-II cured at a) 130°C and b) 200°C.

Furthermore, the results obtained from the thermal characterization of the Ag-II samples, presented in tables 3.6 and 3.7, reinforce these preliminary conclusions. For samples cured at 130°C, it can be seen that the annealing promoted an improvement of the TCR (measured within the linear regime) and compensated the effect of thermal hysteresis in the resistance values. On the other hand, the samples cured at 200°C evidenced a similar behavior whether the annealing process was performed or not. These results indicate that, for samples printed with Ag-II, a curing process at 200°C for 30 minutes is sufficient to stabilize their thermal per-

formance.

Table 3.6: Comparison between the performance of samples not subjected to an annealing process and samples subjected to annealing for resistors printed with Ag-II and cured at 130°C (R_b stands for resistance before heating and R_a for resistance after heating).

Sample	R_b (Ω)	TCR ($^{\circ}\text{C}$)	R^2	R_a (Ω)
AgII_S5	99.7	0.0047 ± 0.0002	0.994	95.0
AgII_S6	123.2	0.0041 ± 0.0002	0.98	114.3
AgII_S7	52.0	0.0054 ± 0.0001	0.996	52.0
AgII_S8	58.2	0.0051 ± 0.0002	0.994	58.2

Table 3.7: Comparison between the performance of samples not subjected to an annealing process and samples subjected to annealing for resistors printed with Ag-II and cured at 200°C (R_b stands for resistance before heating and R_a for resistance after heating).

Sample	R_b (Ω)	TCR ($^{\circ}\text{C}$)	R^2	R_a (Ω)
AgII_S9	53.0	0.0055 ± 0.0001	0.996	53.0
AgII_S10	55.7	0.00594 ± 0.00009	0.9990	55.6
AgII_S11	46.1	0.00602 ± 0.00009	0.9990	46.1
AgII_S12	49.5	0.0052 ± 0.0002	0.994	49.5

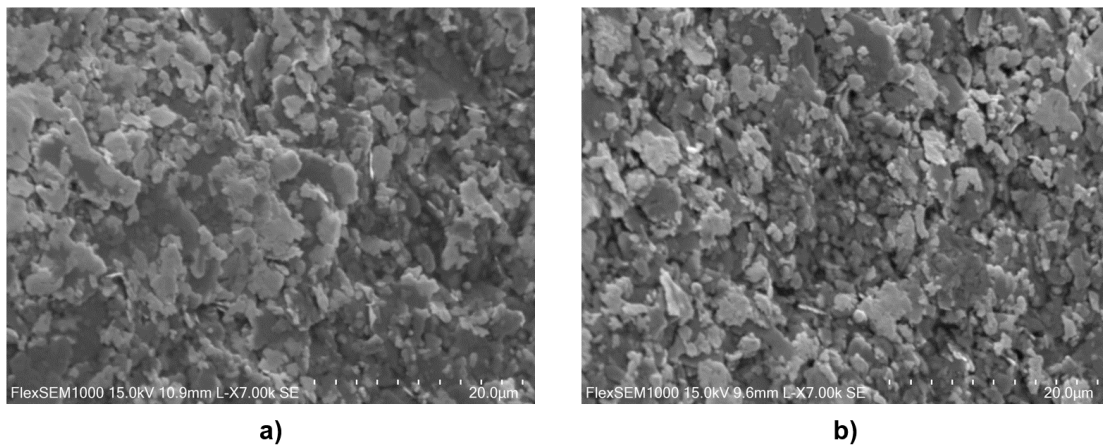


Figure 3.13: SEM images of samples printed with silver ink Ag-II after a) the cure performed at 130°C and b) the annealing.

In figures 3.13 and 3.14, the SEM images for samples printed with Ag-II and cured at 130°C and 200°C, respectively, are presented. By comparing both figures, it is immediate to conclude that no major differences are detected for samples cured at 200°C and then annealed, whilst for samples cured at 130°C it is evident that the annealing process reduced the ink binder between

the film grains.

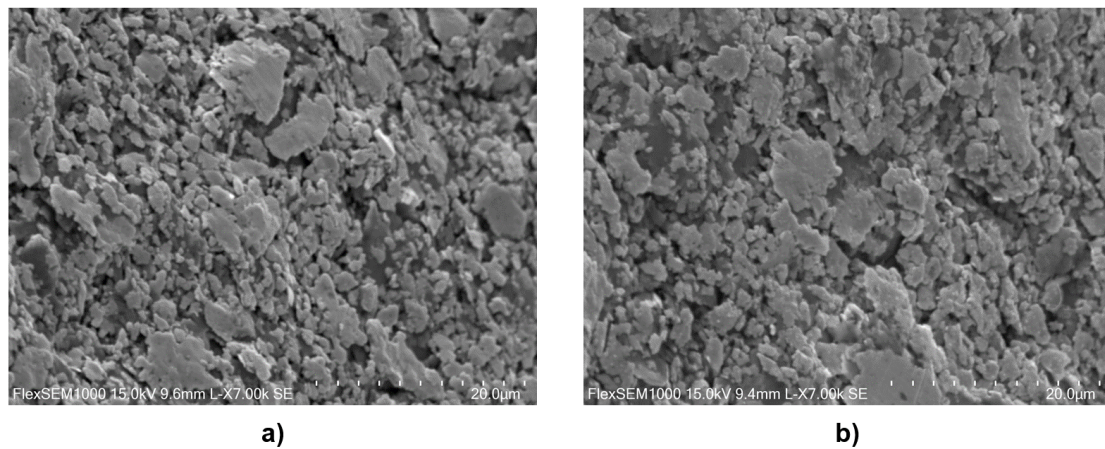


Figure 3.14: SEM images of samples printed with silver ink Ag-II after a) the cure performed at 200°C and b) the annealing.

3.4.3 Final discussion

An obvious conclusion from the comparison of Ag-I and Ag-II results is that the annealing process had different effects in the resistance values. To understand these effects, it is necessary to perceive that the overall resistivity has three main contributions. One of them results from the intrinsic resistivity of the material and has a specific value, ρ_0 . The other two are related to the grain boundaries and to the binder that connects the grains, as presented in the schematic of figure 3.15. When the electrons encounter a grain boundary, they need to surpass the potential barrier by tunneling in order to continue their path; moreover, scattering effects also occur at these boundaries, resulting in an increase of the resistivity. In addition, when in the presence of the binder, that usually is an insulator, another potential barrier is imposed, and the resistivity increases only due to tunneling effects.

The SEM images from samples printed with Ag-I evidenced a decrease on the grain size as a consequence of the annealing process, which was also accompanied by a growth in the number of grains. This result implies that the contribution from the grain boundary resistivity increased and, therefore, an increase of the overall resistivity was promoted.

For samples printed with Ag-II and cured at 130°C, the observation of the respective SEM images suggests that the effects from tunneling were reduced due to the evaporation of the ink

binder. Therefore, the correspondent resistivity decreased and, since the grain size does not suffered major changes, a reduction of the overall resistance was verified. In fact, from these results, it is expected a higher potential barrier of the binder than the one of the grain boundary (figure 3.15b).

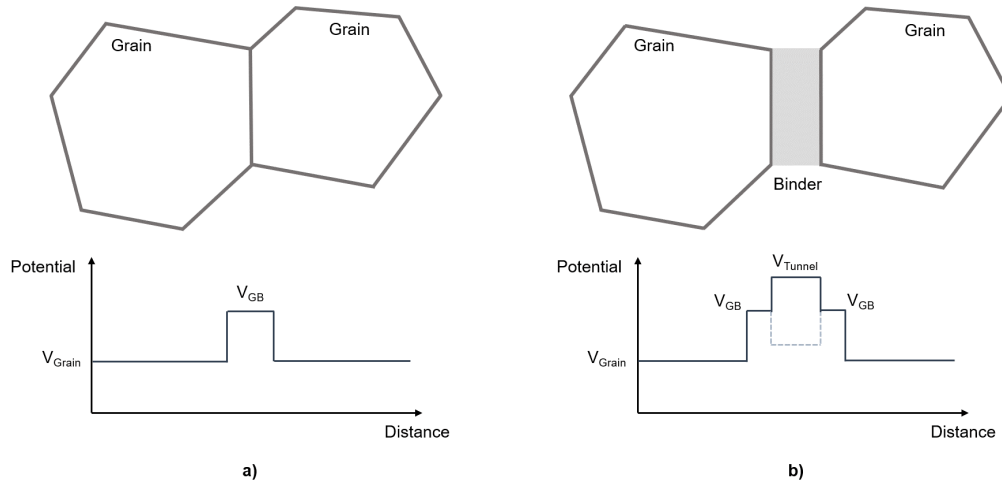


Figure 3.15: Schematic of the potentials related to the a) grain boundary (V_{GB}) and b) ink binder (V_{Tunnel}).

