

# Vanadium Oxide Thin Film Temperature Microsensor

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**Abstract**— Vanadium oxides are known for their thermal properties, such as high temperature coefficient of resistance (TCR). This article presents the development of a low-cost and easy-to-fabricate temperature microsensor, specially designed to be integrated into microfluidic channels, although it can be used in multiple applications. It consists of a thin film of vanadium oxide as a resistive sensor on glass substrates. Two meander-shaped geometries with an area less than 1,5 mm<sup>2</sup> were designed and microfabricated. A steady-state and dynamic thermal characterization was performed to determine its specifications. The sensors demonstrated linear behaviour and an average TCR of 4055 ppm/°C was obtained for a temperature range of 20 to 75 °C.

**Keywords**— microfabrication, sputtering thin film, resistive temperature microsensor, temperature coefficient of resistance (TCR), vanadium oxide.

## I. INTRODUCTION

Temperature is one of the most important magnitudes. Its unit, the kelvin, is one of the seven base units of the International System of Units [1] and can be measured by another temperature-dependent physical quantity.

Thermal sensors are not only used for the analysis of thermal behaviour. They can also be used to measure nonthermal properties like flow velocity [2], radiant heat [3], acceleration [4], heat flux [5], thermal diffusivity [6], etc.

In addition, thin film thermal sensors have some advantages: i) small elements, ii) wide range of temperature, iii) low consumption of materials, iv) the possibility of making custom sensors for specific applications, v) high productivity of already existing equipment and technology for the semiconductor industry, among others.

Nowadays, vanadium oxides are widely used as thermistor material. They have several crystalline phases and some of them show metal-to-insulator phase transitions. This multiplicity of options adds more steps to the fabrication process, making it complex and, therefore, difficult to reproduce. For films grown using the magnetron sputtering technique, this generally means that the deposition of the films must be done while heating the substrates or that post-deposition thermal annealing is needed [6] - [10].

In this work, a thin film thermal microsensor is presented based on the variation of electrical resistance. This sensor will be used to measure temperature in microfluidic systems. Due to

that, both design and manufacturing have to be compatible with the common microfluidic chip fabrication processes and their materials. In this case, the fabrication was carried out at room temperature and without additional thermal treatments, yet still achieving the necessary specifications for the aforementioned application. The design, fabrication, and materials, as simple and inexpensive as possible, will be detailed in sections II and III.

Section IV presents the characterization and results. As the microsensor will be used to measure liquid water temperature, a range of 0 to 100 °C would be sufficient. Considering that the devices will likely to be used for biological applications, the operating range would be narrower. The dynamic response is of particular interest for future applications in flow measurements.

## II. DESIGN

The substrates chosen were 25-mm-wide and 75-mm-long soda-lime glass slides because of their high electrical resistivity (3981  $\Omega \cdot \text{m}$  at 300 °C) and their very low cost. Moreover, they are easy to integrate into microfluidic devices, and their shape is compatible with most laboratory equipment. One disadvantage of this material is its susceptibility to surface degradation in case of long-term exposition to moisture due to its high percentage of Na<sub>2</sub>O. Alkali ions such as sodium can easily move out from glass even at a temperature below the glass transition. This phenomenon can form an interface between the glass and thin film and can also alter the mechanical or electrical properties of the film [11],[12]. Additionally, soda-lime glass has a deviation from flatness of 2  $\mu\text{m}/\text{mm}$  [13] which is high compared to other common substrates such as silicon wafers. In this case, they are

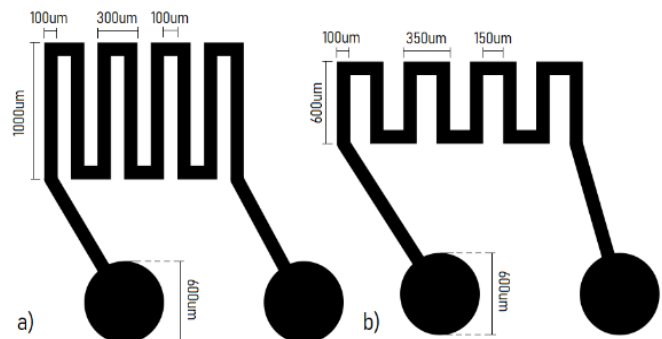


Fig. 1. Mask design for (a) S1 and (b) S2 sensors.

considered flat enough because of the dimensions of the designs presented below.

