

**École polytechnique de Louvain**

# **Study of a piezoresistive pressure sensor with a thin film of vanadium oxide as active layer**

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## ABSTRACT

This master thesis proposes the study of a piezoresistive pressure sensor with a novel design and the piezoresistive characterization of two materials. A first order analytical model of a pressure sensor is studied using *MATLAB*, and a comparison with a numerical model given by *COMSOL Multiphysics* is performed. A new way to concentrate the stresses with a patterning of the piezoresistive layer is studied. A process flow for the fabrication of the sensor of interest has been elaborated and partially conducted in a cleanroom environment. Issues that stopped the process are described and analysed. Thin films of vanadium oxides have been deposited using reactive sputtering with different oxygen concentrations. The impact of the deposition parameters on the residual stress and on the gauge factor of the oxides has been investigated. The piezoresistive properties of vanadium oxides and indium gallium zinc oxide are measured through a bending procedure. The highest gauge factors measured for  $VO_2$  and IGZO are 61.7 and -419.3 respectively.

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Pressure sensors belong to the oldest market in the field of micro electro-mechanical systems (MEMS). Their small size, low power consumption and easy fabrication have made them indispensable, from the automotive to the aerospace industry. Many different transducing techniques are used going from piezoresistive since the 1970's to more recently inductive sensing.

A particular interest is given to **piezoresistive devices** in the course of this work. Such sensors are based on the change of resistance of a material when it is put under strain, arising from a change in its crystalline structure. Strain is applied on the material that is shaped as resistors and deposited on a membrane that will deflect and deform due to the pressure. The most common material used for building such resistors is doped silicon.

This master thesis proposes the **study of a novel design for piezoresistive pressure sensors**. This new layout uses a thick piezoresistive layer deposited on almost the entire surface of the membrane, providing a different stress distribution than usual. The distribution is numerically analysed, and the positions of the piezoresistors on the sensing diaphragm are considered accordingly.

Along with this objective, the **piezoresistive behaviour** of two materials is investigated. Vanadium Oxides ( $VO_X$ ) and Indium Gallium Zinc Oxide (IGZO) are considered, in the form of thin films. As their piezoresistive properties are not well documented in the literature, they have been further explored, measured and discussed throughout this report.

To properly understand the important concepts that drove this work, the present text starts with the state of the art of the most commonly used **pressure sensing techniques** and by a brief **review of the materials** of interest. In addition, the different **figures of merits** used to classify and evaluate pressure sensors and piezoresistive materials are defined.

Once all the basis are set, **design considerations** to take into account when building a piezoresistive pressure sensor are tackled in an **analytical analysis**. This chapter provides a first order model combining mechanical and electrical aspects. It is then compared to a more complete model built using *COMSOL Multiphysics*. Once validated, the **numerical model** is refined to identify the impact of a patterned piezoresistive layer on the sensor performances.

Then, in order to validate the results proposed by the numerical analysis, attempts to **fabricate the sensor** with the patterned layer have been performed in *Winfab* at *UCLouvain*. The idea was to build multiple versions of the device with varying parameters such as the size of the diaphragm or the positions of the resistors to characterize the sensing performances in *Welcome*. Nevertheless, technical issues prevented the completion

of these tests but the associated process flow is still described and analysed.

The **characterisation** of the two materials was finally conducted without applying pressure on a membrane, but rather by the bending of thin films deposited in *Winfab* as well. Some of their piezoresistive properties are extracted, and this work is concluded by some **improvements and perspectives** associated to these results.

The objective of this chapter is to provide the necessary background to understand all the concepts tackled in this master thesis. The topics of interest are divided into different sections which lead together to the application that guided this work: a **piezoresistive pressure sensor with a novel architecture for its active layer**.

The first section defines what a MEMS pressure sensor is and quickly reviews the different **types of sensors** that exist. Particular attention is paid to the configuration of the **sensing membrane** and to their fabrication.

The state of the art of the mostly used **pressure sensing techniques** in the field is then presented in the second section, along with the main concepts needed to understand each technology.

The two next sections provide important tools to understand the specifications of a pressure sensor: the **figures of merits** used to classify and evaluate the performances of a device given by the literature are developed, and three **materials** are studied regarding their piezoresistive properties.

Finally, a **comparison of sensors** found in literature using different sensing principles is completed, demonstrating the variety of specifications achievable depending on the desired application.

## 2.1 Pressure sensors

In this section, the different types of pressure sensors that exist and their applications will be listed. Since all the reviewed sensors use a **membrane**, the first step will be to define clearly what a membrane is and to point out its main characteristics.

### 2.1.1 Defining a membrane in MEMS sensing applications

Most of the pressure sensing devices are based on the **deflection** of a membrane. This is why understanding the different types of membrane that exist is important.

**Definition of a membrane** According to the literature, a membrane is an object where two dimensions are more important than the third one [1]. Its flexural rigidity is very low so it can be neglected, and it can only support tensile load. It can be described only by its mass per unit area and the tension it supports per unit length of boundary.

In the world of micro electro-mechanical systems (MEMS), a membrane is an object that has two dimensions of the same order of magnitude and a third one called the *thickness* that is of a lower order of magnitude. It is a released structure since it has no supporting material underneath and is clamped by its side. It can also be called a *diaphragm* sometimes [2].

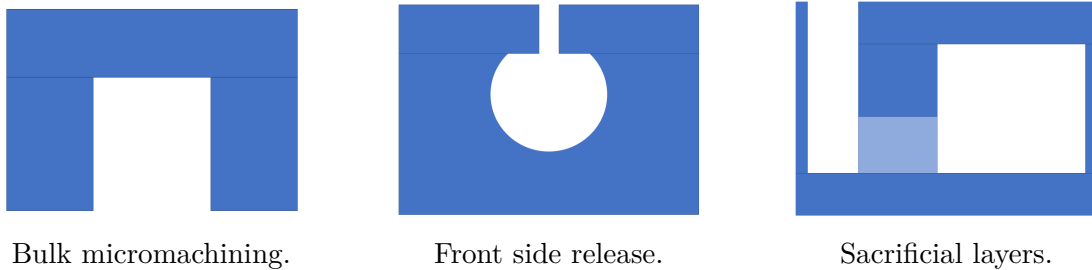


Figure 2.1: Mostly used techniques for building MEMS membranes.

The most common ways to build a membrane for MEMS are presented here after [3]:

- **Bulk micromachining** : The materials composing the membrane are stacked on top of each other. These layers can include the other functional parts of the sensor like the resistors, the wires or the electrodes. When all the layers are fabricated, the membrane is released from the back side with an etching technique.

- **Front side release** : Etching of material under the membrane is done by an isotropic etchant. This involves the appearance of a gap in the membrane that may have to be filled later on.

- **Sacrificial layers** : A material that will be etched later is used to hold the membrane layers together. When all the layers are deposited and designed, the material is etched, thus releasing the membrane.

A schematic representing those three techniques can be found in Figure 2.1.

The two main shapes for the membranes are the **square** and **circular** ones as shown in Figure 2.2.

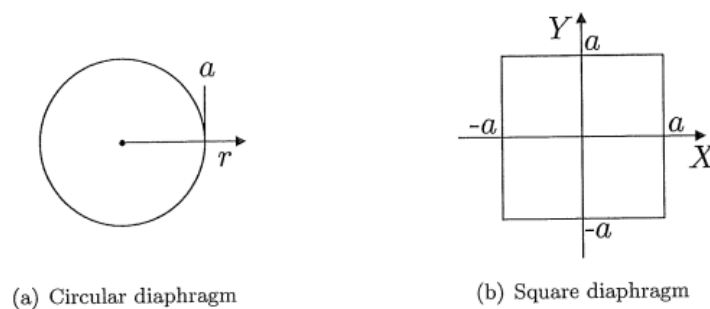


Figure 2.2: Two main membrane geometries [2].

### 2.1.2 Types of pressure sensors

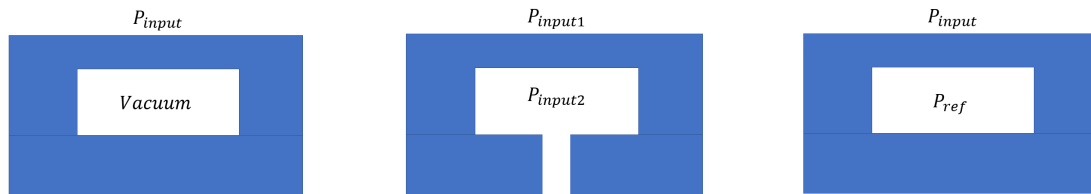
There are 3 main types of pressure sensors. Each one of them will be analysed here after.



**Absolute pressure sensor** This type of sensor measures the absolute pressure, which means that the applied pressure is compared to vacuum. As illustrated in Figure 2.3a, one side of the diaphragm is exposed to the pressure to be measured while the other side is in a sealed cavity where an "almost" vacuum is created. The creation of this vacuum is rather complicated and these devices are mostly used for low pressure sensing applications. One way to do it is to bond two wafers in vacuum conditions [4]. The absence of gas inside the cavity ensures low temperature sensitivity.

**Differential pressure sensor** The basic idea behind this device is that the pressure difference is measured between the two sides of the membrane. These devices are mainly useful when the pressure has to be measured between two different points in a system.

**Gauge pressure sensor** The architecture of this sensor is the same as this of the absolute pressure sensor, but the cavity is at an arbitrary pressure which is not vacuum. A common reference is the atmospheric pressure.



(a) Absolute pressure sensor. (b) Differential pressure sensor. (c) Gauge pressure sensor.

Figure 2.3: Types of pressure sensors.

## 2.2 Pressure sensing principles

There are different pressure sensing methods. This section aims to review some **transducing techniques**, namely capacitive, inductive, piezoelectric and piezoresistive techniques, as well as **other common mechanisms** such as resonating and optical sensing.

### 2.2.1 Capacitive sensors

The structure of a capacitive sensor consists of two conducting plates separated by a dielectric as shown in Figure 2.4. One of the plates is fixed while the other is moving due to the applied pressure. As the capacitance value is a function of the distance between the two plates, the applied pressure can be measured through the variation of capacitance. Many materials such as graphene [6] or polymers [7] can be used as dielectric, but the most common material is air.

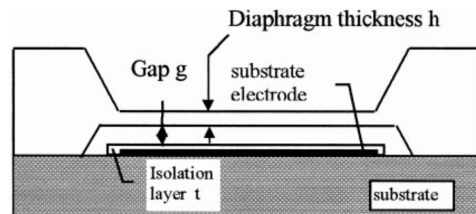


Figure 2.4: Common design of a capacitive pressure sensor [5].

The main equation for capacitive sensing is the following:

$$C = \frac{\epsilon_r \epsilon_0 A}{d} \quad (2.1)$$

When air is used as dielectric, the main contribution to the change of capacitance comes from the variation of distance between the two plates. But when using polymers, the deformation will be reduced since these materials are less flexible than air. That is why micro-machining [8] and adding air voids [7] are done to the lower elastic moduli and increase the deflection. The advantage of adding air voids is that when compressed, the overall permittivity changes as the air is expelled from between the plates.

The advantages of capacitive sensing compared to other techniques are a high pressure sensitivity, a large fabrication area, smaller temperature drifts and reduced power consumption [9]. But on the counterpart, a non-linear response in the change of capacitance with respect to the applied pressure is observed [10]. Such sensors also have a high output impedance, which affects the interface circuit design, and they suffer from parasitic effects. Capacitances due to electrical connections can be of the same magnitude as this of the sensor, and they are prone to electromagnetic interference. Reducing these effects can be performed by reducing the magnitude of the parasitics during sensor design (single-chip integration, physical separation,...) or by minimizing the impact of the effects with circuit design (shielding and grounding, bootstrapping) [2]. Finally, when polymers are used as dielectric, the sensors can have a low time response due to the high elastic modulus.

The main fields of application for capacitive sensing are tyre pressure monitoring for the automotive industry [10], touch sensors for the robotic industry [11], and finally altitude monitoring, weather forecasting, air speed monitoring for the aerospace industry [12].

## 2.2.2 Inductive sensors

Inductive sensing is done by having two spiral square coils, one fixed and one moving on top of the other, separated by a gap. The inductance formed by this structure has two main parts: the fixed inductance formed by the coil structure  $L_s$  and the mutual inductance of the planar coils  $M_s$ . The second one is formed by the varying mutual inductances between the two aligned coils  $M_v(d)$ , which depend on the distance between the two coils [13]. Those devices show an excellent environmental stability, a high sensitivity, and they enable a wireless design. Nevertheless, they are less used than other technologies in the field as they are more difficult to manufacture and tend to have a larger footprint than the other devices [8].

Two different designs has been researched, one is based on the *Villari effect* and the other uses a high permeability *ferrite*.

**Magnetostrictive pressure sensor** The so called *Villari effect* occurs when the permeability of a material changes with applied stress [15]. As presented in Figure 2.5, when pressure is applied on

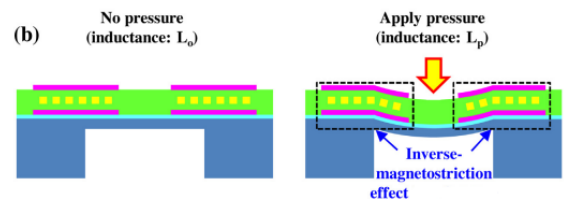


Figure 2.5: Design of a magnetostrictive sensor [14].

the membrane, the magnetic film (in purple) is under a stress denoted  $\sigma$  and the magnetic anisotropy field  $H_s$  of the film changes with respect to [14]:

$$H_s = \frac{3\sigma\lambda}{M_s} \quad (2.2)$$

where  $\lambda$  is the magnetostriction constant and  $M_s$  saturation magnetization. The permeability of the magnetic film will change according to:

$$\mu = \frac{4\pi M_s}{H_0 - H_s} \quad (2.3)$$

where  $H_0$  is the anisotropy field before applying stress on the membrane. Putting these two equations together gives the final relationship between permeability change and applied stress:

$$\mu = \frac{4\pi M_s^2}{M_s H_0 - 3\sigma\lambda} \quad (2.4)$$

The permeability variation translates into a variation of the inductance given by :

$$L = \frac{\mu_0 \cdot W_t^2 \cdot N^2}{2 \cdot T_i + \frac{T_i}{(L_m^2/W_t^2 - 1/2)} + \frac{\sqrt{2} \cdot W_t \cdot L_m}{8 \cdot \mu_{eq} \cdot T_m}} \quad (2.5)$$

where  $T_i$  is the thickness of the insulation,  $W_t$  the width of total coil turns,  $L_m$  the length of the magnetic film and  $T_m$  the thickness of the magnetic film. The variation of inductance is thus related to the stress applied on the magnetic film: the applied pressure on the membrane which will induce stress on the magnetic film and translate into the variation of inductance. To measure this variation, the chip was wire bonded on a printed circuit board (PCB) which was then put in a pressure chamber. The pressure was monitored with a pressure gauge and the PCB was connected to an LCR meter as shown in Figure 2.6. The magnetic films are made of CoFeB as this material has an excellent temperature stability and a very good gauge factor.

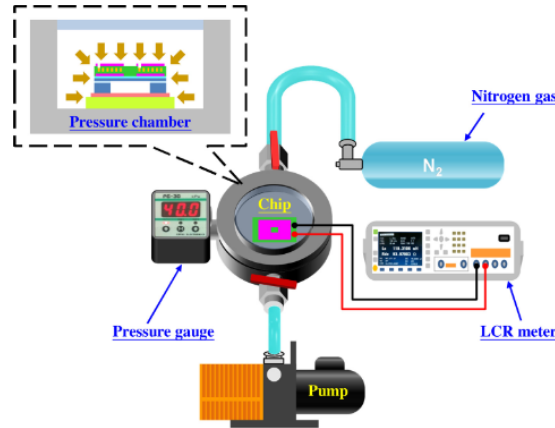


Figure 2.6: Measurement circuit for the magnetostrictive based sensor [14].

**Ferrite films based sensors** Another design uses a ferrite that is attached below a membrane with a planar sensing coil underneath [16]. As the permeability of the ferrite is extremely high, the inductance of the planar coil is significantly increased. The effective inductance  $L_0$  is given by:

$$L_0 = \frac{N^2 \mu_0}{\frac{l_f}{A_f \mu_r} + \frac{l_a}{A_a}} \quad (2.6)$$

where  $N$  is the number of turns of the coil,  $\mu_0$  the permeability of vacuum,  $l_f$  and  $A_f$  the length and area of the magnetic circuit, and  $l_a$  and  $A_a$  the length and area of the magnetic path in the air. When pressure is applied on the membrane, the ferrite film is deflected, leading to a change in inductance:

$$\frac{\Delta L/L_0}{\Delta P} = \frac{a^2(1-\nu^2)}{5ET^3(\frac{1}{t\mu_r+C})} \quad (2.7)$$

The measurement was done by using an impedance analyser to assess the inductance. The pressure was applied by using a mechanical point load on the sensor surface and monitored with a force gauge, where the pressure was equal by the force divided by the surface.

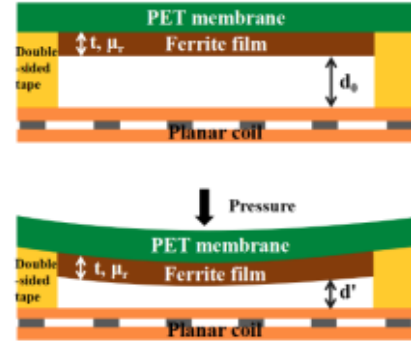


Figure 2.7: Ferrite based sensor [16].

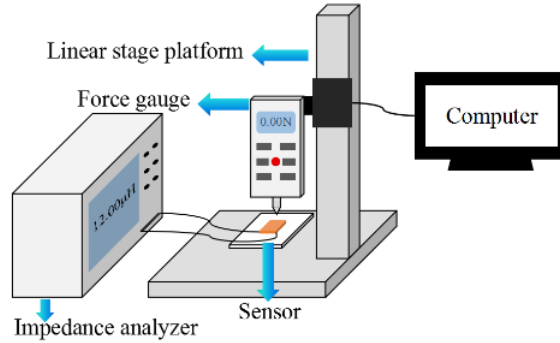


Figure 2.8: Measurement circuit for the ferrite based sensor [16].

### 2.2.3 Piezoelectric sensors

The piezoelectric effect comes from the displacement of ions inside the crystalline structure for *inorganic* materials, and of molecules for *organic* materials. When stress is applied on the structure, the balance of the ions/molecules inside the material changes and dipoles are created, leading to the appearance of an electric charge and polarisation of the material as shown in Figure 2.9. Only non-centrosymmetric structures can exhibit the piezoelectric effect, as the created dipoles cancel out in other structures [17].

Piezoelectric sensors thus convert an applied stress into an electric field. Those sensors are self-generating and do not need to be powered by an external source to generate a signal [2].

One of the main drawbacks however is that it can only measure a *change* in the applied pressure because piezoelectric materials are prone to charge leakage when a static force is applied on them [19]. Another source of concerns is that they are sensitive to temperature because of the pyroelectric effect [20].

They are mostly used for dynamic tactile sensing [21], like for a robotic hand.

One of the most promising material for piezoelectric sensing is based on glycine-chitosan and was used in a flexible pressure sensor [22]. The main interesting property here is that this material is biodegradable and easy to fabricate.

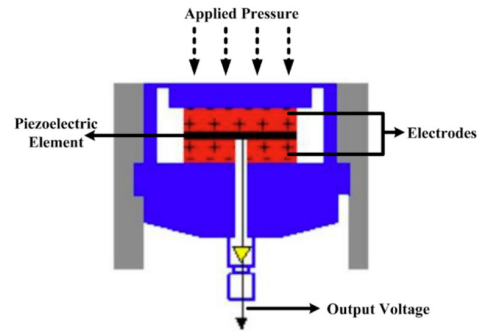


Figure 2.9: Common design of a piezoelectric pressure sensor [18].

## 2.2.4 Piezoresistive sensors

Here the transducing effect is a variation of the transducer resistivity through deformation under mechanical stress. The bandgap of the material changes with the applied strain, influencing the mobility of the carriers and thus its overall electrical resistance. This phenomenon occurs in all materials, but not always in a significant way [2].

In the case of pressure sensors, the pressure acts on a membrane where transducers called *piezoresistors* are implanted, these resistors vary due to the induced stress on the membrane. If a fixed current passes through the varying resistors, the measured voltage will be a function of the applied pressure. The use of piezoresistors has the advantage of producing an output that can easily be read by a simple measuring circuit, with a linear response for *thin diaphragms* and *small deflections* [18]. In addition, the piezoresistors are inherently shielded. But the drawbacks are a large power consumption, a large temperature dependency, and the noise in the resistors can be quite large.

Typical design of a piezoresistive pressure sensor is shown in Figure 2.10. It consists of a diaphragm and four piezoresistors forming a *Wheatstone bridge* to provide an electrical output. The main applications are tyre pressure sensing [23], biomedical applications [24] and ultra high temperature sensing [25]. The last one uses an SOI wafer to reduce the leakage currents that are increased by temperature.

## 2.2.5 Resonating sensors

A resonant sensor does not involve a transducer compared to previous techniques, but is composed of a vibrating element excited at resonance. The applied pressure will result in a change in stress that will induce a variation of the resonant frequency of the device. The

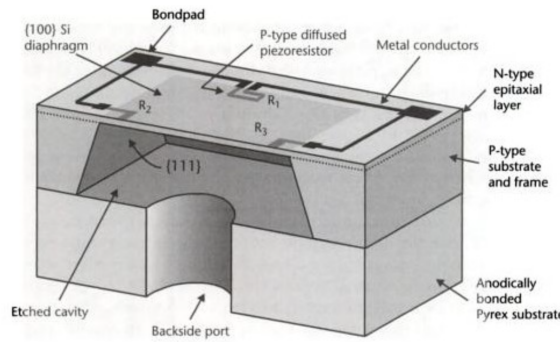


Figure 2.10: Common design of a Si piezoresistive pressure sensor [9].

main equation for those devices is given by [26]:

$$f = f_0(1 + \alpha p^2)^{1/2} \quad (2.8)$$

where  $f_0$  is the resonant frequency without pressure,  $p$  the applied pressure and  $\alpha$  the sensitivity. Those devices are composed of a membrane and two piezoelectric crystals. Both are on the membrane and one acts as an exciter while the other acts as a receptor.

A typical measurement system can be found in Figure 2.11. The working principle

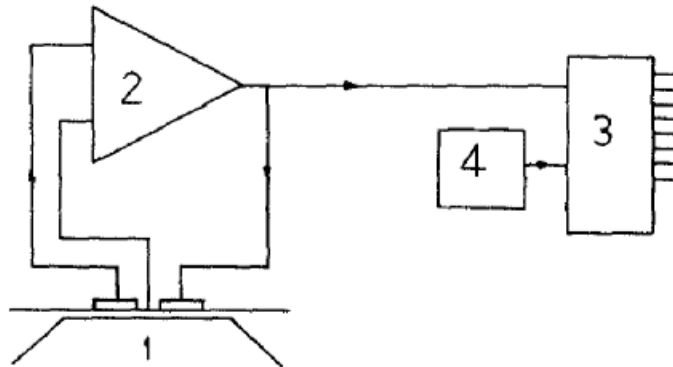


Figure 2.11: Measuring circuit for resonating sensors [26].

is the following; when the exciter is stimulated at a certain frequency, it will contract and elongate. Connected to the membrane, it will also start to vibrate. The receptor crystal will then follow the motion of the membrane and produce a signal proportional to the frequency of the membrane. As pressure acts on it, the resonant frequency of the membrane will change and the more the excitement frequency gets closer to resonance, the bigger the induced motion will be. When the output signal reaches a maximum, resonance has been achieved and pressure can be deduced out of it.

Three basic structures of resonant sensors are presented in Figure 2.12, each with several different resonance modes.

The main drawback of resonant sensors is that high quality material is needed for the vibrator which is the sensing part of the sensor [9].

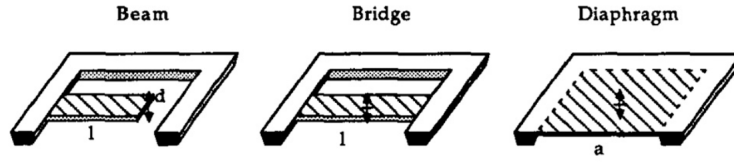


Figure 2.12: Examples of resonant sensor designs [18].

### 2.2.6 Optical sensors

Another technology free from a transducer is optical sensing, which consists in measuring light intensity and wavelength of an applied pressure signal. The working principle is described by Y.Javed *et al.*: "A source focuses light on a membrane and the corresponding change in light intensity due to applied pressure and deflection of membrane is picked up by a detector" as shown in Figure 2.13.

Optical pressure sensors are commonly used in turbomachinery and in touch screen devices.

One of the main advantages of optical sensors is their viability in harsh environments including high-temperature and corrosion conditions. They also provide high resolution, high sensitivity and resistance to electromagnetic interference. However, fabrication of such devices is not easy and involves several materials. Huge stresses are induced in the different materials due to a mismatch in their coefficients of thermal expansion, and another drawback is the difficulty to perform alignment of the optics and calibration of the sensor [18].

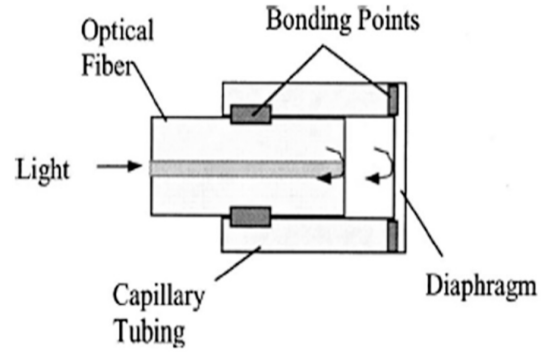


Figure 2.13: Common design of an optical pressure sensor [18].

## 2.3 Figures of merit

The different FoM's of interest for this work are presented in this section. The first part will be about the FoM's used to characterise the piezoresistive potential of a material, and the second about the FoM's related to pressure sensors in general.

### 2.3.1 Piezoresistive figures of merit

**Gauge factor** A useful FoM to characterise the variation of resistance with strain is called gauge factor. It is the normalized resistance variation over the axial strain [27]:



$$GF = \frac{\Delta R/R}{\epsilon_{axial}} = 1 + 2\nu + \frac{\Delta\rho/\rho}{\epsilon_{axial}} \quad (2.9)$$

This formula consists in two different parts: the first one ( $1 + 2\nu$ ) is related to the change in *geometry* of the resistor under strain is applied, and the other ( $\frac{\Delta\rho/\rho}{\epsilon_{axial}}$ ) is related to the change in *conductivity* of the material. The first part is the most critical for **metals** while the second is the dominant one for **semi-conductors**.

As this work will focus on the use of semi-conductors for the transducing layer of the pressure sensor, it is interesting to note for later analyses that the part of the gauge factor related to the properties of the material varies with holes/electrons mobility, impurity concentration, and crystalline direction among other parameters [28].

**Piezoresistive coefficient** The piezoresistive effect is characterised by the piezoresistive coefficients., which are computed as the relative change in conductivity over the applied stress.

$$\pi = \frac{\Delta\rho/\rho}{\sigma} = \frac{\Delta\rho/\rho}{E\epsilon} \quad [1/Pa] \quad (2.10)$$

Those coefficients highly depend on the **crystalline directions** and will be further developed in Chapter 3. It also depends on other parameters like temperature and doping for silicon.

### 2.3.2 Figures of merit for pressure sensors

**Normalized sensitivity** The normalized sensitivity is defined as the slope between the applied pressure and the output signal divided by the bias voltage applied on the sensor:

$$S = \frac{\partial V_{out}/V_{in}}{\partial P} \quad [1/Pa] \quad (2.11)$$

In other words, a sensor with a high sensitivity will have a greater change in its voltage output than a low sensitivity sensor for the same applied pressure.

**Noise** Electrical noise appears as tiny variations of current and voltage affecting the signal read at the output of the sensor. In silicon based sensors, there are two main sources of noise: the **thermal noise** and the **flicker noise**. If a bridge is used to sense the output signal, the spectral density of the noise is denoted as [29]:

$$S_{th} = 4k_bTR \quad [V^2/Hz] \quad (2.12)$$

where  $k_b$  is the Boltzmann constant,  $T$  the temperature in Kelvin and  $R$  the value of the resistor.

The flicker noise is dominant at low frequency and decreases when frequency increases. It is a function of the number of carriers  $N_c$ , a technology coefficient  $\alpha$ , and the bias voltage  $V_s$  [29]:

$$S_{1/f} = \frac{\alpha V_s^2}{N_c f} \quad [V^2/Hz] \quad (2.13)$$



**Limit of detection** The limit of detection is defined as the smallest increase in applied pressure that will produce a **measurable change** in the signal output. It is computed as [29]:

$$LoD = 3 \frac{N}{S} \quad [Pa] \quad (2.14)$$

where  $N$  is the standard deviation of the device under a fixed input and  $S$  the sensitivity computed earlier.

This FOM shows that a high noise in the sensor will lower its ability to sense a low pressure variation. To counteract this, a higher supply voltage  $V_s$  can be used but it will increase the power consumption.

## 2.4 Materials for piezoresistive sensing

As mentioned when reviewing piezoresistive sensors, the piezoresistive effect consists in a variation of the **electrical resistivity** of a given material when this later undergoes mechanical strain. All materials are piezoresistive at some level as the intrinsic properties of every crystalline lattice are modified when the crystal structure is deformed. The change in resistivity comes from the modification of the inter-atomic spacing resulting in a different **bandgap** between the valence and conduction bands [9].

However, piezoresistive sensing requires a transducer whose resistivity varies significantly in order to provide a **measurable output** when pressure is applied on the device. Throughout this work, several materials have been considered as candidates for building a piezoresistive pressure sensor: silicon (Si), Indium Gallium Zinc Oxide (IGZO) and Vanadium Oxides ( $VO_X$ ). Their physical properties of interest are reviewed in the following sections, along with some of their major applications in the literature.

### 2.4.1 Doped Silicon (Si)

Silicon is one of the mostly used material in the field of MEMS, either as a substrate and/or as an active element of the considered device. Its is an hard and brittle semiconductor whose properties have been studied for years.

**Crystalline structure** Silicon has a *cubic* structure, meaning that all directions and planes rotated of  $90^\circ$  are equivalent as shown in Figure 2.15. The unit cell of the silicon cubic crystal is illustrated in Figure 2.14. In *Winfab*, (100) silicon wafers with primary flat in [110] orientation are used, which is important to keep in mind as the crystalline directions of the substrate influence the overall fabrication process. This type of wafer is shown in Figure 2.16.

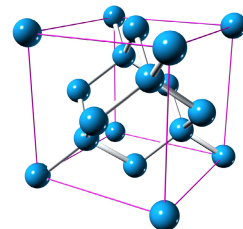


Figure 2.14: Unit cell of silicon cubic crystal [30].

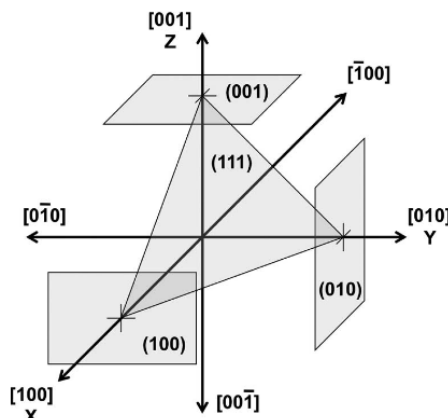


Figure 2.15: Miller indices inside a cubic crystal, with important planes for silicon explicitly shown [31].

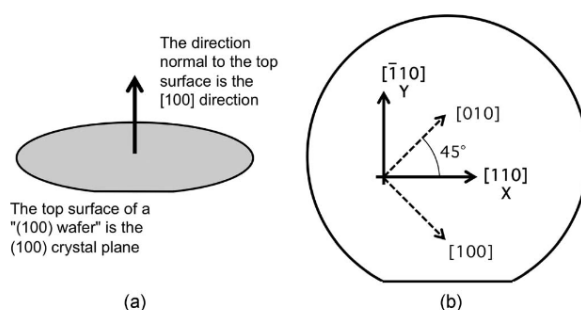


Figure 2.16: Crystal orientation of a standard (100) wafer used in *Winfab*. (a) Crystal plane perpendicular to the [100] direction. (b) Directions inside the (100) plane. [31].

**Mechanical properties** The maximum value for the Young modulus of silicon which has been reported in the literature goes up to **190GPa** [31]. Whereas this property can be very important depending on the application considered, the **piezoresistive properties** of doped silicon will be of a greater interest for this work. The piezoresistive coefficients of n-Si and p-Si can be found in Table 2.1 while their longitudinal and transverse gauge factors are presented in Figure 2.17.

|           | $\pi_l$ | $\pi_t$ |
|-----------|---------|---------|
| n-type Si | -176    | -312    |
| p-type Si | -663    | 718     |

Table 2.1: Longitudinal and transverse piezoresistive coefficients of doped Si [32].

**Common usage** Silicon is widely used in countless applications related to microfabrication. Specific usage in the field of pressure sensing will be detailed in Section 2.5 where modern pressure sensors performances are presented.

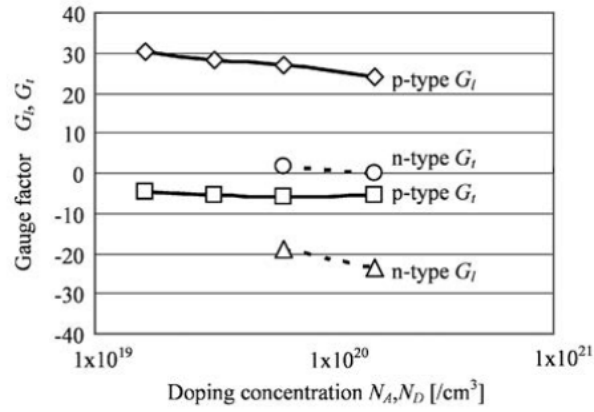


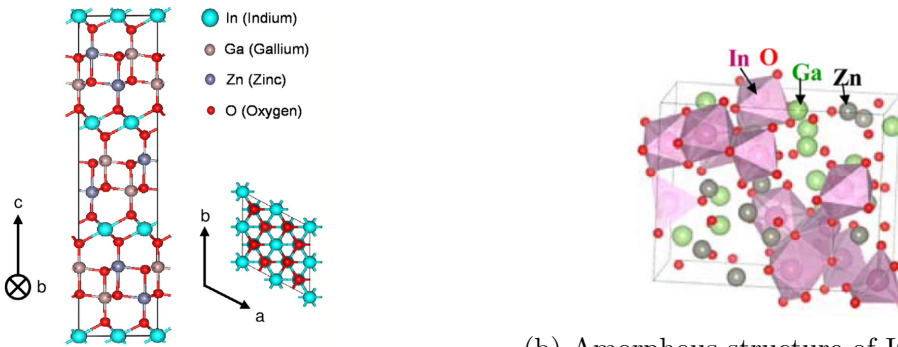
Figure 2.17: Gauge factors of doped silicon [33].

### 2.4.2 Indium Gallium Zinc Oxide (IGZO)

IGZO is a doped version of ZnO on which In and Ga dopants have been added. It is a semiconducting material which is well known for its *piezoelectric* properties. Nevertheless, as mentioned earlier, all materials exhibit a piezoresistive behaviour at some point under deformation. This aspect will be approached in this work.

**Crystalline structure** The crystalline structure of IGZO thin films deposited by sputtering can vary depending on the deposition and post-deposition treatment parameters. The structure can either be *amorphous* (*a*), *nanocrystalline* (*nc*) or *c-axis aligned crystalline* (*CAAC*) [34]. According to different research teams in the field, *CAAC*- and *nc*-IGZO phases should be classified as an intermediate between amorphous and crystal structures because they do not have clear grain boundaries compared to other well-known crystalline materials [35]. This characteristic is actually an advantage for transistor applications as it provides higher carrier mobility due to reduced scattering in the absence of grains compared to polycrystalline silicon for example.

The crystalline structure of the *CAAC*-IGZO organised in layers of  $GaO(ZnO)_n^+$  and  $InO_2^-$  ions [36] compared to the *a*-IGZO structure are represented in Figure 2.18.


 (a) Layered structure of *CAAC*-IGZO [35].

(b) Amorphous structure of IGZO [37].

Figure 2.18: Structures of different crystalline phases of IGZO.

**Mechanical properties** Mechanical properties of IGZO thin films are much less studied in the literature than their electrical and optical properties which are used in many applications as described in the next paragraph. Nevertheless, the Young modulus has been estimated at **137 GPa** for an amorphous structure [38], and gauge factors going up to **536** have been reported [39].

**Common usage** For a few years now, *a*-IGZO films have been used as thin film transistor (TFT) channels due to their high field-effect mobility, their specificity to be deposited at low temperature, and their cost-effective fabrication at large scales. Advantages of *a*-IGZO TFTs are used in the field of pressure sensing and flexible electronics [40],[41].

### 2.4.3 Vanadium oxides ( $VO_X$ )

Vanadium is an average-hard and ductile metal which is electrically conductive and thermally insulating. It has **multiple oxidation states**, ranging from  $VO$  to  $V_2O_5$  [43]. All these vanadium oxides can be found in the phase diagram presented in Figure 2.19.