From the evaluation of the results presented in this section, it can be concluded that the annealing process is particularly advantageous for resistors printed with Ag-I, since it promoted an increase of the resistance, which directly improves the sensitivity expected for the operation of these resistors as flow sensors. Moreover, an improvement of the thermoresistive effect was also achieved. Given these conclusions, this work proceeded with the samples of Ag-I subjected to the annealing (AgI_S11 and AgI_S12, ahead mentioned according to their resistance).

3.5 Constant current sensor characterization

As explained in subsection 2.2.1, the hot-film sensor proposed in this work operated under a constant current mode. Therefore, after choosing the resistors to incorporate in the flow sensor, it was necessary to understand how the temperature changed according to the supplied current, in order to select the adequate values of current to operate in the sensor. The graphics obtained from this characterization are presented in figure 3.16, with the overtemperatures measured with relation to the room temperature (approximately 24°C). Both resistors were characterized twice to examine fluctuations that could occur in the measured temperature. As can be seen, the

variations detected are inferior to 5°C for both samples, which is a reasonable value to work with. Moreover, for low values of current it is verified the resistors are not capable to overcome the room temperature due to conduction phenomena (the samples were placed in a support with low thermal conductivity), but once the threshold is surpassed the increase of temperature is substantial. Furthermore, it is possible to observe that the resistor with higher resistance requires less current to obtain a higher overtemperature, as discussed in section 3.1. After these tests, the respective resistances were measured and it was seen that the values remained the same, which evidenced that this process did not degraded the samples.

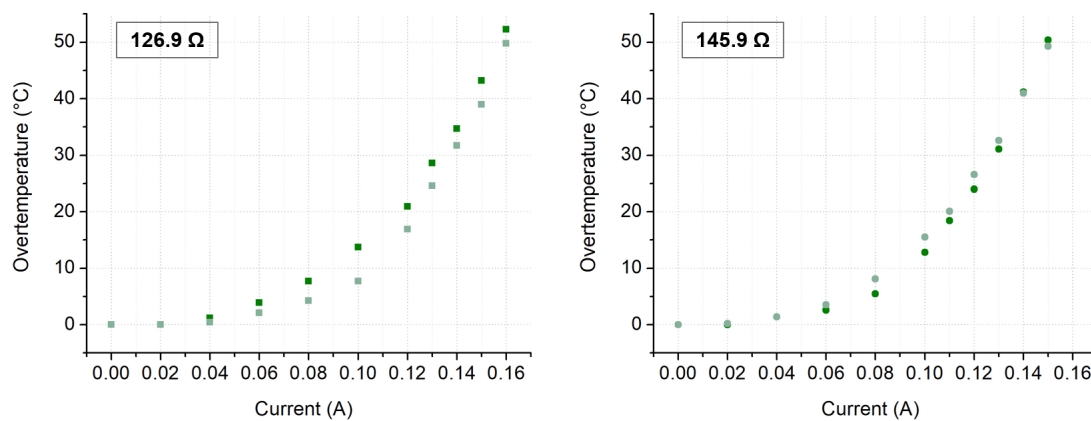


Figure 3.16: Graphic representation of the temperature response in function of the current supplied to the resistors chosen to comprise in the flow sensor.

These results give an indication about the current values required to attain the desired operation temperatures of the sensors. Since the resistance variation achieved is directly dependent of the sensor temperature, it is obvious that a higher overtemperature contributes for the improvement of the measurements sensitivity. However, it is necessary to guarantee that the current supplied does not damage the sensor. On the other hand, a lower overtemperature leads to a rapid saturation of the signal. Therefore, it is necessary to balance both of these considerations when choosing the adequate operation current.

Chapter 4

Flow sensor: test apparatus

This chapter is focused on the final steps concerning the development of the flow sensor, namely the implementation of a circuit for an operation at constant current, and in the experimental setup for tests with flow. In the first section, the current controller assembled to the chosen resistors is presented. The second section discusses the genesis of a wind pipe that was used to performed the flow tests, and the last section explores the final setup for measurements.

4.1 Current controller

As mentioned before, the flow sensor presented in this thesis was projected to operate under constant current, whereby the output signal could be obtained from the voltage variation resultant from the fluid flow. This regime was established with the aid of a current controller, responsible to supply the predefined value of current.

In this work, the current of the sensor was controlled by one LED driver AL8805 provided by Diodes Incorporated. According to the device datasheet, this controller is capable to debit currents up to 1 A with an accuracy superior than 5% and a high efficiency up to 98%. The maximum current supplied is established by an external resistor, whose resistance is defined as $R_{SET} = 0.1/I$, where I is the current value of interest. An internal switch on-and-off cycle is responsible for maintaining the supplied current constant. The device is exhibited in figure 4.1.

Following the results from the current characterization presented in section 3.5, the chosen

values to be operated in the flow sensor were 100 mA and 140 mA, which required resistances of $1\ \Omega$ and $0.7\ \Omega$, respectively.

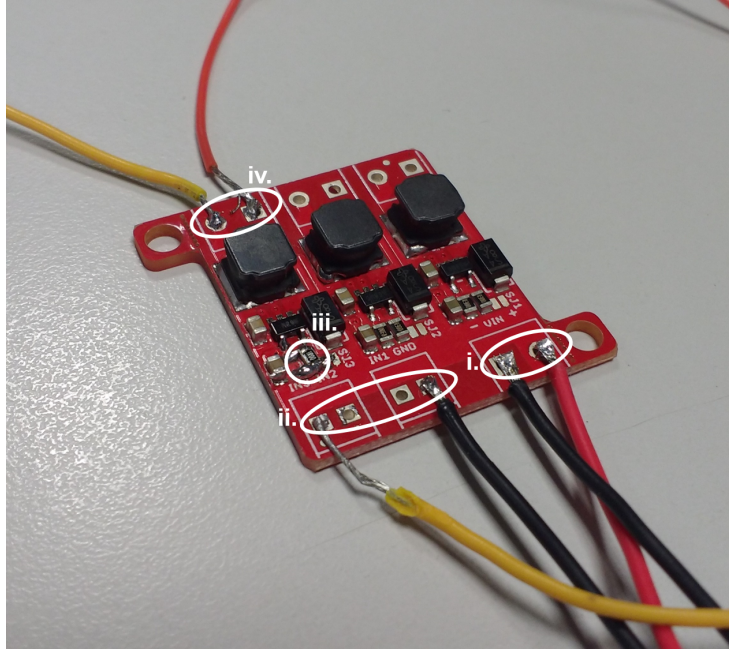


Figure 4.1: Current controller for an operation under constant current of the hot-film sensor, where i) are the input supply pins, ii) corresponds to an on/off control input, iii) refers to the external resistor that defines the current supplied, and iv) are the output signal pins connected to the ends of the sensor resistor.

4.2 Wind pipe construction

For flow measurements, it was chosen to develop a small wind pipe prototype instead of using a typical wind tunnel. This type of approach is common to find in the literature, since its geometry resembles the regular ones used in, for example, HVAC systems, and provides a simple setup to perform these type of tests at a preliminary stage. The projected wind pipe consisted in a tube of small dimensions - diameter of 7.6 cm and 12.5 cm of length - with a fan attached to its entrance, as shown in scheme of figure 4.2. The air flow within the pipe was generated by the power regulated fan. In order to study the wider velocity range possible, two fans of different maximum powers were employed. The final wind pipe is presented in figure 4.3.

The choice of the sensor position in the interior of the wind pipe was made according to considerations related to the behavior of the velocity distribution within the tube. As presented in figure 4.4, the velocity detected by a sensor located at the center of the tube depends on the

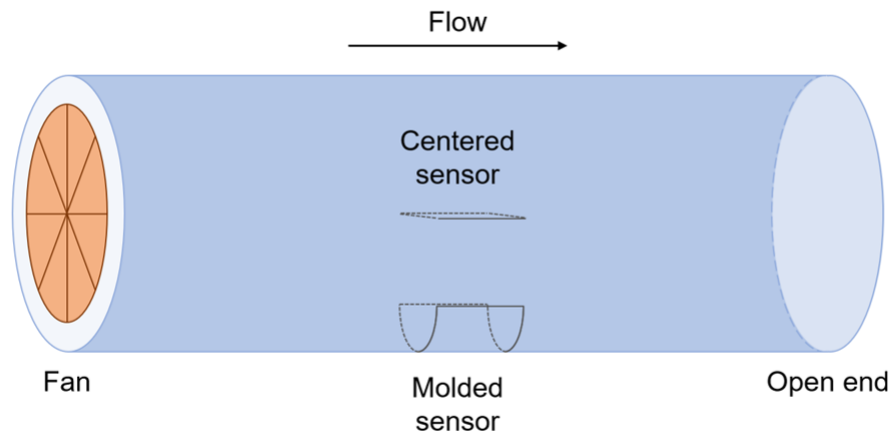


Figure 4.2: Scheme of the projected wind pipe for flow measurements.