As thin film resistor material, a vanadium oxide ( $\text{VO}_x$ ) was chosen because of its high electrical resistivity and temperature coefficient of resistance (TCR). Vanadium oxides have multiple possible stoichiometries and crystalline phases which will grow under different process conditions. Because of that, the properties of the material will vary depending on the fabrication technique and its conditions.

Two meander-shaped geometries (S1 and S2) were proposed for the sensors, and they are shown in Fig. 1. The whole designs measure approximately 2 mm long and 2 mm wide. The meanders were designed to fit below microchannels with widths from 600 to 1200  $\mu\text{m}$ .

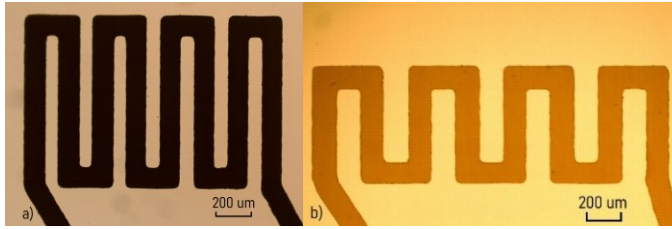


Fig. 2. Microscope images of (a) S1 and (b) S2 meanders.

### III. MICROFABRICATION

The substrates were cleaned in a piranha solution (98 wt %  $\text{H}_2\text{SO}_4$  and 30 wt %  $\text{H}_2\text{O}_2$ , volume ratio of 2:1 for 10 minutes, at room temperature). After that, they were rinsed in deionized water and dried with nitrogen. Then a photolithography process was performed. A photoresist (TI 35E, MicroChemicals GmbH) 4  $\mu\text{m}$  thick layer was coated with a spin coating system (Delta 20 BM, BLE Laboratory Equipment GmbH). After that, the substrates were soft-baked and exposed to 365 nm UV light with a mask aligner (EVG 620, EV Group).

Then a reversal bake was performed to cross-link the exposed areas making them insoluble to the developer. The coated substrates were exposed for the second time without a mask (flood exposure) and the last step was the development.

The  $\text{VO}_x$  thin film was deposited using a magnetron sputtering system (Auto 500, Boc Edwards) with a vanadium dioxide ( $\text{VO}_2$ , 99.9 % purity, Métaux Céramiques Systèmes Engineering) target. The deposition was performed with the following conditions: 200 W of RF power,  $2.7 \times 10^{-3}$  mbar of working pressure, 9 sccm of Ar, 3 sccm of  $\text{O}_2$ , 60 minutes of deposition time, and a substrate temperature below 50  $^\circ\text{C}$ .

The lift-off was performed in an ultrasonic bath using acetone for two minutes leaving the film as patterned (Fig. 2).

### IV. CHARACTERIZATION AND RESULTS

The thickness of the films was characterized with a stylus surface profiler (DektakXT, Bruker Corp.). As it is shown in the measurement of Fig. 3, a 280 nm film was deposited on the substrates.

For thermal evaluation, a temperature calibrator (TC-400, Tek Know Technology) was used. A multimeter (8845, Fluke Corp.) and a switch system (7001, Keithley Instruments) were