Vanadium oxides thin films can be deposited in various ways, but the method used in the scope of this work is **reactive sputtering**. A good understanding of the deposition parameters is important as the oxidation state and stoichiometry of the film depends on them. Calibration of the  $O_2$  concentration to insert in the sputtering chamber compared to this of *Ar* to obtain different vanadium oxide films has been performed in the literature and the results are shown in Figure 2.20.

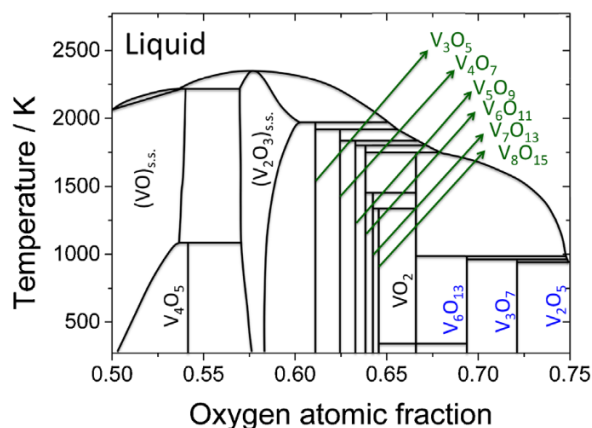


Figure 2.19: V-O phase diagram restricted to oxygen atomic fractions between 0.5 and 0.75 [42].

The V-O system has been studied a lot in the last decades for its very interesting **metal-to-insulator transition** (MIT) behavior. Indeed, under a given temperature, the material will experience a sharp and reversible change of phase, and its physical properties (optical, electrical, thermal,...) are modified drastically. The change of resistivity of  $VO_2$  is illustrated in Figure 2.21 and exhibits *hysteresis* between the two phases. When the material changes of phase, its crystalline structure is modified and going from one state to the other does not require the same energy. When the material is in its insulating phase at low temperature, it has a *monoclinic* structure (M1) which is asymmetric. When it is heated and switches to its metallic phase, the crystalline structure becomes highly symmetric and is called *rutile* (R).

The MIT will not be of much interest in this work, but note that it can also be regulated by strain in  $VO_X$  thin films.

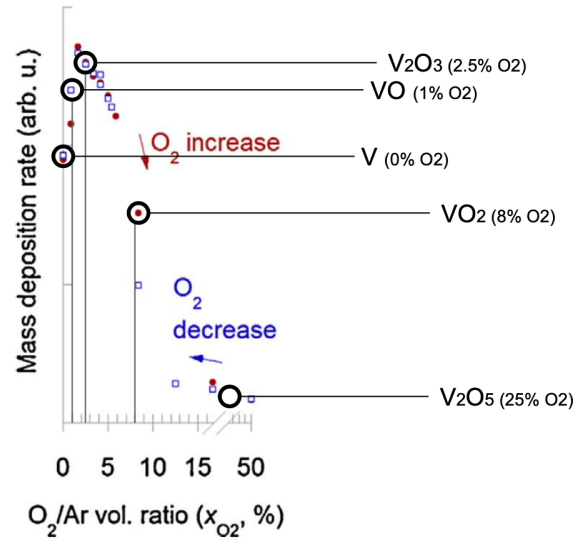


Figure 2.20: Vanadium oxide deposited by reactive sputtering with respect to the oxygen to argon ratio in the chamber. Modified from [44].

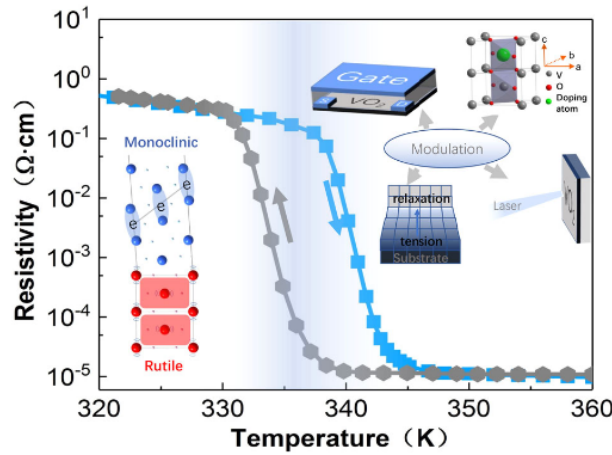


Figure 2.21: Metal-to-insulator transition of  $VO_2$ , with the insulating phase on the left and the metallic phase on the right of the transition [45].

**Crystalline structure** A study of the thermodynamics of vanadium oxides shows that the unit cell of each phase can take multiple *stable* configurations [42]. For example, vanadium dioxide ( $VO_2$ ), which is of high interest for this work, can either have a lattice which is *tetragonal*, *orthorhombic*, or *monoclinic* as illustrated in Figure 2.22. Note that black and grey points on the cells do *not* represent atoms but virtual points surrounding the atoms. These points are not distinguishable from the others, meaning that an observer located on any point would see exactly the same thing as they would on another one.

**Mechanical properties** Each phase of vanadium oxide has its own mechanical properties depending on its structure, determined by the deposition parameters. For example, it has been demonstrated that the Young modulus is very sensitive to stoichiometry: an increase in crystallinity of the  $V_2O_5$  phase deposited by sputtering leads to a higher modulus [47].

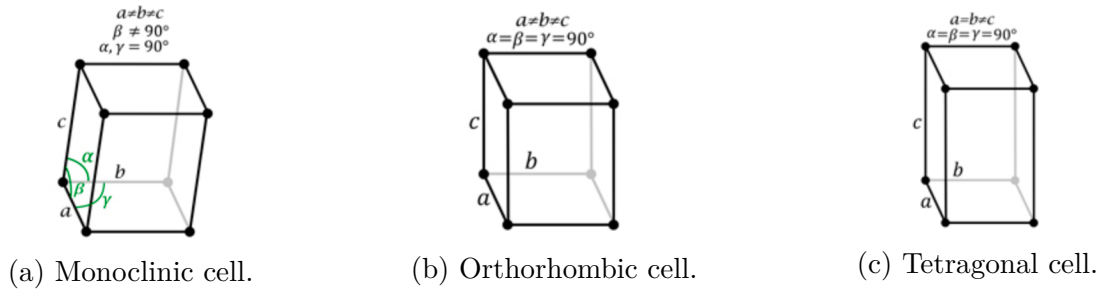


Figure 2.22: Three out of the fourteen Bravais lattices [46].

The Young modulus also depends on the studied shape. For  $VO_X$  nanotubes it can vary between **20** and **80GPa** depending on their length and underlying substrate [48]. For a 100nm polycrystalline thin film investigated using ultra-microindentation on a Si substrate, it is around **170GPa** for a deposition at 300°C [49].

Another relevant mechanical parameter studied in the literature is the gauge factor of the material. The GF of a free-standing  $VO_2$  nanobeam has been evaluated to be of **347** in low strain ranges, with a dominant contribution associated to the *phase transition* of the structure [50]. On the other hand, the GF of  $VO_X$  resistors with a thickness of 370nm on 200nm of  $SiO_2$  over a thick Si membrane has also been measured. The  $VO_X$  film was deposited by reactive sputtering with an oxygen flow of 6%, leading to a 75%  $V_2O_5$  and 25%  $VO_2$  mixture and an estimated GF of **259** [28].

**Common usage** Vanadium oxides are mostly used in other fields than pressure sensing. For example,  $VO_2$  has been studied in the field of temperature sensing for its sharp change in resistivity associated to the MIT transition, or for building infrared microbolometer arrays for its high resistivity, good IR absorption and low flicker noise compared to other commonly used materials such as platinum [51]. Another oxidation state such as  $V_2O_5$  exhibits interesting thermoelectric properties for gas sensing [52].

## 2.5 Modern pressure sensors performances

Now that the main types of pressure sensors have been reviewed along with the different figures of merit used to characterize their **performances**, some of the latest sensors presented in the literature can be compared to better visualise what is achievable with modern fabrication techniques.

As the concept of a sensing membrane has also been introduced, it is important to understand what are the **parameters that enhance or reduce the sensitivity of such diaphragms**, with a particular interest in piezoresistive sensors. This is why the dimensions of the membranes and the shapes and positions of the resistors on top are discussed in this section. The electrical configuration adopted for these resistors to produce a **measurable output signal** is also described afterwards.

### 2.5.1 Comparison of different pressure sensors from the literature

Table 2.2 recapitulates the main characteristics of the pressure sensors found in the literature. Based on this summary, one can notice that comparing pressure sensors from different technologies is not an easy task. Indeed, each technical characteristic is implemented regarding a specific application, and design choices, which also have an impact on the final performances, strongly depend on the **context of the application** (i.e. the available space for the sensor, the environmental conditions, ...).

Nevertheless, some general comparisons can be drawn from these observations. First, the **measurable range** increases with the thickness of the membrane, and the **sensitivity** increases with its area for all types of sensors. Then, it can be seen that piezoresistive sensors have a smaller **footprint** than inductive or capacitive ones. In addition, due to their working principle, piezoresistive based sensors consume more **power** than others, although they are easier to fabricate and provide a more **linear response** because of the use of a full Wheatstone bridge.

### 2.5.2 Influence of the dimensions of the membrane for piezoresistive pressure sensors

As noted from Table 2.2, there are two main parameters related to the dimensions of the membrane that will influence the overall sensor performances: the **thickness** of the diaphragm and its **width**.

Wider diaphragms are used to achieve a bigger sensitivity, but the drawback lies in the sensor taking more space. Thinner diaphragms also provide a better sensitivity, but two main issues have to be addressed. The first one is that thin diaphragms are difficult to fabricate since having a good thickness control is costly. The second one concerns very thin films for which the non linear response zone associated to large deflections is more quickly reached. This means that a wide and thin membrane will increase the sensing sensitivity of the sensor but also the response non-linearity.

### 2.5.3 Influence of the shape and positions of the resistors for piezoresistive pressure sensors

This section will be divided into two parts: the first one will be about the **positioning of the piezoresistors** on the membrane and the second about their **optimal shape**.

**Positioning of the piezoresistors** The most common positions for resistors on a membrane are positions where the **stress** is maximum, i.e. on the membrane mid-side edges as shown in Figure 2.23.







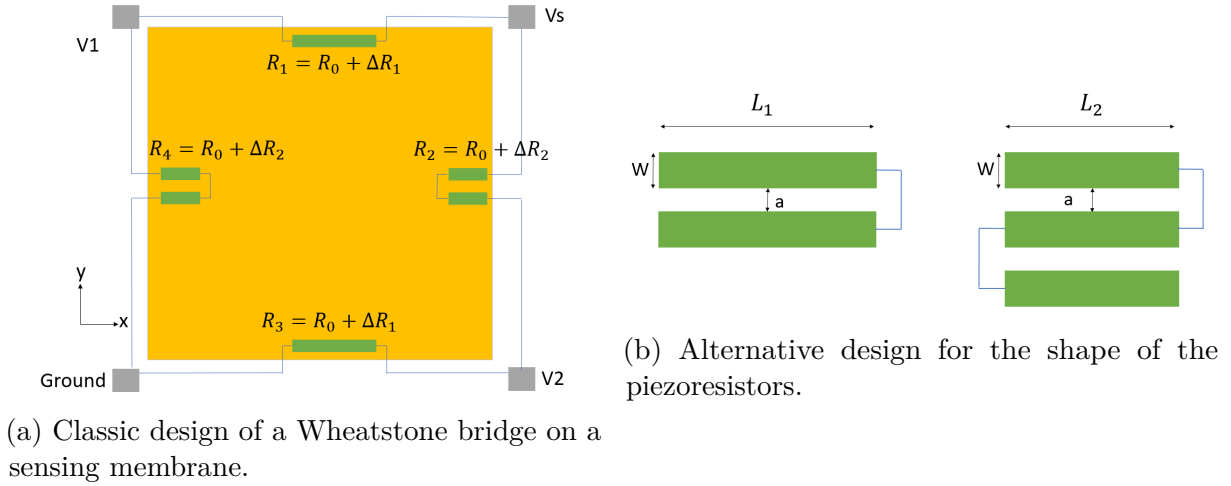


Figure 2.25: Positioning and shape of piezoresistors.

### 2.5.4 Wheatstone bridge measurement

The most common way to measure the output voltage out of resistors positioned on a membrane is to use a so-called **Wheatstone bridge**. Such an architecture is illustrated in Figure 2.25a. On piezoresistive pressure sensors, the *full bridge* configuration is the most standard one. When all the resistors have the same value at rest, under no stress, then  $R_1 = R_2 = R_3 = R_4 = R_0$  and the DC offset is cancelled because  $V_1 = V_2$ .

The output of the bridge is given below, knowing that the values of resistance vary by pair as  $R_1$  and  $R_3$  are in the same configuration and so are  $R_2$  and  $R_4$ :

$$\begin{aligned} V_{out} = V_1 - V_2 &= \left( \frac{R_4}{R_4 + R_1} - \frac{R_3}{R_3 + R_2} \right) V_s \\ &= \left( \frac{R_0 + \Delta R_2}{2R_0 + \Delta R_1 + \Delta R_2} - \frac{R_0 + \Delta R_1}{2R_0 + \Delta R_1 + \Delta R_2} \right) V_s \\ &= \left( \frac{\Delta R_2 - \Delta R_1}{2R_0 + \Delta R_1 + \Delta R_2} \right) V_s \end{aligned} \quad (2.15)$$

The best scenario encountered for a Wheatstone bridge is when two of the four resistors increase with stress while the other two decrease. A good example to illustrate this situation is p-type silicon whose piezoresistive coefficients are of opposite sign. As a reminder, they are given by  $\pi_l = 718 \text{ TPa}^{-1}$  and  $\pi_t = -663 \text{ TPa}^{-1}$ .

But as their absolute values differ, the relative variations of the resistors will not be the same, meaning that the output of the bridge can be increased, and so is the sensitivity. But a drawback of this improvement is that non linearities appear in the response of the sensor due to the variation of the denominator of  $V_{out}$  with the applied strain. In the end, a design choice has to be made to optimize one of these two characteristics. Indeed, the stress inside the resistors allow to tune their variations, and this can be determined by their position on the membrane as explained earlier. If a similar variation is chosen for linearity then  $\Delta R_1 = -\Delta R_2 = \Delta R_3 = -\Delta R_4$  and the output becomes:

$$V_1 - V_2 = \frac{\Delta R}{R_0} V_s \quad (2.16)$$

| Technology          | Ref  | Year | A<br>[mm <sup>2</sup> ] | h<br>[μm] | Sensitivity                  | Range<br>[kPa] | Vs<br>[V] | P<br>[mW] |
|---------------------|------|------|-------------------------|-----------|------------------------------|----------------|-----------|-----------|
| PDMS                | [55] | 2018 |                         |           | 2000<br>[kPa <sup>-1</sup> ] | <0.1           |           | 0.05      |
| PDMS                | [55] | 2018 |                         |           | 491<br>[kPa <sup>-1</sup> ]  | 0.100-1        |           |           |
| PMOS                | [56] | 2019 | 1.44                    | 4.5       | 1.303<br>[mV/V/kPa]          |                | 5         | 0.19      |
| NMOS                | [56] | 2019 | 1.44                    | 4.5       | 0.6<br>[mV/V/kPa]            |                | 5         | 0.395     |
| Si                  | [9]  | 2014 | 0.25                    | 15        | 135.7<br>[mV/V/kPa]          | 0-100          |           | 2         |
| p-type Si           | [57] | 2013 | 0.6131                  | 63        | 6.657<br>[mV/MPa]            | 0-100 000      |           |           |
| p-type Si           | [57] | same | same                    | same      | 1.854<br>[mV/MPa]            | same           |           |           |
| p-type Si           | [57] | same | same                    | same      | 0.562<br>[mV/MPa]            | same           |           |           |
| p-type Si           | [57] | same | same                    | same      | 0.617<br>[mV/MPa]            | same           |           |           |
| p-type Si           | [9]  | 2014 | 1                       | 20        | 200<br>[mV/V/kPa]            | 100            | 3         |           |
| Mono-Si             | [58] | 2017 | 0.71                    | 2         | 3.6<br>[mV/V/kPa]            | 1.2            | 5         | 8.3       |
| Mono-Si             | [59] | 2018 | 8.41                    | 30        | 5.4<br>[mV/V/kPa]            | 5              | 5         | 5.55      |
| Mono-Si             | [54] | 2021 | 4                       | 95        | 0.003779<br>[mV/V/kPa]       | 250-2500       | 5         |           |
| Cap (intrafillable) | [60] | 2020 | 3 × 15                  |           | 220<br>[kPa <sup>-1</sup> ]  | 0.08-360       |           |           |
| Inductive           | [16] | 2019 | 27 × 27                 |           | 1.6<br>[kPa <sup>-1</sup> ]  | 0-0.000 18     |           |           |
| Inductive           | [61] | 2011 | 3.2 × π                 | -         | 3.56<br>[mH/kPa]             | 0.000 3-10     |           |           |
| Inductive           | [14] | 2016 | 0.54 × 0.54             | 20        | 0.04103<br>[nH/kPa]          | 0-300          |           |           |
| Resonant LC         | [62] | 2018 | 3.2 × 3.2               |           | 39.76<br>[kH z/kPa]          | 0-16           |           |           |

Table 2.2: Comparison of different types of pressure sensors from the literature.

## 2.6 Content of this work

Different types of pressure sensors have been reviewed. The **novel architecture** proposed by Meng et al. for a piezoresistive device shows an alternative way to concentrate the stress in the sensing elements with an almost complete piezoresistive layer deposited on the diaphragm. This stress increase led to promising results regarding the improvement of the sensitivity of the sensor.

Regarding **piezoresistive materials**, Inomata et al. demonstrated a high gauge factor of about 295 for  $VO_X$  thin films [28]. They studied a mixture of  $VO_2$  and  $V_2O_5$ , but no article exploring the piezoresistive properties pure  $VO_2$  has been found.

The idea behind this master thesis is then to combine these two discoveries by studying the architecture of Meng et al. with a piezoresistive film of vanadium dioxide. Numerical simulations of the sensor are proposed, along with fabrication and characterization of the material.

## CHAPTER 3

# MODEL DESIGNING AND SIMULATIONS

In this chapter, **two designs** of piezoresistive pressure sensors are compared. The first one, *design 1*, consists of a nitride-oxide (NO) membrane with piezoresistors positioned at the center of each side. The second one, *design 2*, is an innovation proposed by Meng et al. [54]

There are three main differences between the two designs:

- 1) The use of a piezoresistive layer on *design 2* to rigidify the membrane.
- 2) The positions of two of the resistors  $R_1$  and  $R_3$ .
- 3) The shape of the resistors.

An analytical model will only be proposed for *design 1* because the patterning and the shape of resistors cannot be represented correctly by a first order analytical approximation. Only the membrane and the resistors will be considered as they are the core of this type of sensors and all other circuit elements are neglected. Finally, both designs will be simulated via finite element modelling (FEM) to confirm the model.

### 3.1 Analytical model

This section presents a **first order analytical model** to better understand the physical behaviour of the sensing membrane. Firstly, a **low deflection model** will be explained to justify the preliminary design choices. Secondly, an **average layer model** combining properties of the two layers for large deflections will be proposed. Thirdly, the **electrical aspects** of the piezoresistors will be added to have a complete electro-mechanical model. Note that all mathematical expressions in this part are taken from [2].

The top and cross-sectional views of the two designs are illustrated in Figures 3.1 and 3.2 respectively.

#### 3.1.1 Mechanical modelling in small deformation regime

Piezoresistive sensors are based on stress/strain induced resistance variations. The shape of the membrane shape influences these parameters so a comparison between a **circular** and a **square membrane** is useful. It has to be noted that the two models detailed below are only valid in the **small-deflection regime**, i.e. when the deflection is small compared to the diaphragm thickness. This is why a more complete model will be elaborated afterwards.

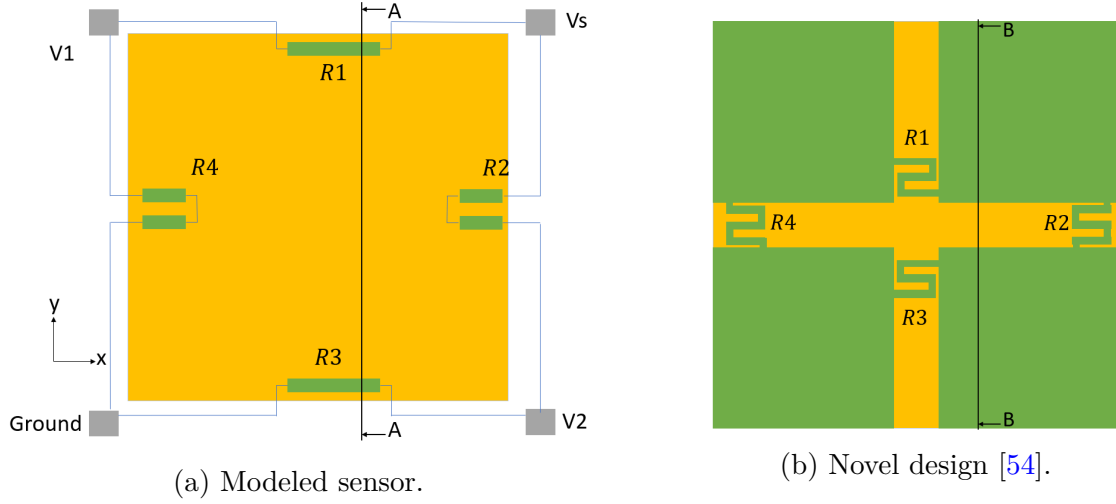


Figure 3.1: Top views of the studied designs.

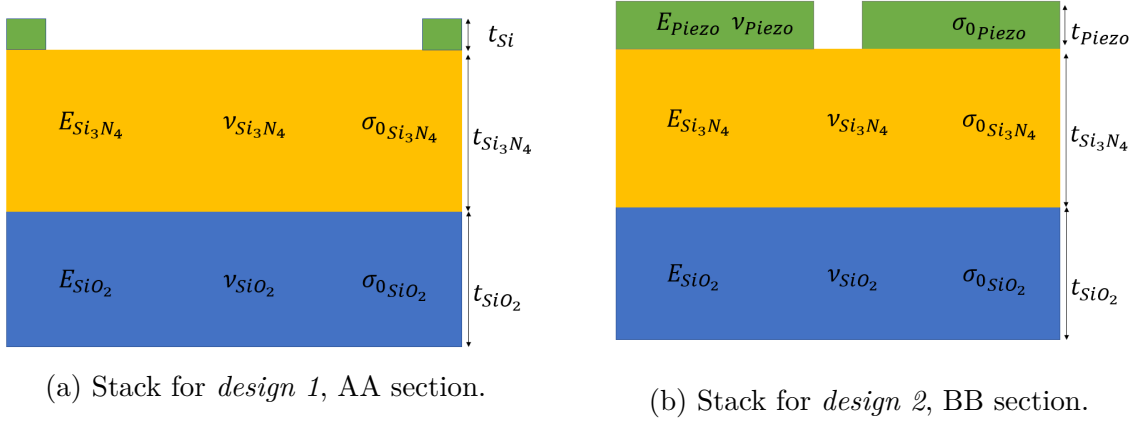


Figure 3.2: Cross-sectional views for the studied designs.

### 3.1.1.1 Equations for the circular membrane

The displacement of a circular membrane under pressure without any residual stress can be expressed in polar coordinates according the following equation [63]:

$$w(r) = \frac{pa^4}{64D} \left(1 - \frac{r^2}{a^2}\right)^2 \quad (3.1)$$

where  $a$  is the radius,  $p$  the applied pressure,  $h$  the membrane thickness,  $E$  the Young modulus,  $\nu$  the Poisson coefficient and  $D = \frac{Eh^3}{12(1-\nu^2)}$  the flexural rigidity. From this, the radial ( $T_r$ ) and tangential ( $T_t$ ) stress are given by:

$$T_r(r) = \pm \frac{3pa^2}{8h^2} \left[ (3 + \nu) \frac{r^2}{a^2} - (1 + \nu) \right] \quad (3.2)$$

$$T_t(r) = \pm \frac{3pa^2}{8h^2} \left[ (3\nu + 1) \frac{r^2}{a^2} - (1 + \nu) \right] \quad (3.3)$$

According to these equations, the maximal stresses are concentrated all around the membrane edges and are given by:

$$T_r(a) = \frac{3}{4} \frac{a^2}{h^2} p \quad (3.4)$$

$$T_t(a) = \frac{3}{4} \nu \frac{a^2}{h^2} p \quad (3.5)$$

### 3.1.1.2 Equations for the square membrane

Displacement for the square membrane under pressure without residual stress, if the origin is taken at the center of the membrane, can be expressed as [63]:

$$w(x, y) = \frac{1}{47} p \frac{a^4}{D} \left(1 - \frac{x^2}{a^2}\right)^2 \left(1 - \frac{y^2}{a^2}\right)^2 \quad (3.6)$$

The stresses are given by :

$$T_{XX}(x, y) = \pm 0.51 p \frac{a^2}{h^2} \left[ \left(1 - \frac{y^2}{a^2}\right)^2 \left(1 - 3 \frac{x^2}{a^2}\right) + \nu \left(1 - \frac{x^2}{a^2}\right)^2 \left(1 - 3 \frac{y^2}{a^2}\right) \right] \quad (3.7)$$

$$T_{YY}(x, y) = \pm 0.51 p \frac{a^2}{h^2} \left[ \left(1 - \frac{x^2}{a^2}\right)^2 \left(1 - 3 \frac{y^2}{a^2}\right) + \nu \left(1 - \frac{y^2}{a^2}\right)^2 \left(1 - 3 \frac{x^2}{a^2}\right) \right] \quad (3.8)$$

with the positive sign for the lower surface and the negative sign for the top surface.

The maximal stresses are located in the middle of each side and are given by:

$$T_{XX}(\pm a, 0) = 1.02 \frac{a^2}{h^2} p \quad (3.9)$$

$$T_{YY}(\pm a, 0) = \nu T_{XX} \quad (3.10)$$

$$T_{YY}(0, \pm a) = 1.02 \frac{a^2}{h^2} p \quad (3.11)$$

$$T_{XX}(0, \pm a) = \nu T_{YY} \quad (3.12)$$

As shown by Equations 3.4 and 3.9, the squared shape has a more important stress than the circular one for the same membrane dimensions (half-side length and thickness) and under the same applied pressure. This is interesting since the main interest for building a piezoresistive pressure sensor is the stress concentration. Consequently, a **square membrane** design has been chosen for this master thesis.

### 3.1.2 Models for large deflection of membranes with residual stresses

#### 3.1.2.1 Residual stresses

**Residual stresses** are a central part of the subject in both modelling and design as well as in fabrication. Initially for the model as they influence the membrane displacement, and later on during the fabrication as they can cause buckling, rupture in the membrane, or even destroy the stack. They mainly arise from the lattice mismatch between materials. Table 3.1 summarises the different stresses according to the material and the deposition techniques used. WTO and PECVD techniques were chosen for multiple reasons:

- 1) Accessibility
- 2) Well known
- 3) Repeatability

| Material  | Deposition process          | Expected residual stress [MPa] |
|-----------|-----------------------------|--------------------------------|
| $SiO_2$   | Wet thermal oxidation (WTO) | -300                           |
| $Si_3N_4$ | PECVD                       | 300                            |

Table 3.1: Materials and available processes with expected residual stress [64],[3].

#### 3.1.2.2 Building the average layer

Models for the displacement are made for membranes with a single layer in the literature [65],[66]. Since the NO membrane is composed of two layers, a new model is required. It consists of an **equivalent single layer** with the thickness of the two layers and material equivalent characteristics. Average Young modulus  $E_{NO}$ , Poisson coefficient  $\nu_{NO}$  and residual stress  $\sigma_{0,NO}$  will be computed by a weighted average:

$$\sigma_{0,NO} = \frac{\sum_{i=1}^n \sigma_{0,i} t_i}{\sum_{i=1}^n t_i = t_{NO}} \quad (3.13)$$

$$E_{NO} = \frac{\sum_{i=1}^n E_i t_i}{\sum_{i=1}^n t_i = t_{NO}} \quad (3.14)$$

$$\nu_{NO} = \frac{\sum_{i=1}^n \nu_i t_i}{\sum_{i=1}^n t_i = t_{NO}} \quad (3.15)$$

where  $E_i, \nu_i$  and  $\sigma_{0,i}$  are respectively the Young modulus, Poisson coefficient and residual stress of each layer, and  $t_i$  the thickness of the considered layer. Table 3.2 presents the values for the separate layers and for the average layer.

The considered thicknesses have been taken from another master thesis as the design presented in this work is a thin  $Si_3N_4$ - $SiO_2$  membrane with controlled and low tensile stress [3].

|           | $Si_3N_4$ | $SiO_2$   | Average |
|-----------|-----------|-----------|---------|
| t [nm]    | 600       | 400       | 1000    |
| E [GPa]   | 250 [67]  | 70 [68]   | 178     |
| $\nu$ [/] | 0.23 [67] | 0.17 [68] | 0.207   |

Table 3.2: Mechanical properties of each layer and of the average layer.

### 3.1.2.3 Displacement of the average layer

The deflection at the center of a square membrane  $w_0$  can be modelled by [65]:

$$P = \frac{3.41t_{NO}}{a^2}\sigma_{0,NO}w_0 + \frac{1.37(1.446 - 0.427\nu_{NO})t_{NO}}{a^4}\frac{E_{NO}}{1 - \nu_{NO}}w_0^3 \quad (3.16)$$

where  $P$  is the applied pressure,  $t_{NO}$  the total thickness of the membrane and  $a$  the half length of the membrane. This model is implicit and needs to be numerically solved in order to obtain the displacement at the center,  $w_0$ . The coefficients 3.41, 1.37, 1.446 and 0.427 are fitting parameters obtained through finite element modelling in [65]. Figure 3.3 shows the displacement at the center of the membrane for different pressures.

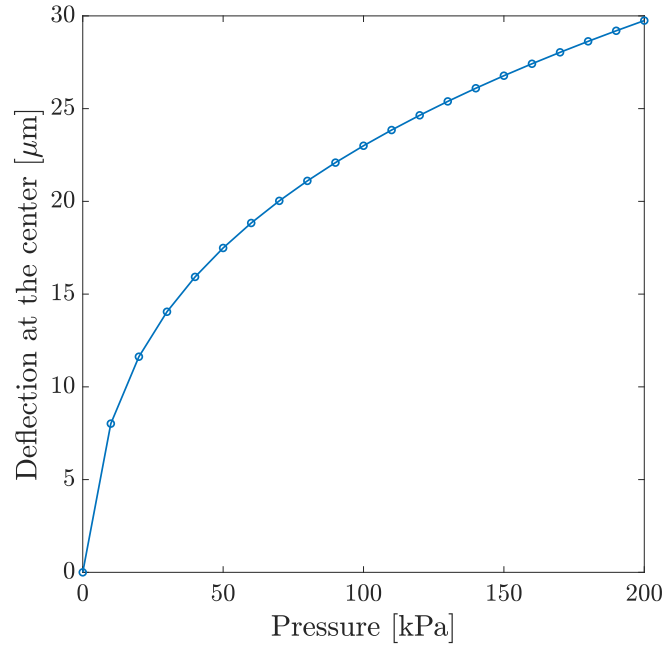


Figure 3.3: Maximal deflection of the membrane computed with Equation 3.16.

Once  $w_0$  is obtained, the deflection of the membrane along the z-axis, noted  $w(x, y)$ , is needed across the entire membrane from one edge to the other to compute the stress. Three models extracted from the literature each propose an approximation of this displacement that can be compared to the others,  $\hat{w}_1$  [53],  $\hat{w}_2$  [69],  $\hat{w}_3$  [2] :



$$\hat{w}_1(x, y) = \frac{w_0}{4} \left( 1 + \cos\left(\frac{\pi x}{a}\right) \right) \left( 1 + \cos\left(\frac{\pi y}{a}\right) \right) \quad (3.17)$$

$$\hat{w}_2(x, y) = w_0 \left( 1 + 0.401 \left( \frac{x^2 + y^2}{a^2} \right) + 1.1611 \frac{x^2 y^2}{a^4} \right) \cos\left(\frac{\pi x}{2a}\right) \cos\left(\frac{\pi y}{2a}\right) \quad (3.18)$$

$$\hat{w}_3(x, y) = w_0 \left( 1 - \left( \frac{x}{a} \right) \right)^2 \left( 1 - \left( \frac{y}{a} \right) \right)^2 \quad (3.19)$$

As can be seen in Figure 3.4, Figure 3.5 and Figure 3.6, the computed displacement differs in shape for each approximation. The deflection of the membrane is wider with  $\hat{w}_2$  as compared to  $\hat{w}_1$  and  $\hat{w}_3$ .

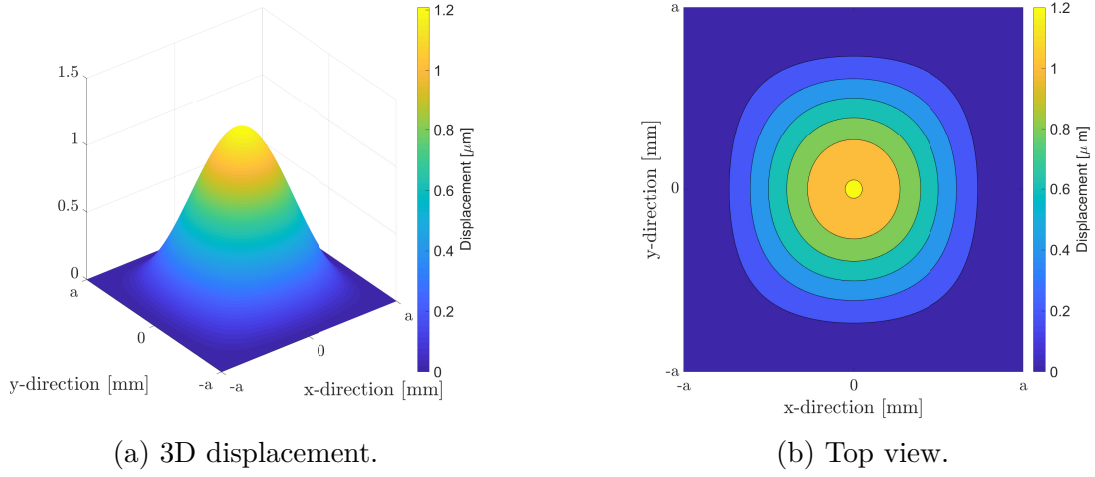


Figure 3.4: Deflection with model  $\hat{w}_1$  for  $a = 0.5\text{mm}$  and  $p = 1000\text{Pa}$ .

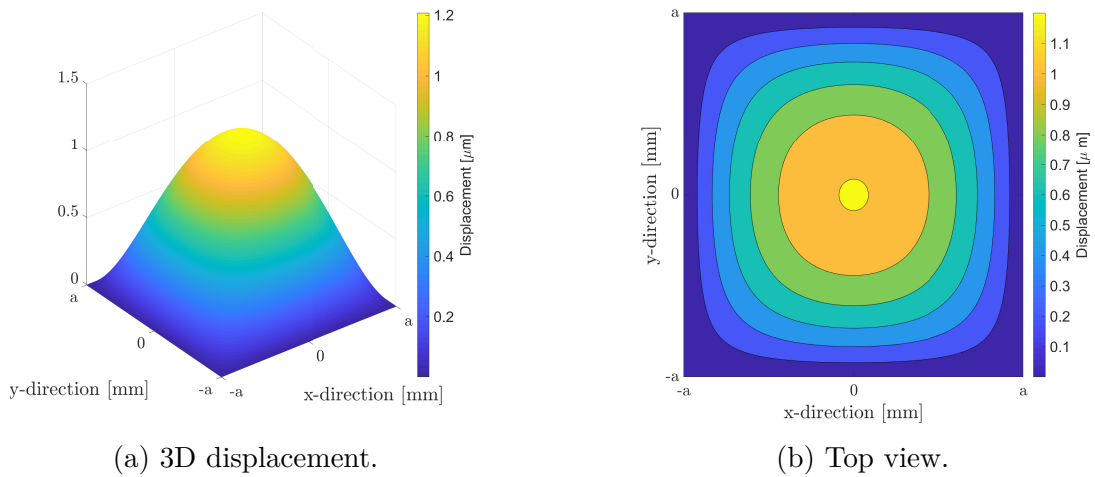


Figure 3.5: Deflection with model  $\hat{w}_2$  for  $a = 0.5\text{mm}$  and  $p = 1000\text{Pa}$ .

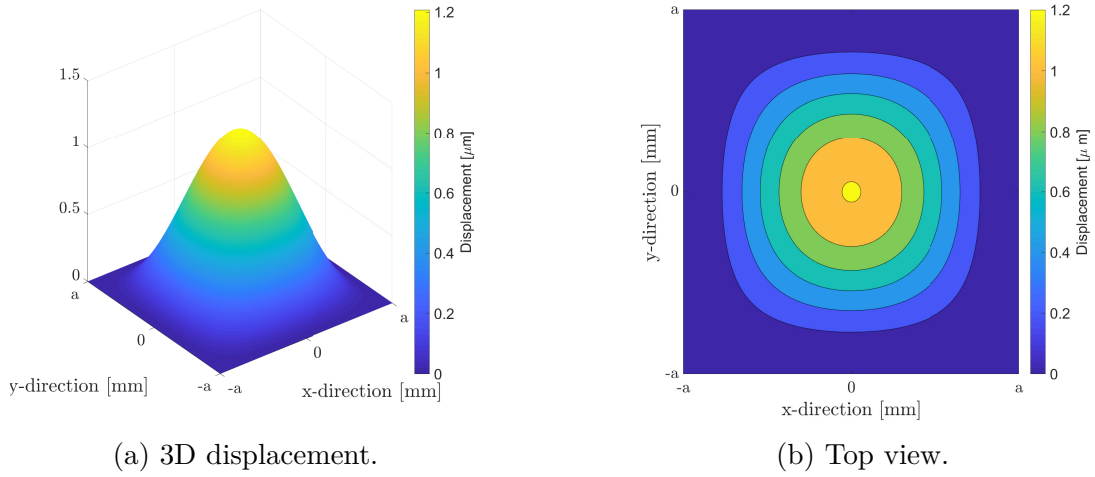


Figure 3.6: Deflection with model  $\hat{w}_3$  for  $a = 0.5\text{mm}$  and  $p = 1000\text{Pa}$ .