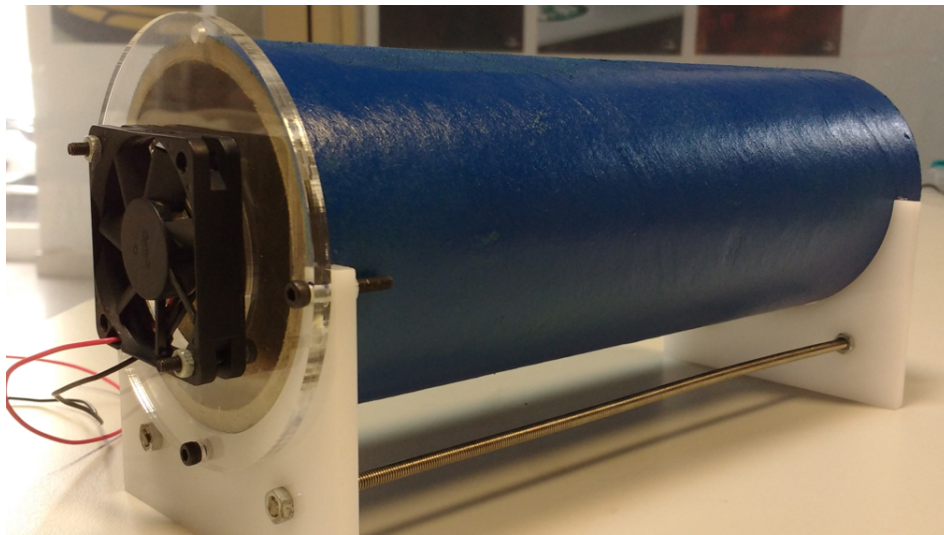


Figure 4.3: Wind pipe developed to perform flow measurements with the sensor proposed in this work.

velocity distribution and an accurate output can only be obtained if the sensor is located in the fully developed region. However, this region is attained at a certain distance from the entrance of the tube, where the fan is positioned, being dependent of the flow-inlet conditions [10]. In fact, this distance, denominated as hydrodynamic entry length, is proportional to the stream velocity for both laminar and turbulent flows [54]. Thus, an alternative to guarantee more stable results consists in the placement of the sensor in the wall surface of the tube, as also seen in the work of Tan et al. [10], since this is the first point where the velocity achieves a constant value. This configuration is possible due to the flexibility of the sensor and allows to shorten the required distance between the sensor position and the tube entrance, which is a fundamental aspect to the purpose of having a setup of small dimensions. Therefore, following this discussion, the

tests were both performed with the sensor molded to the interior wall of the tube (figure 4.5a), and with the sensor located at the center of the tube (figure 4.5b), in order to study how the signal output changed. With respect to the entrance of the wind pipe, the sensor was located at the middle of the tube.

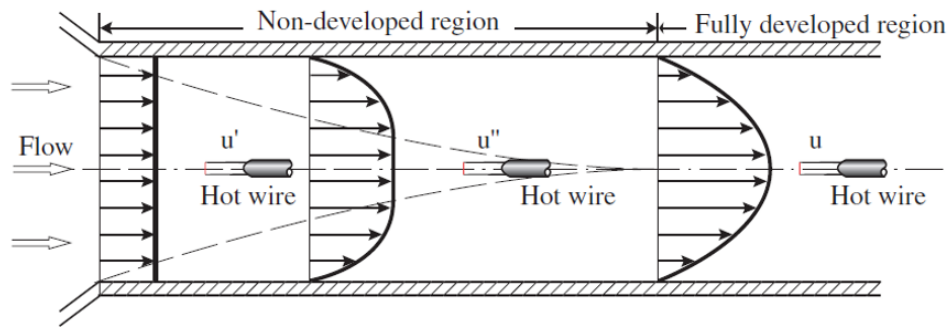


Figure 4.4: Schematic of the flow development in the interior of a circular tube (from Tan et al. [10]).

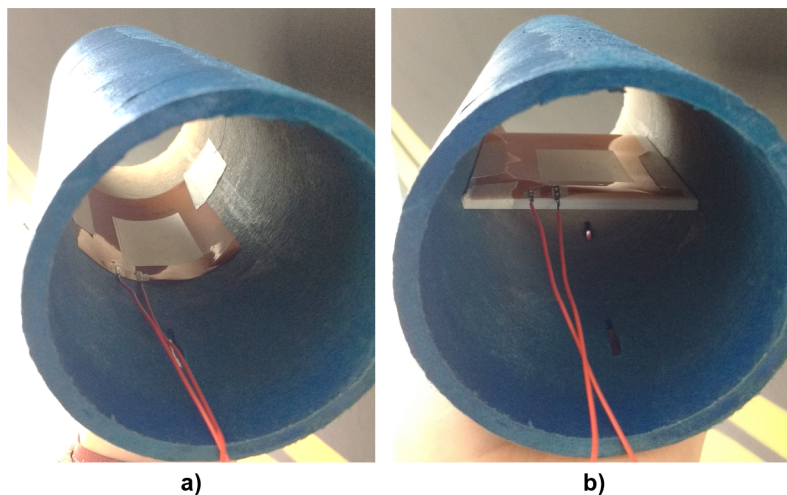


Figure 4.5: Sensor position a) molded to the surface and b) centered in the interior of the tube.

4.3 Flow measurement setup

Once both the constant current controller was connected to the sensor and the wind pipe was built, the flow sensor was prepared to be tested. The completed setup for flow measurements is presented in figure 4.6, and the principal aspects of the procedure are mentioned below.

- The wind pipe fan and the current controller were both supplied by power sources (HQ Power Adjustable DC Power Supply);
- Since the sensor worked at constant current, a decrease of the resistance promoted by the passage of flow resulted in a decrease of the potential difference (according to the Ohm's law) and the obtained voltage variation was considered as the output signal of the sensor;
- These voltage variations were obtained with a multimeter (Agilent 34410A) via an automatic data acquisition;
- The respective flow velocity for each value of power supplied to the fan was measured with a reference anemometer (Air Velocity Transducer 8475 Series) with an operation range of 0 to 2.5 m/s, according to its datasheet;
- The reference sensor was located at the center of the wind pipe, in the same place as the sensor was positioned with respect to the tube entrance.

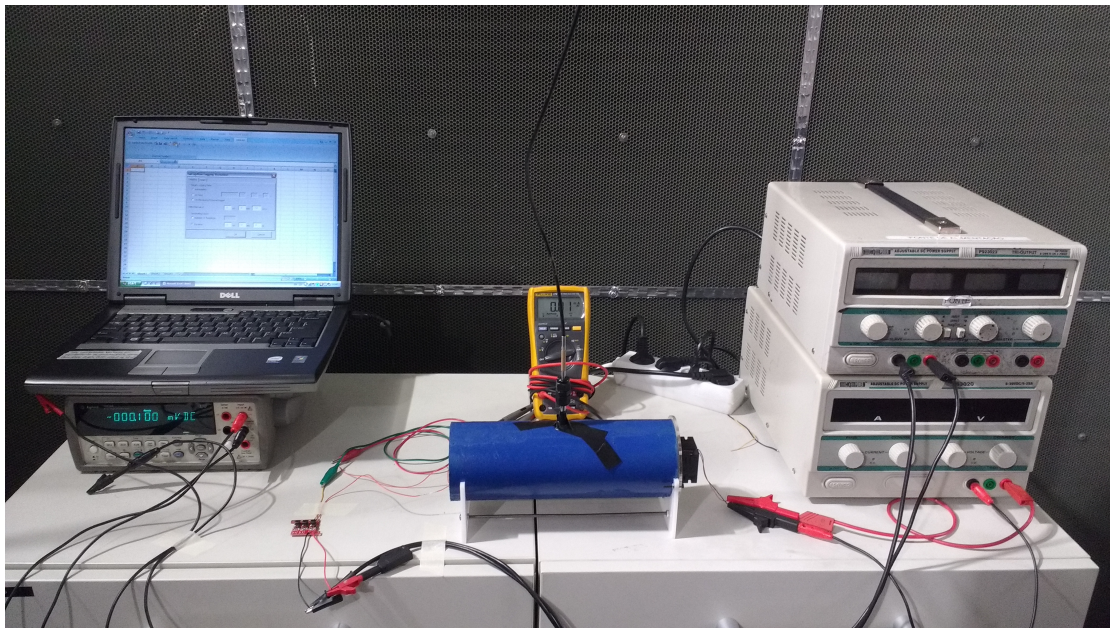


Figure 4.6: Final experimental setup for flow measurements.