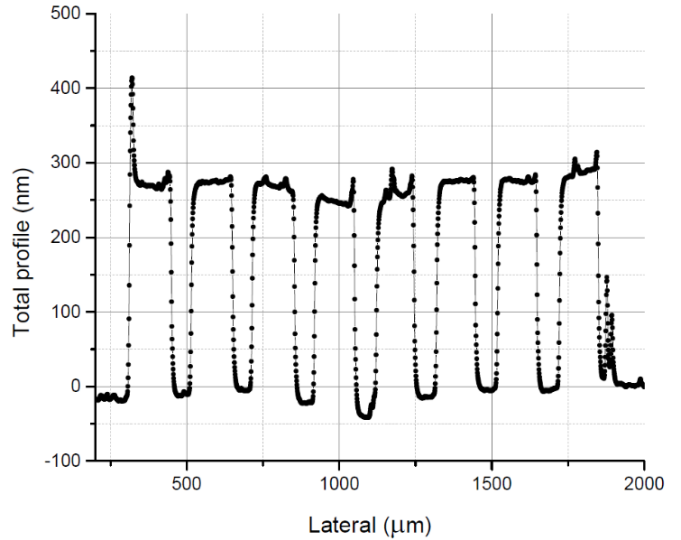


Fig. 3. Thin film step height measurement on top of the meander design of S1.

connected to the sensors to measure them by a four-lead method. A program in LabVIEW software was used to register the values. A diagram of the characterization set-up is shown in Fig. 4.

For the steady-state characterization, averages of the measured electrical resistances ( $R$ ) for both designs at different temperatures in the range of interest (from 20 to 75  $^\circ\text{C}$ ) are shown in Fig. 5. The results indicate that the resistance is linearly proportional to the temperature. In addition, Fig. 6 shows the changes in the resistance of the S1 device by increasing the temperature. All measured points were made after 30 minutes to allow the system to reach a thermal steady state. In addition, the sensors were tested with the same procedure but for a broader range of temperature (from -10 to 120  $^\circ\text{C}$ ), as shown in Fig. 7. It is observed that the curve fits slightly better with an exponential equation than with a linear one. This behaviour was expected due to the semiconductor characteristics of the vanadium oxide [14], and it is prone to increase with a

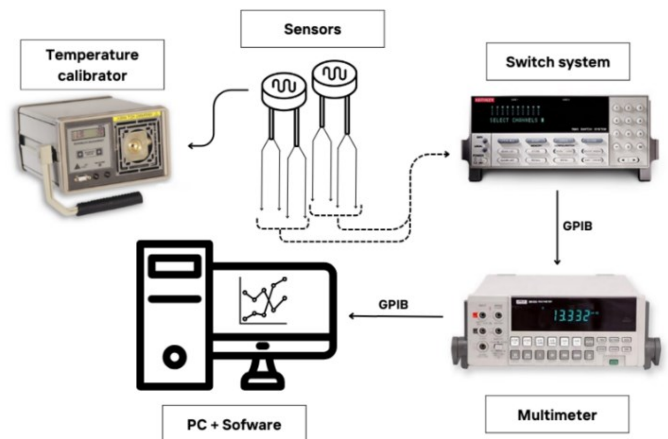


Fig. 4. Set-up for sensors characterization.

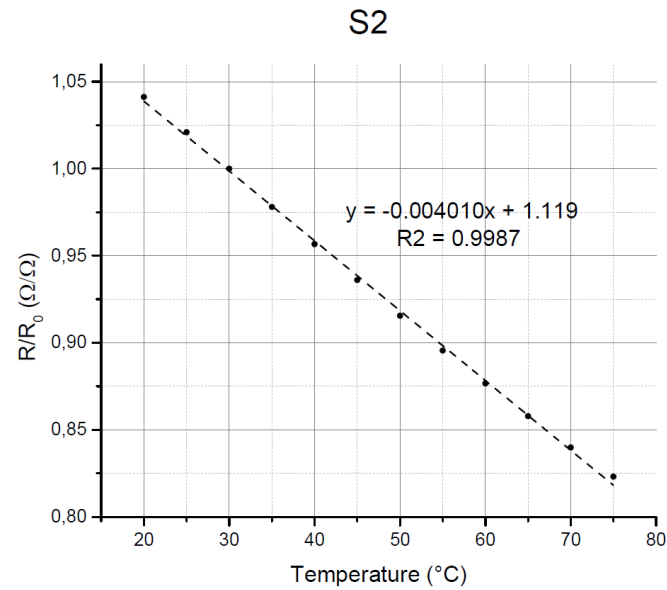
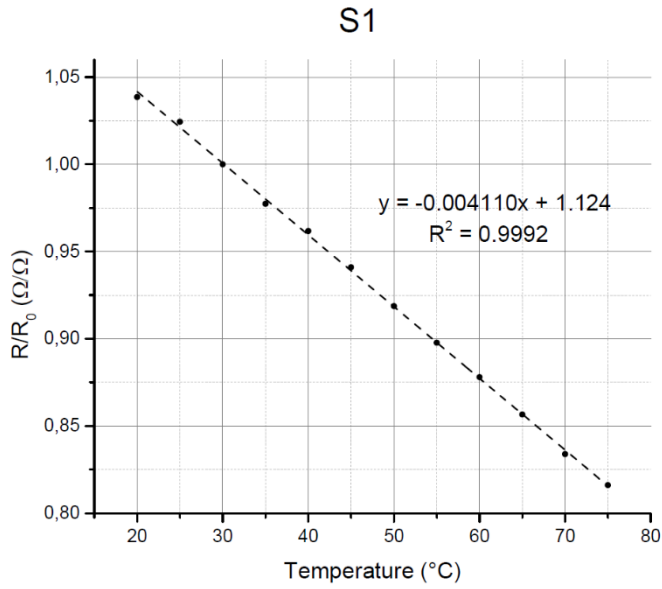