### 3.1.2.4 Uniaxial stress computation on the top of the average layer

The main equation for the stress on the surface of a membrane is given by [70]:

$$\frac{E_{NO}t_{NO}^3}{12(1-\nu^2)} \left[ \frac{\partial^4 w(x,y)}{\partial x^4} + 2\frac{\partial^4 w(x,y)}{\partial x^2 \partial y^2} + \frac{\partial^4 w(x,y)}{\partial y^4} \right] = P \quad (3.20)$$

Along with the expressions of  $\sigma_{XX}$  and  $\sigma_{YY}$  derived from Equation 3.20:

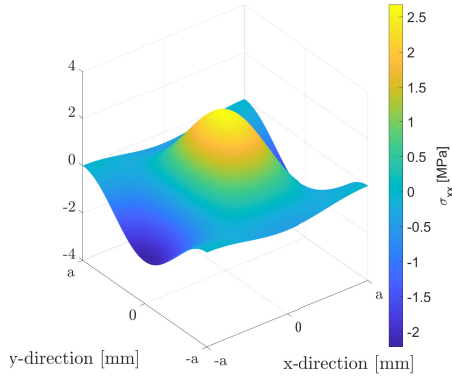
$$\sigma_{XX} = -\frac{E_{NO}t_{NO}}{2(1-\nu^2)} \left( \frac{\partial^2 w(x,y)}{\partial x^2} + \nu_{NO} \frac{\partial^2 w(x,y)}{\partial y^2} \right) \quad (3.21)$$

$$\sigma_{YY} = -\frac{E_{NO}t_{NO}}{2(1-\nu^2)} \left( \frac{\partial^2 w(x,y)}{\partial y^2} + \nu_{NO} \frac{\partial^2 w(x,y)}{\partial x^2} \right) \quad (3.22)$$

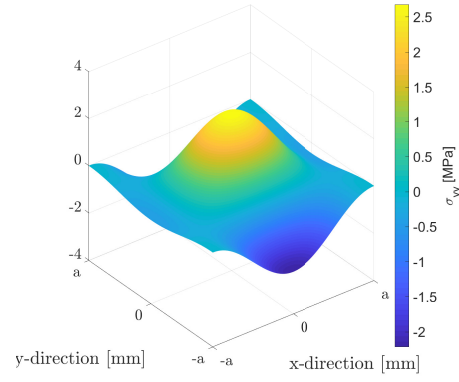
The stress distribution of  $\sigma_{XX}$  and  $\sigma_{YY}$  were computed by injecting the models  $\hat{w}_1$ ,  $\hat{w}_2$  and  $\hat{w}_3$  into Equation 3.21 and Equation 3.22. Results can be found in Figure 3.7, Figure 3.8, and Figure 3.9.

The stress distributions associated to models  $\hat{w}_1$  and  $\hat{w}_3$  show a compressive stress concentration on each mid-side of the membrane edges ( $x = \pm a, y = 0$  and  $x = 0, y = \pm a$ ), whereas the ones associated to model  $\hat{w}_2$  show a tensile stress on the membrane sides ( $x = \pm a, y = \pm \frac{a}{2}$  and  $x = \pm \frac{a}{2}, y = \pm a$ ).

With the model analysed in Section 3.1.1, the stress was maximum on the membrane mid-sides. It is not the case for the stress computed using  $\hat{w}_2$ . Hence, it will not be used for the rest of this work. Since  $\hat{w}_1$  and  $\hat{w}_3$  gave approximately the same results,  $\hat{w}_1$  has been chosen to follow through.

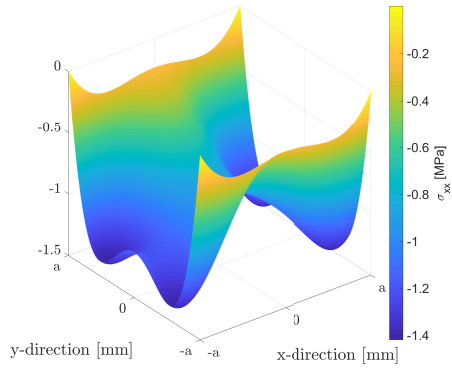


(a) 3D representation of  $\sigma_{XX}$ .

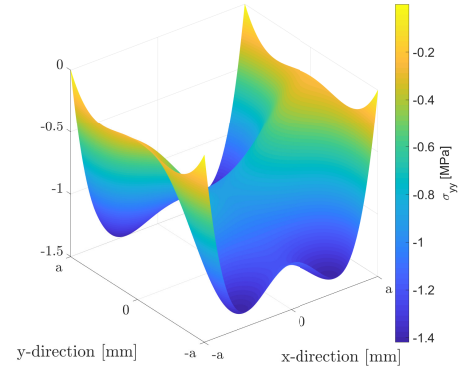


(b) 3D representation of  $\sigma_{YY}$ .

Figure 3.7: Computed stresses with the  $\hat{w}_1$  model for  $a = 0.5\text{mm}$  and  $p = 1000\text{Pa}$ .

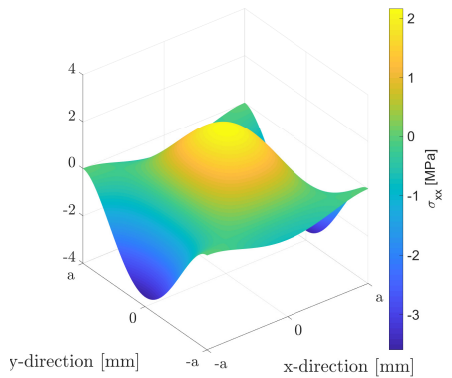


(a) 3D representation of  $\sigma_{XX}$ .

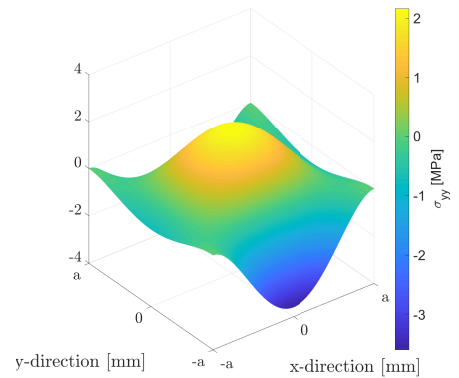


(b) 3D representation of  $\sigma_{YY}$ .

Figure 3.8: Computed stresses with the  $\hat{w}_2$  model for  $a = 0.5\text{mm}$  and  $p = 1000\text{Pa}$ .



(a) 3D representation of  $\sigma_{XX}$ .



(b) 3D representation of  $\sigma_{YY}$ .

Figure 3.9: Computed stresses with the  $\hat{w}_3$  model for  $a = 0.5\text{mm}$  and  $p = 1000\text{Pa}$ .

### 3.1.3 Electro-mechanical modelling for the average layer

The next step consists in coupling the stress to the variation of electrical properties of the resistors. First, general theory about piezoresistors is reviewed. Then, the coupling between the stress on the membrane and the piezoresistivity is added to the current stress model. Finally, an additional explanation regarding the Wheatstone bridge configuration is provided to present how the stress is converted into a measurable voltage.

#### 3.1.3.1 Modelling of a piezoresistor

The variation of resistance induced by stress can be modeled as follows:

$$\frac{\Delta R}{R} = (1 + 2\nu)\epsilon + \frac{\Delta\rho}{\rho} \quad (3.23)$$

where  $R$  is the resistance,  $\rho$  is the resistivity,  $\nu$  is the Poisson coefficient, and  $\epsilon$  is the strain applied on the resistor. The first term is related to geometry changes of the component, while the second is due to the piezoresistive effect. In a semiconductor like silicon, the first term is negligible [63].

The *stress* acting on a solid can be written as 6 independent terms using the *force balance condition*. Using the Onsager's theorem [71], the *resistivity* of a single-crystal material can be written as a second-rank tensor with 6 independent components. This tensor is referred to as the **piezoresistive tensor** and is written  $\langle\pi\rangle$ . The relation between these two can be written as:

$$\begin{bmatrix} \frac{\Delta R_1}{R_1} \\ \frac{\Delta R_2}{R_2} \\ \frac{\Delta R_3}{R_3} \\ \frac{\Delta R_4}{R_4} \\ \frac{\Delta R_5}{R_5} \\ \frac{\Delta R_6}{R_6} \end{bmatrix} = \begin{bmatrix} \pi_{11} & \pi_{12} & \pi_{13} & \pi_{14} & \pi_{15} & \pi_{16} \\ \pi_{21} & \pi_{22} & \pi_{23} & \pi_{24} & \pi_{25} & \pi_{26} \\ \pi_{31} & \pi_{32} & \pi_{33} & \pi_{34} & \pi_{35} & \pi_{36} \\ \pi_{41} & \pi_{42} & \pi_{43} & \pi_{44} & \pi_{45} & \pi_{46} \\ \pi_{51} & \pi_{52} & \pi_{53} & \pi_{54} & \pi_{55} & \pi_{56} \\ \pi_{61} & \pi_{62} & \pi_{63} & \pi_{64} & \pi_{65} & \pi_{66} \end{bmatrix} \begin{bmatrix} \sigma_1 \\ \sigma_2 \\ \sigma_3 \\ \sigma_4 \\ \sigma_5 \\ \sigma_6 \end{bmatrix}$$

For silicon, the piezoresistive matrix can be simplified using symmetric operations. This leaves only 3 independent coefficients:

$$\begin{bmatrix} \frac{\Delta R_1}{R_1} \\ \frac{\Delta R_2}{R_2} \\ \frac{\Delta R_3}{R_3} \\ \frac{\Delta R_4}{R_4} \\ \frac{\Delta R_5}{R_5} \\ \frac{\Delta R_6}{R_6} \end{bmatrix} = \begin{bmatrix} \pi_{11} & \pi_{12} & \pi_{12} & 0 & 0 & 0 \\ \pi_{12} & \pi_{11} & \pi_{12} & 0 & 0 & 0 \\ \pi_{12} & \pi_{12} & \pi_{11} & 0 & 0 & 0 \\ 0 & 0 & 0 & \pi_{44} & 0 & 0 \\ 0 & 0 & 0 & 0 & \pi_{44} & 0 \\ 0 & 0 & 0 & 0 & 0 & \pi_{44} \end{bmatrix}$$

This relation is derived with respect to the crystallographic axes  $\langle 100 \rangle$ . But changing the orientation of the resistors compared to these directions can give rise to an increase of the piezoresistive effect. For example, the most common orientation for resistors is  $\langle 110 \rangle$  on a (100) silicon wafer. To account for the associated changes, the piezoresistive

coefficients are noted  $\pi'$  instead of  $\pi$ .

Figure 3.10 shows a resistor with an arbitrary direction and current passing through it. Considering that the thickness of the piezoresistor is very small compared to its length and width, the stress in the z-direction can be neglected. The relative variation of resistance is then given by [63]:

$$\frac{\Delta R}{R} = \pi'_{11}\sigma'_1 + \pi'_{12}\sigma'_2 = \pi_l\sigma_l + \pi_t\sigma_t \quad (3.24)$$

where  $\pi'_{11}(\pi_l), \pi'_{12}(\pi_t)$  are the longitudinal and transverse piezoresistive coefficient respectively and  $\sigma'_1(\sigma_l), \sigma'_2(\sigma_t)$  are the longitudinal and transverse stresses.  $\pi_l$  is the relative variation in the direction of the current while  $\pi_t$  the variations perpendicular to the current.

It can be proven [63] that  $\pi_l$  and  $\pi_t$  can be expressed with:

$$\pi_l = \pi_{11} - 2(\pi_{11} - \pi_{12} - \pi_{44})(l_1^2 m_1^2 + l_1^2 n_1^2 + m_1^2 n_1^2) \quad (3.25)$$

$$\pi_t = \pi_{12} + (\pi_{11} - \pi_{12} - \pi_{44})(l_1^2 l_2^2 + m_1^2 m_2^2 + n_1^2 n_2^2) \quad (3.26)$$

where  $(l_1, m_1, n_1)$  and  $(l_2, m_2, n_2)$  are the sets of directional cosines between the longitudinal resistor and the crystal orientation. Resistors with a  $\langle 110 \rangle$  orientation have  $l_1 = m_1 = m_2 = \frac{\sqrt{2}}{2}$  and  $l_2 = -\frac{\sqrt{2}}{2}$ . The coefficients are in that case equal to:

$$\pi_{l,[110]} = \frac{1}{2}(\pi_{11} + \pi_{12} + \pi_{44}) \quad (3.27)$$

$$\pi_{t,[110]} = \frac{1}{2}(\pi_{11} + \pi_{12} - \pi_{44}) \quad (3.28)$$

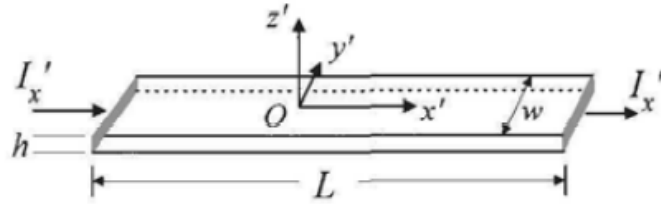


Figure 3.10: Piezoresistor with an associated coordinate system [63].

### 3.1.3.2 Connecting the membrane and the piezoresistors

The schematics used for modelling are given in Figure 3.11b.

One can now compute the resistance variation by computing the average stress applied on the different resistors. The two contributions are given by:

$$\sigma_{XX,average} = \frac{1}{A} \iint_A \sigma_{XX}(x, y) dx dy \quad (3.29)$$

$$\sigma_{YY,average} = \frac{1}{A} \iint_A \sigma_{YY}(x, y) dx dy \quad (3.30)$$

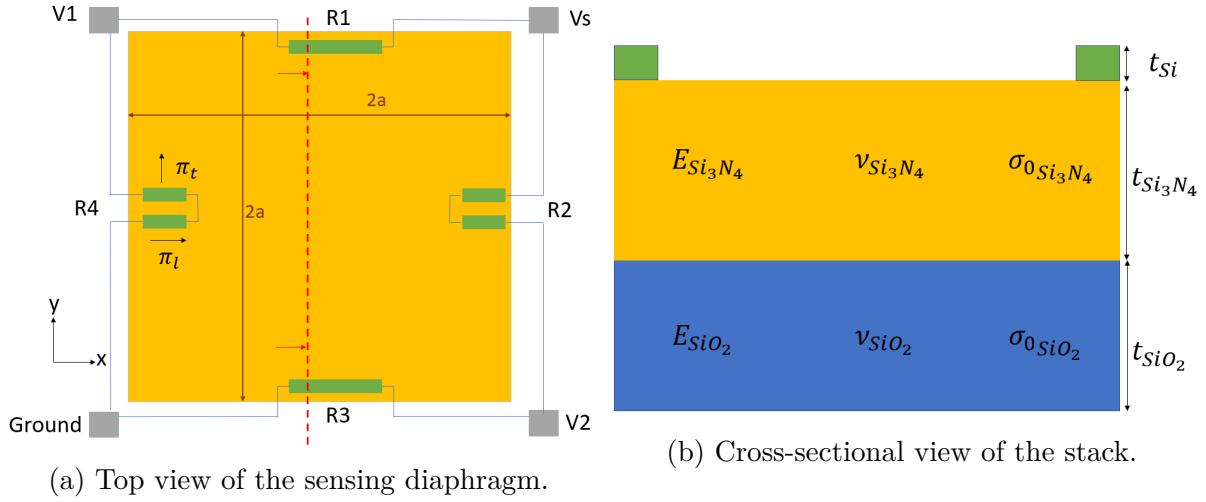


Figure 3.11: Schematics of the classic pressure sensor.

Since the thickness of the resistors is very small compared to the membrane thickness ( $t_{resi} = 100\text{nm}$  and  $t_{NO} = 1\mu\text{m}$ ) in the model considered, the stress on the surface of the membrane is assumed to be the same as the one inside the resistors.

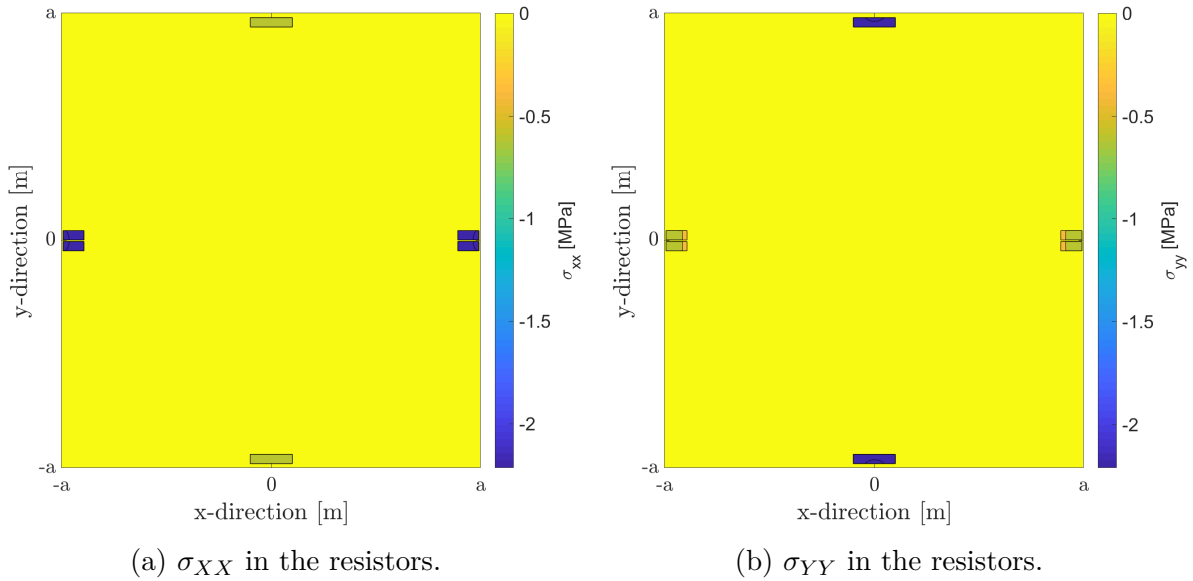


Figure 3.12: Stress distribution in the membrane.

The newly obtained relations can be injected in Equation 3.24:

$$\frac{\Delta R}{R} = \pi_l \sigma_{XX,average} + \pi_t \sigma_{YY,average} \quad (3.31)$$

### 3.1.3.3 Wheatstone bridge measurement

As presented in Chapter 2, the circuit shown in Figure 3.1a is called a Wheatstone bridge and its differential structure provides a greater output than a singular ratiometric mea-

surement. Assuming that all resistors have the same dimensions and that the conductivity is uniform, all resistors are equal when no pressure is applied:  $R_1 = R_2 = R_3 = R_4 = R_0$ . When pressure is applied, the resistors will vary with stress and the relative variations are given by:

$$\frac{\Delta R_1}{R_0} = \pi_l \sigma_{XX,averageR1} + \pi_t \sigma_{YY,averageR1} = \alpha_1 \quad (3.32)$$

$$\frac{\Delta R_2}{R_0} = \pi_l \sigma_{XX,averageR2} + \pi_t \sigma_{YY,averageR2} = \alpha_2 \quad (3.33)$$

$$\frac{\Delta R_3}{R_0} = \alpha_1 (by \text{ symmetry}) \quad (3.34)$$

$$\frac{\Delta R_4}{R_0} = \alpha_2 (by \text{ symmetry}) \quad (3.35)$$

If those relations are injected in the Wheatstone bridge measurement, the output is now given in terms of stress by:

$$V_{out} = V_1 - V_2 = \frac{R_2 R_4 - R_1 R_3}{(R_1 + R_4)(R_2 + R_3)} V_s \quad (3.36)$$

$$= \frac{R_0(1 + \alpha_2)R_0(1 + \alpha_2) - R_0(1 + \alpha_1)R_0(1 + \alpha_1)}{(R_0(1 + \alpha_1) + R_0(1 + \alpha_2))(R_0(1 + \alpha_1) + R_0(1 + \alpha_2))} V_s \quad (3.37)$$

$$= \frac{1 + 2\alpha_2 + \alpha_2^2 - 1 - 2\alpha_1 - \alpha_1^2}{(\alpha_1 + \alpha_2 + 2)^2} V_s \quad (3.38)$$

$$= \frac{\alpha_2 - \alpha_1}{2 + \alpha_2 + \alpha_1} V_s \quad (3.39)$$

When looking at the stress distributions, one can see that  $\sigma_{XX,averageR2} \gg \sigma_{XX,averageR1}$  and that  $\sigma_{YY,averageR2} \ll \sigma_{YY,averageR1}$ , which allows to simply rewrite Equation 3.39 as:

$$V_{out} \approx \frac{\pi_l \sigma_{XX,averageR2} - \pi_t \sigma_{YY,averageR1}}{2 + \pi_l \sigma_{XX,averageR2} + \pi_t \sigma_{YY,averageR1}} V_s \quad (3.40)$$

## 3.2 COMSOL simulations

The goal of this section is to present the simulations done using the *COMSOL Multiphysics* software. It allows to perform finite-element method (FEM) modelling and to couple different physics on the same project.

In this present work, all the simulations were performed in **vacuum**. The impact of air is supposed to be negligible as only stationary simulations are implemented. It allowed to isolate the effects of the stack on the behaviour of the membrane.

For starters, the **displacement** and the **stress** between two different membranes are compared. The first one is composed of an "average" material similar to this of the analytical model while the second is made of two distinct layers, a silicon nitride ( $Si_3N_4$ ) film and a silicon dioxide ( $SiO_2$ ) film on top of each other. For the rest of this work, the average model will be referred to as *model A* and the bilayer has *model B*.

Afterwards, a **stress analysis** close to the membrane edges is performed. Once completed, **piezoresistors** are added on the top layer, on the mid-side edges of the membrane. Their variation under stress will be analysed and compared with the results of the analytical modelling. Finally, the complete patterning of the piezoresistive layer is implemented and the associated stress distribution studied. The effect of patterning the piezoresistors is analysed and the optimal positioning for those is discussed.

### 3.2.1 Building the averaged and bilayer membrane

A review of how the two models were defined in *COMSOL* is described. The geometric elements considered and the corresponding boundary conditions are defined. Then, the different materials used and the procedure to create an average material are described. Furthermore, the different meshes for *model A* and *B* are discussed.

#### 3.2.1.1 Geometry and boundary conditions

As shown by the red box in Figure 3.13a, each layer is composed of a single block. For example, as *model A* is composed of one layer, only one block "average" is defined. *Model B* on the other hand has two blocks, "SiO<sub>2</sub>" and "Si<sub>3</sub>N<sub>4</sub>".

A *Work Plane* is built around the membrane to attach the boundary condition (BC) "fixed constraint". It simulates the fact that the membrane is part of the wafer.

The pressure was applied on the bottom side as a "boundary load" BC. Since the internal stresses are different for each layer, three distinct "Initial stress and strain" conditions are created into the "Linear elastic material" window.

#### 3.2.1.2 Materials

There are three materials used for the membranes,  $Si$ ,  $SiO_2$  and  $Si_3N_4$ . Silicon dioxide and nitride are needed to build the bilayer model. An average material was created as explained in Section 3.1.2.2. All of the needed material characteristics are detailed in



Table 3.3. For the piezoresistive layer, *p-type mono-crystalline Silicon* is chosen to match the design extracted from the literature [54].

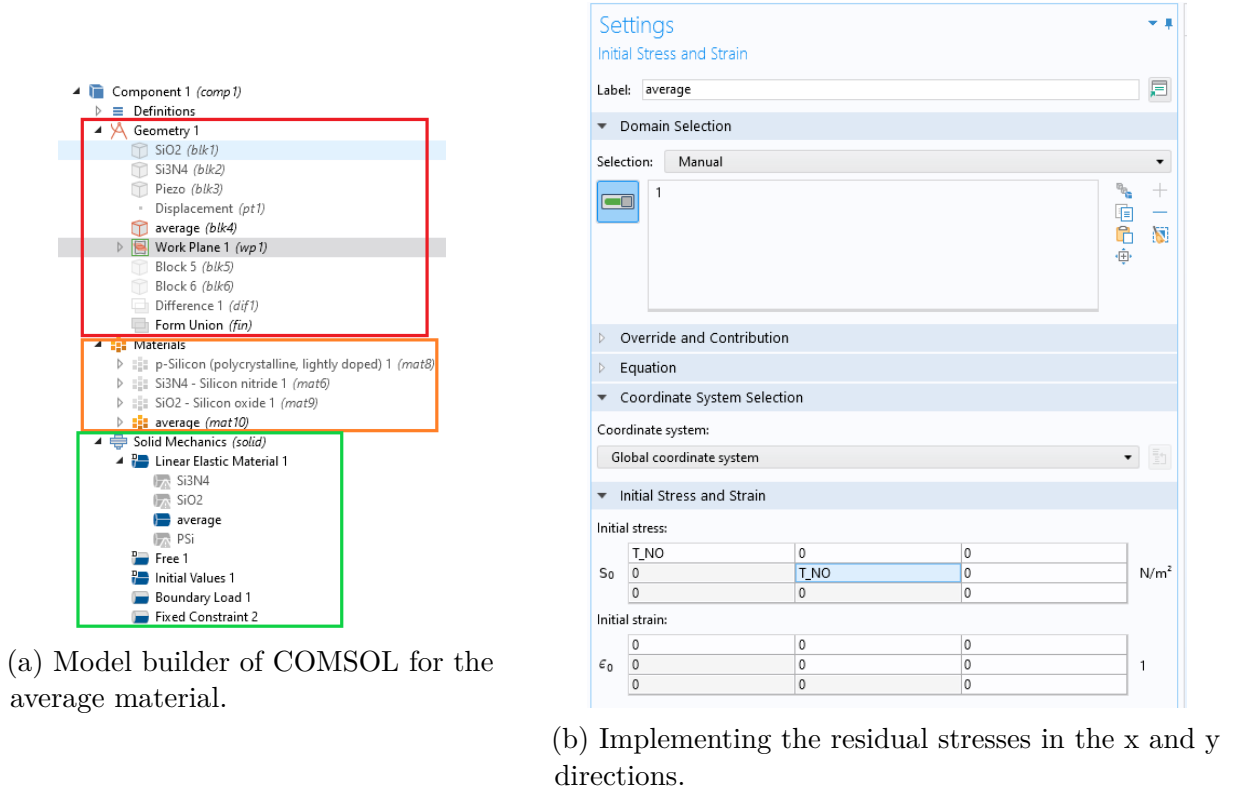


Figure 3.13: COMSOL environment and design for the single-layer simulation.

|                      | Averaged layer | $Si_3N_4$ layer | $SiO_2$ layer |
|----------------------|----------------|-----------------|---------------|
| Halfsize length [mm] | 0.5            | 0.5             | 0.5           |
| Total thickness [um] | 1              | 0.6             | 0.4           |
| E [GPa]              | 178            | 250             | 70            |
| $\nu$ [-]            | 0.206          | 0.23            | 0.17          |
| Density [ $kg/m^3$ ] | 2740           | 3100            | 2200          |
| $\sigma_0$ [MPa]     | 60             | 300             | -300          |

Table 3.3: Mechanical properties of the used materials.

### 3.2.1.3 Mesh generation for thin membranes

Each model has its own mesh in which each layer is meshed using free tetrahedrals as illustrated in Figure 3.14. The difference between the two models consists in *model A* having approximately twice less elements than *model B* since it consists of only one layer. Mesh refinements are done in different sections afterwards for precision purposes.

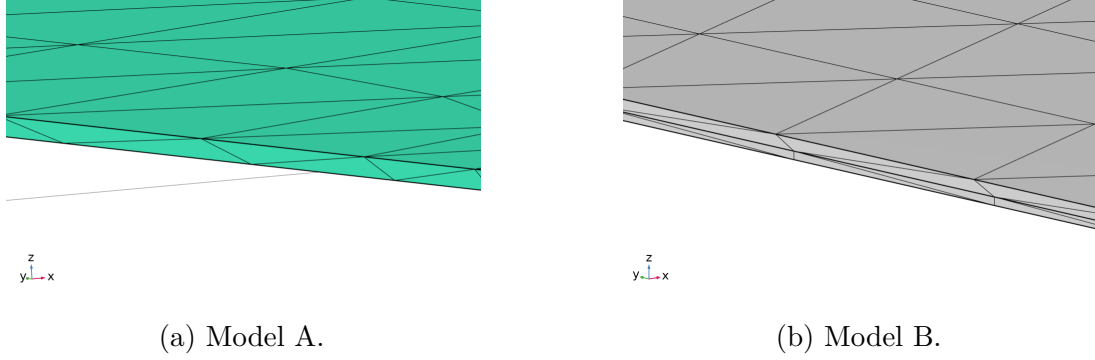


Figure 3.14: Initial meshing.

### 3.2.2 Comparison of the deflection for model A and B

The first comparison between *models A* and *B* explores the deflection under a given pressure. The principal results of each simulations are:

- 1) A comparison of the maximal displacement,
- 2) Mid-side to mid-side displacement comparison.

#### 3.2.2.1 Maximal displacement comparison

The **maximal displacement** occurs at the center of the membrane as shown in Figure 3.15. It was computed for each model with **pressures going from 0 to 200kPa** in steps of 10kPa, once including residual stresses and once excluding them.

Figure 3.16 shows that, without residual stress, both models present the same deflection. If residual stress is taken into account, it is no longer the case. The difference can come from the meshing. For *model A* the tetrahedrons go from the top surface to the bottom without intermediary steps as shown in Figure 3.14a. On the contrary, *model B* has tetrahedrons in each of its layers as shown in Figure 3.14b.

To verify this, the mesh of the average layer has been modified by putting a free triangular mesh on the top surface and then repeating it into the structure with a swept as shown in Figure 3.17. The results are shown in Figure 3.18 and it can be seen that the difference between the two is reduced by a factor 100. To quantify the difference in displacement, the mean square error between *models A* and *B* is computed. Results are shown in Table 3.4.

|     | Without swept | With swept |
|-----|---------------|------------|
| MSE | 5.643e-13     | 5.5499e-15 |

Table 3.4: MSE for the maximal deflection computed with and without a swept in the mesh.

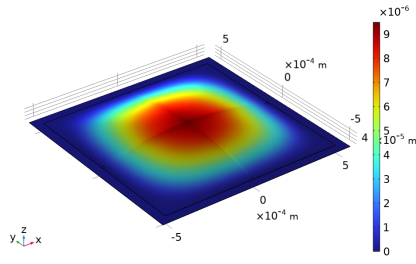


Figure 3.15: Deflection of a membrane.

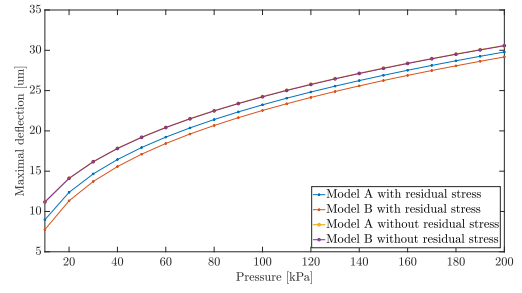


Figure 3.16: Maximal deflection for both models, with and without residual stresses.

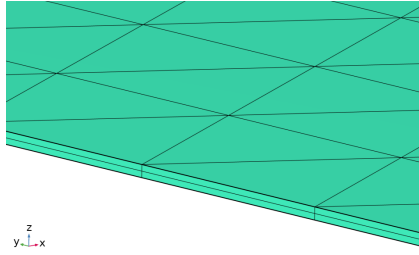


Figure 3.17: Sweeping.

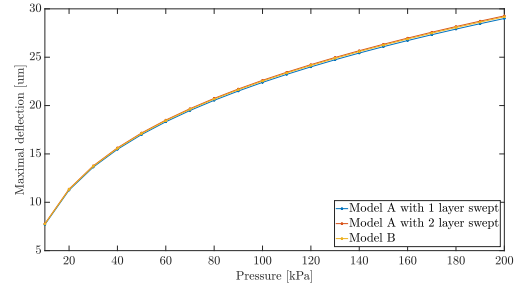


Figure 3.18: Maximal deflection for the bi-layer and the averaged layer with swept.

A zoom between zero and 10kPa was made in Figure 3.19 to have a better visualisation of what happens in this pressure range. *Model A* was meshed with a 2 layers swept, while *model B* still has the same meshing.

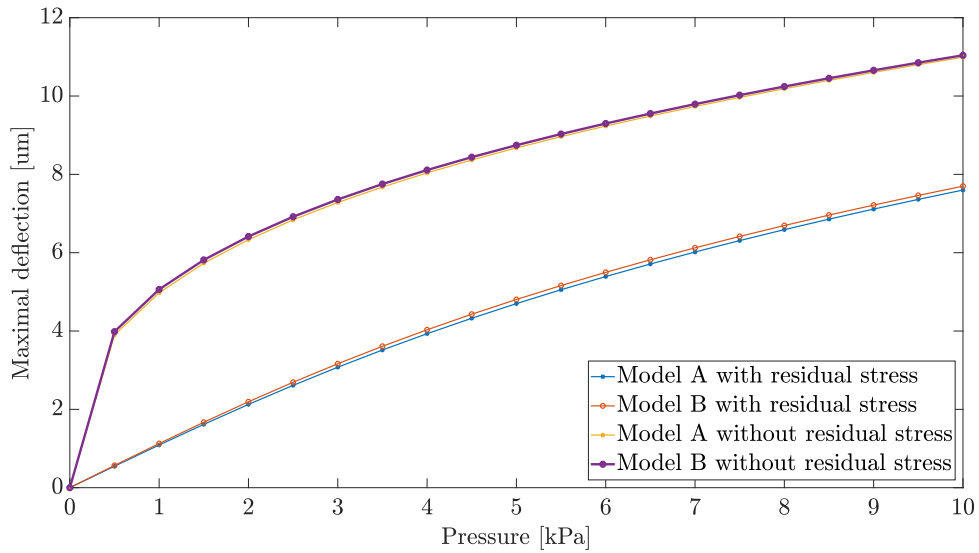


Figure 3.19: Zoom in the 0-10kPa zone.

### 3.2.2.2 Mid-side to mid-side comparison

The displacement is taken from the mid-side of an edge to the center as shown on Figure 3.20. Free tetrahedrons are again used for meshing, but they will be replaced for *model A* afterwards.

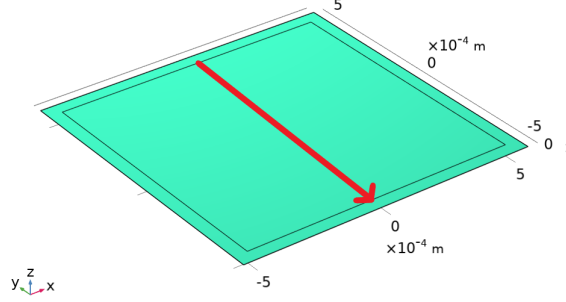


Figure 3.20: Axis on which the displacement was taken.

The results shown in Figure 3.21a exhibit the same behaviour as previously. A difference appears when the internal stresses are taken into account. To counterbalance this, the same type of meshing for *model A* as in Section 3.2.2 is applied. The outcome is displayed in Figure 3.21b, showing that the difference between *models A* and *B* is again smaller.

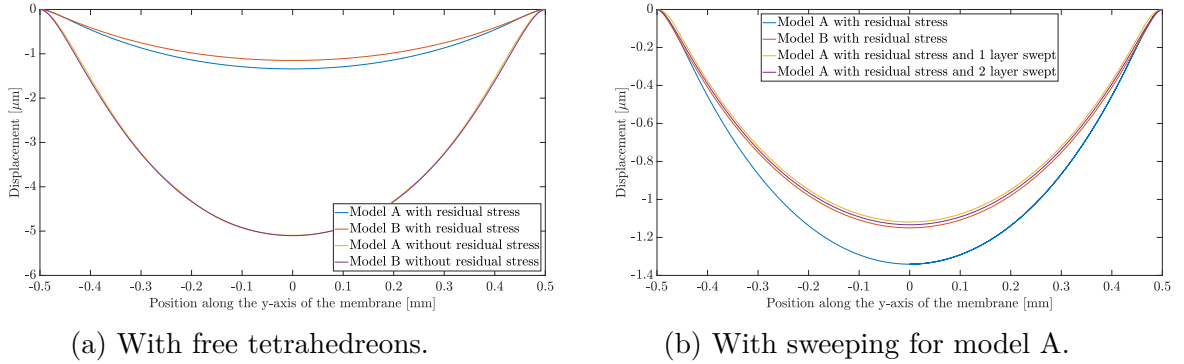


Figure 3.21: Mid-side to mid-side displacement at 1000Pa.