The preliminary tests were performed inside the CeNTI's climatic chamber, since the operation of the available reference anemometer was dependent of the entire chamber operation. Due to the impossibility to work with the chamber over the required period of time to realize all the

essential measurements, the calibration curves between the power supplied to the fan and the respective velocity were determined during these initial tests, under controlled room temperature and relative humidity (24°C and 50%). Thus, the resultant output signals obtained with the flow sensor were combined with the velocity values of these curves.

Chapter 5

Flow sensor evaluation tests

This chapter summons the results obtained with the operation of the airflow sensor developed in the previous chapters and their subsequent discussion. The velocity calibration curves mentioned in the previous chapter are presented in the first section of this chapter. In the second section, the discussion focus on the operations of four different tests of flow sensor devices. The results being presented show the signal and the respective characteristic responses under different flows. The final discussion debates the fundamental conclusions from the sensors operation.

5.1 Velocity calibration curves

As mentioned in section 4.3, the reference anemometer was responsible for measuring the velocity of the flow in the interior of the wind pipe. This flow was generated by the fan located at the entrance of the pipe and the respective velocity was controlled by the power supplied to it. The use of two different fans allowed the study of a wider range of velocities; however the maximum value was defined by the operation range of the reference anemometer, whereas the minimum value was limited by the lower velocity provided by the fan with less power.

The measurements obtained with the airflow sensor were based in the calibration curves exhibited in figure 5.1. These curves resulted from the conjugation of tests performed with both fans and represent the average velocity obtained for each value of supplied voltage. As a result, the

operation of the purposed airflow sensor was studied between 0 m/s and 2.506 m/s, with 0.569 m/s as the minimum non-zero velocity measured.

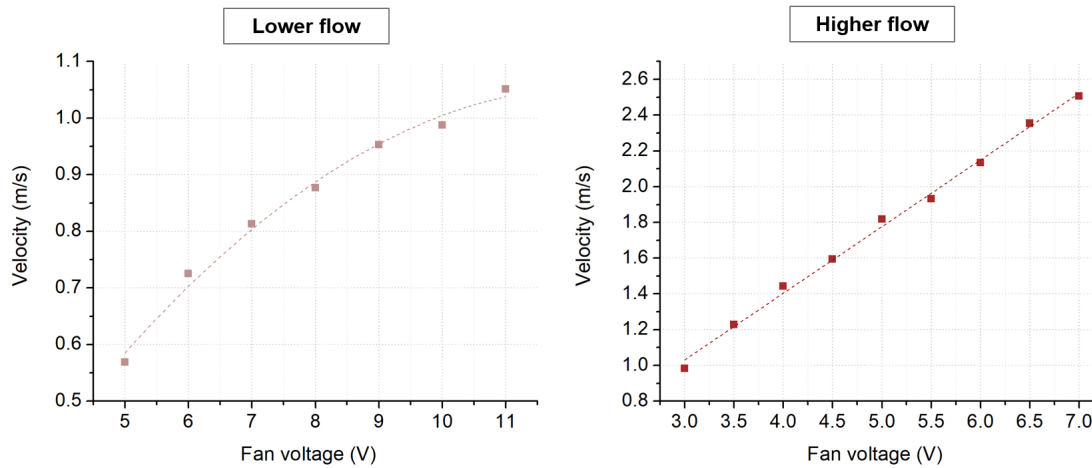


Figure 5.1: Calibration curves of the flow velocity measured in relation to the voltage provided to the wind pipe fans.

For higher flow rates the results were fitted to a linear curve, with an error associated to the slope of 2.1%. However, the same linear behavior was not suitable for lower rates, so a parabolic fit was applied with an error of 25.0% relative to the quadratic term.

5.2 Operation of the flow sensor

In order to inquire about the airflow sensor model created in this work, its operation was studied for different sensor characteristics. The chosen characteristics were the sensor resistance, the applied current and the position at which the sensor is located in the wind pipe, since they are directly related to the sensitivity and stability of the obtained output signal. As discussed in subsection 1.2.3, the coefficients of the sensor response change according to these parameters and the sensor performance is also influenced by them. The different parameters considered were studied in different tests. The conditions of each test are represented in the table 5.1. For comparative terms, the sensor of Test I was considered the reference one, since a higher sensitivity was expected due to its characteristics.

For the sensors of Test I, II and III, the voltage signal was studied in conditions of zero flow and in the presence of flow, in order to understand how the signal behaved. The respective considerations are listed below. The consequent discussion is presented in subsections 5.2.1

and 5.2.2.

Table 5.1: Conditions of each developed sensor considered for the experimental tests with flow.

Test	Resistance	Current	Position
I	145.9 Ω	140 mA	Molded
II	145.9 Ω	100 mA	Molded
III	145.9 Ω	140 mA	Centered
IV	126.9 Ω	140 mA	Molded

1. **Signal in the absence of flow:** When supplying current to the sensor, an increase of the voltage signal is expected in the absence of flow, due to the resultant increase of temperature. Thus, the sensors were tested during a certain period of time with zero flow in order to ascertain if the difference between values was negligible.

2. **Signal in the presence of flow:**

- **Stabilization time:** When the fan is switched on, the voltage is expected to decrease until a steady state is achieved (figure 5.2). Therefore, the purpose of this study was to understand how the voltage signal changed over time under a flow system. The voltage signals for the chosen velocities of 0.983 m/s and 2.354 m/s were evaluated during a period of 8 minutes, to guarantee that the stability state was attained. These velocities were selected due to their proximity to the velocity range limits. To inquire about the evolution of the signal, the respective voltage variations were measured 60 seconds and 120 seconds after connecting the fan (it was expected a good stabilization at these intervals). Then, the obtained values were compared to the total variation read at the steady state.
- **Alternate flow:** The flow was changed between zero and a fixed velocity (0.983 m/s and 2.354 m/s) in intervals of 120 seconds. In this analysis, it was of interest to understand how the signal behaved with the alternate conditions of flow. To evaluate the changes that occurred, a comparison between the values obtained in the absence of flow was performed, as well as in the presence of constant flow.

Once these tests were concluded, the characteristic response of each of the four sensors presented in table 5.1 was obtained and discussed. The room temperature in all these tests was

approximately 24°C and the relative humidity was maintained constant during the experiment.

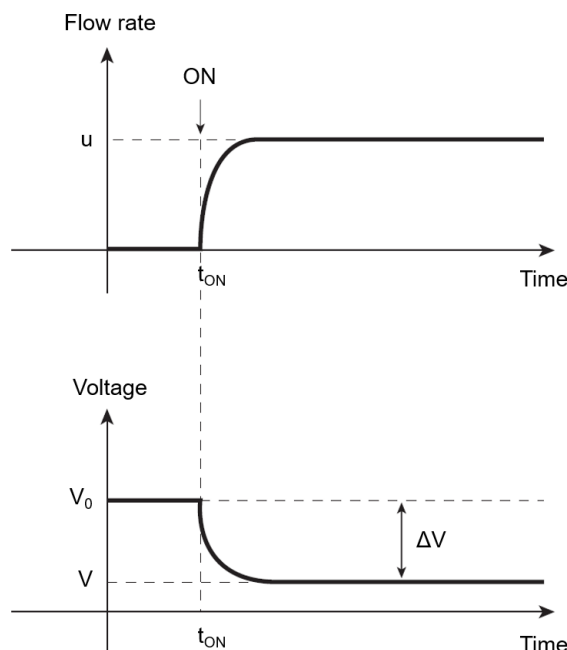


Figure 5.2: Variation of the voltage signal in function of time for an applied flow (adapted from Tan et al. [10]).

5.2.1 Signal in the absence of flow

The voltage signals for the sensors of Test I, Test II and Test III are presented in figure 5.3. All three sensors were tested in the absence of flow for a period of 13 minutes, in order to the stabilization to occur. Given the values of applied current, the temperatures obtained with the resistance were approximately 78.9°C, 47.8°C and 78.6°C, respectively, with a major deviation of 4°C. A first observation of the results evidences similar values between the voltages obtained for Test I and Test III, since the resistance of the device and the applied current were the same. In contrast, the values measured with the sensor from Test II are clearly inferior, resulting from the decrease of the current value.