Fig. 5. Steady-state fitting curves for S1 and S2 sensor designs.

further increase of the temperature range. This must be considered for applications requiring higher or lower temperatures.

$TCR$  is defined with (1). When the film is thicker than a few hundred angstroms it can be assumed that  $dR/dT$  is independent of temperature. Because of that, (2) can be used instead, where  $R_0$  is the resistance at 30 °C ( $T_0$ ) and  $R_n$  is the resistance at some other temperature ( $T_n$ ). With the results of these experiments  $TCR$  was calculated in ppm/°C for both designs and it is shown in Table I. This property depends on the material structure, composition, and thickness of the film, so it is almost the same for both designs. In contrast, there is a difference in the resistance at ambient temperature. The S1 design has a higher resistance because of its meander length. This implies a higher output signal, which results in enhanced sensitivity.

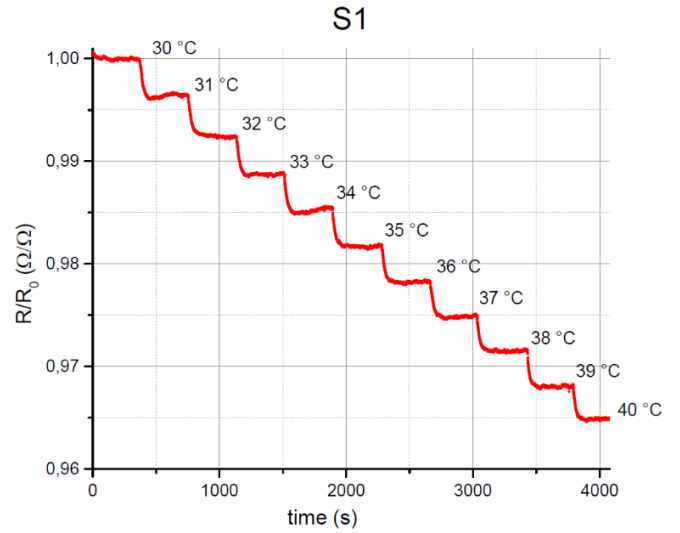


Fig. 6. Changes in relative resistance of S1 sensor by increasing 1 °C (from 30 to 40 °C) versus time.

$$TCR = \frac{1}{R} \frac{dR}{dT} \quad (1)$$

$$TCR \approx \frac{(R_n - R_0)}{R_0 (T_n - T_0)} \quad (2)$$

TABLE I. ELECTRICAL PROPERTIES OF THE FABRICATED SENSORS

Sensor	R@25 °C (kΩ)	TCR (ppm/°C)
S1	77.2	-4110
S2	72.2	-4010

The  $TCR$  values resulted to be lower than others specified in previous works from other authors. For example, in [15] -7 %/°C