A final comparison at 200kPa to ensure that the displacements still coincide after modifications is presented in Figure 3.22. The less than 1% difference in the mid-side to mid-side displacement allows to conclude that *models A* and *B* are close equivalents if a two layers swept mesh is used in both.

### 3.2.3 Comparison of the stress difference $\sigma_{XX} - \sigma_{YY}$ for models A and B

The second point to check is the stress distribution between *models A* and *B* on the top surface, as the piezoresistors are placed on it. It has been shown in the analytical

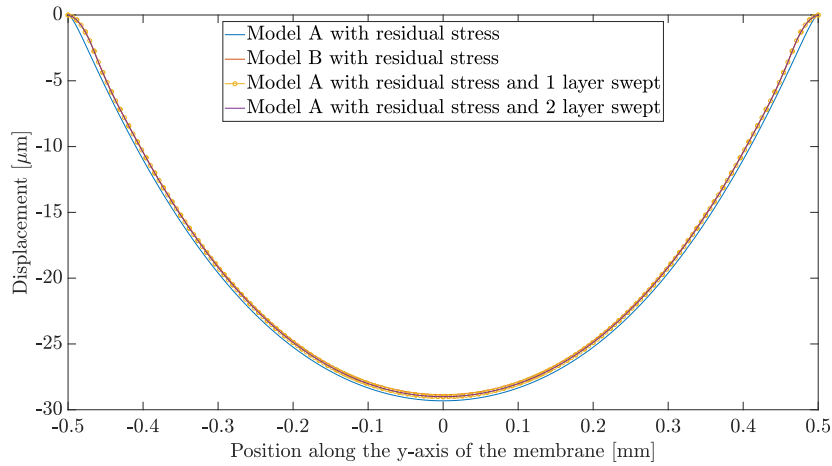


Figure 3.22: Mid-side to mid-side displacement with corrections at  $p = 200\text{kPa}$ .

development in Section 3.1.3.2 that the most critical stress for the piezoresistors is the stress difference  $\sigma_{XX} - \sigma_{YY}$ .

Two types of figures will be presented to analyse the results. The first one will be a top view of the membrane to have a clear vision of the stress difference distribution. The second will be the stress difference along two axes as shown in Figure 3.23.

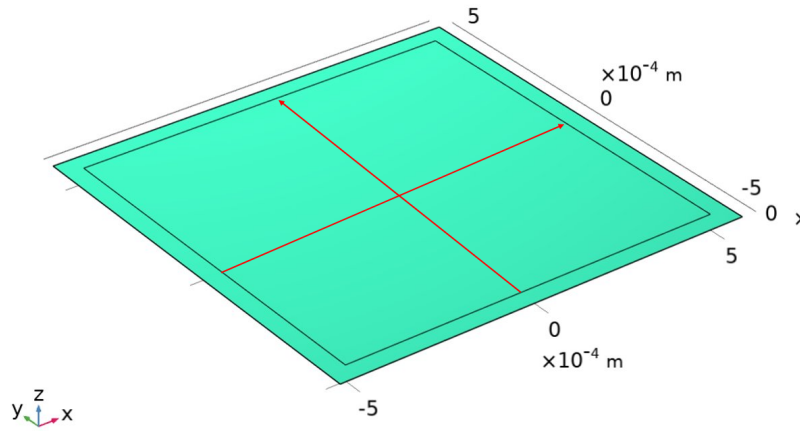


Figure 3.23: Axes along which  $\sigma_{XX} - \sigma_{YY}$  was taken.

### 3.2.3.1 Top view of $\sigma_{XX} - \sigma_{YY}$ at 1000Pa

The top views are presented in Figure 3.24 for *models A* and *B*. It can be seen that the stress is concentrated on the edges of the membrane as expected. The maximal stress difference ( $\sigma_{XX} - \sigma_{YY}$ ) on the edge decreases by approximately 15MPa if the residuals stresses are considered. The residual stresses stiffen the membrane which will deflect less as shown in Figure 3.21. But less deflection translates into a reduction of the induced stress.

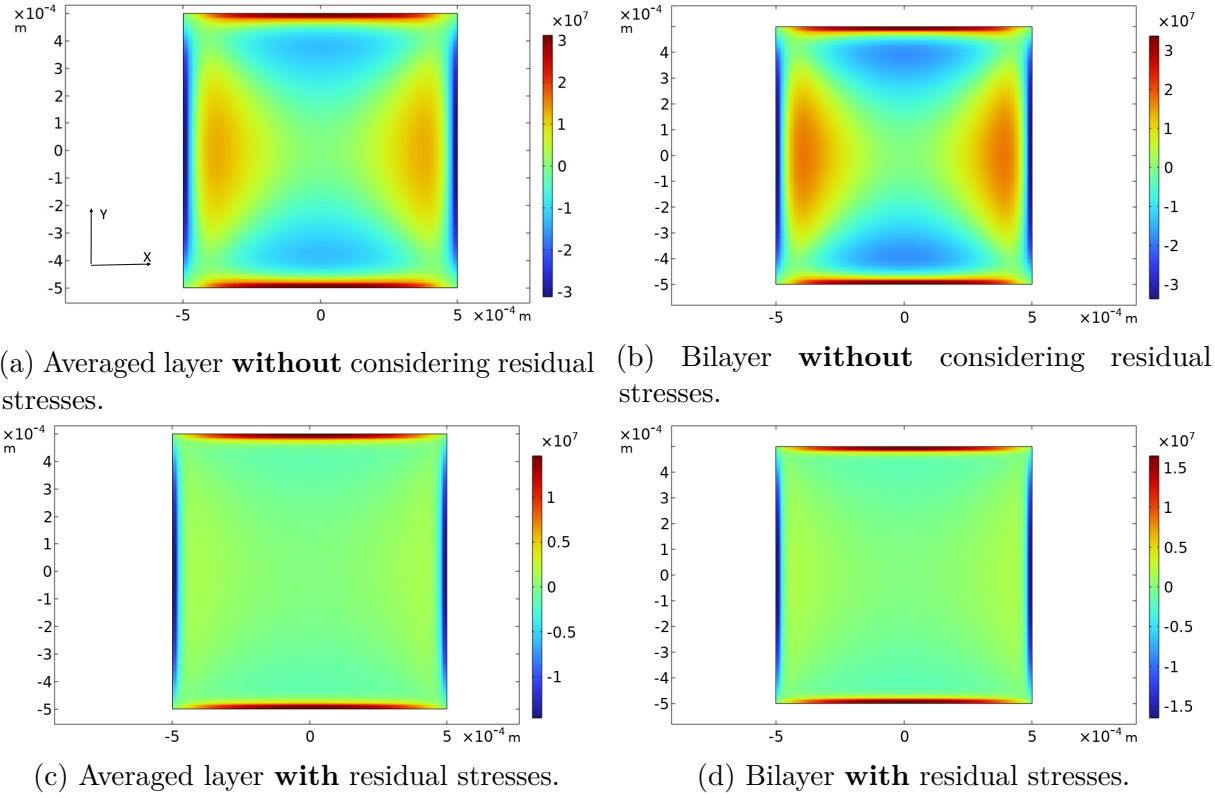


Figure 3.24: Top view of  $\sigma_{XX} - \sigma_{YY}$  distribution at  $p = 1000\text{Pa}$ .

### 3.2.3.2 Evolution of $\sigma_{XX} - \sigma_{YY}$ along two axes at 1000Pa

To more precisely compare the stress distributions, it was decided to follow the same method as for the displacement. The stress difference is taken along two axes as symmetry is a key concept in piezoresistive based pressure sensors. It is the same simulation as in Figure 3.24, the only difference is that the stresses are taken along two lines and not on the whole membrane. Results can be found in Figure 3.25 and Figure 3.26.

The variations stated earlier about the maximal stress difference are again found here. Another difference appears between *models A* and *B*; the maximal stress close to the clamping sides is lower for *model A* by a factor of 1.4 approximately.

When going from one edge towards the center, the stress goes from being tensile (compressive) near the edge to being compressive (tensile) further away before reaching zero at the center.

The average layer shows an imprecision in the stress computation near the clamping sites. A corrective factor can be applied to counter this but will not be done in this work.

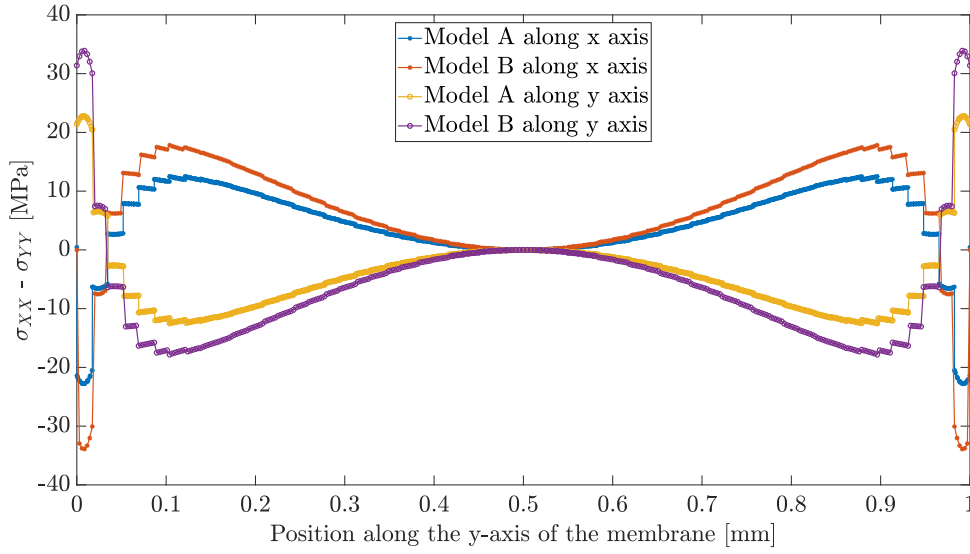


Figure 3.25:  $\sigma_{XX} - \sigma_{YY}$  without residual stresses at  $p = 1000\text{Pa}$ .

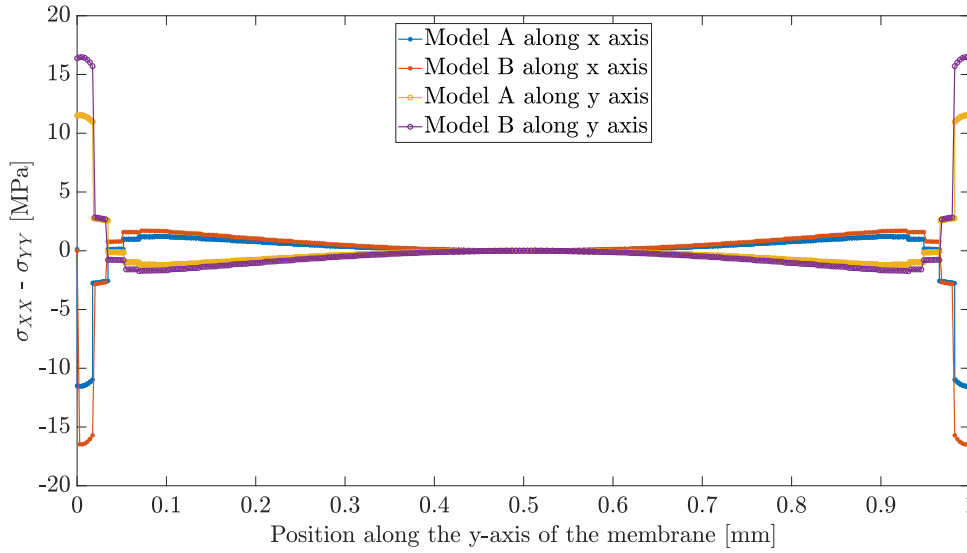


Figure 3.26:  $\sigma_{XX} - \sigma_{YY}$  including residual stresses at  $p = 1000\text{Pa}$ .

### 3.2.3.3 Top view of $\sigma_{XX} - \sigma_{YY}$ at 200kPa

The same stress verification protocol is applied for a pressure of 200kPa to see if this factor between *models A* and *B* is still there.

Top views of the stress difference at 200kPa are showed in Figure 3.27 for each case considered. The shape of the stress distribution is the same for both models.

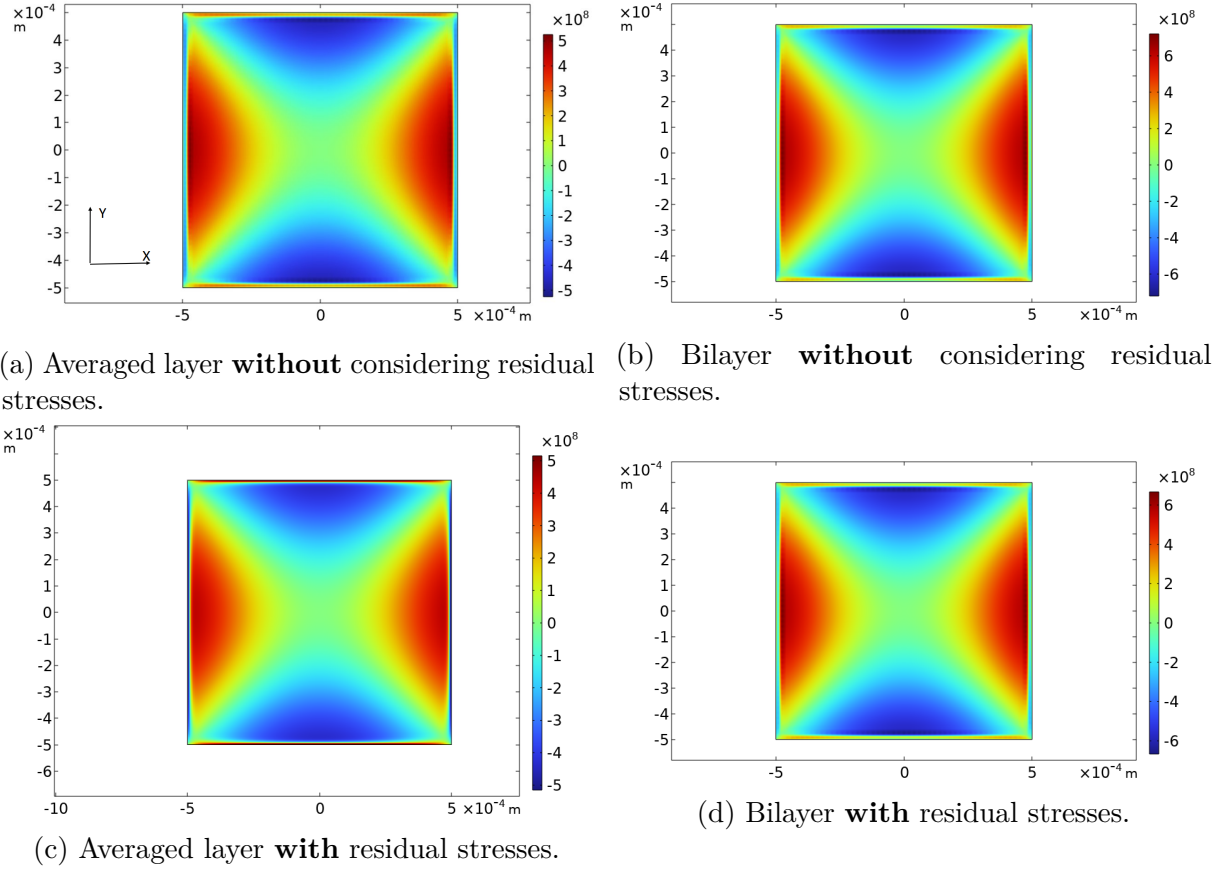


Figure 3.27: Top view of  $\sigma_{XX} - \sigma_{YY}$  distribution at  $p = 200\text{kPa}$ .

### 3.2.3.4 Evolution of $\sigma_{XX} - \sigma_{YY}$ along two axes at 200kPa

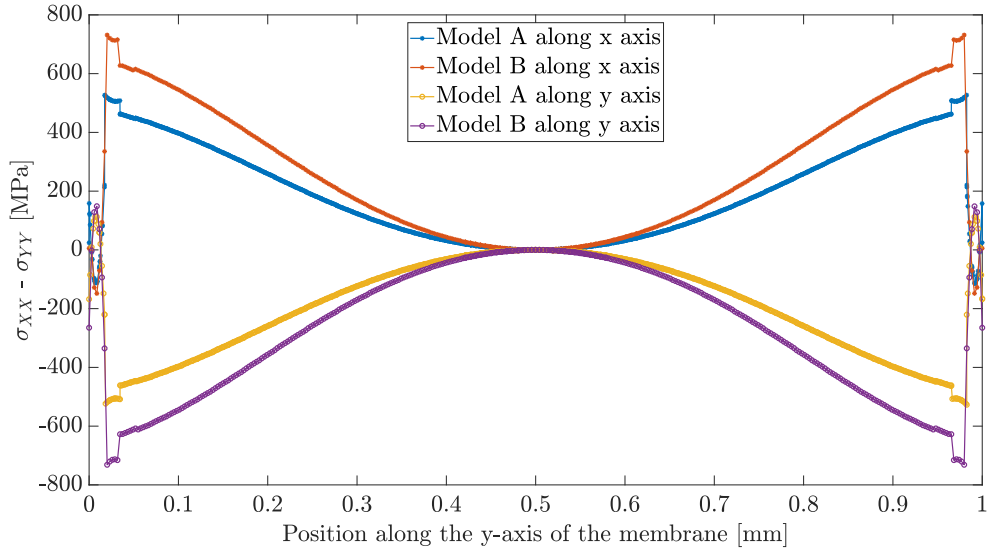
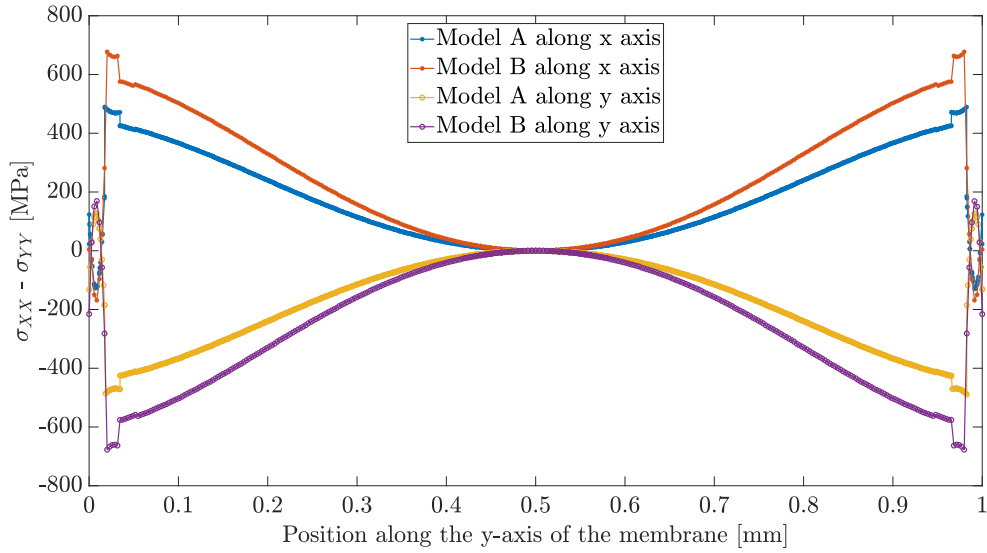
Figure 3.28 and Figure 3.29 display the results. There is still a 1.4 factor between the maximal stress between *models A* and *B*. An imprecision in the stress computation appears near the clamping sites. It can arise from the a too coarse mesh, but it will not be further studied.

*Model A* is used for the rest of this work although it has to be noted that it presents a limitation for the stress computation.

## 3.2.4 Comparison of the deflection for model A and for the analytical model

Now that the average layer model has been compared to the more complete bilayer model and validated, it has to be compared to the analytical modelling done previously. The first comparison point is the deflection. The methodology is the same as in Section 3.2.2, and the FoMs considered are the maximal deflection and the MSE between the mid-side to mid-side displacements of both models.




 Figure 3.28:  $\sigma_{XX} - \sigma_{YY}$  without residual stresses at  $p = 200\text{kPa}$ .

 Figure 3.29:  $\sigma_{XX} - \sigma_{YY}$  including residual stresses at  $p = 200\text{kPa}$ .

### 3.2.4.1 Maximal deflection comparison

Figure 3.30 presents a pressure sweep between 0 and 10kPa with steps of 500Pa while Figure 3.31 displays a coarser sweep from 10kPa to 200kPa with steps of 10kPa. Both sweeps permitted to see the contrast between *model A* and the analytical one on two different scales.

A consistent difference of around  $0.5\mu\text{m}$  between the curves of *model A* and the analytical ones is observed, whether the residual stress is included or not.

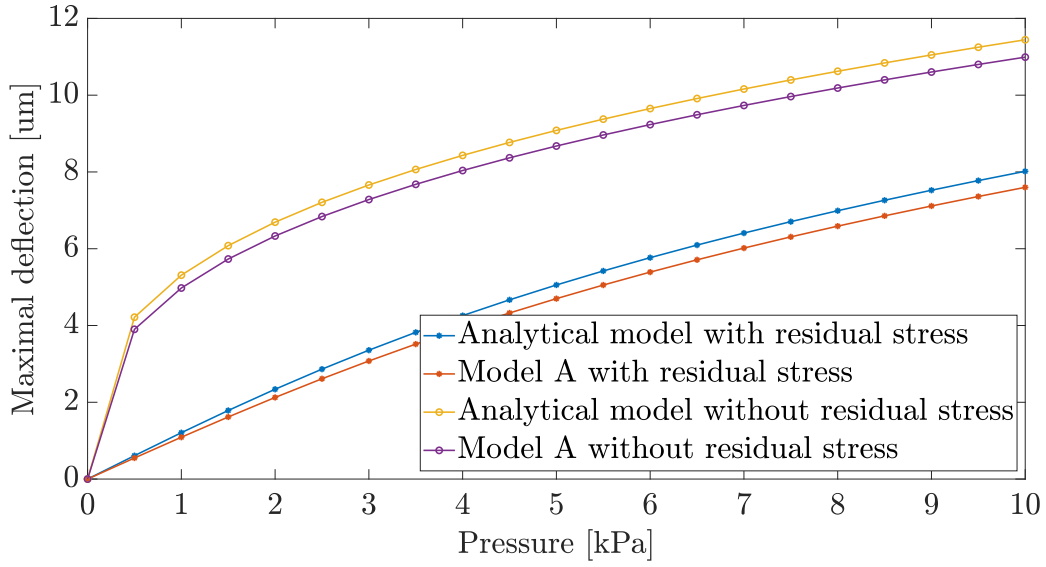


Figure 3.30: Maximal deflection from 0 to 10kPa.

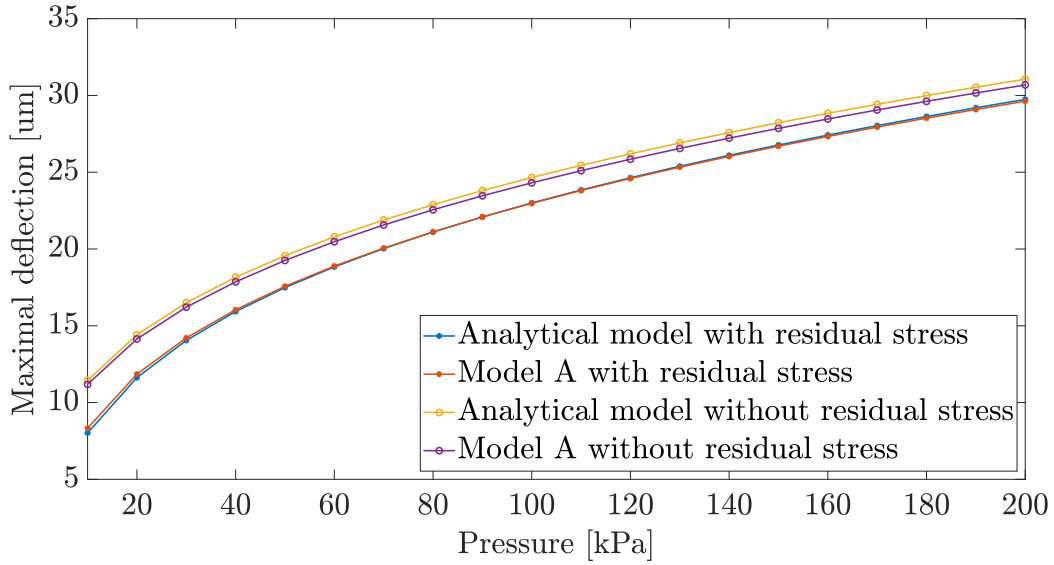


Figure 3.31: Maximal deflection from 10 kPa to 200kPa.

### 3.2.4.2 Mid-side to mid-side comparison

As a reminder, the models used to approximate the displacement of the membrane are:

$$\hat{w}_1(0, y) = \frac{w_0}{2} \left( 1 + \cos \left( \frac{\pi y}{a} \right) \right) \quad (3.41)$$

$$\hat{w}_2(0, y) = w_0 \left( 1 + 0.401 \left( \frac{y}{a} \right)^2 \right) \cos \left( \frac{\pi y}{2a} \right) \quad (3.42)$$

$$\hat{w}_3(0, y) = w_0 \left( 1 - \left( \frac{y}{a} \right)^2 \right)^2 \quad (3.43)$$

As shown in Figure 3.32, the general shape of the displacement is best approximated by  $\hat{w}_2$ . A zoom in the red dotted circle near the clamping edges in Figure 3.33 shows however that  $\hat{w}_1$  and  $\hat{w}_3$  provide a better correspondence with the physical reality near the membrane edges than  $\hat{w}_2$ .

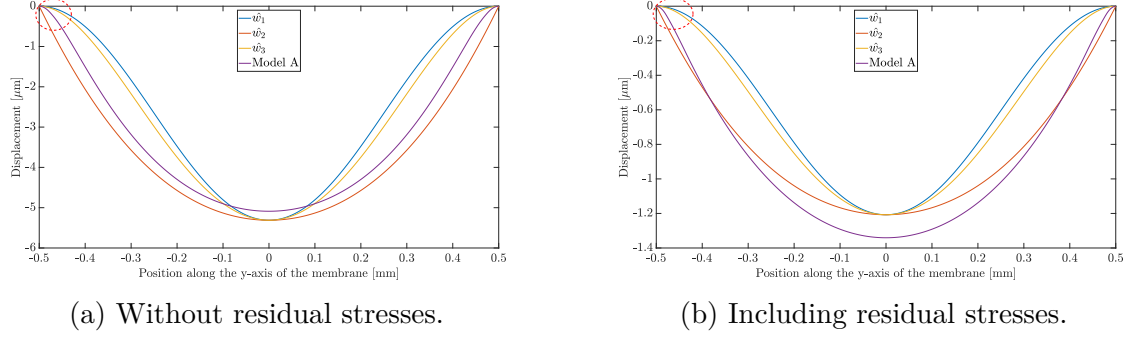


Figure 3.32: Mid-side to mid-side displacement at 1000Pa.

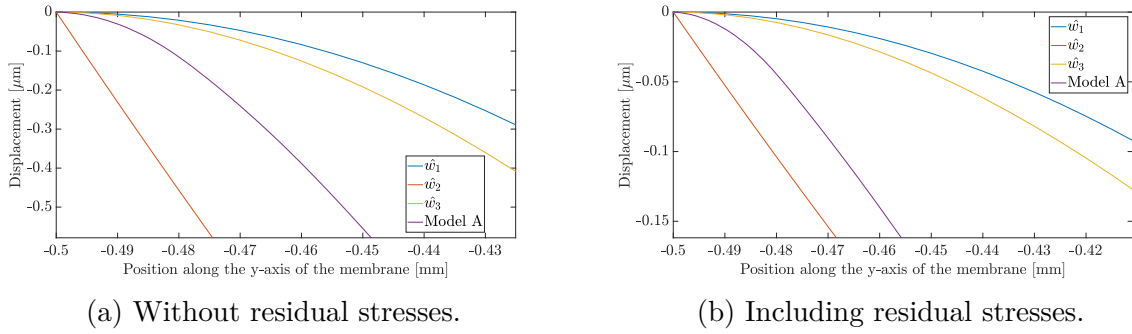


Figure 3.33: Zoom on the clamping sides.

When looking at the MSE of the mid-side to mid-side displacements in Table 3.5, none of the proposed models is really in accordance with *model A*. The general shape is best given by  $\hat{w}_2$  while the other two give better results near the clamping sites.

|     | $\hat{w}_1$ | $\hat{w}_2$ | $\hat{w}_3$ |
|-----|-------------|-------------|-------------|
| MSE | 9.6079e-14  | 7.9172e-15  | 6.8955e-14  |

Table 3.5: MSE of the mid-side to mid-side comparison between the displacements given by *COMSOL* and by  $\hat{w}_1, \hat{w}_2, \hat{w}_3$ .

Another analytical formula which is only valid for a mid-side to mid-side displacement *with* residual stresses was found in the literature [72]:

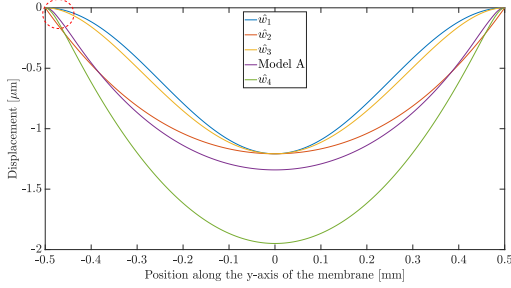
$$\hat{w}_4(y) = \frac{p(2a)^4}{16u^3 D \tanh(u)} \left( \frac{\cosh(u(1 - y/a))}{\cosh(u)} \right) + \frac{p(2a)^2(2a - y)y}{8u^2 D} \quad (3.44)$$

where  $u = \sqrt{\frac{S(2a)^2}{4D}}$  and  $S$  is an axial distributed force.

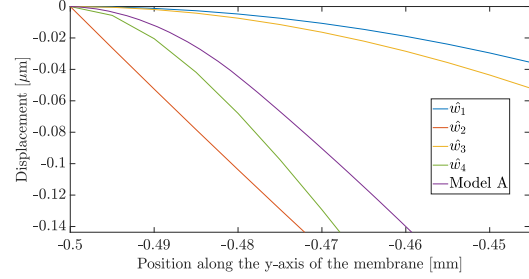
The deflections are displayed in Figure 3.34 and the MSE in Table 3.6.

|     |             |
|-----|-------------|
|     | $\hat{w}_4$ |
| MSE | 1.7586e-13  |

Table 3.6: MSE of the mid-side to mid-side comparison between the displacement given by *COMSOL* and by  $\hat{w}_4$ .



(a) Displacement mid-side to mid-side.



(b) Zoom on the clamping sites.

Figure 3.34: Simulation including the new model.

Even if the maximal displacement produced with  $\hat{w}_4$  is less similar to these produced by *model A* and by the other analytical models, its shape near the clamping sides is closer to what is computed by *COMSOL* as seen in Figure 3.34a. Since this model was found quite late during this research work, it has not been further investigated.

### 3.2.5 Comparison of the stress distribution for model A and for the analytical model

Two comparisons are done using same protocol as in Section 3.2.3. The figures containing the stress distribution of *model A* can be found in this same section. Since the second derivative of the displacement  $w(x, y)$  is needed to compute the stress, the deflection is given by  $\hat{w}_1$  as in the analytical modeling.

#### 3.2.5.1 Top view of $\sigma_{XX} - \sigma_{YY}$

The following results are obtained with a pressure of 1000Pa. By comparing Figure 3.35a to Figure 3.24a and Figure 3.35b to Figure 3.24c, it can be seen that the stress difference is symmetric along the  $x$  and  $y$  axes as for *model A*. However, the maximal values found on the edges differ by one order of magnitude.

A possible explanation for this disparity resides in the approximation used to compute the stress. As seen in Figure 3.33,  $\hat{w}_1$  is not really concordant with the results given by *COMSOL* for *model A*, especially along the clamping sides. The second derivative of the displacement along the  $y$ -axis is therefore greater using the displacement provided by *COMSOL*.

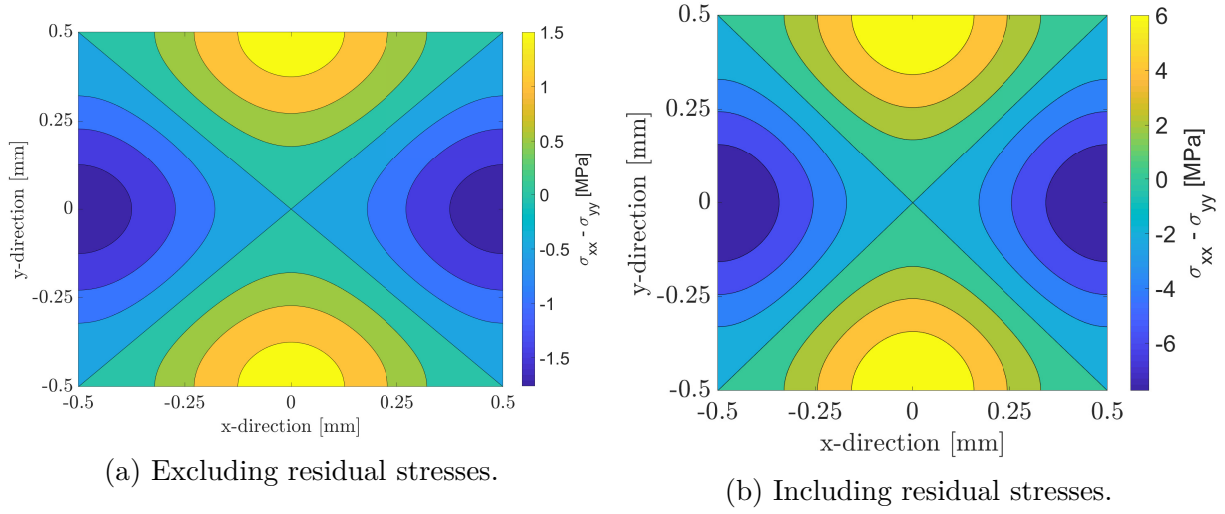


Figure 3.35:  $\sigma_{XX} - \sigma_{YY}$  computed with  $\hat{w}_1$ .

### 3.2.5.2 Mid-side to mid-side comparison

Since  $\hat{w}_1$  did not give good results, a mid-side to mid-side comparison of the stress distributions was done using  $\hat{w}_1$ ,  $\hat{w}_3$  and  $\hat{w}_4$ , as well as results from *model A*. For the stresses computed with  $\hat{w}_1$  and  $\hat{w}_3$ , the displacement is known everywhere so the stress model is given by:

$$\sigma_{XX} = -\frac{E_{NO}t_{NO}}{2(1-\nu^2)} \left( \frac{\partial^2 w(x,y)}{\partial x^2} + \nu_{NO} \frac{\partial^2 w(x,y)}{\partial y^2} \right) \quad (3.45)$$

But for  $\hat{w}_4$ , only the mid-side to mid-side displacement is known, and the derivative can only be done following one axis. The stress model is then given by:

$$\sigma_{YY} = -\frac{E_{NO}t_{NO}}{2(1-\nu^2)} \left( \frac{\partial^2 w(x,y)}{\partial y^2} \right) \quad (3.46)$$

The results are displayed in Figure 3.36. As expected, the stress difference computed with  $\hat{w}_1$  is very different from *model A*, and so is the one computed with  $\hat{w}_2$ . With  $\hat{w}_4$  however, the computed stresses are higher than the ones given by *model A*. This is maybe due to the second derivative missing along the other axis as the displacement was not known everywhere.

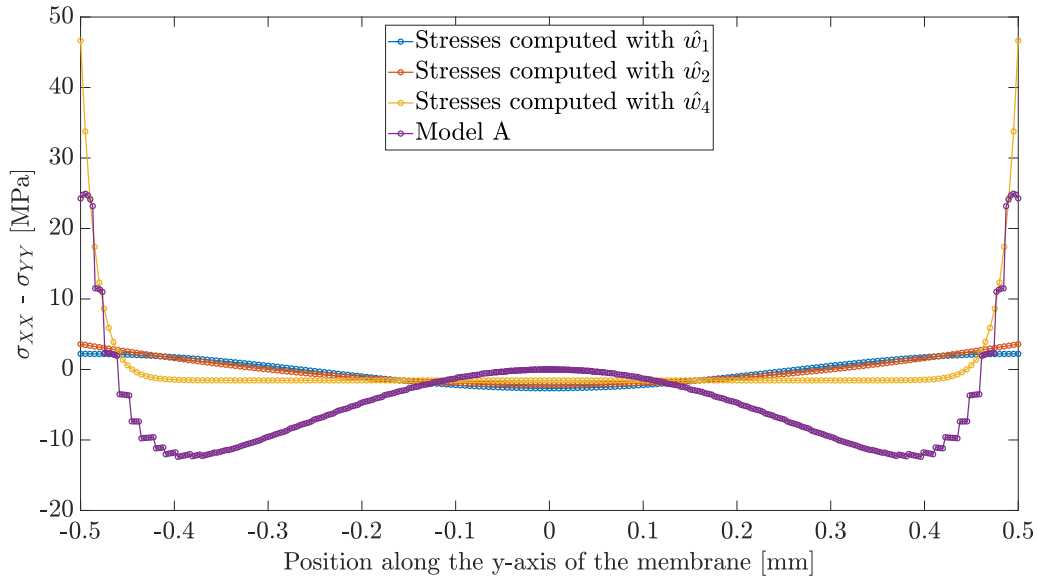


Figure 3.36: Comparing the stress difference between the analytical models and model A.