From the analysis of the respective responses, an increase of 2.5%, 2.3% and 5.8% between the first and the last value measured was obtained for the sensors of Test I, Test II and Test III, respectively. The obtained percentages evidence that the variation of values for the signals in the absence of flow are relatively small and could be neglected without prejudice to the results

presented in the next subsections.

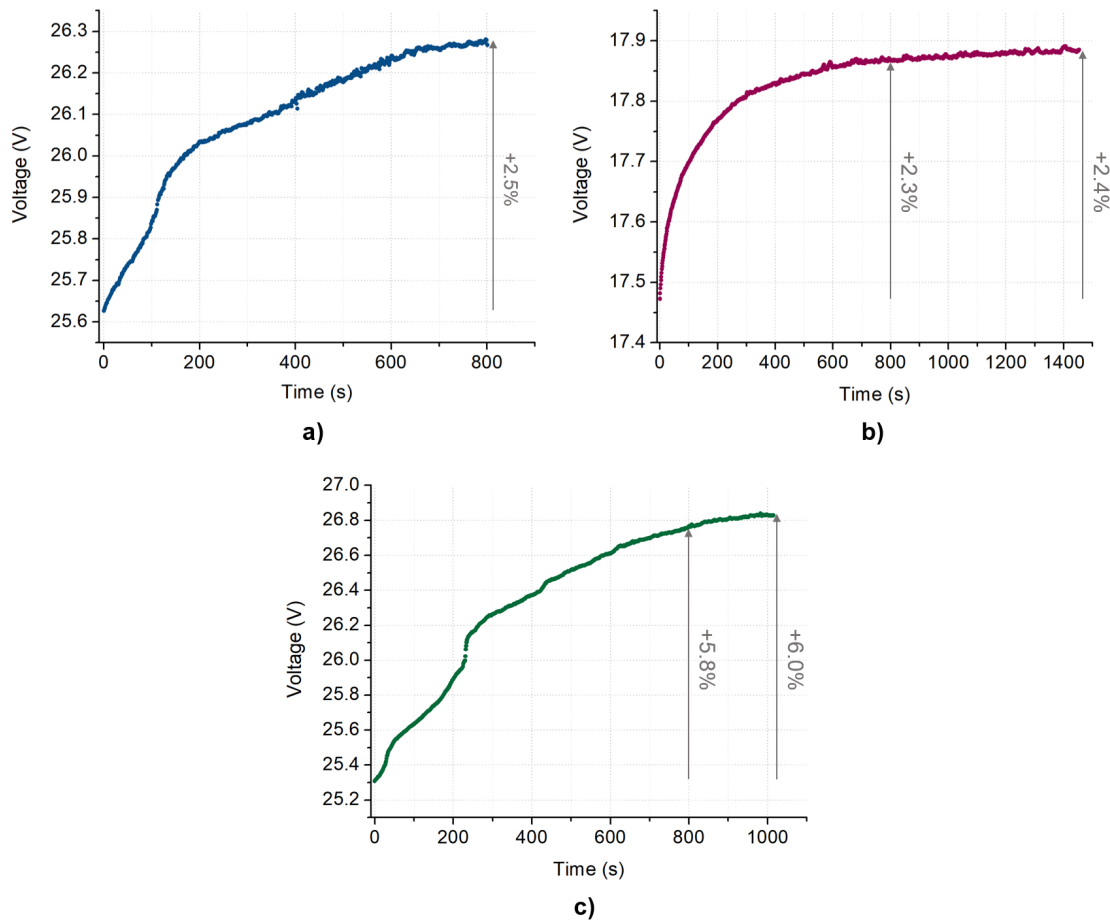


Figure 5.3: Voltage signal measured in the absence of flow for the sensor of a) Test I, b) Test II and c) Test III.

5.2.2 Signal in the presence of flow

The results concerning the signals under the presence of flow for the sensor of Test I are exhibited in figure 5.4. From the analysis of the stabilization times for a velocity of 0.983 m/s, it can be seen that 60 seconds after switching the fan the measured voltage corresponded to 45.1% of the asymptote value, and 60 seconds after that the percentage ascended to 56.4%; however, the discrepancy detected at the overall 600 seconds, that is probably related to movements in the air or to a change in the flow temperature, clearly influenced these results. In fact, for 2.354 m/s the same percentages increased to 78.4% and 85.5%, respectively, thus presenting more reliable values. Moreover, by inspecting the below row of graphs in figure 5.4, it is seen that the

most significant changes of values were 1.0% for 0.983 m/s and 0.93% for 2.354 m/s, which reveal that the signal followed a regular behavior and no relevant changes were detected.

A comparison of both signal variations was also performed, in order to verify how the fluctuations that occur between different measurements of the sensor could impact its operation. For this discussion, the voltage variation measured 120 seconds after the switch of the fan was considered the comparative term. As a result, a maximum deviation of 14.4% was obtained for 0.983 m/s, while for 2.354 m/s this percentage was fixed in 15.0%. Therefore, deviations in the order of these percentages must be expected when operating with the flow sensor.

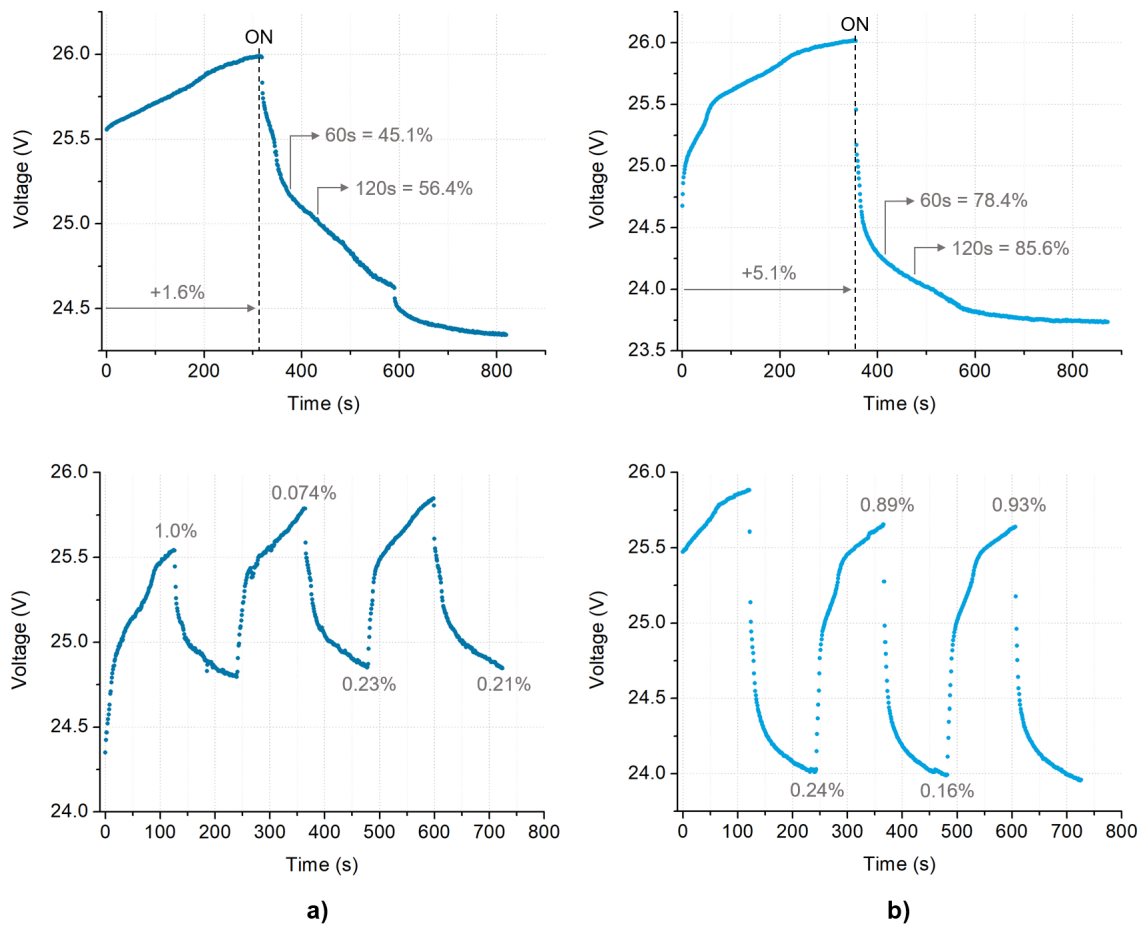


Figure 5.4: Voltage signal evolution for a fixed applied flow (above) and in the presence of an alternate flow (below) for velocities of a) 0.983 m/s and b) 2.354 m/s, both measured with the sensor of Test I.