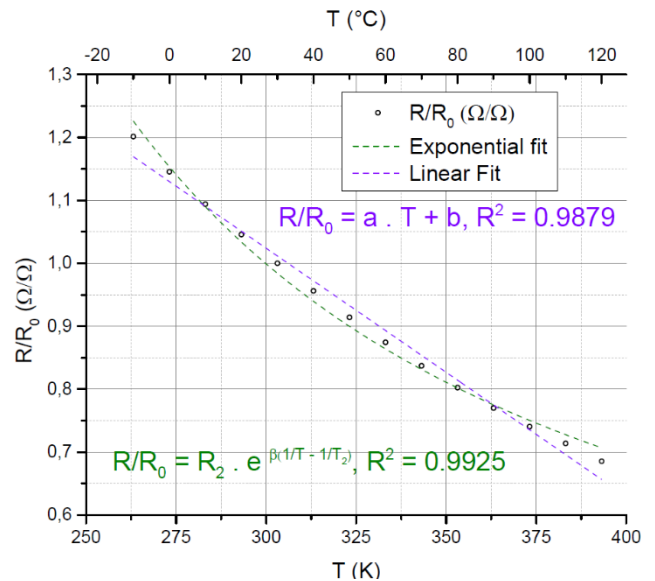


Fig. 7. Steady-state fitting curves for S2 with different equations for a wider range of temperature (from -10 to 120 °C).

TCR is reported for a VO<sub>2</sub>(B) thin film deposited via magnetron sputtering. Intrinsic structure optimization of the resistive material (e.g., crystal morphology) leads to an improved TCR. One way to achieve this is by adjusting the thin film deposition parameters. Another way is adding post-deposition thermal annealing to obtain a different stoichiometric ratio or crystalline phase.

In this work, the resistance curves showed a good linearity for the specified temperature range. Also, the film growth was made with no need of heating the substrates so the film can be structured via lift-off, which is simpler and cheaper than an etching process.

In addition, the same experiment was carried out cooling down the sensors. The curves are shown in Fig. 8 for S2. The difference between the points at the same temperature varies from 0.06 to 0.2 %. These curves indicate that this difference increases with the temperature although they are very similar.

For the dynamic performance characterization, the step response of the devices was studied to characterize its sensing velocity. First, the sensors were left in air at room temperature