### 3.2.6 Refining the mesh for stress calculation

With *model A* validated for displacement and stress over the whole membrane, a *Work Plane* (WP) that has the size of a piezoresistor ( $40\mu m \times 10\mu m$ ) was defined as shown in Figure 3.37. It allows to have a more precise insight on the stress distribution. The stress was taken along the dotted line with its beginning being at the membrane edge.

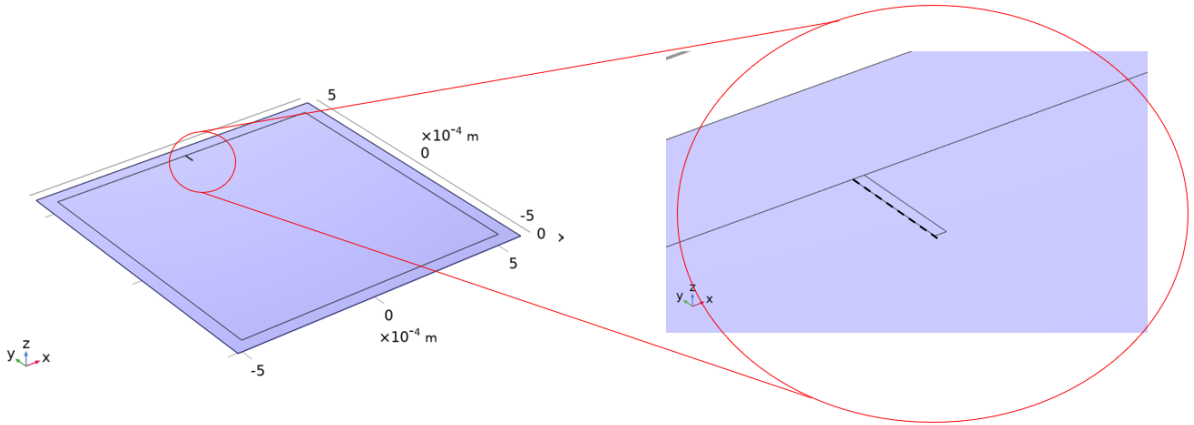


Figure 3.37: Work plane with size of the piezoresistor.

The first attempt to compute the stress with the coarse mesh used earlier as illustrated in Figure 3.38a led to jagged results. As can be seen in Figure 3.39, there are well defined stress levels inside the WP. Those come from the meshing, each step corresponding to a change of element.

Using a finer mesh as shown in Figure 3.38b gives better results, even if a discontinuity appeared around  $1\mu m$ . The hypothesis to explain this discontinuity is also related to the

mesh size since  $1\ \mu\text{m}$  corresponds to the size of the smallest element of the mesh. No further investigations related to this were done because not enough RAM memory were available to support a finer mesh.

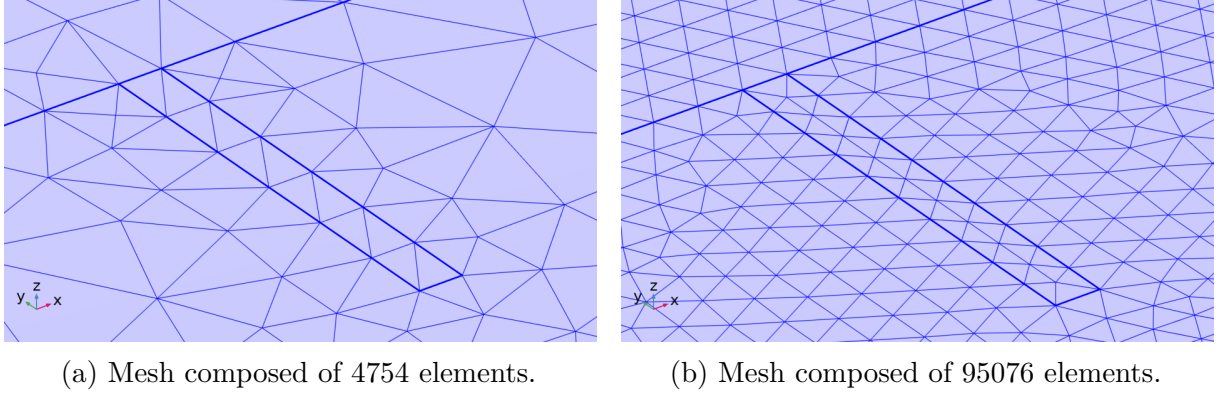


Figure 3.38: Coarser and finer meshes

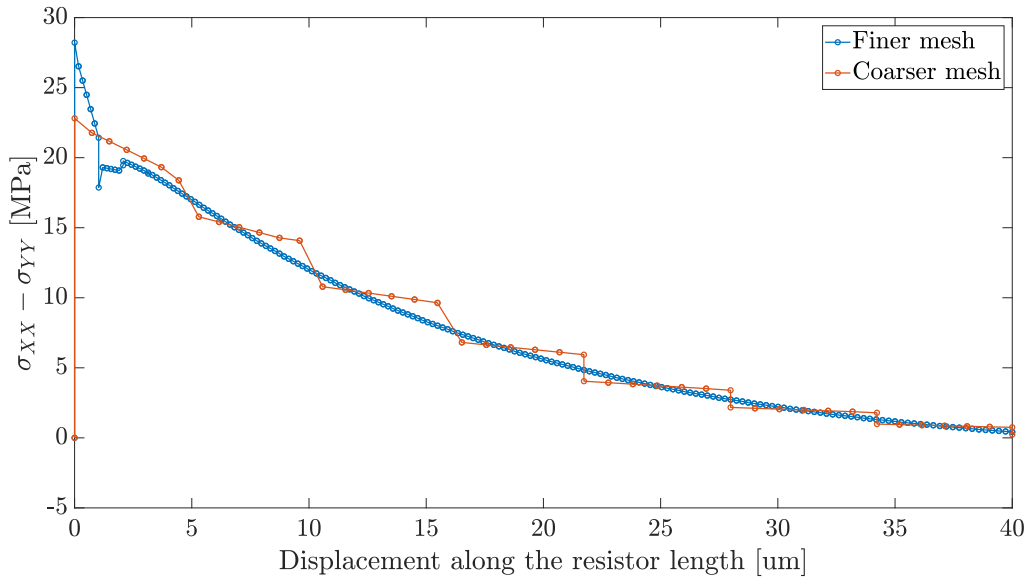


Figure 3.39:  $\sigma_{xx} - \sigma_{yy}$ .

### 3.2.7 Verification of the piezoresistive physics of COMSOL

This section proposes a verification of the piezoresistive physics. Two piezoresistors made of p-type mono-crystalline silicon (Si) were placed on an edge of the membrane with a 5V voltage difference on their sides. The direction of the piezoresistors is chosen to be  $\langle 110 \rangle$  as it is the best orientation for such devices [25]. This is achieved by defining a new coordinate system for the p-type silicon with a  $45^\circ$  rotation as shown in Figure 3.41. One is longitudinal with regards to the membrane side while the other is transverse.

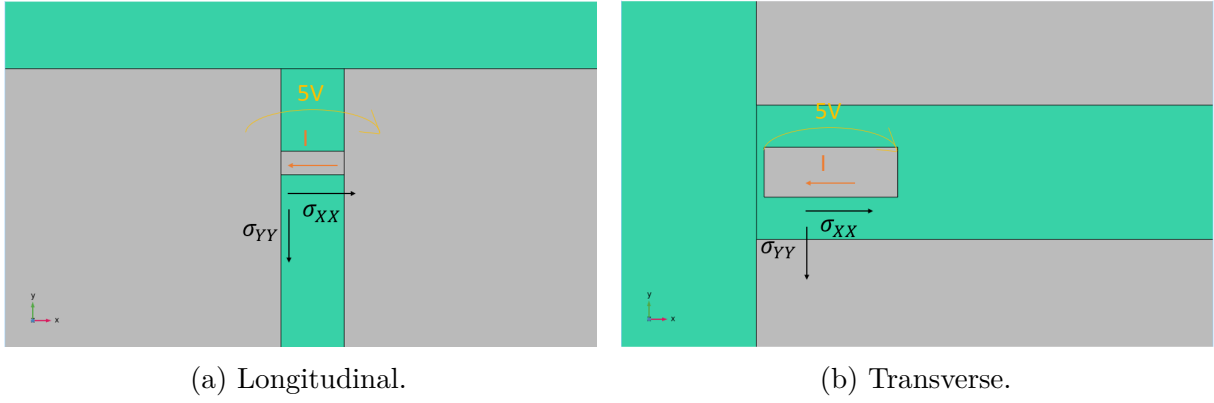


Figure 3.40: Building of piezoresistors.

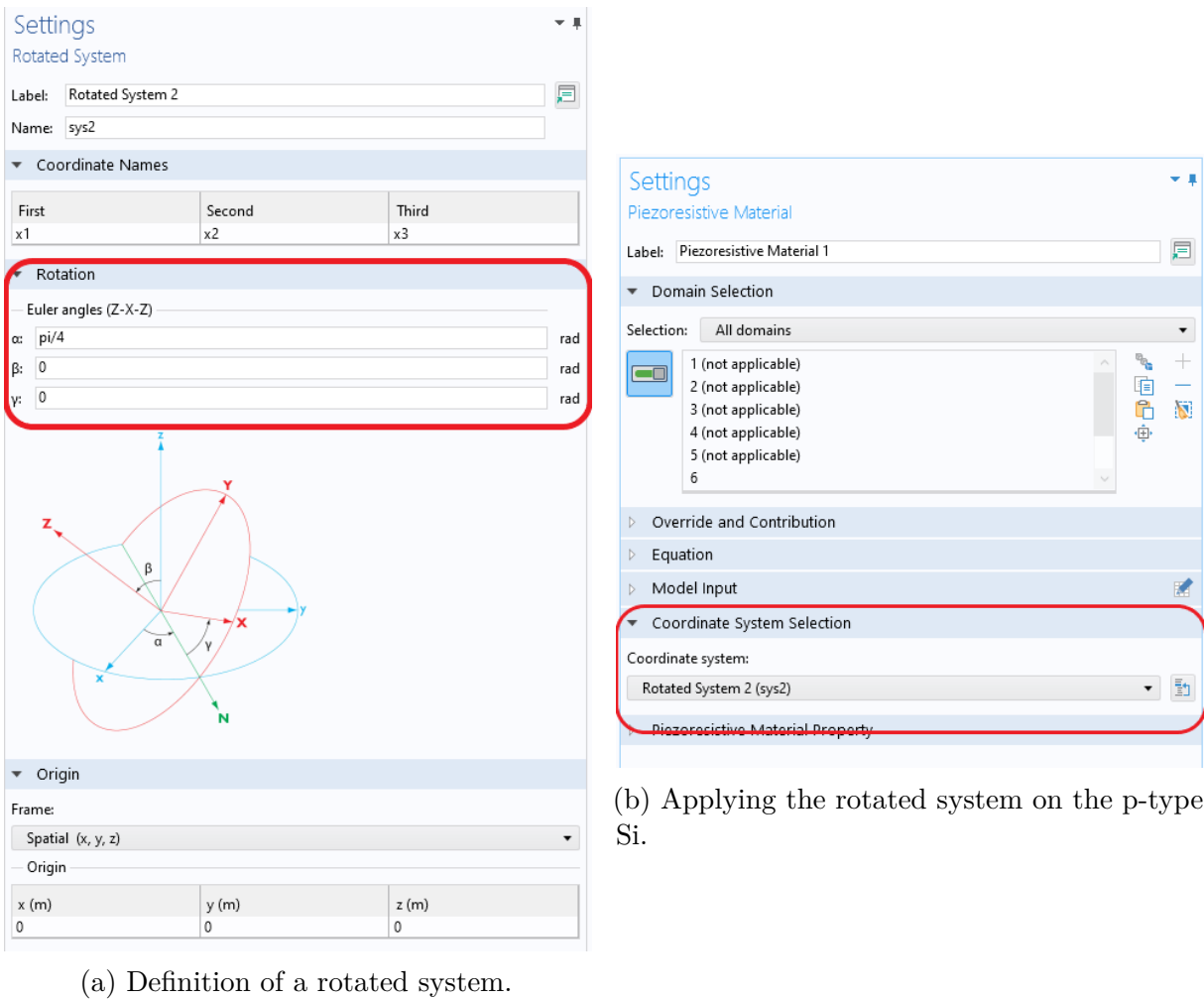


Figure 3.41: Rotated systems for the piezoresistors orientation.

The piezoresistive effect is compared using two methods for a pressure sweep from 0 to 1000Pa. The first method consists in measuring the current through the resistors and



computing the relative resistance variation with :

$$\frac{R(\sigma) - R_0}{R_0} = \frac{\Delta R}{R_0} = \frac{I(\sigma) - I_0}{I_0} * 100[\%] \quad (3.47)$$

To verify these results, the mean stresses ( $\sigma_{XX,av}$  and  $\sigma_{YY,av}$ ) inside the resistors are also measured. Knowing the orientation and the used type of material, the piezoresistive coefficients are given by:

$$\pi_l = \frac{1}{2}(pi_{11} + pi_{12} + pi_{44}) = 71.8 * 10^{-11}[Pa^{-1}] \quad (3.48)$$

$$\pi_t = \frac{1}{2}(pi_{11} + pi_{12} - pi_{44}) = -66.3 * 10^{-11}[Pa^{-1}] \quad (3.49)$$

As developed in Section 3.1.3.3, the relative variation of the resistors is given by :

$$\alpha = (\pi_l \sigma_{XX,av} + \pi_t \sigma_{YY,av}) * 100[\%] \quad (3.50)$$

The theoretical relative resistance variation computed with the stresses and the one computed by *COMSOL* with the current variation are shown in Figure 3.42.

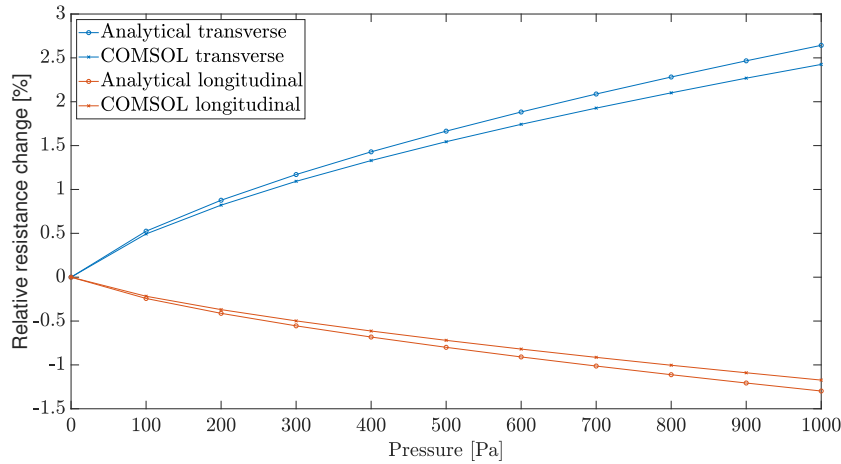


Figure 3.42: Variation of the resistors.

The results show a maximal variation of 1.5% at 1000Pa, meaning that the piezoresistive variation of *COMSOL* is validated.

### 3.2.8 Effect of the patterned piezoresistive layer on the stress

The final design will have a patterned piezoresistive layer on top of the silicon nitride layer. A 100nm thick layer is added on the top of the membrane and patterned without the piezoresistors. This influences the overall stress distribution. Since the zone of interest lies in the patterning, the mesh has to be modified as shown in Figure 3.43 to have a enhanced precision on the patterned zone.

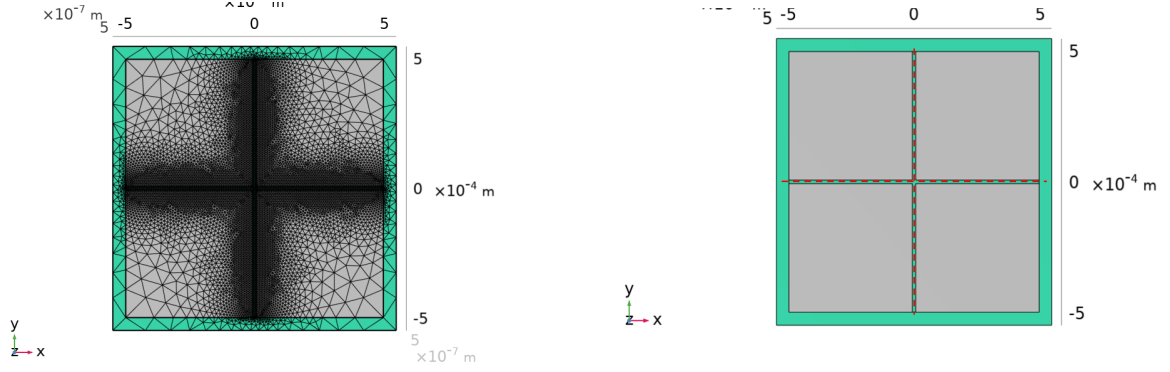


Figure 3.43: New mesh with the patterning.

Figure 3.44: Lines on which  $\sigma_{XX} - \sigma_{YY}$  was

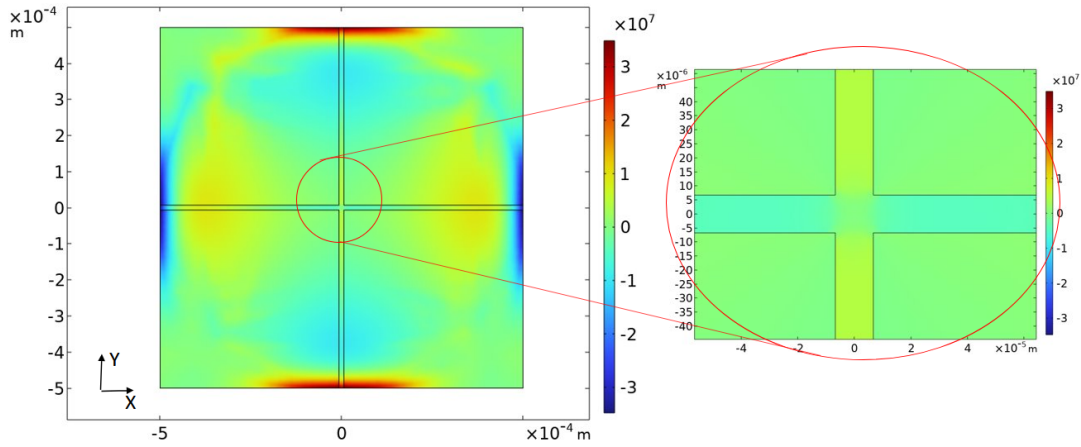


Figure 3.45:  $\sigma_{XX} - \sigma_{YY}$  with the patterning.

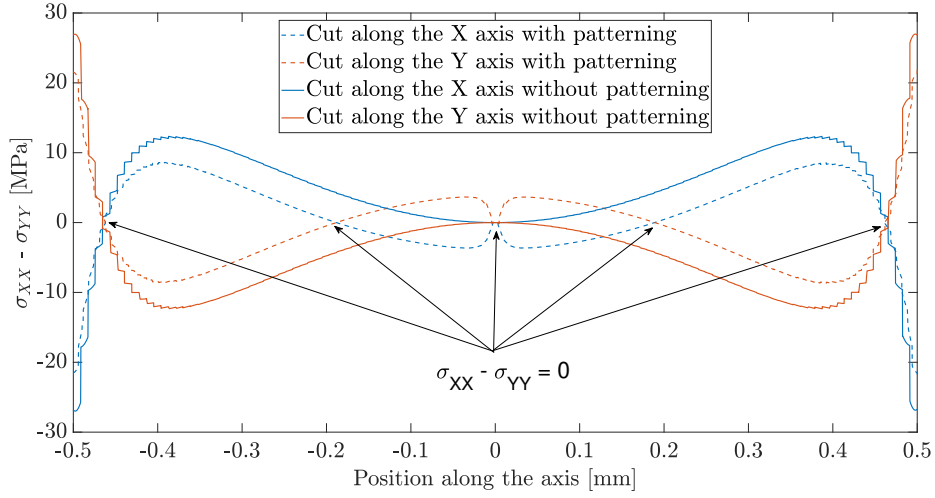
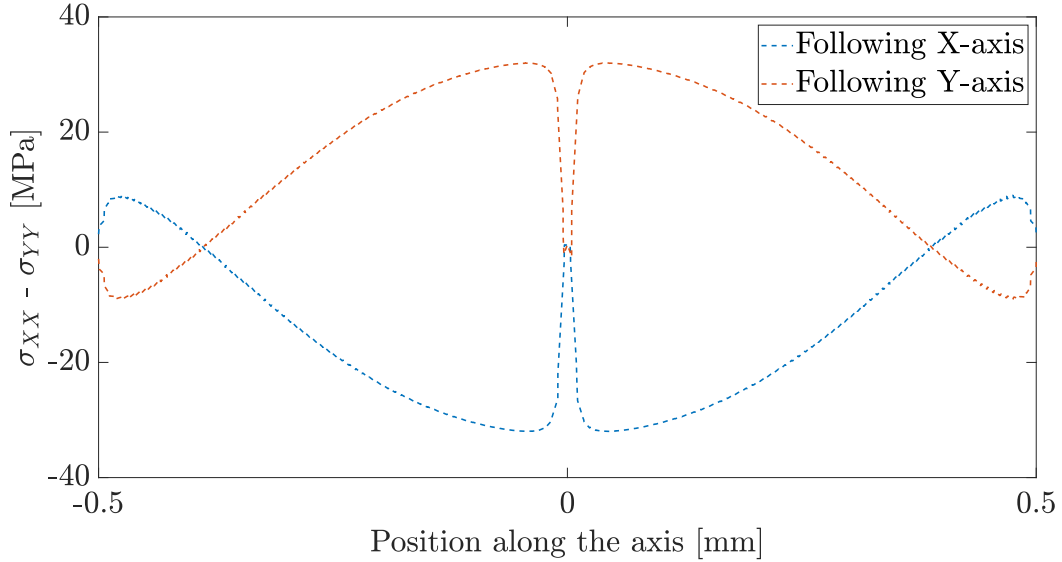
Figure 3.45 shows a top view of the stress difference  $\sigma_{XX} - \sigma_{YY}$  distribution. A zoom in the middle of the membrane shows the appearance of one zone where this difference is positive and another where it is negative.

The stress difference on the top of the nitride layer is taken along two axes as displayed in Figure 3.44, once with the patterning and once without for comparison.

The results are shown in Figure 3.46 and allow to see that the patterning decreases the stresses close to the membrane edges but increases when approaching the center of the membrane. This is also called a *stress concentration*. Another phenomenon is the appearance of zero stress at  $x = \pm 0.2\text{mm}$  and  $y = \pm 0.2\text{mm}$  with the 100nm patterned layer.

The thickness of the piezoresistive layer is increased to  $1\mu\text{m}$  to see the effect on the stresses.

Expanding the piezoresistive layer thickness will concentrate the stress even more near the center of the membrane as shown in Figure 3.47. An optimal thickness could be found to have the greatest stress concentration with the largest sensitivity in future works.


 Figure 3.46:  $\sigma_{XX} - \sigma_{YY}$  with and without patterning.

 Figure 3.47:  $\sigma_{XX} - \sigma_{YY}$  Along X and Y axes for  $1\mu m$  thick piezoresistive layer.

### 3.2.9 Finding the best positions for the resistors

This part studies the influence of the position of the resistors on their relative resistance variations. As shown in Figure 3.48, the resistors are moved at 3 different locations. The results in Figure 3.47 state that increasing the thickness of the piezoresistive layer concentrates the stresses in the middle of the membrane. At a certain thickness, it becomes more interesting to place the resistors in position 1.

To verify this, two simulations, each one with a different piezoresistive thickness, are performed. The relative resistance is computed for each position. With a 100nm thickness, position 3 is best for both resistors as shown in Figure 3.49. However, when the thickness increases to 1000nm as displayed in Figure 3.50, position 1 becomes the most interesting one for both resistors.

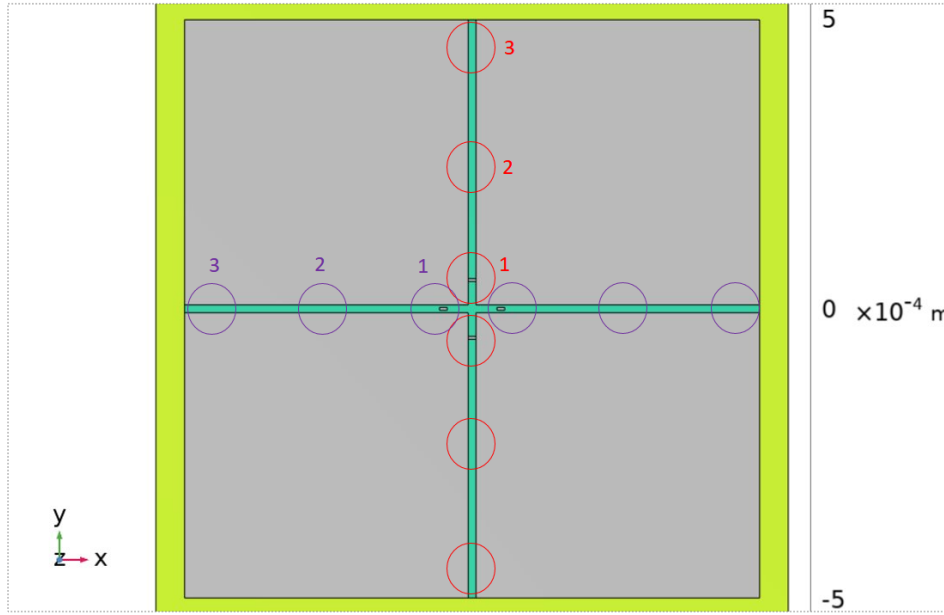


Figure 3.48: Studied resistor positions.

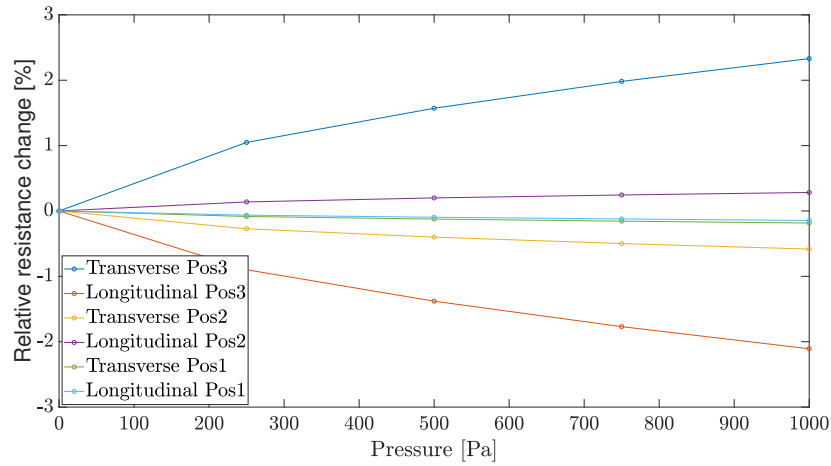


Figure 3.49: Transverse and longitudinal relative resistance variation with a 100nm piezoresistive layer.

### 3.2.10 Converting the resistance variation into voltage

The last point is about converting those resistance variations in a measurable voltage. For this, a full Wheatstone bridge is proposed to increase the sensitivity.

The voltage difference with 100nm thickness is displayed in Figure 3.51. Position 3 is the most interesting one as expected since the stress difference is the biggest near the edges. Figure 3.52 shows the voltage difference with a 1000nm piezoresistive layer. Position 1 becomes the best one as expected but the sensitivity decreases compared to the 100nm thick layer.

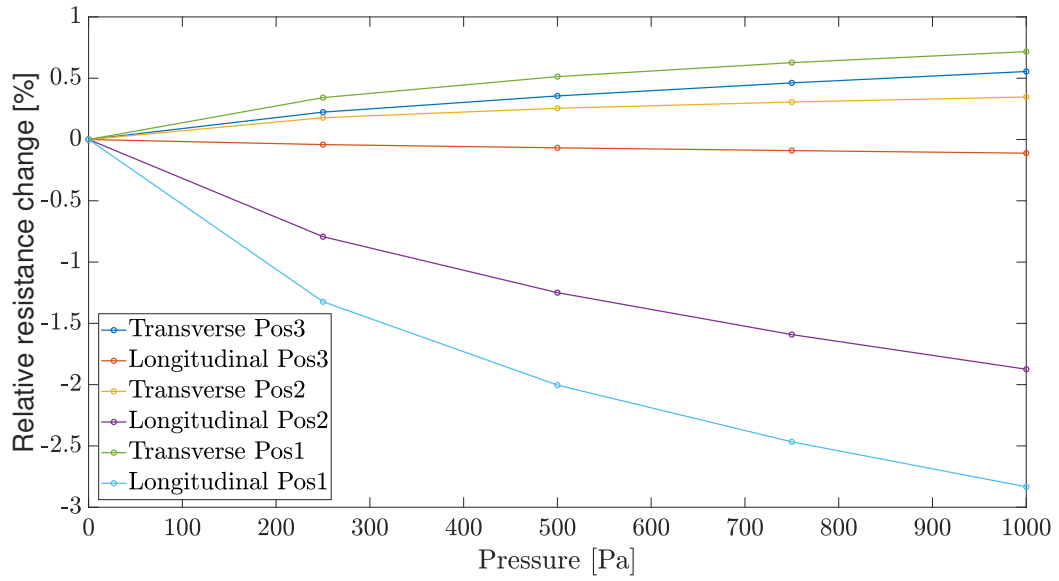


Figure 3.50: Transverse and longitudinal relative resistance variation with a 1000nm piezoresistive layer.

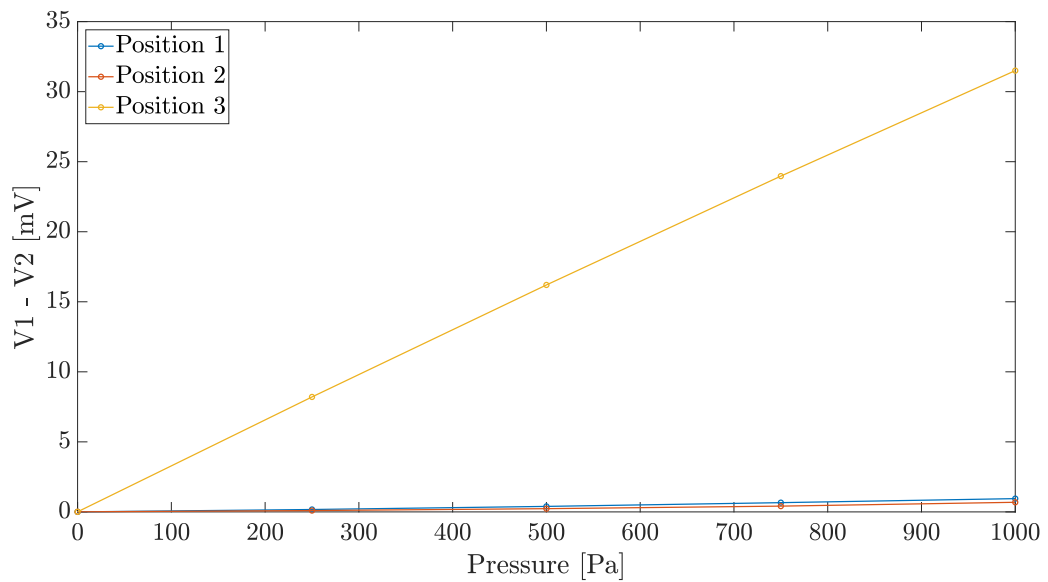


Figure 3.51:  $V1 - V2$  for a 100nm thick piezoresistive layer.

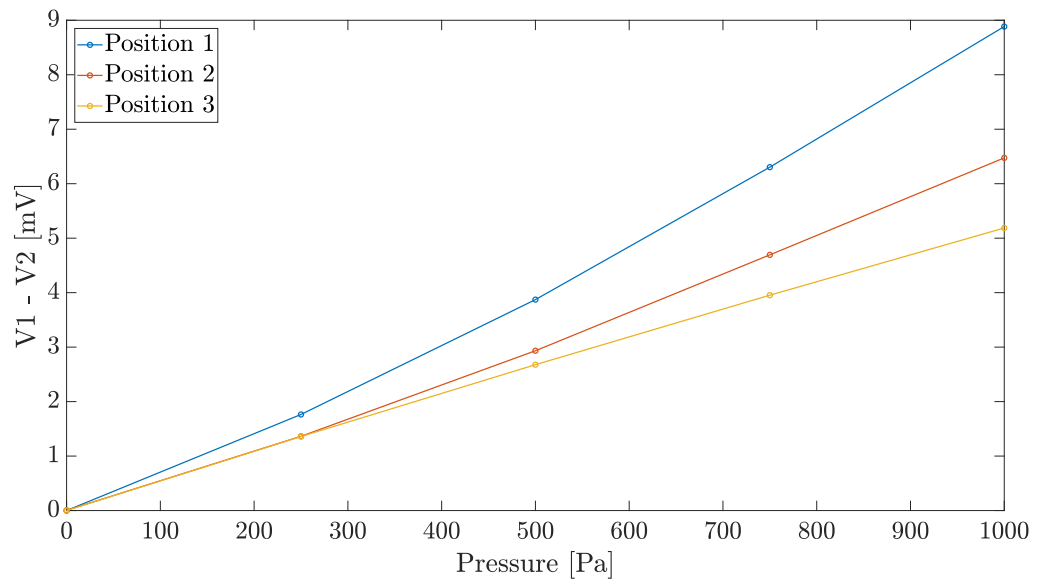


Figure 3.52:  $V1 - V2$  for a 1000nm thick piezoresistive layer.

This section articulates all the work that has been performed in *Winfab* throughout this research. Indeed, one of the main objectives of this thesis was to **manufacture the pressure sensor** of interest from scratch. The description of the fabrication process elaborated to reach this goal is described, along with the different fabrication techniques.

However, it was not possible to go through the entire process as planned due to mechanical cracking of the different layers deposited. These issues are explained and illustrated, and eventually some solutions are proposed for future work.

## 4.1 Process description

The aim of this section is to present a summarized version of the process flow followed to fabricate the pressure sensors.

### 4.1.1 Overall process flow

The process sheet summarizing the different fabrication steps can be found in Table 4.1. Measurements that have been performed to calibrate the process are indicated in italic, and the diverse reasons that lead to the choice of these steps will be detailed later on.

The wafers used throughout this process were standard and double-polished 7 inches silicon wafers available in *Winfab*. The average height of the silicon layer on each wafer is  $380\mu m$ .

The process consists in **4 main stages**: the fabrication of the part that has been called the *base stack*, the deposition and patterning of the *piezoresistive layer*, the deposition of the *contacts* and finally the fabrication and release of the *membranes*. Unfortunately, it was not possible to achieve all the steps and reach the final device. Indeed, an important step of the process consists in the annealing of the piezoresistive layer in order to make its structure crystalline and improve its piezoresistive behavior, but mechanical issues with one of the layers of the base stack made it impossible to provide a functional stack to proceed with the etching of the membranes. The process has thus been stopped before the second photolithography scheduled for depositing the contacts. This challenge will be further detailed in the following sections, and possible solutions proposed at the end of the chapter.

Note that the 7th step is not part of the final process but has been performed to calibrate the nitride layer before annealing with the piezoresistive layer on top.

|                      | #   | Step description                                | Thickness   | Time   | Equipment       |
|----------------------|-----|-------------------------------------------------|-------------|--------|-----------------|
| Base stack           | M1  | Thickness measurement                           | 380 $\mu m$ | -      | Palmer          |
|                      | M2  | Curvature measurement                           | -           | -      | Dektak          |
|                      | 1   | Standard cleaning                               | -           | 1h     | Wet bench       |
|                      | 2   | <b>Wet oxidation of <math>SiO_2</math></b>      | 400 nm      | 3h30   | Koyo furnace    |
|                      | M3  | Ellipsometry                                    | -           | -      | Ellipsometer    |
|                      | M4  | Curvature measurement                           | -           | -      | Dektak          |
|                      | 3   | Protective blue resist coating                  | -           | 90"    | Wet bench       |
|                      | 4   | Backside $SiO_2$ stripping                      | -           | -      |                 |
|                      | 5   | Protective resist stripping                     | -           | -      |                 |
|                      | 6   | <b>PECVD of <math>Si_3N_4</math></b>            | 600 nm      | 25-30' | Oxford          |
|                      | M5  | Ellipsometry                                    | -           | -      | Ellipsometer    |
|                      | M6  | Curvature measurement                           | -           | -      | Dektak          |
|                      | 7   | Thermal treatment                               | -           | -      |                 |
|                      | M7  | Curvature measurement                           | -           | -      | Dektak          |
| Piezoresistive layer | 8   | <b>Reactive sputtering of <math>VO_2</math></b> | 100 nm      | 10'30" | AJA             |
|                      | M8  | Average step height                             | -           | -      | Dektak (Kapton) |
|                      | M9  | Curvature measurement                           | -           | -      | Dektak          |
|                      | 9   | <b>Annealing</b>                                | -           | 30'    | HBn furnace     |
|                      | M10 | Visual inspection                               | -           | -      | Microscope      |
|                      | M11 | Curvature measurement                           | -           | -      | Dektak          |
|                      | 10  | Frontside resist spin coating                   | 1 $\mu m$   | -      | SUSS            |
|                      | 11  | <b>Photolithography</b>                         | -           | -      | MA6             |
|                      | M12 | Visual inspection                               | -           | -      | Microscope      |
|                      | 12  | <b>RIE</b>                                      | 110 nm      | 20"    | Oxford          |
|                      | M13 | Visual inspection                               | -           | -      | Microscope      |
|                      | M14 | Average step height                             | -           | -      | Dektak          |
|                      | M15 | Ellipsometry                                    | -           | -      | Ellipsometer    |
|                      | 13  | PR removal                                      | -           | -      |                 |
| Contacts             | 14  | <b>Photolithography</b>                         | -           | -      | MA6             |
|                      | M16 | Visual inspection - alignment                   | -           | -      | Microscope      |
|                      | 15  | <b>Metallisation</b>                            | 105 nm      | -      | Vacotec         |
|                      | 16  | Lift-off                                        | -           | -      |                 |
|                      | M17 | Visual inspection                               | -           | -      | Microscope      |
|                      | M18 | Ellipsometry                                    | -           | -      | Ellipsometer    |
|                      | M19 | Average step height                             | -           | -      | Dektak          |
|                      | 17  | Frontside resist coating                        | 1 $\mu m$   | -      | SUSS            |

Table 4.1: Process sheet.