The voltage signals relative to Test II are presented in figure 5.5. As mentioned before, the decrease of the applied current is reflected in the decrease of the voltage values compared to

the ones obtained with the sensor of Test I. From these results, it is also evident that the signal variations are less significant, which directly decreases the sensitivity of this sensor.

From the evaluation of the stabilization times, it can be seen that for a velocity of 0.983 m/s, the voltage variation measured at 120 seconds attained 76.0% of the total variation, whereas for 2.354 m/s the same percentage was 91.4%. These values indicate that an interval of 120 seconds is sufficient to guarantee that the voltage variation already achieved an accurate value. Furthermore, the responses with an alternate flow evidenced that the maximum variation observed had a percentage of 1.1%, which revealed the conformity of the measured signals.

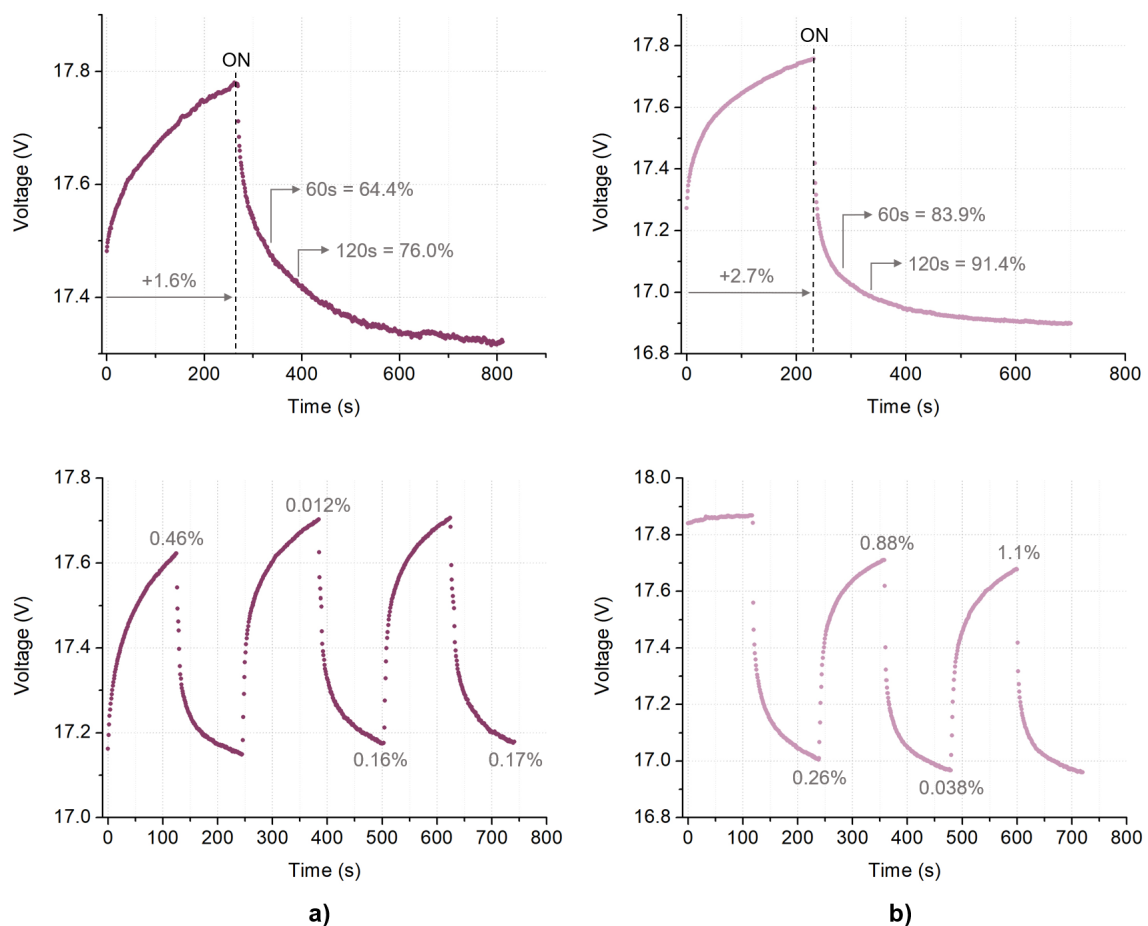


Figure 5.5: Voltage signal evolution for a fixed applied flow (above) and in the presence of an alternate flow (below) for velocities of a) 0.983 m/s and b) 2.354 m/s, both measured with the sensor of Test II.

The voltage signals in figure 5.6 refer to the results of Test III. As mentioned above, the voltage values are similar to the ones obtained with the sensor of Test I. The analysis of the stabiliza-

tion times revealed that this sensor achieved 80.5% and 92.8% of the total voltage variation for 0.983 m/s and 2.354 m/s, respectively, 120 seconds after switching on the wind pipe fan. Moreover, in the alternate response the higher variation obtained presented a percentage of 0.26%, evidencing an excellent agreement between the signals.

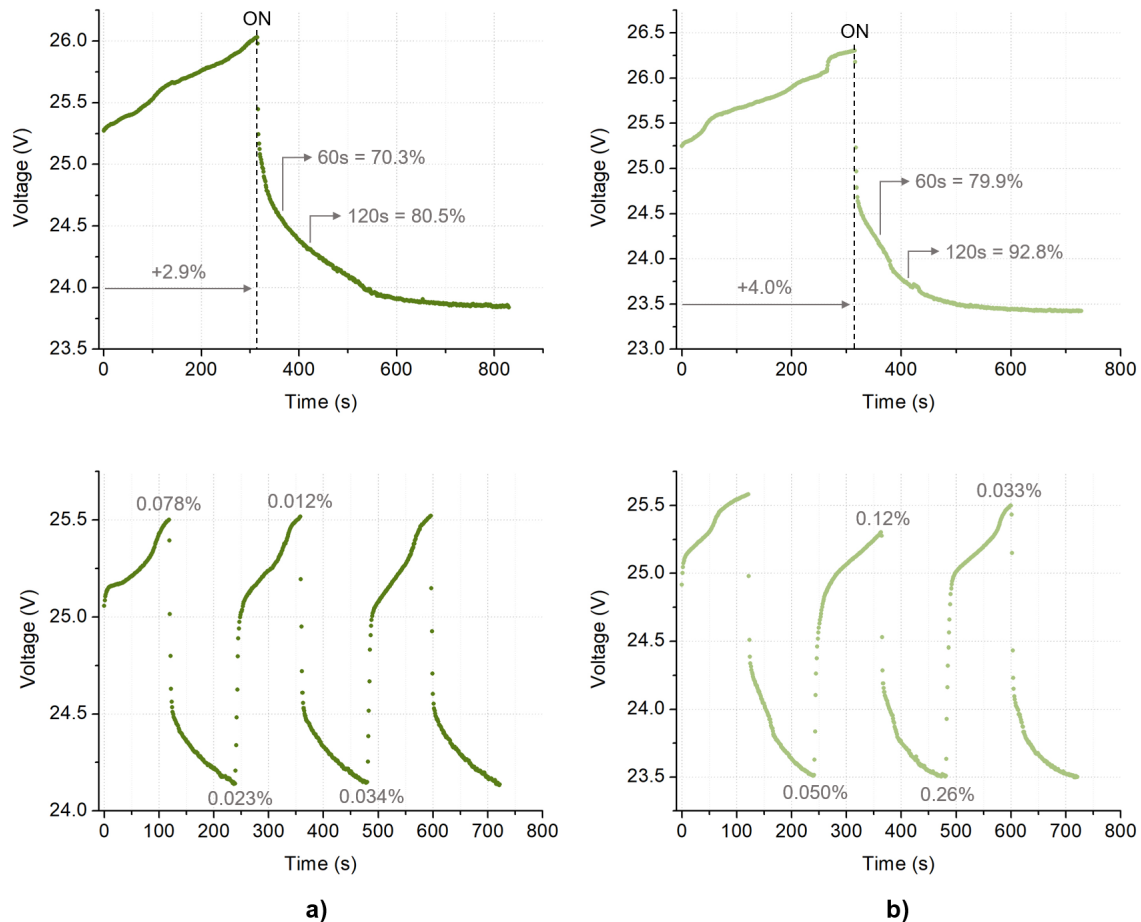


Figure 5.6: Voltage signal evolution for a fixed applied flow (above) and in the presence of an alternate flow (below) for velocities of a) 0.983 m/s and b) 2.354 m/s, both measured with the sensor of Test III.