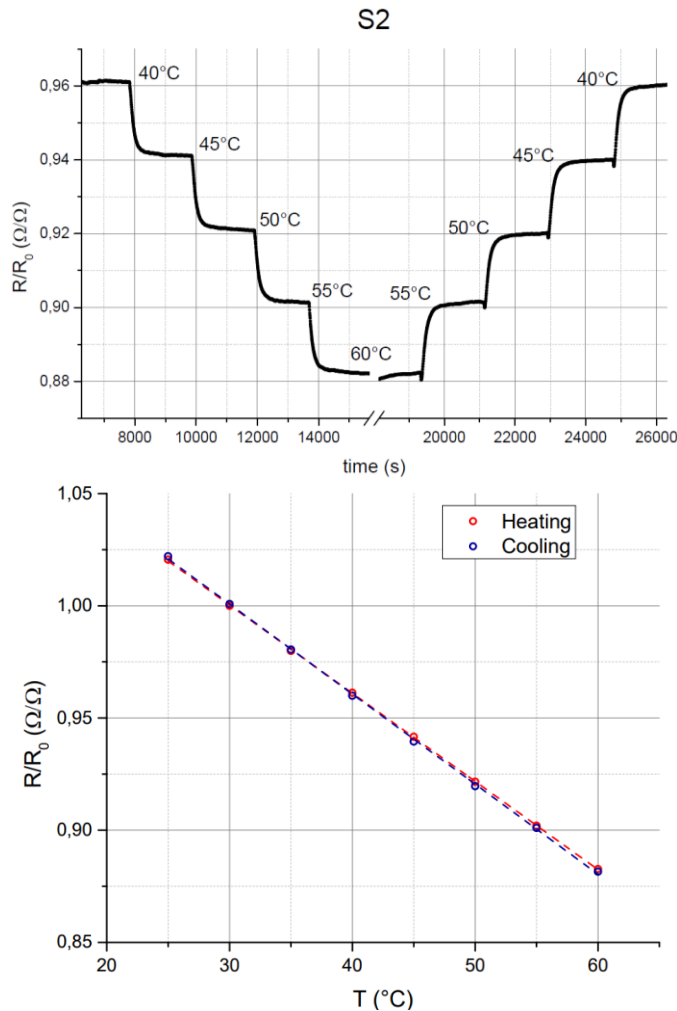


Fig. 8. Thermal characterization of S2 sensor from 40 to 60 °C. (a) Variation of resistance, by increasing (left) and decreasing (right) the temperature 5 °C each time. (b) Average resistance for different temperatures for heating and cooling processes.

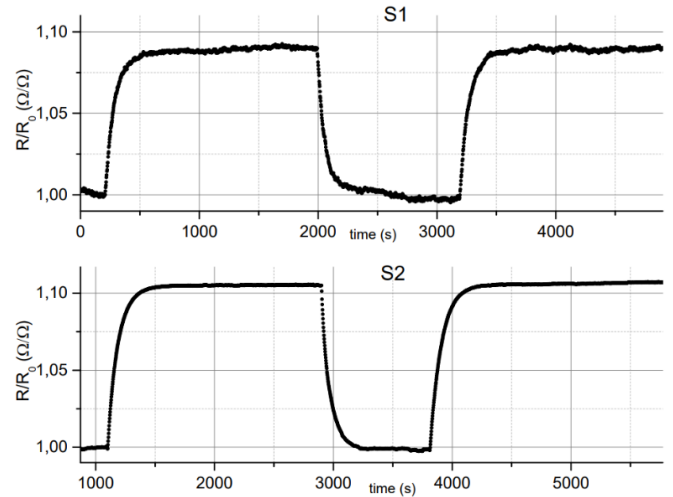


Fig. 9. Relative resistance variations for step changes from ambient temperature to 4°C.

until they came to equilibrium. Then, they were placed inside the temperature calibrator at 4 °C. The results of this experiment are presented in Fig. 9 for both sensors.

In Table II the required times for the sensors to change from their initial value to 63.2 % and 95 % of the final resistance are shown. According to international standards [16], as its ratio lies between 3.0 and 3.7 it can be assumed that the sensors exhibit a single exponential response. Hence, the time constant can be considered as the 63.2 % response time. Considering that, the time constants for S1 and S2 are 81 s and 92 s, respectively. These results could be improved by reducing the thermal mass of the sensors using another substrate with higher thermal conductivity. For the intended application, the time constants obtained are acceptable. A summary of the obtained specifications is presented in Table III.

TABLE II. DYNAMIC RESPONSE CHARACTERIZATION PARAMETERS

Sensor	63.2 % response time (s)	95 % response time (s)	Ratio of 95 % to 63.2 % response time
S1	81	284	3.53
S2	92	282	3.05

TABLE III. SENSORS SPECIFICATIONS

	S1	S2
Temperature range	20 to 75 °C	20 to 75 °C
Nominal R – T characteristic	$R = m T + b$	$R = m T + b$
m	-0.04110 Ω/°C	-0.04010 Ω/°C
b	1.124 Ω	1.119 Ω
R@25 °C	77.2 kΩ	72.2 kΩ
TCR	-4110 ppm/°C	-4010 ppm/°C
Time constant	81 s	92 s

## V. CONCLUSIONS

Vanadium oxide thin film temperature microsensors were designed and microfabricated without thermal post-processing. A steady-state and dynamic characterization was performed.

A linear response was observed for the temperature range of interest (from 20 to 75°C). A TCR from -4010 to -4110 ppm/°C and an average electrical resistance of 75 kΩ were obtained. The dynamic response yielded a time constant of 81 to 92 s. These results are very promising for the intended applications, like sensor integration in microfluidic devices.

For a wider range of temperatures, an exponential response should be considered. Given that the results were satisfactory, a miniaturized version or the use of other substrates, such as borosilicate glass or silicon, could be considered for more restricted applications.

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