The main steps of the process flow are illustrated in Figures 4.1 and 4.2. The cross sectional views are not scaled, and the top views only aim to show how the top layer has been patterned on *one* specific design. The dotted square represents the moving diaphragm while the dashed line identifies the cross sectional view. The other designs on the photomask will be presented in Section 4.1.2.

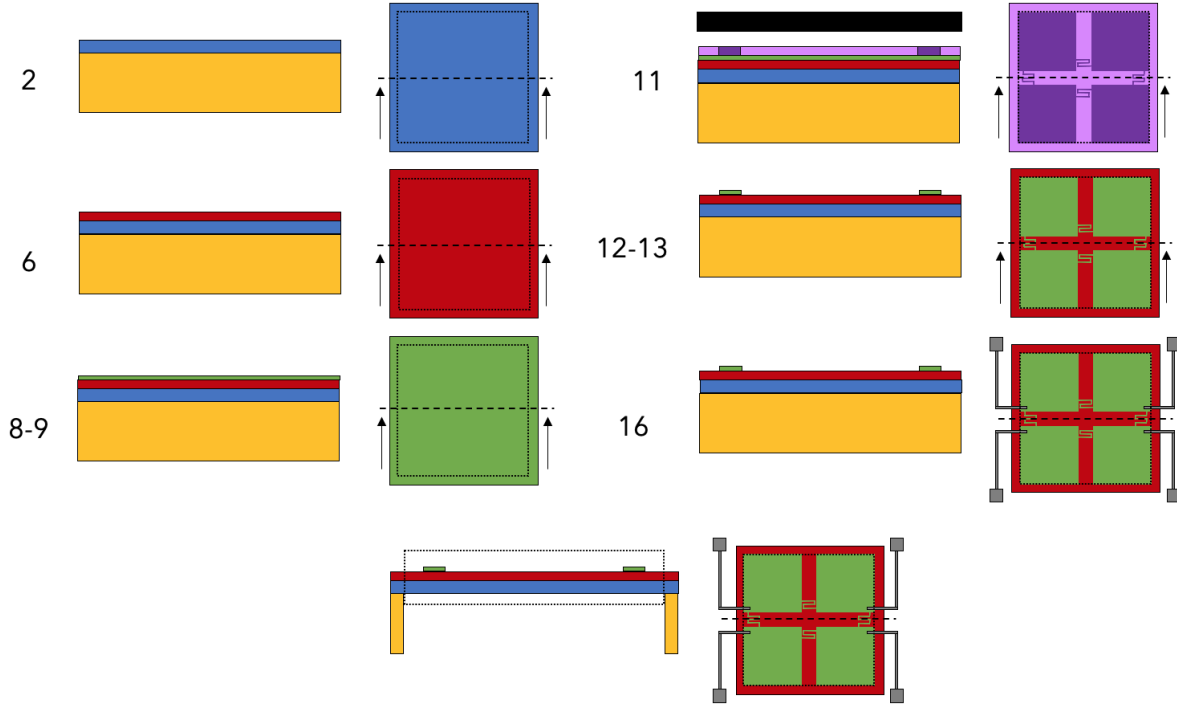


Figure 4.1: Cross sectional and top views of the key steps of the fabrication process.

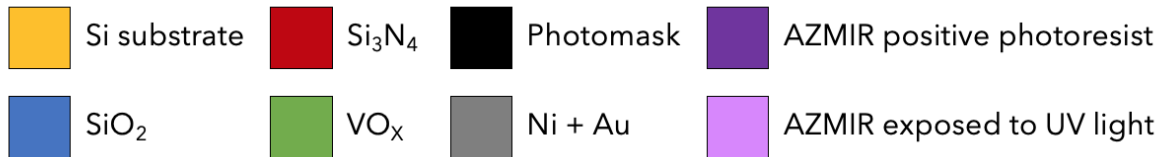


Figure 4.2: Legend of the process flow from Figure 4.1.

The last cross and top views without numbering represent the etching of the membranes under the diaphragms. The associated steps are not detailed on the *process sheet* as they were not realised due to the aforementioned issues, but will be briefly discussed in Section 4.2.5.

### 4.1.2 Mask design

Different masks have been used or were supposed to be used throughout the process. On one hand, three *photomasks* were needed: one for patterning the piezoresistive layer, one for depositing the electrical contacts around the membrane, and one for etching the

membranes on the backside of the wafers. On the other hand, *shadow masks* for depositing the contacts on wafers that are used to characterise the material in Section 5.

The design of the photomasks comes from another project related to pressure sensors and conducted in *Winfab*, which is quite similar to this of the present work: membranes of different sizes and orientations are studied, as well as top layers with different sizes and shapes of resistors. Only the mask used to pattern the vanadium oxide top layer has been modified according to the design proposed by Meng et al. for the resistors [54], and these modifications have been performed using *KLayout*. The entire mask consists of multiple blocks of 9 designs with 3 different sizes each time, and the positions and shapes of the resistors vary from one batch to another. Original designs only have the resistors made of the piezoresistive material, while modified designs for this thesis are almost entirely covered by this layer. Examples of the designs on the masks are shown in Figures 4.3 and 4.4. The red designs correspond to the mask for patterning the piezoresistive layer, while purple designs correspond to the mask for etching the membranes. The mask to deposit the contacts around the membranes is not shown.

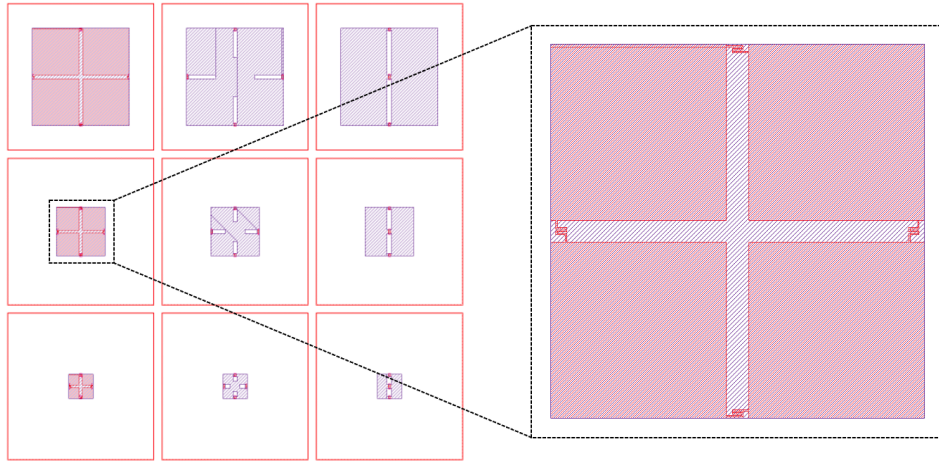


Figure 4.3: Batch of 9 designs from the photomasks on the left. Zoom of a modified design with S-shaped piezoresistors on the right.

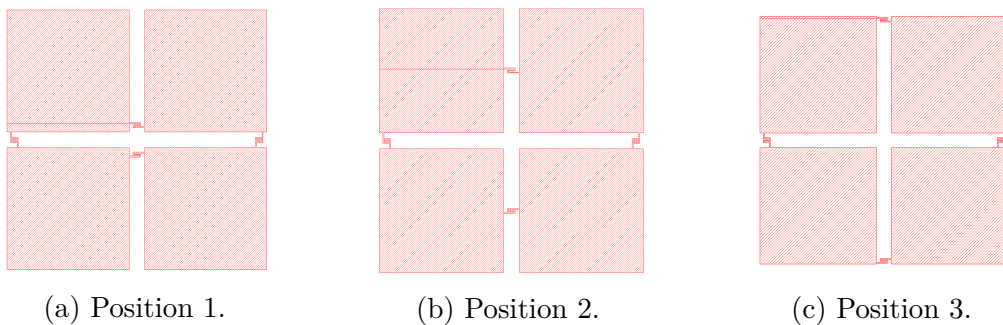


Figure 4.4: Examples of the different positions of interest for the piezoresistors. Only the mask for patterning the piezoresistive layer is represented.

The shadow masks used to deposit contacts to perform the two-point measurements under bending presented in Chapter 5 were fabricated with a laser and an example of design is shown in Figure 4.5. Unlike photomasks, shadow masks are not made to be exposed to UV lights and their maximum resolution is much lower. Using a shadow mask can be compared to lift-off but by removing the mask itself manually after the deposition of a metal for example, and not with any chemical agent. Any deposition technique can be performed over a shadow mask, but the quality of the deposition is mainly determined by the distance between the mask and the wafer: the smaller the gap, the better the deposition.

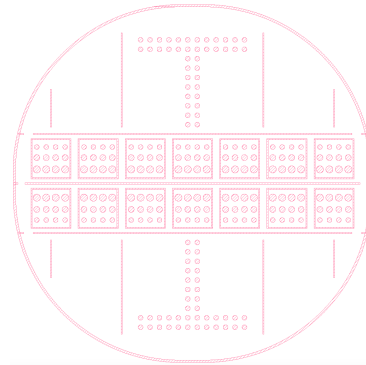


Figure 4.5: Shadow mask designed to deposit contacts on standard wafers.

## 4.2 Fabrication steps

### 4.2.1 Base stack fabrication

**Objective** The first stage of the process was to fabricate the **base stack** on which the transducing layer will be deposited and patterned. This stack is composed of bulk silicon on which silicon dioxide and silicon nitride is deposited. The thickness of the layers and their residual stress are measured at each step and adapted to obtain an overall stress that is *tensile* in the structure and below the thresholds for *elastic breaking*.

This condition is wanted to avoid a physical phenomenon called *buckling* that occurs under compressive stress [73][74]. Indeed, under uniform compression, when the stress experienced by the membrane becomes greater than a critical stress, the structure will deform in order to compensate the load. First order buckling is illustrated in Figure 4.6, along with its two stable states.

Both these states are a problem when performing pressure sensing as the initial deformation will bias the voltage output. If the membrane is not tensile and thus flat at rest, the observed displacement of the moving diaphragm is not representative of the applied pressure and the sensor **loses in sensitivity**. This is the reason why the tensile nitride layer should compensate the compressive stress of the thermally grown silicon dioxide layer.

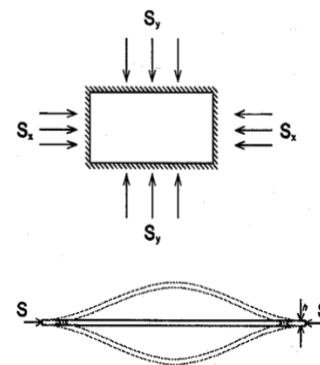


Figure 4.6: Buckled membrane and its two stable states [73].

**Standard cleaning** This first step is a standard cleaning of the wafers aiming to remove the native oxide and any other unwanted organic compounds on top of the silicon substrate.

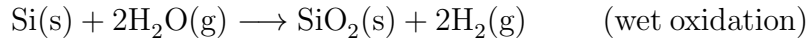
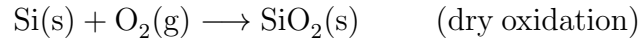
The process is standardized in order to provide repeatability.

The procedure is the following: the wafer is put in a *Piranha* bath, a mixture of  $H_2SO_4$  and  $H_2O_2$ , at  $100^\circ C$  for 10 minutes, and then it is rinsed a first time for 10 minutes in demineralised water before repeating these two steps. After the second rinsing, the wafer is put in a bath of a solution of HF 2% for 15 seconds. Once that is done, an hydrophobic check is performed to ensure that all the oxide has been removed. Finally, the wafer is rinsed a last time with  $N_2$  for 5 minutes before drying.

**Wet thermal oxidation** The second step consists in growing a layer of silicon dioxide with a targeted thickness of **400nm** in the Koyo furnace. This layer is expected to be under a *compressive* stress of about **250 to 350 MPa** according to the literature [75],[64].

The deposition was performed at  $1000^\circ C$  for 1 hour and 30 minutes, with an overall duration in the oven of 3 hours and a half.

Wet oxidation has been chosen over dry oxidation as it is faster to grow a relatively thick layer. Indeed, the reactions occurring during dry and wet oxidation are the following:



In which hydroxide diffuses faster than dioxide at high temperatures.

**PECVD** The third step is a *Plasma Enhanced Chemical Vapor Deposition* (PECVD) of a silicon nitride layer with a desired thickness of **600nm**, performed in the Oxford chamber. The *tensile* stress in the layer is expected to be around **300MPa** to compensate the stress of the previous layer [3].

The deposition parameters can be found in Table 4.2.

| $SiH_4$ [sccm] | $NH_3$ [sccm] | $N_2$ [sccm] | $N_2O$ [sccm] | T [ $^\circ C$ ] | P [Torr] | Power [W] |
|----------------|---------------|--------------|---------------|------------------|----------|-----------|
| 600            | 21            | 700          | 0             | 300              | 1        | 30        |

Table 4.2: Deposition parameters for the layer of  $Si_3N_4$ .

Note that the film has been deposited using PECVD for technical issues with the LPCVD chamber, but LPCVD would have been preferred for the film quality [76]. The poorly deposited nitride will, in fact, be the major source of problem encountered throughout this work. Issues related to the quality of the film will be discussed in the following paragraph.

### 4.2.2 Piezoresistive layer deposition and patterning

**Objective** The second stage of the process aims to deposit a piezoresistive layer that will be patterned to form a membrane with an arrangement of resistors that is innovative compared to what is usually done in the literature. Several piezoresistive layers have been considered for the final device: p-type Si, Indium Gallium Zinc Oxide (IGZO) and vanadium oxides ( $VO_X$ ). Indeed, as highlighted by Inomata et al., vanadium oxides thin films exhibit a piezoresistive character that is even higher than this of silicon [28]. This behaviour is not extensively studied in the literature yet, but promising values have been proposed and further experiments on the topic are of interest in the scope of this work. The piezoresistive response of vanadium oxides will be discussed in Chapter 5.

**Reactive sputtering** The final layer selected to build the sensor is made of a **vanadium oxide**, whose oxidation state is determined by the parameters of the deposition performed in *Winfab* in the AJA chamber through reactive sputtering. This technique belongs to physical vapor deposition (PVD) techniques and consists in sputtering with oxygen intentionally added into the atmosphere, allowing to deposit oxide thin films [77]. In this case, a DC power supply has been used and several oxygen concentrations have been tested to obtain the desired vanadium oxide. Finally, a concentration of 12.5% of oxygen has been selected to deposit **vanadium dioxide** ( $VO_2$ ) and the remaining parameters of the deposition can be found in Table 4.3.

| $O_2$ [sccm] | Ar [sccm] | DC power [W] | P [mTorr] | T [°C] | Time   | Thickness [nm] |
|--------------|-----------|--------------|-----------|--------|--------|----------------|
| 6.25         | 43.75     | 200          | 5         | 21     | 10'30" | 100            |

Table 4.3: Deposition parameters for  $VO_2$ .

Once the deposition is over, it is common to measure the **thickness** of the new layer by ellipsometry. But unfortunately, the ellipsometer available in *Winfab* does not provide an optical model for  $VO_2$ . A trick had to be used in order to measure the thickness of the layer: before the deposition, a thin stripe of Kapton was applied on the edge of the wafer, protecting the previous layer against the metal. Once the deposition was over, the Kapton was removed and a Dektak measurement performed to extract the thickness of the step between the previous layer, nitride in this case, and the layer of  $VO_2$ . Results of this measurement show an average height of 96nm, which is close to the expected 100nm. Another method consists in a virtual measure of the thickness of the layer using a SEM cross section. This technique is more accurate than the previous one but requires to cut the wafer in small pieces to be put on the microscope.

**Thermal treatment** As reactively sputtered films are often non-stoichiometric and amorphous when deposited [77], an **annealing** step is needed to improve the film quality and obtain a crystalline version of vanadium dioxide. Multiple tests have been performed in *Winfab* at 300, 400, 500 and 600°C respectively for a duration of 60 minutes in a tubular furnace with controlled atmosphere and pressure. Note that the oven is hermetic to oxygen to avoid further oxidation of the vanadium oxide once deposited. Raman spectroscopies

of the samples demonstrated that 300°C was not sufficient to obtain a poly-crystalline structure. These results will be further analysed in Chapter 5.

Annealing parameters have thus been investigated with the objective to reach **poly-crystalline**  $VO_2$  while preserving the integrity of the nitride layer deposited right under. But experiments on several wafers presented in Table 4.4 have shown that the nitride layer does not support some conditions of thermal treatments and cracks under **excessive stress**<sup>1</sup>.

| Wafer |    | Top layer | T<br>[°C] | Time<br>[min] | Furnace       | Ramp | Result                                           |
|-------|----|-----------|-----------|---------------|---------------|------|--------------------------------------------------|
| A     |    | $Si_3N_4$ | 500       | 60            | Hydrox        | No   | Broken                                           |
| B     | B1 | $Si_3N_4$ | 400       | 60            | SiC           | Yes  | Micro cracks                                     |
|       | B2 | $Si_3N_4$ | 400       | 30            | SiC           | Yes  | Delamination                                     |
| C     |    | $Si_3N_4$ | 400       | 30            | Hermetic oven | Yes  | Micro cracks where nitride had defects           |
|       |    | $VO_2$    | 400       | 30            | Hermetic oven | Yes  | Cracks and delimitation where Kapton was applied |
| D     |    | $VO_2$    | 400       | 30            | Hermetic oven |      | Not annealed                                     |

Table 4.4: Annealing parameters.

Indeed, it was visible by naked eye that the nitride layer of wafer *A* was broken when removed from the oven. Other conditions were then tested on wafer *B* which had been cut in four equal pieces with a diamond tip. For these tests, a ramp to reach the annealing temperature was applied once the wafer in the furnace to **avoid a thermal shock** that could be a source of cracks. The piece annealed for 1 hour showed micro cracks all over its surface under the microscope, whereas the piece annealed for 30 minutes showed cracks only on its edges with delamination of the nitride where the layer appeared to be not uniformly deposited. Pictures of these wafers are gathered in Figures 4.7 to 4.9.

As annealing a fourth of a wafer is not the same as annealing an entire one, the assumption that cutting the wafer could weaken its structure under high temperatures was made and the two remaining pieces were not annealed. Nevertheless, wafer *C* annealed without being cut showed micro cracks as well under the microscope, but only at locations where the nitride had defects and on its edges as shown in Figure 4.10.

Another thermal treatment has then been performed in the hermetic furnace over wafer *C* on which amorphous  $VO_2$  had been deposited, with a stripe of Kapton tape at its base for height measurement. It resulted in micro cracks and delamination on the top layer where the Kapton had been stripped out, as illustrated in Figures 4.11 and 4.12.

<sup>1</sup>Note that to be put in the hermetic furnace, the wafer must still be covered by silicon dioxide all over in order to avoid any release of pure silicon which is a contaminant for the oven, which is the reason why the first tests have been conducted in different machines.



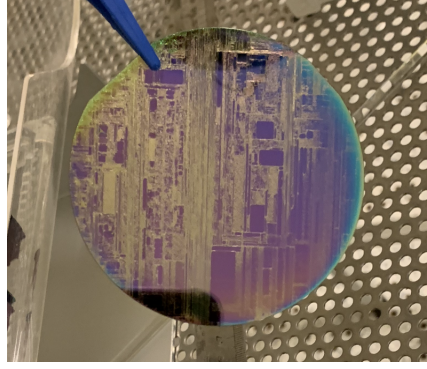
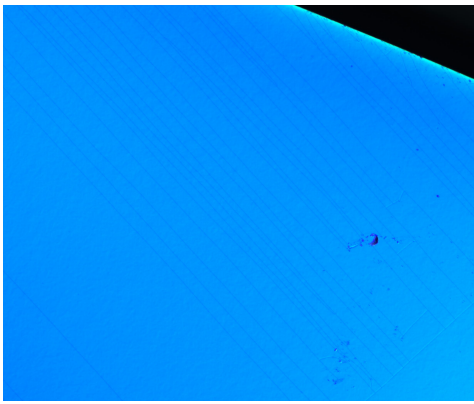


Figure 4.7: Visual inspection of wafer *A* after annealing under  $N_2$  in the Hydrox furnace at  $500^\circ\text{C}$  for 60 minutes.



(a) Cracks on the edges of the wafer.

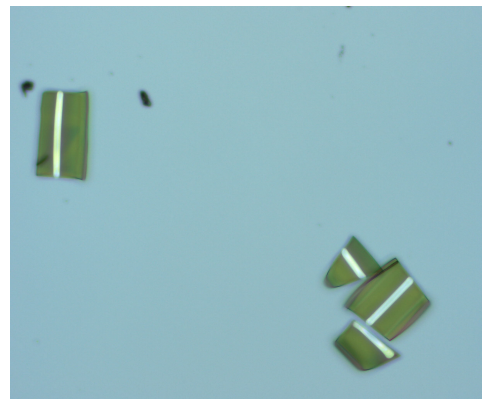


(b) Cracks in the middle of the wafer, following crystalline directions.

Figure 4.8: Visual inspection of wafer *B1* after annealing under  $N_2$  in the SiC furnace at  $400^\circ\text{C}$  for 60 minutes.



(a) No appearing cracks on the edges of the wafer.



(b) Delamination in the middle of the wafer.

Figure 4.9: Visual inspection of wafer *B2* after annealing under  $N_2$  in the SiC furnace at  $400^\circ\text{C}$  for 30 minutes.

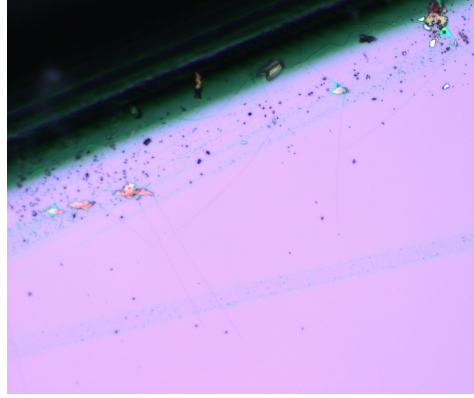
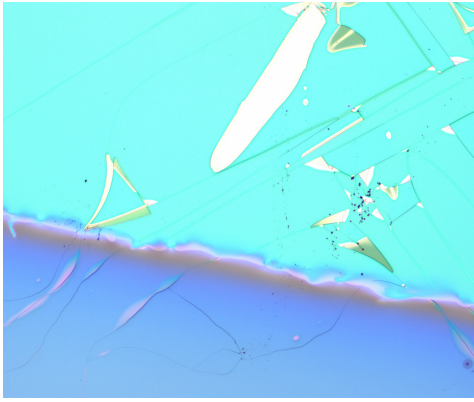
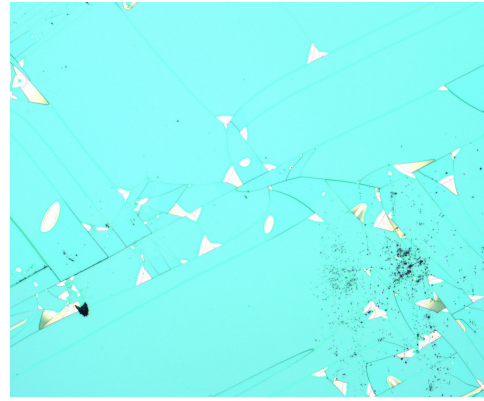


Figure 4.10: Visual inspection of wafer  $C$  with  $Si_3N_4$  after the *first* annealing under  $Ar$  in the hermetic furnace at  $400^\circ C$  for 30 minutes.

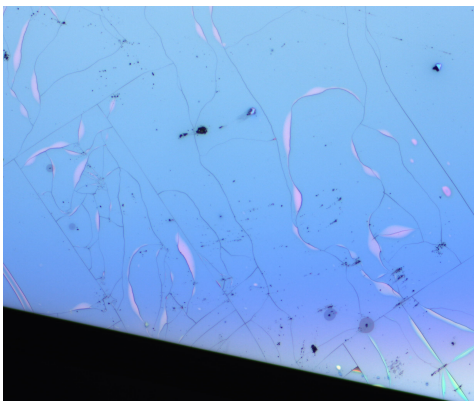


(a) Cracks at the step between the  $Si_3N_4$  and  $VO_2$  layers.



(b) Cracks on the  $Si_3N_4$  layer.

Figure 4.11: Visual inspection of wafer  $C$  with  $Si_3N_4$  and  $VO_2$  where the Kapton has been removed, after the *second* annealing under  $Ar$  in the hermetic furnace at  $400^\circ C$  for 30 minutes.



(a) Cracks on the edges of the wafer.



(b) Cracks on the middle of the wafer.

Figure 4.12: Visual inspection of the weakened  $VO_2$  layer on  $C$  after the *second* annealing under  $Ar$  in the hermetic furnace at  $400^\circ C$  for 30 minutes.



As wafer *C* had been heated two times and as removing the Kapton before annealing was certainly a source of pre-cracks on the crystalline structure, other tests have been performed and will be analysed in Section 4.2.2 to further investigate the trade-off between the annealing conditions required to obtain poly-crystalline  $VO_2$  and the cracking of the nitride layer under excessive stress.

**Photolithography** Photolithography is a **patterning technique** for which a photomask as presented in Section 4.1.2 is used to expose certain parts of a photosensitive film to UV lasers. The light exposure is achieved by the patterns that are drawn on the photomask to block light at certain areas.

Two photolithography steps are required on the *front side* of the wafer during the full process; a first one to pattern the piezoresistive layer, and a second one to deposit the contacts around the membranes. These steps require **alignment** between the different masks, which represents a critical step in the fabrication process as misalignment will give rise to undesired contacts. Another photolithography is then required on the *back side* of the wafer to fabricate the membranes. The process flow of the photolithography is not detailed in the process sheet but is quickly reviewed hereafter.

First, a positive photoresist (i.e. a resist whose parts exposed to light will become removable) is spin coated on the top layer for 90 seconds at  $90^\circ\text{C}$ . Adhesion promotion of the resist is then performed for 28 minutes in the LPIII oven before being exposed to light. Exposure is done with hard contact between the mask and the wafer using a common recipe of the Süss MA6 mask aligner with a power of  $180\text{mJ}/\text{cm}^2$ . Follows a post exposure bake of 90 seconds at  $100^\circ\text{C}$  to amplify the photoaction. Once the key sports of the resist have been exposed, development is performed to remove the soluble parts. A hard bake is then achieved to solidify the rest of the resin for 120 seconds at  $130^\circ\text{C}$ . The thin film can finally be etched, being partially protected by the resist, which will be in turn etched away at the end of the step.

The quality of the photolithography is assessed by inspecting the **quality marks** under the microscope, as well as the desired patterns. Pictures of the top layer covered with resist are shown in Figures 4.13 and 4.14, where we can see that the lithography is of good quality.

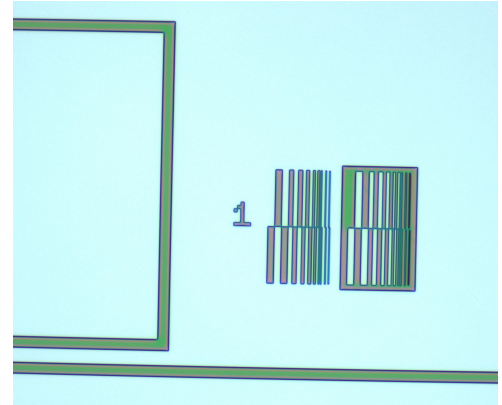


Figure 4.13: Quality marks showing the precision of the lithography.

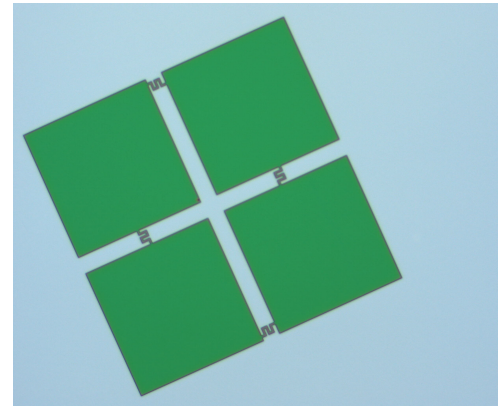


Figure 4.14: Accuracy of one of the patterns designed on the photomask.

**RIE** *Reactive Ion Etching* (RIE) is a plasma etching technique used to **pattern the  $VO_2$  layer**. A recipe proposed in the literature to etch  $VO_X$  deposited by reactive sputtering with a good **selectivity** with regards to  $Si_3N_4$  (5:1) and  $SiO_2$  (10:1) substrates involves a  $Cl_2/N_2$  plasma [79]. For practical reasons related to the use of the machines in *Winfab*, this recipe has not been tested in the scope of this work.

However, the parameters of a **Bosch process** have been studied and modified to etch the top layer in the Oxford chamber. These parameters can be found in Table 4.5. The Bosch process is a well known technique in which alternative steps of passivation film deposition and etching are repeated in a cyclic way as illustrated in Figure 4.15.

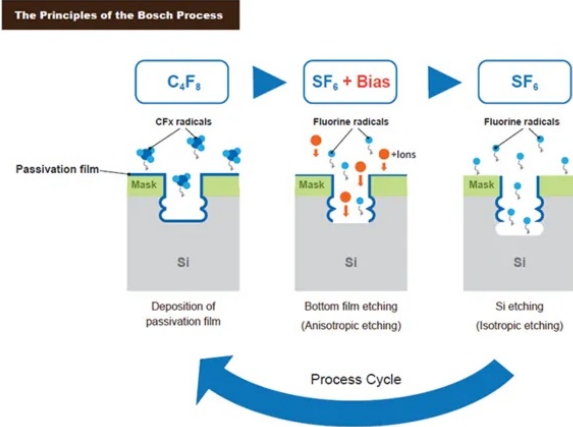


Figure 4.15: Bosch process steps [78].

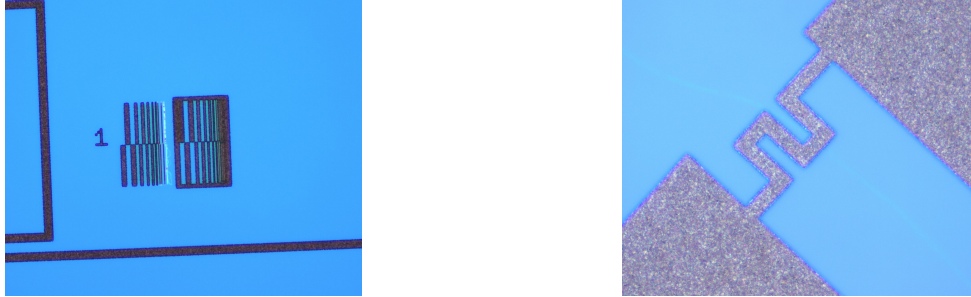
The wafer used for these experiments, wafer *C*, has been cut in small pieces to test the different parameters on a few membranes only. Etching recipes on wafers *C1* to *C4* all provoke **over-etching** of the layers under the  $VO_2$ , as measured by ellipsometry over the  $Si_3N_4/SiO_2$  layers.

The final recipe consists of 100 sccm of  $SF_6$ , 40W of RF power and no ICP during a single cycle of 20 seconds. As  $SF_6$  does not exhibit any selectivity between  $VO_2$  and  $Si_3N_4$ , the parameters were tuned to stop at about 10nm in the nitride layer to make sure that all the  $VO_2$  was etched. Visual checks have been performed under the microscope to confirm the quality of the patterning, and the results are shown in Figures 4.18.

| Wafer | C4F8 [sccm] | SF6 [sccm] | RF power [W] | ICP | Cycles | Time [s] | Results                                                                                 |
|-------|-------------|------------|--------------|-----|--------|----------|-----------------------------------------------------------------------------------------|
| C1    | 140         | 120        | 100          | Yes | 10     | 120      | VO2 fully etched<br>Si3N4 fully etched<br>SiO2 85nm etched                              |
| C2    | 0           | 100        | 100          | No  | 1      | 60       | VO2 fully etched<br>Si3N4 225nm etched                                                  |
| C3    | "           | "          | "            | "   | "      | 20       | VO2 fully etched<br>Si3N4 52nm etched                                                   |
| C4    | "           | "          | 50           | "   | "      | "        | VO2 fully etched<br>Si3N4 15nm etched                                                   |
| C5    | "           | "          | 40           | "   | "      | "        | VO2 fully etched<br>Si3N4 10nm etched                                                   |
| C6    | "           | "          | "            | "   | "      | "        | <i>With resist:</i><br>Si3N4 12nm etched<br><i>Without resist:</i><br>Si3N4 10nm etched |

Table 4.5: Parameters of the tuned Bosch process for  $VO_2$  etching.

Note that without the use of  $C_4F_8$  in the process, the etching becomes isotropic and also spreads under the material protected by the resist. This might be a source of modification of the true stress felt by the piezoresistors in the final design, which could result in a reduced sensitivity of the sensor.



(a) Quality marks showing a loss in resolution with etching. (b) Etching around one of the piezoresistors, with over-etching until the  $SiO_2$  layer.

Figure 4.16: Visual inspection of the etching of the  $VO_2$  layer on wafer  $C1$ , with resist on the wafer.



(a) Example of a piezoresistive pattern.

(b) Resolution of the piezoresistor.

Figure 4.17: Visual inspection of the etching of the  $VO_2$  layer on wafer  $C3$ , with resist on the wafer.

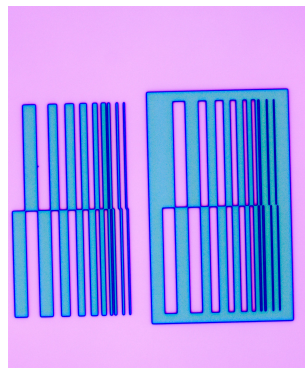


Figure 4.18: Visual inspection of the etching of the  $VO_2$  layer on wafer  $C6$ , without resist.

### 4.2.3 Testing with calibrated parameters

Once the annealing and etching parameters have been determined, new tests were performed on wafers *E*, *F* and *G* to validate the procedure. These wafers are **double-polished wafers**, and the *base stack* has been fabricated following the previously presented process sheet. The thickness of each layer measured by ellipsometry can be found in Table 4.6.

| Wafer | $h_{Si} [\mu m]$ | $h_{SiO_2} [nm]$ | $h_{Si_3N_4} [nm]$ | $h_{VO_2} [nm]$ |
|-------|------------------|------------------|--------------------|-----------------|
| E     | 378              | 445              | 711                | $\sim 100$      |
| F     | 378              | 440              | 567                | $\sim 100$      |
| G     | 378              | 438              | 564                | $\sim 100$      |

Table 4.6: Thickness of the different layers deposited on double-polished wafers.

It can be noticed that the thickness of the different layers does not perfectly match those originally planned, i.e. 400nm of  $SiO_2$  and 600nm of  $Si_3N_4$ . The first variation can be explained by the fact that the **resistivity** of doped double-polished wafers is not the same as the one of standard wafers, and that the Koyo furnace is calibrated to grow oxide on standard wafers. Furthermore, the **crystalline direction** of the Si substrate plays a major role in the oxidation rate: if the crystallographic planes are not in the [110] orientation, then the speed can be increased for both dry and wet oxidation, leading to a slightly thicker layer of silicon dioxide [80] [81].

Regarding the second variation for the nitride layer, the **deposition timing** has been calculated based on previous depositions performed on wafers *C* and *D* to run a first deposition on wafer *E*. But once again, as the wafers of this batch are not standard, different results were observed. The timing has then been reduced for wafers *F* and *G* that were treated together in the Oxford chamber, but the desired thickness of 600nm was still not reached.

A single **annealing** under *Ar* in the hermetic furnace was then performed on the batch for 30 minutes at 400°C with a ramp of 30 minutes to heat the samples in the furnace. This resulted in **significant cracks** in the different layers as well as **delamination** of the top layer. Figure 4.19 shows the gradient of deposition of the layers from the  $Si_3N_4$  on the left to the  $VO_2$  on the right, and it is clear that both layer have been damaged. Figure 4.20 shows more cracks in the middle of the  $VO_2$  layer, whereas Figure 4.21 shows the delamination of this same layer above the silicon nitride. Finally, Figure 4.22 explicitly shows the breaking of the layers where the edges of the AJA holder formed the deposition gradient between the two materials.