A common behavior in the results of all three tests is related to the time required for the signal to achieve a steady state for each of the studied velocities. A comparison between the signals of both velocities evidences that the equilibrium is attained more rapidly for a velocity of 2.354 m/s when compared to the case of a velocity of 0.983 m/s. This result is also supported by the percentages obtained in the following 60 and 120 seconds after the switch of the wind pipe fan, which were always superior for a velocity of 2.354 m/s. Then, it is possible to assume that the

increase of the flow velocity promotes a better response of the voltage signal.

5.2.3 Characteristic response

After evaluating the behavior of the voltage signals in the previous sections, the characteristic responses of the sensors presented in table 5.1 were studied for the velocity range between 0 and 2.506 m/s. To guarantee reliable results within a reasonable period of operation, and given the previous results, the voltage value for each flow velocity was measured 120 seconds after the switch of the fan in the respective rate. The characteristic curves presented below follow the relation $-\Delta V$ vs Velocity, with the voltage variation being read in function of the value obtained in the absence of flow.

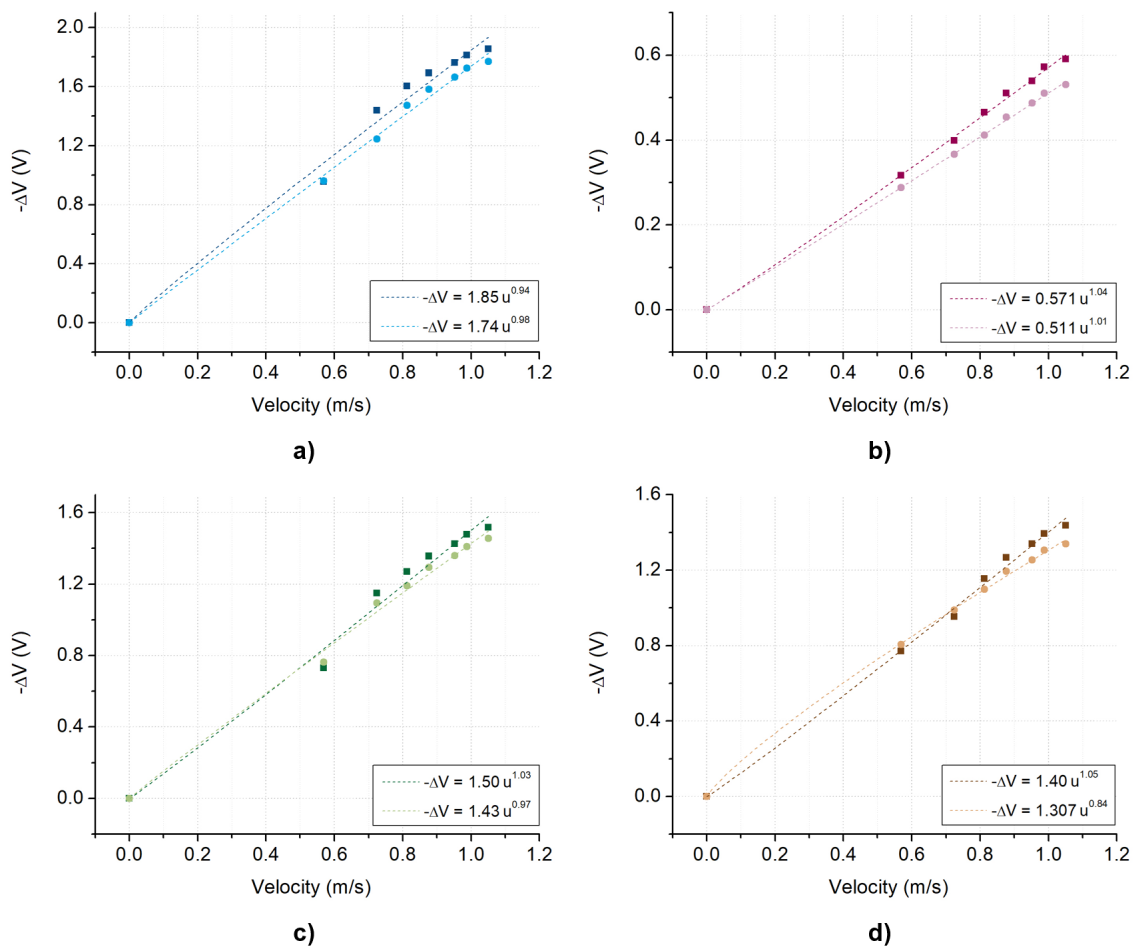


Figure 5.7: Characteristic responses measured for low flow rates with sensors from a) Test I, b) Test II, c) Test III and d) Test IV.

The respective characteristic curves for the tested sensors are presented in figure 5.7 for the lower flow rates and in figure 5.8 with respect to the higher flow rates (see figure 5.1). These results are presented separately since the wind pipe fans operated independently and the sensor monitoring had to be restarted between measurements. For each case, the sensors were tested twice to understand the behavior between different operations. The obtained temperatures at zero flow were approximately 80.0°C, 49.0°C, 78.5°C and 70.0°C for Test I, Test II, Test III and Test IV, respectively, with fluctuations of 4°C. The power of operation was fixed between 3 W and 4 W. The respective experimental data was fitted according to the modified King's law (equation 1.6).

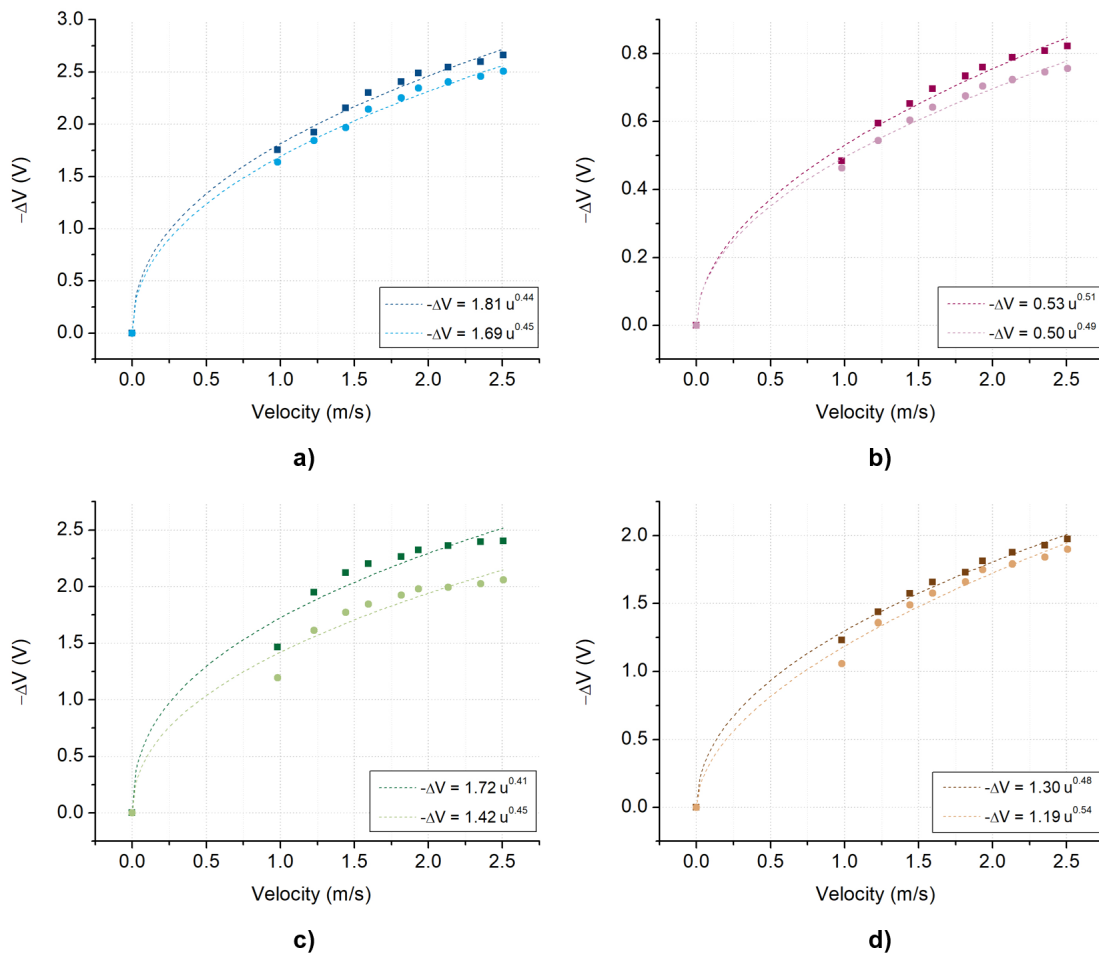


Figure 5.8: Characteristic responses measured for high flow rates with sensors from a) Test I, b) Test II, c) Test III and d) Test IV.