It can be concluded that the base stack did not support the **excessive residual stress** provoked by the thermal treatment, and a stress analysis will be performed in the following section to tackle this issue and propose alternative solutions at the end of this chapter.



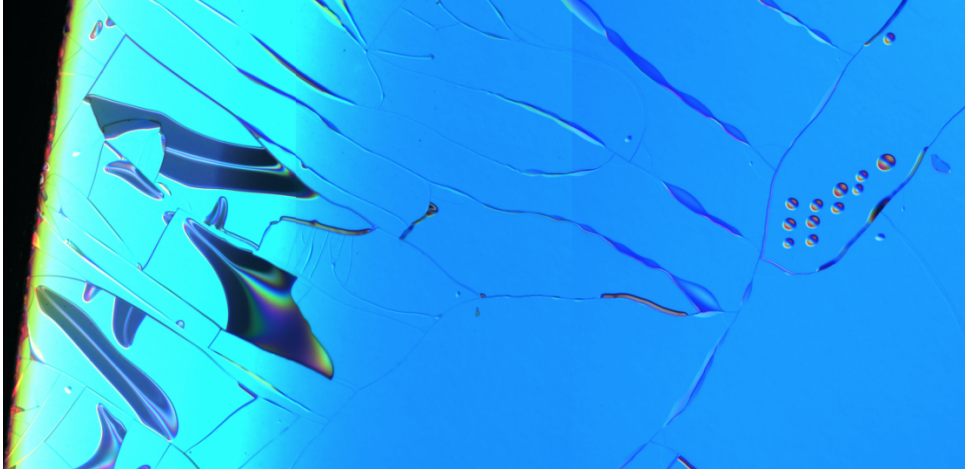
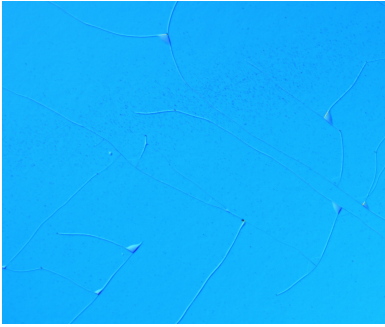
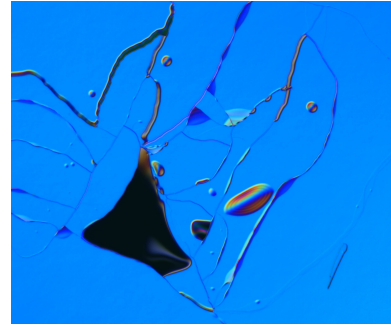


Figure 4.19: Deposition gradient on wafer  $F$  after annealing in the hermetic furnace for 30 minutes at  $400^{\circ}\text{C}$ .

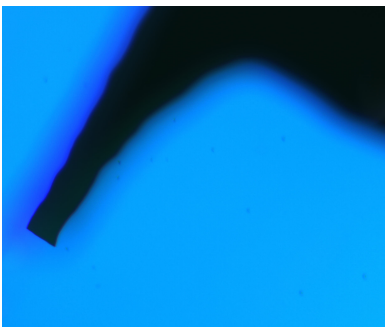


(a) Cracks in the middle of the layer.

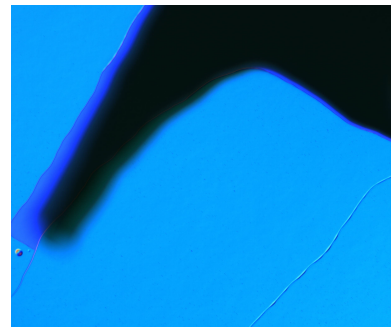


(b) Cracks and delamination in the middle of the layer.

Figure 4.20: Visual inspection of wafer  $6$  after annealing in the hermetic furnace for 30 minutes at  $400^{\circ}\text{C}$ .



(a) Delamination with focus of the  $\text{VO}_2$  film.



(b) Delamination with focus on the  $\text{Si}_3\text{N}_4$  film exposed.

Figure 4.21: Visual inspection of the delamination on wafer  $6$  after annealing in the hermetic furnace for 30 minutes at  $400^{\circ}\text{C}$ .

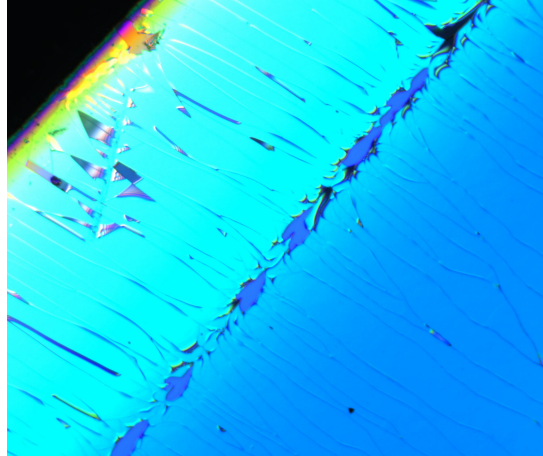


Figure 4.22: Cracks at the edges of wafer  $F$  where the wafer was supported by the AJA holder.

#### 4.2.4 Contacts deposition

**Objective** Once the piezoresistive layer has been deposited and patterned, adding **electrical contacts** is required to measure the response of the Wheatstone bridge formed by the piezoresistors as explained in Section 3.2.10.

**Metallisation** Pads made of gold with a thin layer of nickel for adhesion have been chosen for contacting the thin films of vanadium oxide. The electrodes should be fabricated after photolithography and alignment with the previous patterns to avoid poor contacts with the resistors. The metals are deposited by electron beam (E-beam) evaporation in the Vacotec chamber, with thicknesses of 5nm and 200nm for the Ni and Au layers respectively. Once the evaporation is over, unwanted metal over the resist is removed by lift-off.

#### 4.2.5 Membrane etching

**Objective** Finally, once the front side of the wafer has been treated, membranes can be etched on the Si substrate. This step is very important as it will determine the quality and final thickness of the sensing diaphragm.

**DRIE** Previous works conducted in *Winfab* reported the fabrication of such membranes using *Deep Reactive Ion Etching* (DRIE) in the Oxford chamber. First, a back side photolithography is needed to prepare the substrate for etching, and alignment of the associated mask with the patterns on the front side is a critical to ensure the accuracy of the sensors. Then, DRIE is performed and the membranes are finally released. Parameters of these three steps are not summarized in this work as the process was stopped before, but more information can be accessed through the *Winfab* staff.

### 4.3 Stress analysis of the base stack layers

As it has been observed that the tested base stacks crack under thermal treatment, a stress analysis of the different layers has been conducted in this section.

#### 4.3.1 Stress control by stacking

When a material is deposited in a thin film over a substrate or another film using any fabrication technique, it undergoes **residual stress** in its structure. This stress mainly comes from lattice mismatch at the interface between the stacked materials, and it propagates inside the whole crystalline structures. The layers are then under stress constraints, even at rest. Examples of the magnitude that the residual stress can take depending on the material and the technique used can be found in Table 3.1. Positive and negative values correspond to *tensile* and *compressive* stress respectively, as illustrated in Figure 4.23 for thin films.

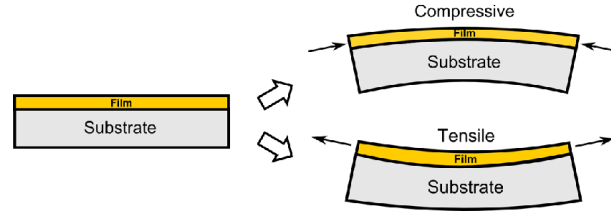


Figure 4.23: Residual stress *inside* thin films [82].

As different materials deposited on top of each other can be under opposite stress with different magnitudes, it is possible to **tune the overall residual stress** of the stack by adjusting the parameters of the deposited layers such as their thicknesses, deposition temperatures,...

The tuning can be quantified using **Stoney's formula**, along with measurements of the curvature of the wafer *before* and *after* the deposition of each film. The formula is given by Equation 4.1 and is used under the following hypothesis taken from [83]:

- the thicknesses of the substrate and the coating are smaller than the lateral dimensions;
- deformations and rotations are infinitesimal;
- the thickness of the coating is smaller than the thickness of the substrate;
- the substrate and the coating are homogeneous, isotropic and linear elastic;
- the radius of curvature is equal in all directions (spherical deformation);
- the stress and the radius of curvature are constant on the whole surface of the plate.

$$\sigma_f = \frac{E_s}{6(1 - \nu_s)} \frac{h_s^2}{h_f} \left( \frac{1}{R} - \frac{1}{R_0} \right) \quad (4.1)$$

where  $\sigma_f$  is the average residual stress in the film  $f$ ,  $E_s$  is the Young modulus of the substrate,  $\nu_s$  is the Poisson coefficient of the substrate,  $h_s$  is the thickness of the substrate,  $h_f$  is the total thickness of the deposited films, and  $R_0$  and  $R$  are the curvature radii of the wafer before and after manipulation respectively.

Note that the Stoney formula is frequently used even if all the hypothesis are not fully respected. Based on curvature measurements made with the Veco Dektak 150 profilometer, the curvature radii are calculated using Equation 4.2:

$$R = \frac{(\frac{L}{2})^2}{2h_{max}} \quad (4.2)$$

where  $L$  is the measurement length and  $h_{max}$  the highest curvature point.

Analysing the curvature graphs to extract the stress requires the use of some **conventions** to determine the signs of the radii in Stoney's equation. When the curvature is concave, with a maximum, then the sign of the radius is negative and associated with compressive stress. On the opposite, when the curvature is convex, with a minimum, then the sign is positive and associated with tensile stress. As long as the curvatures of the different layers are in the same direction, the signs should be the same in the formula.

Another version of Stoney's formula has been used as well to better approximate the residual stress when the measured curvatures were not entirely concave or convex. This alternative method requires the second derivative of a second order polynomial  $P$  used to fit the curves, as given by Equation 4.3:

$$\sigma_f = \frac{E_s}{6(1 - \nu_s)} \frac{h_s^2}{h_f} \frac{d^2 P}{dx^2} \quad (4.3)$$

### 4.3.2 Measurements and results

The thickness of the wafers before any treatment has been measured using a Palmer instrument. The thickness of the different layers has then been measured by ellipsometry at each step and are summarised in Tables 4.6 and 4.7.

The residual stress after deposition of each layer has been computed using *MATLAB*, both with the regular Stoney formula and with the polyfit, using a value of 183GPa for the biaxial elastic modulus of silicon given by  $E_s/(1 - \nu_s)$  [84]. Results are given in Table 4.8, and Figure 4.25 illustrates the curvatures measured for the different layers along with their polynomial fitting compared to the initial curvature of bulk silicon.

Multiple conclusions can be drawn from these computations. First, it is established that

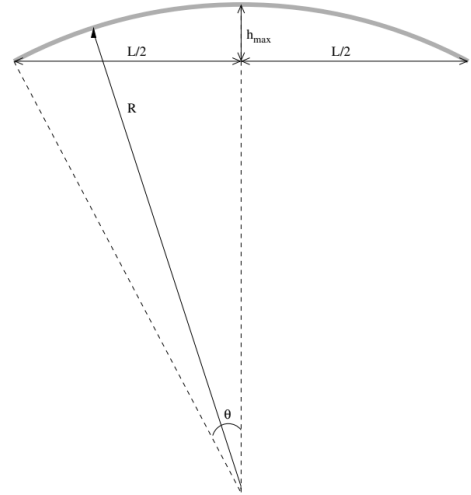


Figure 4.24: Evaluation of the radius curvature [64].

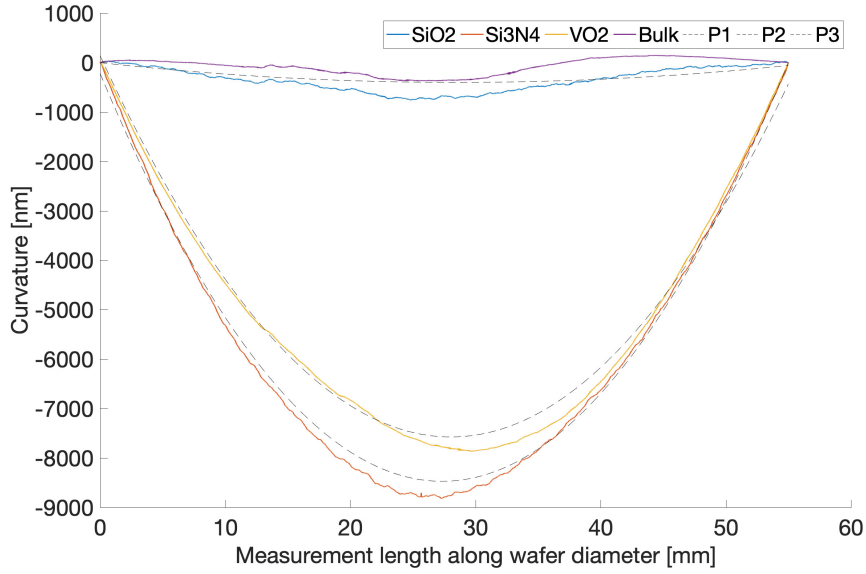


| Wafer | $h_{Si}$ [ $\mu m$ ] | $h_{SiO_2}$ [nm] | $h_{Si_3N_4}$ [nm] | $h_{VO_2}$ [nm] |
|-------|----------------------|------------------|--------------------|-----------------|
| A     | 383                  | 408              | 594                | Broken          |
| B     | 376                  | 408              | 628                | Cut             |
| C     | 382                  | 410              | 493                | $\sim 100$      |
| D     | 394                  | 410              | 497                | $\sim 100$      |

Table 4.7: Thickness of the different layers deposited on standard wafers.

| Wafer | $SiO_2$ [MPa] |         | $Si_3N_4$ [MPa] |         | $VO_2$ [MPa] |         | $VO_{2_{ann}}$ [MPa] |         |
|-------|---------------|---------|-----------------|---------|--------------|---------|----------------------|---------|
|       | Regular       | Polyfit | Regular         | Polyfit | Regular      | Polyfit | Regular              | Polyfit |
| A     | -308.22       | -301.11 | -44.98          | -36.53  | Broken       |         |                      |         |
| B     | -309.12       | -318.22 | -34.77          | -35.49  | /            | /       | /                    | /       |
| C     | -14.36        | 2.41    | 194.76          | 195.33  | 3136.8       | 3106    | 3772                 | 3736.7  |
| D     | 11.79         | 10.51   | 212.75          | 195.89  | 937.85       | 864.92  | /                    | /       |
| E     | -217.33       | -189.86 | 116.44          | 116.46  | 1071.4       | 1055    | Broken               |         |
| F     | 63.24         | 92.21   | 149.32          | 153.25  | 785.35       | 814.11  | Broken               |         |
| G     | -177.06       | -123.6  | 40.64           | 56.31   | 279.09       | 377.63  | Broken               |         |

Table 4.8: Residual stress in each layer computed using Stoney's formula, "/" entries meaning that the step has not been performed for diverse reasons.


 Figure 4.25: Dektak measurements of wafer  $D$  in solid lines, along with fitting polynomials in dashed lines.

vanadium dioxide thin films are significantly *tensile* when deposited by reactive sputtering. Indeed, an increase in the residual stress is observed each time between the  $Si_3N_4$  and  $VO_2$  layers, and standard wafer  $D$  which has not been annealed at all shows that the deposition of  $VO_2$  on the base stack with aforementioned dimensions is sufficient to reach tensile conditions.

Then, excessive stress and cracking due to annealing conditions can be quantified.

Wafers  $A$  and  $B$  could not be measured because of delamination, but respectively indicate that annealing for one hour at  $500^{\circ}\text{C}$  and at  $400^{\circ}\text{C}$  is too long and too hot. On the other hand, wafer  $C$  shows two major augmentations of stress: one related to the annealing of the already tensile nitride layer on which tensile  $\text{VO}_2$  is deposited, and a second one related to the annealing of this stack. As notable cracks were already visible after the first annealing and the resulting stress is estimated to be around  $3\text{GPa}$ , it can be concluded that annealing of the nitride layer before depositing the piezoresistive layer is highly not recommended.

However, even without an intermediate thermal treatment, analysis of double-polished wafers  $E$ ,  $F$  and  $G$  shows that the residual stress generated by the proposed configuration is too high to survive annealing at  $400^{\circ}\text{C}$  for 30 minutes.

Further stress analyses will be performed in Chapter 5 on wafers without the nitride layer between  $\text{SiO}_2$  and  $\text{VO}_2$ .

## 4.4 Results of the process and perspectives

Attempts to fabricate an innovative piezoresistive sensor with a patterned top layer have been made in *Winfab*, but could not be conducted until the final device.

The **limiting step** that stopped the process is the annealing of the piezoresistive film that resulted in the breaking of the stack to be patterned before etching of the membranes. This problem mainly occurred because of the poor quality of the silicon nitride film deposited by PECVD which cannot withstand excessive stress. This stress is generated by the tensile layer of vanadium oxide on one hand, and is amplified by the thermal treatment on the other hand.

The issue could not be solved simply by reducing or avoiding the annealing step as it is mandatory to obtain a poly-crystalline version of the vanadium oxide with enhanced piezoresistive properties. In addition, the smallest temperature and heating time used have been reported to be insufficient for a good crystallisation of the oxide, meaning that breaking is unavoidable if the proposed stack and parameters are maintained.

Nonetheless, several leads have been theoretically investigated to propose an **alternative solution** to the original plan elaborated to fabricate the sensor.

First, membranes without the  $\text{Si}_3\text{N}_4$  layer have been considered. The idea is to etch only part of the silicon substrate to have a membrane thick enough to endure the applied pressure, while trying to reach a neutral stress state to avoid buckling as previously mentioned. Otherwise, if the nitride is kept, the dimensions of the layers should be reviewed to better tune the stress with regards to the weakness of the film.

Another solution consists in depositing the nitride layer by *Low Pressure Chemical Vapor Deposition* (LPCVD) to have a much better film quality than this of the film deposited by PECVD [76]. This could also provide a better repeatability of the process as the deposition would be more uniform, even though parameters such as the base curvature of the wafers or the physical origins of the residual stress would still alter the different results obtained.

Characterization of the **piezoresistive properties** of IGZO and  $VO_x$  is desired to attest if one of the two candidates could be suitable to replace doped silicon in a piezoresistive pressure sensor.

Resistivity measurements of the materials under bending are performed to determine their **gauge factors**. The study of IGZO is limited to two samples as the main focus of this work has been oriented towards  $VO_x$  thin films whose deposition parameters have been studied in *Winfab*.

As there are several oxidation states possible for vanadium, the need to identify which phase is measured is required. This is why this chapter presents spectroscopy techniques used to **identify the oxide**. An analysis of the thin film properties such as the grain size of the sputtered oxide or the residual stress that can alter the crystalline structure and thus the piezoresistive behaviour is also proposed.

## 5.1 Characterization of vanadium oxides deposited by reactive sputtering

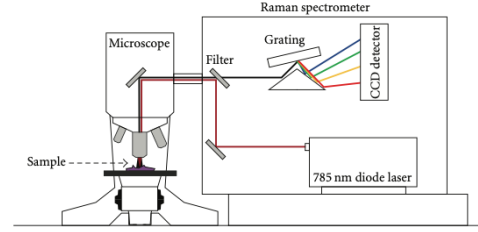
Due to the numerous oxidation states of vanadium presented in the phase diagram in Figure 2.19, it is important to identify which phase is under test when measuring the mechanical properties of a given wafer. To do so, two measurement techniques have been used and will be discussed in the following sections: Raman spectroscopy and X-ray Photoelectron Spectroscopy (XPS).

### 5.1.1 Raman spectroscopy

Raman spectroscopy is a technique relying on the **scattering** of light to characterise the constituents of a sample both qualitatively and quantitatively. Indeed, it provides the "fingerprint" of the molecules under measurement, along with an indication of their concentration.

**Description of the technique** The base principle consists in illuminating the sample with monochromatic light and measuring the energy of the photons scattered by the material with a setup as shown in Figure 5.1. Most have the same energy as the incident photons, which is associated to *elastic* or *Rayleigh scattering*, while a small percentage have an energy slightly different, which is associated with *inelastic* or *Raman scattering*.

This difference in energy comes from a shift in the **frequency** of the photons after they have been scattered. All these shifts, also called *Raman shifts* expressed in  $[cm^{-1}]$ , form a *frequency spectrum* which is unique for each molecule due to its band structure [86].



**Measurements and results** Raman spectroscopy has been performed using the *LabRamHR* equipment from Horiba on  $VO_x$  layers deposited by reactive sputtering over a layer of thermal silicon dioxide, with a specific oxygen concentration each time. All the wafers have been annealed for one hour at  $500^\circ C$  in the hermetic furnace described in Chapter 4. The phase of each sample has been identified in Table 5.1 based on the peaks in the frequency spectra observed. The values of the frequency shifts gathered from the literature and the plots of the different measurements can be found in Appendix A.

Figure 5.1: Example of a typical setup for Raman spectroscopy [85].

| Wafer | $O_2$ flow [%] | Main peaks $[cm^{-1}]$ |     |     |     |     |       | Phase           |
|-------|----------------|------------------------|-----|-----|-----|-----|-------|-----------------|
| 1     | 7              | 194                    | 223 |     |     | 618 |       | $VO_2$          |
| 2     | 9              | 194                    | 223 |     |     | 618 |       | $VO_2$          |
| 3     | 11             | 194                    | 223 |     |     | 618 |       | $VO_2$          |
| 4     | 12             | 194                    | 223 |     |     | 618 |       | $VO_2$          |
| 5     | 13             | 194                    | 223 | 308 | 386 | 616 |       | $VO_2$          |
| 6     | 14             | 195                    | 223 | 308 | 389 | 618 |       | $VO_2$          |
| 7     | 15             | 194                    | 223 | 308 | 387 | 617 | (881) | $VO_2 (V_3O_7)$ |
| 8     | 16             | 194                    | 223 | 309 | 389 | 617 | (881) | $VO_2 (V_3O_7)$ |

Table 5.1: Raman shifts observed on different vanadium oxides deposited by reactive sputtering and identification of their oxidation state.

Another Raman measurement has been performed to determine the quality of the crystalline structure of  $VO_2$  deposited and annealed on top of a layer of  $Si_3N_4$ . Figure 5.2 shows the calibration peak related to the silicon substrate at a shift of around  $500 cm^{-1}$ , and small peaks around 194, 223 and  $618 cm^{-1}$  corresponding to the spectrum of vanadium dioxide<sup>1</sup>. It can be concluded from this measurement that  $VO_2$  is the dominant phase deposited, but the annealing conditions of  $400^\circ C$  for 30 minutes are not enough to create a clear poly-crystalline structure.

<sup>1</sup>The peak under  $100 cm^{-1}$  should not be considered as it is below the cut-off frequency of the device.

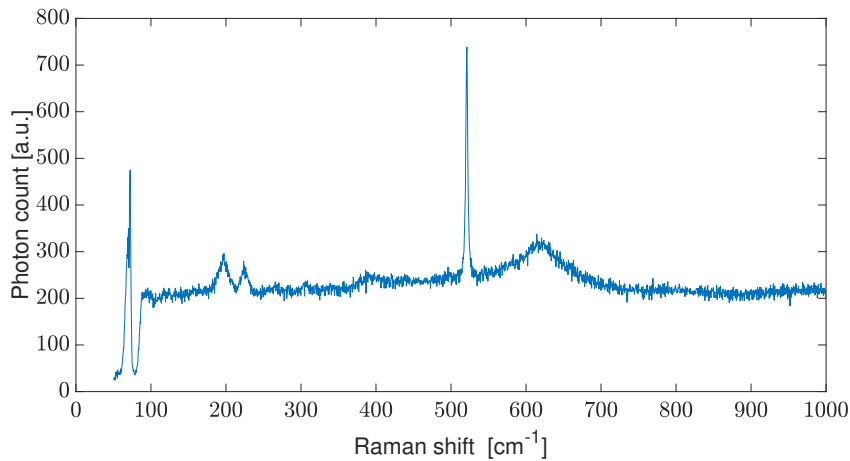


Figure 5.2: Frequency spectrum of  $VO_2$  deposited with 12.5% of  $O_2$  and annealed on top of  $Si_3N_4$ .

### 5.1.2 XPS measurements

X-ray photoelectron spectroscopy is a technique used to analyse the **surface chemistry** of a given material and provide a proper characterisation of the top atomic levels to perform surface engineering for example. Indeed, atoms on the top of a sample are far more reactive than bulk atoms as they are free to bond to other atoms. This is why being able to measure their physical and chemical properties accurately is important [88].

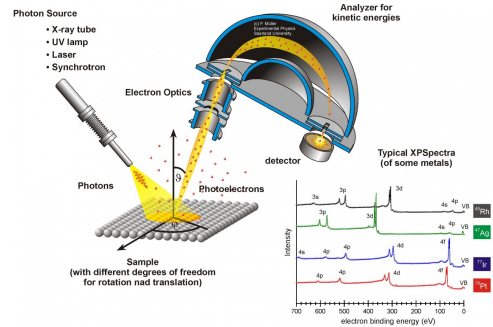


Figure 5.3: Example of a typical setup for XPS [87].

**Description of the technique** Based on the *photoelectric effect*, the idea of XPS is to put a sample under X-rays knowing the kinetic energy of the photons that are sent, and to measure the kinetic energy of the photoelectrons emitted by the surface of the material after exposure. The difference of energy corresponds to the ionisation energy of the atoms, and the crystal structure and properties can be identified based on the number of electrons detected at a specific level.

The interest of using this technique in addition to Raman spectroscopy is its ability to provide clear results even if the structure of the sample is not crystalline. Indeed, XPS measurements are mainly independent of the crystal lattice.

**Measurements and results** Test wafers used to calibrate the parameters for depositing  $VO_2$  in the AJA chamber have been characterized by XPS to obtain a quantitative ratio between the different oxidation states of the samples.

Parameters used to produce these tests wafers and XPS results are presented in Table 5.2. Measurements have been taken before and after annealing of the samples for 30 minutes at 400°C in the SiC furnace.

| Wafer  | $SiO_2$ [nm] | $O_2$ flow [%] | $VO_2$ [nm] | Composition                                                                         |                          |
|--------|--------------|----------------|-------------|-------------------------------------------------------------------------------------|--------------------------|
|        |              |                |             | Before annealing                                                                    | After annealing          |
| Test 1 | 200          | 0.5            | 97          | All chemical states but mostly $VO_2$ and $V_2O_5$                                  | Only $VO_2$ and $V_2O_5$ |
| Test 2 | 200          | 3              | 88          | Mostly $VO_2$ , noticeable presence of $V_2O_5$ , and traces of other species       | Only $VO_2$ and $V_2O_5$ |
| Test 3 | 200          | 5.5            | 86          | Mostly $VO_2$ , noticeable presence of $V_2O_5$ , and fewer traces of other species | Only $VO_2$ and $V_2O_5$ |
| Test 4 | 200          | 7              | 129         | Mostly $V_2O_5$ , $V_2O_3$ in depth                                                 | Unchanged                |

Table 5.2: Deposition parameters of the test wafers and XPS results before and after annealing at 400°C for 30 minutes.

Compositions reported in Table 5.2 only account for approximately 5 nanometers beneath the surface of the samples. Indeed, it has been observed that passed this distance, the remaining layer was only composed of pure vanadium without any trace of oxidation states. These poorly performed depositions have been explained by issues related to the usage of the AJA system that have now been fixed in *Winfab* to produce homogeneous depositions of vanadium oxides from the surface to the substrate. XPS measurements of accurately deposited wafers have been performed in *Welcome* but will not be presented in this work.

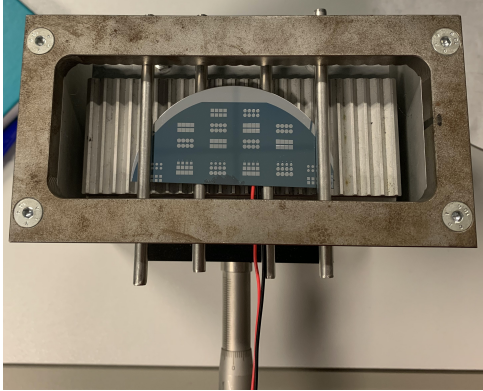
## 5.2 Study of the gauge factor of vanadium oxides and IGZO by bending measurements

The goal of the bending procedure is to characterise the piezoresistivity of a given material when it is put under strain. The FoM of interest to quantify the relative change of resistivity of the deformed material with respect to the applied strain is the **gauge factor** which has been defined in Section 2.3.1.

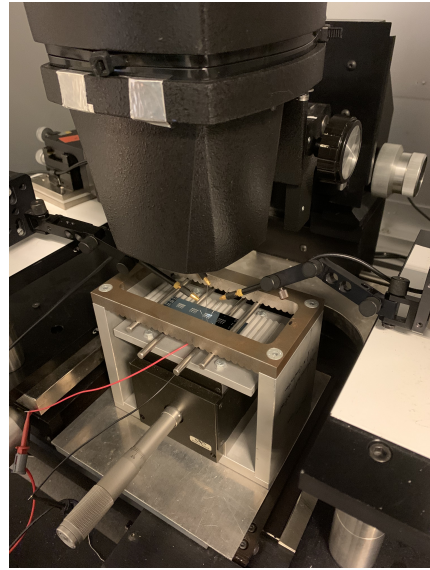
### 5.2.1 Electrical resistivity measurement

One of the simplest ways to measure the **electrical resistance** of a sample is the **2-probe method**. Basically, it consists in measuring the voltage difference and current between two metallic contacts deposited on the material under test and compute the value of the resistance from these observations [89]. A major advantage of this technique is that it is easily reproducible.

**Experimental set up** First, the contacts are deposited in *Winfab* using the shadow masks described in Section 4.1.2. The results of the metallisation are visible in Figures 5.4a and 5.5a for the two different masks. Then, a reference gauge from *Micro-Measurements*<sup>2</sup> is glued under the wafer to provide a quantification of the applied strain. This gauge has a resistance of  $350\Omega$  at rest and is connected by two wires to an ohmmeter, a Keithley2000 multimeter from *Keithley*. Finally, the wafer is fixed in the bending block within 4 metal rods, the structure is put under the microscope, and the probes are positioned right over the contacts as shown in Figure 5.4b. These probes are connected to a B1500 semiconductor design analyser from *Agilent Technologies* to extract the I-V curves between the electrodes.



(a) Top view with the metallic bars to hold the wafer.



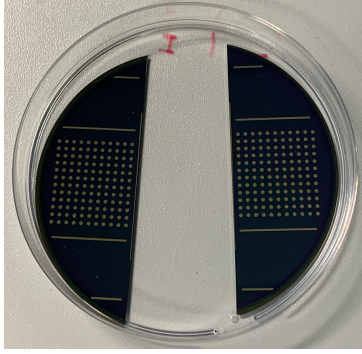
(b) Wires connected to the ohmmeter and probes in position.

Figure 5.4: Bending set up under the microscope.

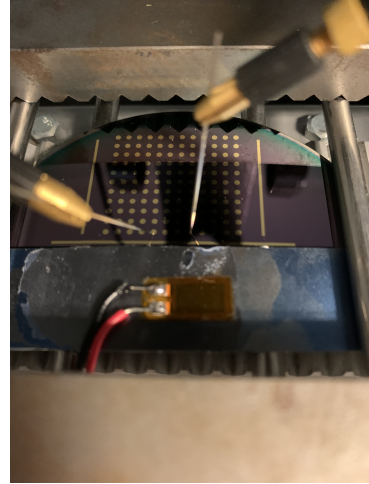
**Hypotheses** The 2-probe technique is effective for thin films with a **large resistance** to overcome the influence of the series resistance of the contacts, which is the case in this work [89]. Nevertheless, some hypotheses have been made to simplify the procedure and the data analysis afterwards.

<sup>2</sup>The gauge can be found on the website of the manufacturer under the following designation: CEA-05-250UWA-350.





(a) Cleaved wafers with contacts in Au.



(b) Set up with the gauge on a previous IGZO slice.

 Figure 5.5: Bending set up for characterization of  $VO_X$  slices.

First, the deformation is **elastic** as the silicon substrate is an elastic material, and breaking of the wafers should not be reached as the maximum strain applied is of  $500\mu strain$ .

Then, in order to avoid the manipulation and gluing of all the wafers by hand, an IGZO tab with a gauge has been reused to perform several measurements. The assumption that **the gauge undergoes the same deformation as the wafer** is then made, once the gauge has been aligned close to the sample under test as demonstrated in Figure 5.5b. However, the **alignment** of the two pieces under the rods is not always perfectly horizontal, which leads to small errors in the final results. These errors are considered small and thus ignored.

Finally, a strong hypothesis is made regarding the **current lines** around the contacts on the thin film. When current is injected in a contact, it spreads radially in all the directions until it reaches another contact on which it can be measured as depicted in Figure 5.6. But in this analysis, edge effects are ignored and the current is assumed to be straight between pairs of pads. This is the **main source of error** in the computation of the gauge factors, but it is difficult to properly quantify these effects without performing additional measurements on resistive bars of  $VO_X$ .

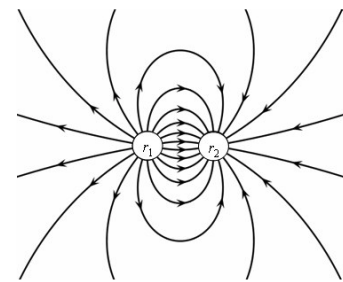


Figure 5.6: Current lines around two contacts placed on a thin film.

**Measurements** The resistivity measurements are performed by first applying different **strain levels** on the wafer. The deformation is enforced by pressing the metallic rods against its surface on opposite directions as pictured in Figure 5.7, and the strain is quantified by the change of resistivity measured at the terminals of the gauge. This change is either positive or



negative depending on the tensile or compressive stress applied *on the gauge*. If the gauge on the tab is in the same direction as the front side of the wafer like in Figure 5.5b, then, as its gauge factor is positive, tensile stress is measured. If it is aligned with the back side of the sample, the gauge undergoes a compressive stress and its resistivity will be reduced. For example, a level of  $50\mu strain$  corresponds to a positive or negative variation of  $35m\Omega$  as given by Equation 5.1:

$$\Delta R = GF R_0 \epsilon \quad (5.1)$$

where  $R_0 = 350\Omega$  is the resistance of the gauge at rest,  $GF = 2$  its gauge factor, and  $\epsilon$  the applied strain.

Then, a fixed voltage is applied through one of the probes on a contact that is chosen as a reference for the entire duration of the measurements, while the other probe applies a voltage varying between -2 and 2V on another contact. The current between the two probes is finally measured, and the next level of strain can be applied.

Note that most of the measures have been conducted in **dark** conditions as light can have a non-negligible impact on the current. The noise it generates can be of the same order of magnitude as the current in highly resistive materials ( $\approx nA$ ).

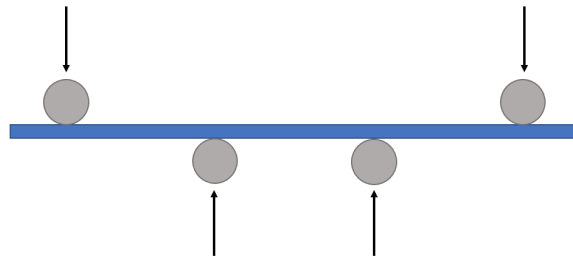


Figure 5.7: Schematic of the wafer within metallic rods.

### 5.2.2 Extraction of the gauge factor

During the bending procedure, I-V curves between the different electrical contacts on the wafer are measured under multiple strain levels going from 0 to  $500\mu strain$ . The goal is then to extract the gauge factor of each material along the **longitudinal** and **transverse** directions.

For any material put under stress, the data processing performed with *MATLAB* is the same and consists in the following steps:

1. Apply a *moving average filter* on raw data to smooth out the curves that can be noisy due to connection cables or environmental factors such as light.
2. Perform a *linear regression* on the smoothed data to obtain a single line for each level of strain and extract the slopes to obtain each variation of current  $dI/dV$ .
3. Invert these results to obtain the resistance  $R$  of the material at each level of strain in  $\Omega$  and perform another *linear regression* to obtain  $dR/d\epsilon$ .

Examples of the collected data and treatments applied to obtain the values of resistance are shown in Appendix B.

Once this processing is done, the *gauge factor*  $GF$  of the material can be easily extracted by dividing  $dR/d\epsilon$  by the initial resistance value  $R_0$  measured before applying any strain. Results are shown in Table 5.3 for IGZO and in Table 5.4 for vanadium oxides.

| Material      | Gauge factor [/ $\epsilon$ ] |            |               |
|---------------|------------------------------|------------|---------------|
|               | Longitudinal                 | Transverse | $ \Delta GF $ |
| a-IGZO        | -170.6                       | -112.2     | <b>58.4</b>   |
| Annealed IGZO | -419.3                       | -375.6     | <b>43.7</b>   |

Table 5.3: Gauge factors of IGZO extracted from bending measurements.

| Wafer  | Oxygen flow [%] | Gauge factor [/ $\epsilon$ ] |            |               |
|--------|-----------------|------------------------------|------------|---------------|
|        |                 | Longitudinal                 | Transverse | $ \Delta GF $ |
| 1      | 7               | 61.7                         | 60.8       | <b>0.9</b>    |
| 2      | 9               | 35.6                         | 35.9       | <b>0.3</b>    |
| 3      | 11              | 39.5                         | 36.1       | <b>3.4</b>    |
| 4      | 12              | 5                            | 0.65       | <b>4.35</b>   |
| 5      | 13              | 18.8                         | 8.6        | <b>10.2</b>   |
| 6      | 14              | 24.8                         | 14         | <b>10.8</b>   |
| 7      | 15              | 31.7                         | 38.9       | <b>7.2</b>    |
| 8      | 16              | 32.2                         | 38.5       | <b>6.3</b>    |
| 9      | 15              | 30.4                         | 27.5       | <b>2.9</b>    |
| 10     | 12.5            | 54.2                         | 51.6       | <b>2.6</b>    |
| 11     | 9               | 26.3                         | 36.5       | <b>10.2</b>   |
| Test 3 | 5.5             | -3.5                         | -4.2       | <b>0.7</b>    |

Table 5.4: Gauge factors of vanadium oxides extracted from bending measurements according to the the oxygen concentration used for the deposition in the AJA chamber.

Figure 5.8 shows the variation of  $R_0$  with the oxygen concentration for wafers 1 to 8. As the thin films are highly resistive for wafers 7 and 8, it was particularly important to take the measurements in the dark to avoid parasitic noise as mentioned earlier.

### 5.2.3 Analysis of the results

**IGZO** It can be seen in Table 5.3 that amorphous IGZO (a-IGZO) exhibits an *anisotropic* behaviour with regards to the direction in which the gauge factor is extracted compared to the applied deformation. Both longitudinal and transverse gauge factors can be amplified when the material is annealed. It is assumed that this is related to the lattice structure becoming poly-crystalline, and that changes occur in the band structure of the material leading to higher variations of its resistivity.

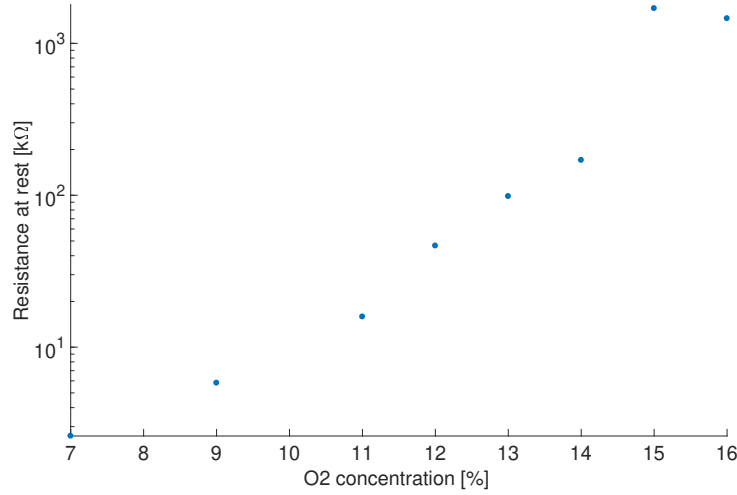


Figure 5.8: Variation of  $R_0$  with the oxygen concentration in semi-log scale, extracted from I-V curves as described in Section 5.2.2.

**Vanadium oxides** Wafers 1 to 8 presented in Table 5.4 are the same as these for which the phase has been identified in Table 5.1. Wafers 9 to 11 come from another batch for which the oxygen concentration used during the deposition of the  $VO_X$  has also been varied, and Test wafer number three comes from a deposition batch for which the AJA chamber was not properly calibrated. This poorly calibrated deposition led to a layer mostly metallic with only a few nanometers of oxide, which is in accordance with the gauge factors measured that commonly range around 2 for metals [2].

It can be noticed from these measurements that both the longitudinal and transverse gauge factors vary with the oxygen concentration, and thus with the phase of the oxide deposited on the wafer. But by comparing wafers 2 and 11 and wafers 7 and 9 that have been deposited with 9 and 15 % of  $O_2$  respectively, it can be noticed that the gauge factors differ in the two directions for both pairs. This means that the **oxygen concentration is not the only parameter having an impact on the magnitude of the gauge factor**, whatever the direction under test.

Other parameters such as the thickness of the layer or its main crystalline directions should be explored to better understand this difference in gauge factors. In the following sections, the **grain size** of the oxide or its **residual stress** have partially been studied.

### 5.3 Stress analysis

This section aims to analyse the residual stress inside the  $VO_X$  layers of wafers 1 to 11 that have been identified as  $VO_2$  using the method detailed in Section 4.3 based on Stoney's formula. However, as the base curvatures of the wafers before the growth of  $SiO_2$  were not measured, only the **difference of polynomial fits** have been used for the computations.

Once again, results presented in Table 5.5 show that the vanadium dioxide is tensile compared to the compressive layer of silicon dioxide. In addition, Figure 5.9 exhibits

a **decreasing trend for the residual stress** with the  $O_2$  concentration used during fabrication. Comparison with wafers 9 to 11 shows that the residual stress at a given concentration of  $O_2$  can vary from one wafer to another, but the general trend of decreasing stress with an increased flow of  $O_2$  is confirmed.

| Wafer | Oxygen flow [%] | $VO_2$ [MPa] | $VO_{2_{ann}}$ [MPa] |
|-------|-----------------|--------------|----------------------|
| 1     | 7               | 36.86        | 473.03               |
| 2     | 9               | 64.61        | 436.06               |
| 3     | 11              | -21.46       | 386.63               |
| 4     | 12              | 40.91        | 319.95               |
| 5     | 13              | 34.57        | 273.96               |
| 6     | 14              | 15.02        | 182.01               |
| 7     | 15              | -29.91       | 90.21                |
| 8     | 16              | 3.73         | 152.8                |
| 9     | 15              | -13.46       | 150.04               |
| 10    | 12.5            | 29.66        | 230.7                |
| 11    | 9               | 75.82        | 301.86               |

Table 5.5: Residual stress estimated by Stoney's formula with polynomial fitting.

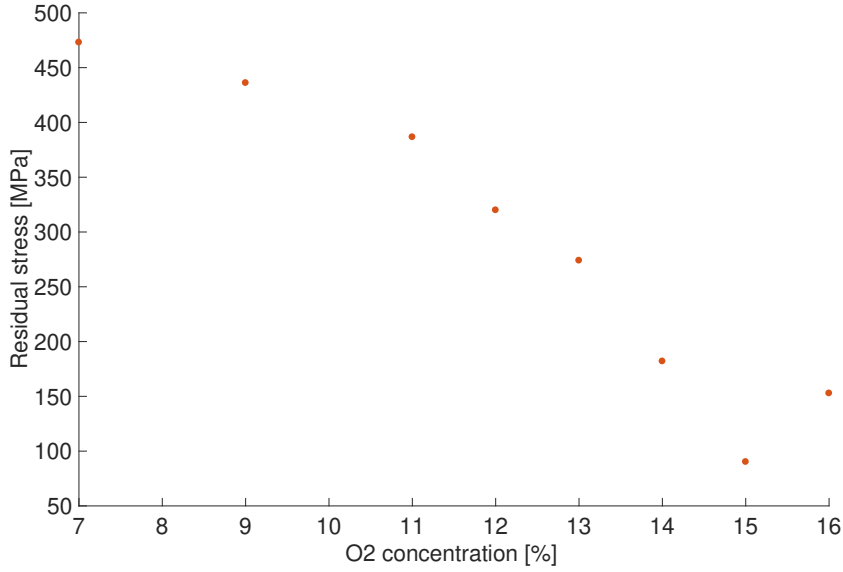
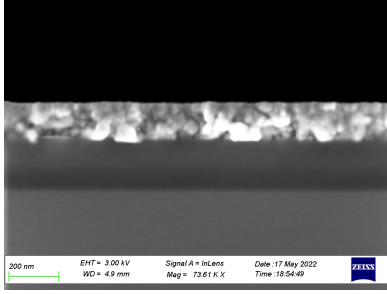


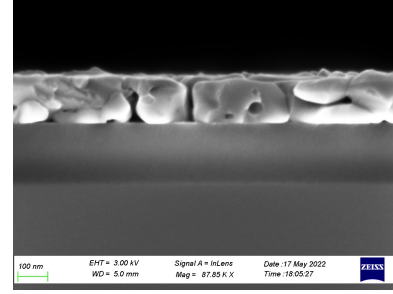
Figure 5.9: Residual stress in the  $VO_2$  layer of wafers 1 to 8 after annealing in the hermetic furnace for one hour at  $500^\circ\text{C}$ .

## 5.4 Quality of the vanadium oxides

Even though the main oxidation state of wafers 1 to 8 has been identified as  $VO_2$ , the flow of oxygen used during the deposition plays a major role in the physical structure of the layer: the **grain size** of the sputtered oxide increases with the flow. Figures 5.10 and 5.11 show the SEM pictures of the  $VO_2$  layers deposited with the two extreme concentrations studied in this work, 7 and 16 % of  $O_2$ , while Figure 5.12 highlights the evolution of the grain size with respect to this parameter.

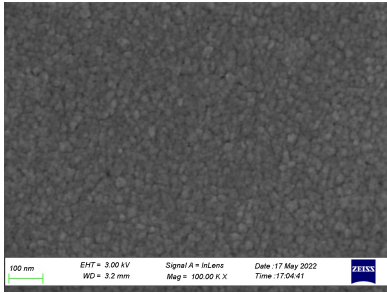


(a) Oxygen flow of 7%.

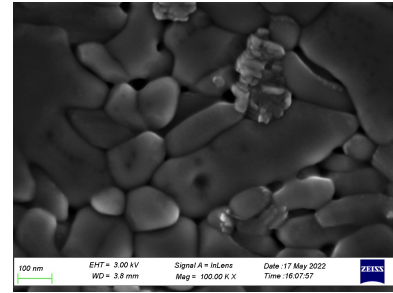


(b) Oxygen flow of 16%.

Figure 5.10: SEM cross-sectional views of wafers 1 (a) and 8 (b) after annealing.



(a) Oxygen flow of 7%.



(b) Oxygen flow of 16%.

Figure 5.11: SEM top views of wafers 1 (a) and 8 (b) after annealing.

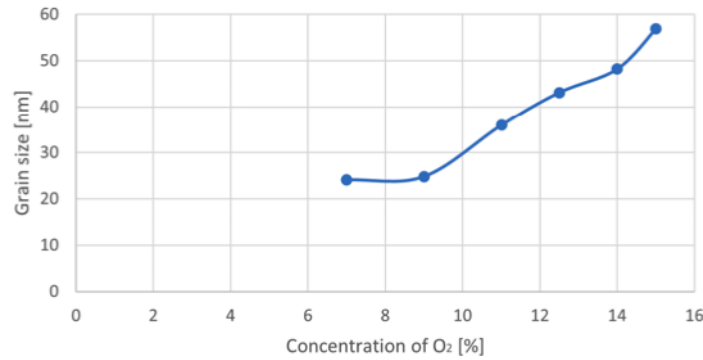


Figure 5.12: Grain size of sputtered vanadium oxide with respect to the oxygen flow used for the deposition [90].

## 5.5 Impact of the gauge factors on the sensitivity of the pressure sensor

Results from Tables 5.3 and 5.4 show the **absolute difference between the longitudinal and transverse gauge factors** of IGZO and vanadium oxides respectively. This difference is particularly interesting when working with a **Wheatstone bridge**. Indeed, this configuration takes advantage of the variation of resistance of its components to provide a measurable output voltage as explained in Sections 2.5.4 and 3.1.3.3.

The previously analysed output of the bridge can now be expressed as a function of the gauge factor of the resistors associated to their orientation:

$$\left. \begin{array}{l} GF = \frac{1}{\epsilon} \frac{\Delta R}{R_0} \\ R = R_0 + \Delta R \end{array} \right\} \longrightarrow R = R_0(1 + GF\epsilon) \quad (5.2)$$

$$\begin{aligned} V_{out} = V_1 - V_2 &= \left( \frac{R_4}{R_4 + R_1} - \frac{R_3}{R_3 + R_2} \right) V_s \\ &= \left( \frac{1 + GF_l \epsilon}{(1 + GF_l \epsilon)(1 + GF_t \epsilon)} - \frac{1 + GF_t \epsilon}{(1 + GF_t \epsilon)(1 + GF_l \epsilon)} \right) V_s \end{aligned} \quad (5.3)$$

where  $GF_l$  and  $GF_t$  represent the longitudinal and transverse gauge factors respectively.

With this expression, it is possible to estimate the outputs of a Wheatstone bridge using the materials studied in this chapter. Figure 5.13 shows the outputs for wafers 1 to 8 with a  $VO_2$  layer, while Figure 5.14 shows the outputs for annealed IGZO.

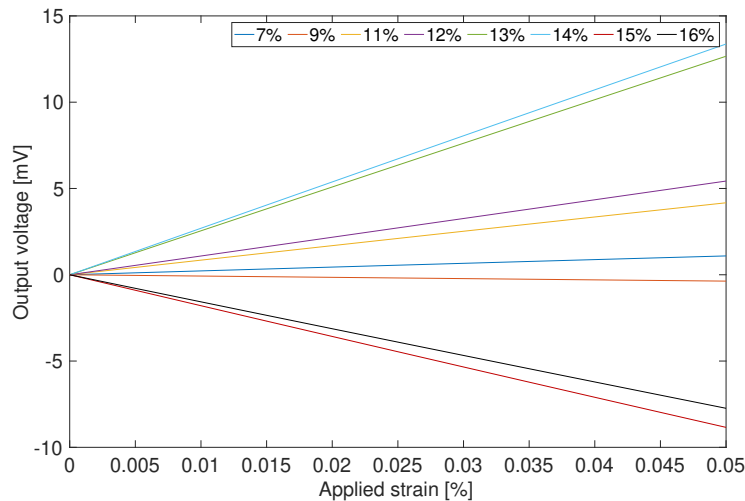


Figure 5.13: Voltage output of a Wheatstone bridge with four piezoresistors made of  $VO_2$ .

It is pretty straightforward to notice that some deposition parameters for vanadium oxides are to avoid for building a Wheatstone bridge. But as discussed earlier, varying the flow of  $O_2$  for sputtering is not enough, more variables have to be taken into account such as the base curvature of the wafers and the residual stress in the thin films, certainly

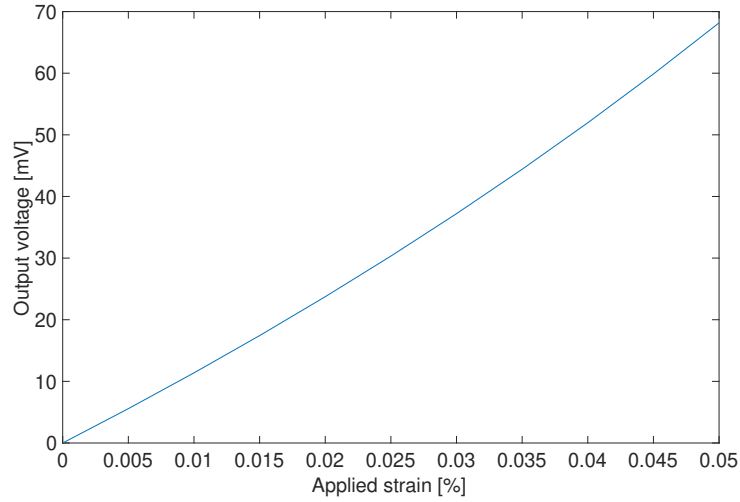


Figure 5.14: Voltage output of a Wheatstone bridge with four piezoresistors made of IGZO.

as well as other parameters which could not have been identified within the scope of this work.

On the other hand, annealed IGZO shows a higher sensitivity but only one wafer has been characterized so no other parameters regarding this material have been investigated.

## 5.6 Improvements and perspectives

In order to go further with the characterization of the aforementioned materials, some additional experiments are proposed.

First, **higher strain levels** can be explored to identify what is the reachable maximum before breaking, and the highest gauge factors achievable for both materials.

Then, additional measurements should be performed on **newly deposited IGZO films** to dig into the promising results presented in this section.

Finally, characterization of vanadium oxides should be continued to reach **new oxidation states** and compare their gauge factors to those of the vanadium dioxide, such as  $V_2O_5$  which seems to have a gauge factor of 295 according to Inomata et al. [28]

This chapter gathers the perspectives and suggestions that could be useful for future work requiring the concepts and achievements presented in this thesis.

## 6.1 Improvements of the numerical simulations and models

Inaccuracies are pointed out in the use of the average layer model for the *COMSOL* simulations, and improvements are proposed hereafter.

A **corrective factor** can be applied for the stress computation of the average layer model. This provides a stress distribution that is closer to the one of the more realistic bilayer model. If such a factor is added in the model, then it can be interesting to check if this it varies with other membrane side lengths and thicknesses.

Moreover, all the simulations have been performed without including **time dependency**. The time response of the membrane is thus not taken into account. This characteristic should be included in the study of the device if a non-stationnary pressure needs to be measured for whatever reason. Indeed, if the period of the varying pressure is smaller than the time response of the sensor, then the measured pressure will not be correct and the outcomes of this error can be disastrous depending on the application.

Furthermore, the **optimal thickness of the piezoresistive layer** should be investigated. Indeed, it has been demonstrated that a thicker layer concentrates the stress at the center of the membrane where the patterning ends, but analysis of the output of the Wheatstone bridge showed that a thicker membrane has a smaller voltage output than the thinner one for the same applied pressure. This trade-off may hide the fact that it is not possible to have better sensitivity using the patterned design compared to the unpatterned one.

Another lead concerns the studied materials. As the longitudinal and transverse gauge factors of  $VO_X$  and IGZO films have been investigated, a good improvement would be to compute their piezoresistive coefficients using  $\pi = \frac{GF}{E}$  and inject those values into a piezoresistive matrix in *COMSOL* to visualise their influence on the sensor performances. Nevertheless, doing so requires to build a new material in the simulated stack with some mechanical and electrical properties that are not especially well known in the literature like the Young modulus.



## 6.2 Perspectives for the fabrication process

Possible solutions to overcome the issues encountered during the fabrication process have been proposed at the end of Chapter 4 and are summarised here.

The base stack is mainly weakened by the  $Si_3N_4$  layer deposited in *Winfab*. As its stoichiometry is not exactly known and PECVD nitride films are well-known to be of poor quality, the main focus should be on finding an **alternative solution that does not include this layer**. A configuration without it has been proposed and consists in etching only part of the silicon substrate to have a membrane thick enough to support the applied pressure, while trying to reach a neutral stress state to avoid buckling. Another solution would be to deposit a nitride layer by LPCVD which provides films of better quality according to the literature.

Finally, as the annealing step cannot be avoided for the  $VO_x$  layer, better **stress tuning** could be performed by playing on the dimensions of the layers or by adding another compressive layer to the base stack. The order of the fabrication steps could also be discussed to find a way to deposit the vanadium oxide film and anneal it before depositing the nitride layer.

## 6.3 Improvements for studying the piezoresistive properties of different materials

Once again, perspectives have been discussed earlier in Chapter 5 and are summarised in this section, with a few additional improvements proposed.

Firstly, **higher strain levels** can be explored to identify what is the reachable maximum before breaking and to see if a linear variation of the gauge factor is reached under increasing strain.

Then, **additional measurements** should be performed on **IGZO thin films** deposited with controlled parameters and environment to confirm the promising results observed in this work.

The study of **other vanadium oxides** should also be conducted to compare their gauge factors to those of the vanadium dioxide measured.

Finally, in order to fully characterise the piezoresistive properties of the materials, the Young modulus of the materials should be extracted to be able to compute their **piezoresistive coefficients** and plug these values into the numerical simulations.

This work was oriented around the study of a **novel design for a piezoresistive pressure sensor** and the **characterisation of two materials** that could possibly be used as transducers for this device. The main interest that guided this research was to find out whether the design could lead to an **increase in the sensing performances** compared to what is usually done in the literature or not, and if other materials have **better piezoresistive properties** than classic silicon.

With that in mind, the principal **transducing techniques** for pressure sensing technology have been reviewed, along with the theory behind **sensing membranes**. The impact of shape and size of a membrane on the stress have been modeled at the first order to justify the use of a square membrane rather than a circular one. The models showed a higher stress under deflection for a square shape. Different **figures of merit** were defined and discussed in order to characterise and classify sensors.

A **first order analytical model for a bilayer membrane** has then been proposed to complete the theory of monolayer membranes found in the literature. The model was built by creating an "average layer" based on the weighted average of the parameters of originally separated layers.

Two **numerical models** were also built using *COMSOL Multiphysics*, a finite element solver allowing to perform the coupling of different physics. The first one is a complete version of the stack using two distinct layers to build the membrane, and the other uses the average layer technique. Specific **mesh refinement** has been performed to reduce the computational time while maintaining a satisfying precision to simulate the physical distribution of the stress in the stack. Both models were compared and it has been showed that the average layer needed a corrective factor to accurately compute the maximum stress on its surface. Another comparison with the analytical model was done to further validate the numerical model.

The **effect of the patterned piezoresistive layer** was then included to approach the results proposed in the literature, and it has been shown that it is possible to concentrate the stress in the middle of the membrane by increasing the thickness of the piezoresistive layer. All the simulations were conducted with p-type silicon as a top layer but can easily be modified to test another material if all the necessary coefficients and parameters are provided. The numerical models are available for future work in the field.

Besides these theoretical studies supported by finite element modelling, the **materials** of interest, namely Indium Gallium Zinc Oxide (IGZO) and Vanadium Oxides ( $VO_X$ ), have been manipulated in *Winfab*. More specifically, the **deposition parameters** of  $VO_X$  thin films have been investigated and the associated piezoresistive properties measured. The **tuning of the stress** inside a stack of thin films has also been studied but did not led

to a viable solution for going through the entire fabrication process of the newly designed sensor.

In the end, **two major conclusions** can be drawn out of this work. Firstly, it has numerically been demonstrated that it was in fact possible to **concentrate the stress** inside a deflected membrane at specific locations by patterning its top layer. The stress distribution is modified compared to general membrane theory, and zones of high stress can be found around the center of the diaphragm instead of being mostly located in the middle of its edges. This observation opens the door to new possibilities in the field of pressure sensing, with an adequate tuning of the sensing layer.

And secondly, this work also highlighted **promising piezoresistive properties** of IGZO and of vanadium oxides which are usually studied for other of their intrinsic properties. High longitudinal and transverse gauge factors of -419 and -376 respectively have been reported for IGZO, whereas the piezoresistive behaviour of  $VO_x$  films has been observed but still requires further analyses to reach values comparable to the one found in the literature.

APPENDIX A \_\_\_\_\_

\_\_\_\_\_ RAMAN MEASUREMENTS

### A.1 Frequency spectra of different vanadium oxides

The Raman shifts characterizing the different oxidation states of vanadium have been collected from the literature and are presented in Figure A.1.

[illegible]

Figure A.1: Raman shifts of different vanadium oxides extracted by Raman spectroscopy measurements.

## A.2 Identification of the deposited vanadium oxides

The results of the Raman measurements performed on the wafers studied in this work are plotted in Figures A.2 and A.3.

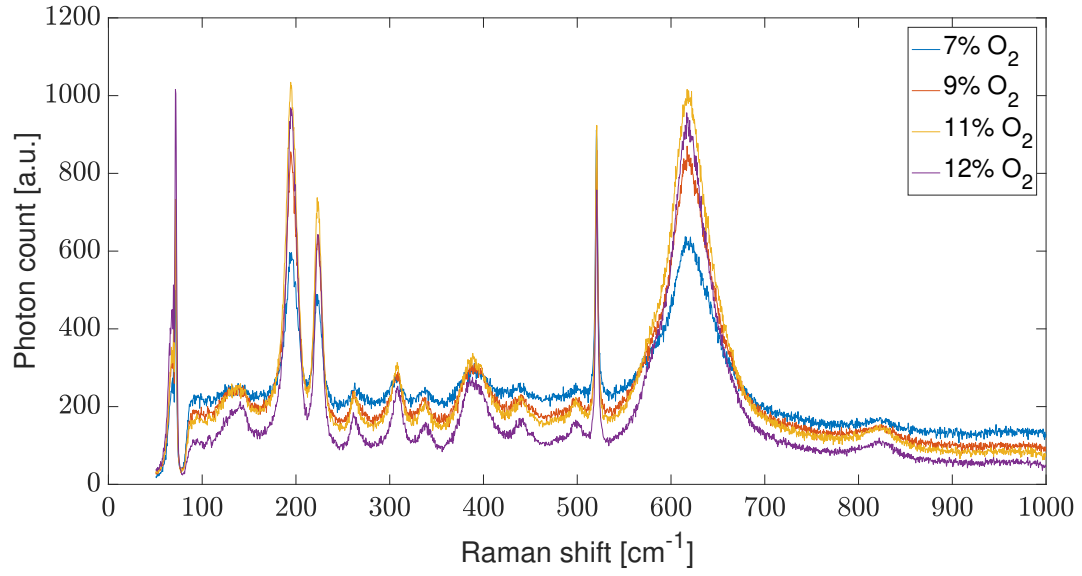


Figure A.2: Frequency spectra of wafers 1 to 4.

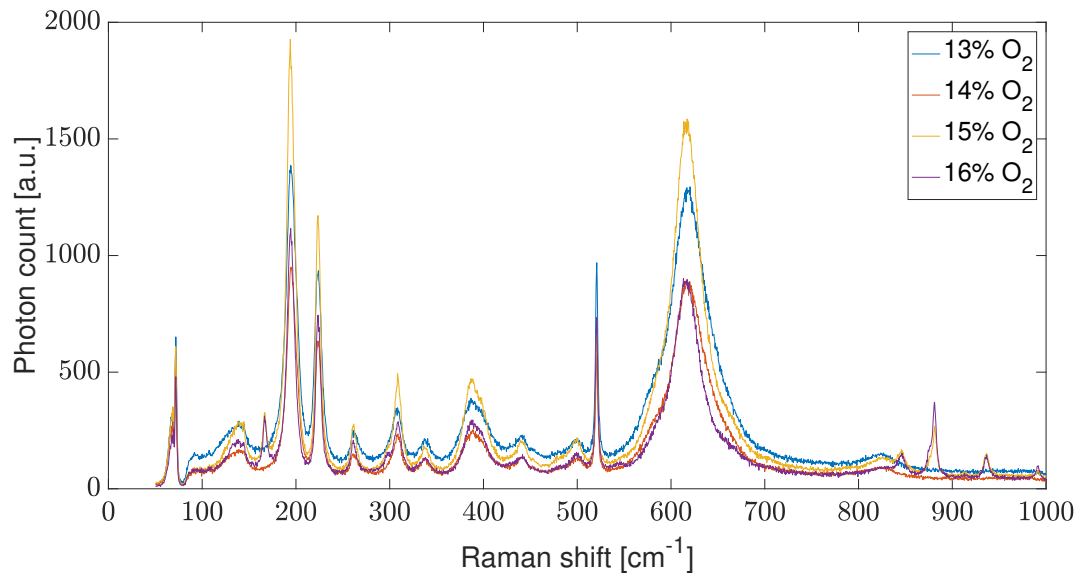
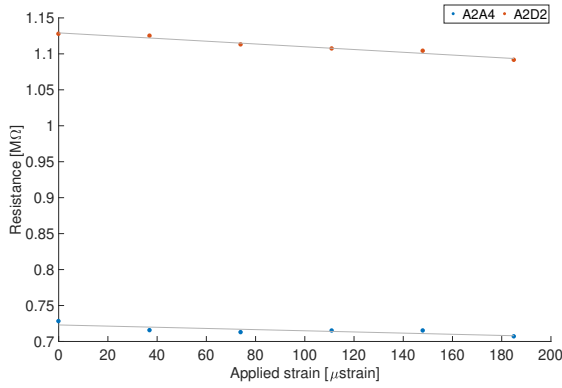


Figure A.3: Frequency spectra of wafers 5 to 8.

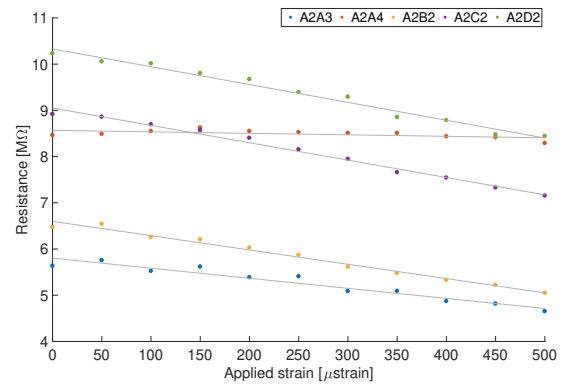
## APPENDIX B

## BENDING MEASUREMENTS

Figures B.1 to B.4 show the computed values of the resistance for each strain level, along with the linear regression showing its variation under deformation, for each pair of pads probed during the bending procedure.

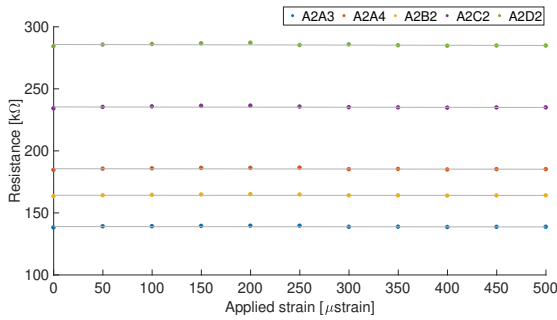


(a) Amorphous IGZO.

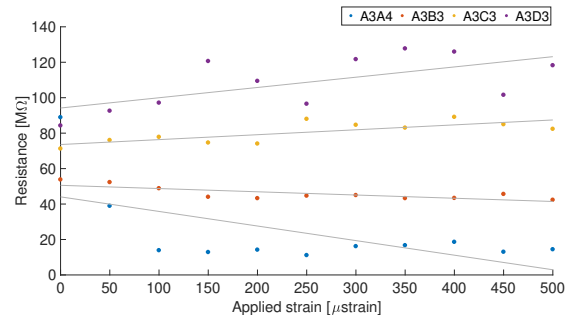


(b) Annealed IGZO.

Figure B.1: Linear regression over the measured resistivities of IGZO slices under strain.



(a)  $VO_2$  deposited with 5.5% of  $O_2$ .



(b)  $VO_2$  deposited with 7% of  $O_2$ .

Figure B.2: Linear regression over the measured resistivities of *poorly deposited*  $VO_2$  slices under strain.

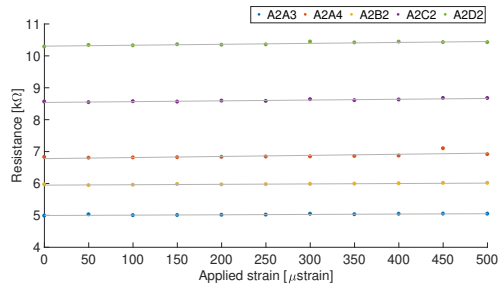
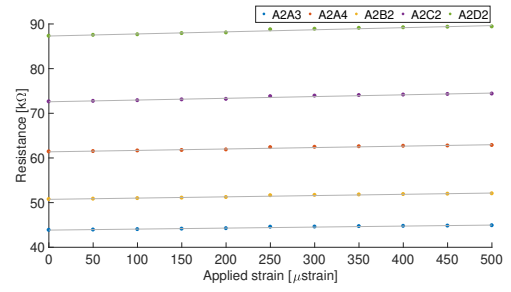
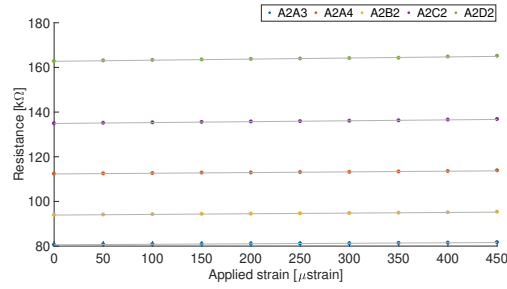

 (a)  $VO_2$  deposited with 9% of  $O_2$ .

 (b)  $VO_2$  deposited with 12.5% of  $O_2$ .

 (c)  $VO_2$  deposited with 15% of  $O_2$ .

 Figure B.3: Linear regression over the measured resistivities of  $VO_2$  slices under strain.

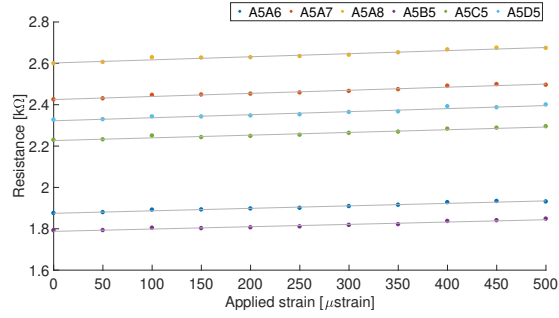
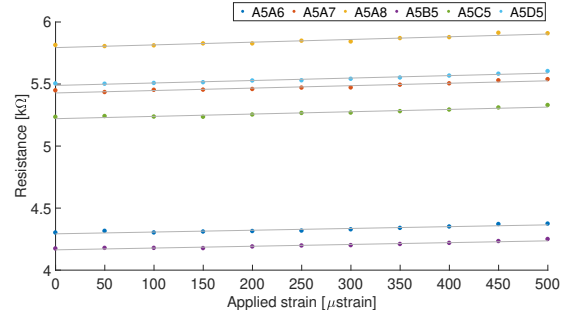
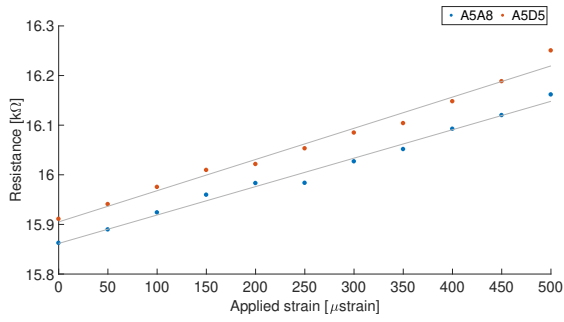
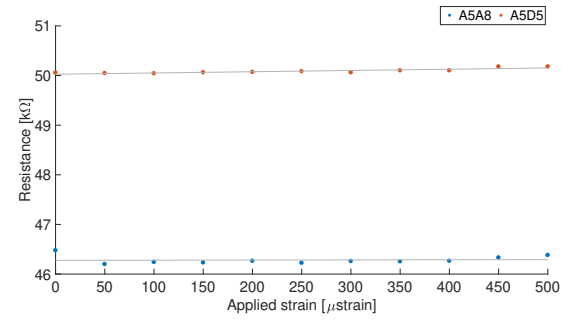
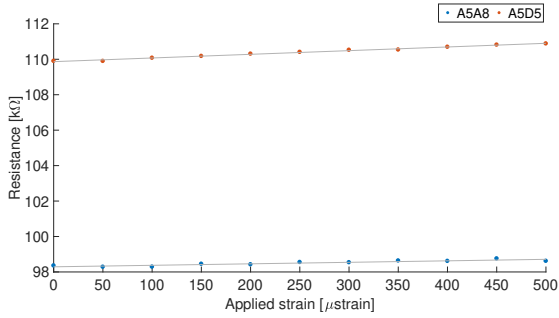
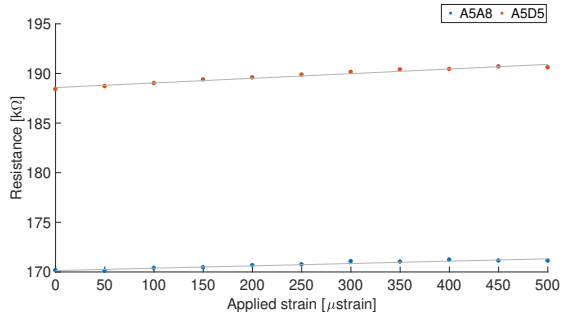
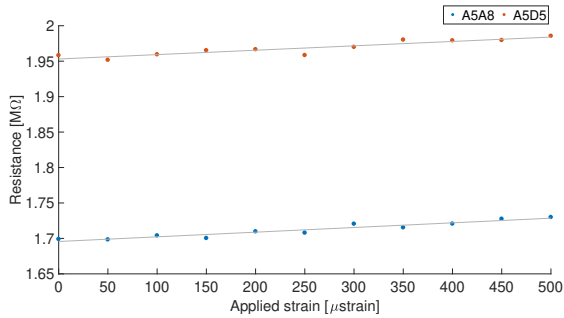
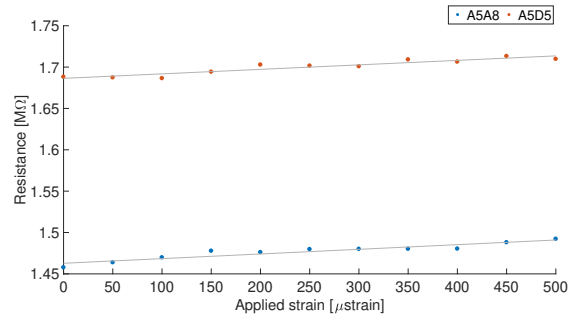

 (a)  $VO_X$  deposited with 7% of  $O_2$ .

 (b)  $VO_X$  deposited with 9% of  $O_2$ .

 (c)  $VO_X$  deposited with 11% of  $O_2$ .

 (d)  $VO_X$  deposited with 12% of  $O_2$ .

 (e)  $VO_X$  deposited with 13% of  $O_2$ .

 (f)  $VO_X$  deposited with 14% of  $O_2$ .

 (g)  $VO_X$  deposited with 15% of  $O_2$ .

 (h)  $VO_X$  deposited with 16% of  $O_2$ .

Figure B.4: Liner regression over the measured resistivities of different vanadium oxide slices under strain.



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