The first aspect to retain from the comparison of both curves is related to the sensitivity of the

sensors. As can be seen, the response for low flow velocities presents a higher sensitivity (the curve is practically linear), whereas for higher velocities the sensitivity decreases with the increase of the flow rate (a regime similar to the square-root is approached). This behavior is characteristic of anemometers, as predicted in subsection 1.2.3. The results of the experimental fitting evidence that the exponent values present a good agreement for all the tested sensors. For low flow rates the characteristics reflected exponents between 0.84 and 1.05, while a variation from 0.41 to 0.54 was obtained for high flow rates.

From the evaluation of the characteristic responses for each tested sensor, it is immediate to conclude that a higher sensitivity was obtained for the sensor of Test I (higher voltage variation). In contrast, Test II presented a lower sensitivity as already expected, since the sensitivity is strongly dependent of the current value applied in the sensor operation. A comparison between the obtained coefficients b validate this assumption, since this parameter is directly related to the sensitivity. For Test I, this parameter varied between 1.69 and 1.85 (error of 3.0%), whilst for Test II the same values decreased for a range between 0.50 and 0.571 (with 2.0% of error), a result that arises from the lower overtemperature achieved with the respective applied current. On the other hand, the sensors from Test III and Test IV evidence a slight decrease of the sensitivity in relation to Test I: the former due to the instability of the signal and the last as a consequence of the decrease in the resistance of the sensor. The values of b varied from 1.42 to 1.72 and 1.19 to 1.40, respectively. For the sensor of Test IV, the maximum error detected for the entire range of studied velocities was 3.4%, while for Test III the same error was 1.4% for lower rates and 49.3% for higher rates.

For low flow rates, the experimental fit revealed a maximum error of 10.7% with respect to the exponent n . The more prominent errors were found for the response of Test III, which can be associated to the poorer stability of the signal. In fact, for the operation of this sensor at higher rates, the exponent n exhibited a major error of 17.1%, a result that, combined to the values of b discussed above, evidence the instability promoted by the device position. Between the sensors of Test I, Test II and Test IV, the maximum errors of the coefficient n reached a percentage of 9.3% for higher flows.

In table 5.2 the maximum deviations obtained between the experimental data of the characteristic responses for each sensor are presented. For the sensors of Test I, Test II and Test IV the deviations decreased with the increase of velocity, which can be related to the improvement

of the voltage signal with the flow rate, as mentioned in the end of the previous subsection. However, for Test III a significant increase in the signal deviation is observed. From the discussion in section 4.2, this result is probably associated to the hydrodynamic entry length; since this length increases with velocity, this indicates that the fully developed region of the flow is achieved successively farther from the sensor position and, consequently, the read output signal is progressively less accurate.

Table 5.2: Percentages of the characteristic response deviation between operations for each tested sensor.

Test	Lower flow	Higher flow
I	15.5%	9.6%
II	12.8%	9.3%
III	6.8%	22.6%
IV	7.3%	5.7%

5.3 Final discussion

The responses obtained in figures 5.7 and 5.8 represent the calibration curves for the operation of each sensor of table 5.1. However, their application is only valid for measurements in the same conditions as the ones used in the calibration process, in particular the flow temperature and the sensor position inside the pipe. Therefore, whenever these parameters change a new calibration curve is required.

Moreover, from the evaluation of the characteristic responses, it is evident that the sensor of Test I presents the most interesting performance for operation. In addition, the results for the sensor molded to the pipe exhibit more stability than the ones obtained with the centered sensor, therefore proving the assumptions discussed in subsection 4.2.

However, the deviation results evidenced that the flow monitoring with these sensors is not absolutely accurate. As a consequence, these sensors are not appropriate for measurements that require a high precision and/or for systems where the flow varies rapidly. In addition, the high power consumption is a parameter that has to be optimized.

In conclusion, these sensors can provide an adequate option for applications that require robust

devices adaptable to non-planar surfaces and with an easy implementation, without demanding an excellent precision. The combination of these conditions with the need of cost-effective devices is also an advantage of the devices presented here, since the process of fabrication is simple and involve low cost materials.

Chapter 6

Conclusions and future work

Flow sensors are devices usually required for the monitoring and control of flow systems, with the main functionalities being the optimization of the energy consumption and/or the evaluation of the quality of those systems.

In this thesis, the challenge was focused on the development of an innovative flexible flow sensor based on thermal principles. One of the main requests was the effective fabrication costs of the sensor. From the state of the art review, and due to the necessity of a simple methodology, the proposed sensor was fabricated by screen printing techniques and followed the hot-film configuration.

To fabricate and study this device, four steps were considered: 1) the geometry of the resistors, 2) the ink formulation, 3) the current controller to operate the sensor under constant current, and 4) the flow system for experimental measurements.

Concerning the combined study between geometries and ink formulations, four different types of formulations (two based on silver, one of nickel and one of carbon) were tested. It was concluded that a serpentine resistor printed with the two different silver inks, Ag-I and Ag-II, provided the best option since it presented a high resistance variation between room temperature and 70-80°C. It was stated that Ag-I evidenced a **TCR** ranging from 0.0051 to 0.0057 (maximum error of 3.9%), whereas for Ag-II the studied samples exhibited a **TCR** between 0.0044 and 0.0045 (error of 2.7%). In order to improve the thermal performance of the resistors printed with these inks, an annealing process was also performed, where a resistance increase up to 53.4% was

observed for the ink Ag-I. This change seemed to arise from the reduction of the film grains promoted by the annealing, which in turn resulted in an increase of the grain boundary resistivity and increased the overall resistivity. In addition, the linear behavior of the resistance response in function of temperature was improved (from room temperature until 90°C). The obtained **TCR** values did not suffered major changes, ranging from 0.0058 to 0.0060 with an error of 1.6%.

A current controller and a wind pipe setup with 12.5 cm of length and a diameter of 7.6 cm were constructed for the flow studies. The overall flow system operated in a range of velocities between 0.569 m/s and 2.506 m/s. The measurements were performed for four different sensors. The variables studied were the device resistance, the applied current and the sensor position in the pipe. Regarding the characteristic responses, the signal followed the expected square-root behavior. For low flow rates, where the response was practically linear, the exponents varied between 0.84 and 1.05 with 10.7% of error. In contrast, for higher flow rates a variation from 0.41 to 0.54 was obtained with an error of 9.3% for sensors molded to the pipe and 17.1% for the centered sensor.

The sensor of Test I (145.9 Ω , 140 mA, molded to the pipe) presented the best results in terms of sensitivity. The coefficient b , that can be seen as a qualitative parameter of the sensitivity, varied between 1.69 and 1.85 with a major error of 3.0%. Moreover, it was seen that a reduction in the applied current (100 mA) promoted a greater loss in sensitivity - reduction higher than 50% of b - than the decrease in the sensor resistance (126.9 Ω). For the sensor located at the center of the pipe, a slight decrease on the sensitivity was also verified.

6.1 Future work

Although the promising results obtained with the presented flow sensor, there are relevant improvements to be performed in future work:

- Numerical simulations could be an important asset, in particular using COMSOL multiphysics that uses the finite element method and gives the ability to combine thermal models with electrical models. A study of new geometries or configurations should be performed in order to optimize the sensor behavior.

- Thermogravimetry and differential scanning calorimetry should be performed to understand the mechanisms on samples subjected to annealing processes, as mass release, optimal temperatures of annealing, and others.
- One of the main issues of this work was the measurement of the samples thickness. Thus, an optimized and methodical study using a profilometer should be performed.
- The annealing process can also be tested for samples printed with nickel and carbon, in order to observe if it is possible to decrease their resistance to values of interest. Other materials and inks with different formulations can be tested to study how they can be used to improve the performance of the sensors.
- With respect to the printing techniques, the resistors can be screen printed with thicker meshes to reduce the resistance obtained for highly resistive materials. In addition, other printed electronic techniques can be tested. For example, inkjet is an interesting option, since it provides a lower resolution than screen printing and therefore allows the reduction of the designs dimensions. This technique also decreases the material waste and allows the test of other materials.
- Finally, the operation of the sensor can be tested for different flow temperatures and other gases than air. Also, the model of the presented sensor has to be optimized in order to advance with the reproduction in large scale and test the product scalability. The studies of the sensor performance can also proceed with endurance tests, for repeated heating and cooling cycles.

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